



INVESTIGATION ON SINGLE FIBRE STRENGTH FROM RECLAIMED CARBON FIBRE (rCF) FOR SECONDARY APPLICATION

Submitted in accordance with the requirement of the Universiti Teknikal Malaysia Melaka (UTeM) for the Bachelor Degree of Manufacturing Engineering with honours

by

NORSYAFIQAH BINTI ADNAN

B051510180

960701106042

FACULTY OF MANUFACTURING ENGINEERING

2019

BORANG PENGESAHAN STATUS LAPORAN PROJEK SARJANA MUDA

Tajuk: **INVESTIGATION ON SINGLE FIBRE STRENGTH FROM RECLAIMED CARBON FIBRE (rCF) FOR SECONDARY APPLICATION**

Sesi Pengajian: **2018/2019 Semester 2**

Saya **NORSYAFIQAH BINTI ADNAN (960701-10-6042)**

mengaku membenarkan Laporan Projek Sarjana Muda (PSM) ini disimpan di Perpustakaan Universiti Teknikal Malaysia Melaka (UTeM) dengan syarat-syarat kegunaan seperti berikut:

1. Laporan PSM adalah hak milik Universiti Teknikal Malaysia Melaka dan penulis.
2. Perpustakaan Universiti Teknikal Malaysia Melaka dibenarkan membuat salinan untuk tujuan pengajian sahaja dengan izin penulis.
3. Perpustakaan dibenarkan membuat salinan laporan PSM ini sebagai bahan pertukaran antara institusi pengajian tinggi.

SULIT (Mengandungi maklumat yang berdarjah keselamatan atau kepentingan Malaysiasebagaimana yang termaktub dalam AKTA RAHSIA RASMI 1972)

TERHAD (Mengandungi maklumat TERHAD yang telah ditentukan oleh organisasi/badan di mana penyelidikan dijalankan)

TIDAK TERHAD

Disahkan oleh:

Alamat Tetap:

Tarikh: _____

Cop Rasmi:

Tarikh: _____

*Jika Laporan PSM ini SULIT atau TERHAD, sila lampirkan surat daripada pihak berkuasa/organisasi berkenaan dengan menyatakan sekali sebab dan tempoh laporan PSM ini perlu dikelaskan sebagai SULIT atau TERHAD.

DECLARATION

I hereby, declared this report entitled “Investigation on single fibre strength from reclaimed carbon fibre (rCF) for secondary application” is the results of my own research except as cited in reference.

Signature :

Author's name : NORSYAFIQAH BINTI ADNAN

Date :

APPROVAL

This report is submitted to the Faculty of Manufacturing Engineering at Universiti Teknikal Malaysia Melaka as a partial fulfilment of the requirements for the degree of Bachelor of Manufacturing Engineering with Honours

The members of the supervisory committee are as follow:

.....

(DR. ZURINA BINTI SHAMSUDIN)

ABSTRAK

Gentian karbon merupakan bahan yang sering digunakan di dalam komposit oleh kebanyakan industri disebabkan sifat mekanikalnya yang unggul tetapi penghasilan gentian karbon yang terlalu banyak telah menyebabkan pembaziran dan memberi kesan pada alam sekitar. Maka, kajian terkini telah mencadangkan dan melakukan proses pirolisis sebagai salah satu cara untuk menuntut semula gentian karbon. Walau bagaimanapun, suhu dan masa pirolisis yang berbeza memberi kesan pada kualiti gentian karbon (rCF). Oleh itu, analisis ujian tegangan dan analisis statistik digunakan untuk mengetahui sifat mekanikal gentian karbon (rCF). Tujuan kajian ini adalah untuk mengetahui sifat mekanikal seperti kekuatan dan kekuatan anjal gentian karbon yang telah melalui pirolisis proses. Ujian tegangan gentian tunggal telah dilakukan pada gentian karbon (rCF) dan gentian karbon (rCF) berskala industri mengikut piawaian ASTM C1557 pada panjang tolok 25 mm dan 40 mm. Pada analisis tegangan-tekanan, kekuatan gentian karbon (rCF) untuk kedua-dua panjang tolok adalah antara 1.23-2.48 GPa. Taburan kekuatan gentian karbon (rCF) telah menguatkan korelasi dengan garis pusat dan nilai kekuatan taburan pada gentian yang diuji, dan daripada analisis Weibull adalah antara 1.4-2.8 GPa. Variasi parameter Weibull bergantung pada panjang tolok. Penilaian kegagalan tekanan melalui Pengimbasan mikroskop electron (SEM) telah mendedahkan adanya patah yang menyebabkan kecacatan terletak pada permukaan atau di dalam gentian.

ABSTRACT

Carbon fibres is the most recent popular material use for composites purpose by many industries due to its superior mechanical properties but due to massive production in industry has created waste and environmental effect. Hence, recent study suggested and carried out the pyrolysis process as one of the method to reclaimed the carbon fibre. However, different temperature and dwelling time of pyrolysis affect the quality of rCF. Therefore, tensile testing analysis and statistical analysis was use to determine the mechanical properties of the reclaimed carbon fibre. The aim of this study is to evaluate the mechanical strength and modulus of the received pyrolysed reclaimed carbon fibre (rCF). The single fibre tensile testing was carried out on the rCF and rCF industry scale via standard ASTM C1557 at the gage length of 25 mm and 40 mm. From the stress-strain analysis, the strength of rCF for both gauge length was between 1.23-2.48 GPa. The rCF strength distributions were strongly correlated with the diameter with distribution strength values of the tested fibres, and from Weibull analysis is between 1.4-2.8 GPa. The variation of the Weibull parameter depended on the gauge length. Scanning electron microscopy (SEM) evaluation of the failure stress revealed the presence of fracture inducing flaws located at the surface or in the interior of the fibre.

DEDICATION

Only

My beloved father, Adnan Bin Yaakob

My appreciated mother, Kamsiah Binti Kemen

My sister and brother, Amir, Nurfarahani and Nurhazirah

For giving me moral support, cooperation, encouragement and understandings

Thank You So much

ACKNOWLEDGEMENT

In the name of ALLAH, the most gracious, the most merciful, with the highest praise to ALLAH that I manage to complete this final year project (FYP) successfully with the help of my supervisor and friends. I would like thank you so much to my respected supervisor, Dr. Zurina Binti Shamsudin for the great mentoring and help throughout this project. I appreciate all the lesson and help from the supervisor that guide me throughout the journey of completing this report. I also would like to thank you to my friend Hawa Bt Ahmad Fuzi and Lau Sie Lin for kind motivation in completing this FYP report.

TABLE OF CONTENT

ABSTRAK	i
ABSTRACT	ii
DEDICATION	iii
ACKNOWLEDGEMENT	iv
TABLE OF CONTENT	v
LIST OF TABLES	vii
LIST OF FIGURES	viii
LIST OF ABBREVIATIONS	x
LIST OF SYMBOLS	xi
CHAPTER 1: INTRODUCTION	1
1.1 Background of Study.....	1
1.2 Problem Statement	4
1.3 Objectives.....	7
1.4 Scope of Research work.....	7
1.5 Significant/ Important of Study.....	7
1.6 Organization of the report	8
1.7 Summary	8
CHAPTER 2: LITERATURE REVIEW	9
2.1 Introduction	9
2.2 Carbon Fibre.....	11
2.3 Reclaimed carbon fibre	12
2.3.1 Process	13
2.4 Pyrolysis process	14
2.4.1 Temperature.....	15
2.4.2 Atmosphere.....	17
2.4.3 Dwelling Time.....	19
2.5 Mechanical Properties	20
2.6 Characterization of carbon fibre strength.....	22

2.6.1 Preparation of Sample	22
2.6.1 Measurement of fibre diameter	24
2.7 Strength of Fibre.....	25
2.7.1 An overview of reclaimed carbon fibre strength analysis	25
2.7.2 Weibull Analysis	27
2.7 Fractography.....	28
2.8 Summary	31
CHAPTER 3: METHODOLOGY	32
3.1 Flowchart.....	33
3.2 Materials.....	35
3.2.1 Chemical treatment of reclaimed carbon fibre (rCF)	36
3.3 Single fibre tensile testing	37
3.3.1 Mechanical Strength & Elastic modulus	38
3.4 Weibull Analysis	39
3.5 Fractography of rCF.....	39
3.6 Summary	40
CHAPTER 4: RESULTS AND DISCUSSIONS	41
4.1 Characterization of reclaimed carbon fibre	42
4.1.1 Physical observation	42
4.1.2 Measurement of fibre diameter	46
4.2 Tensile strength	47
4.2.1 Fibre Strength	47
4.2.2 Weibull analysis	50
4.3 Fractography of reclaimed carbon fibre	52
4.4 Correlation of Weibull analysis with fractography	53
CHAPTER 5: CONCLUSION AND RECOMMENDATION	54
5.1 Conclusion.....	54
5.2 Sustainability.....	55
5.3 Complexity	55
5.4 Life-long learning.....	56
REFERENCES	57

LIST OF TABLES

Table 2.1 Operating parameters for pyrolysis process	15
Table 2.2 Mechanical properties of carbon fibre tow	21
Table 4.1 Average diameter of reclaimed carbon fibre	47
Table 4.2 Tensile properties and young modulus of reclaimed carbon fibres.....	49
Table 4.3 Weibull distribution of reclaimed carbon fibres.....	51

LIST OF FIGURES

Figure 1.1 The part of composite with silica coating	2
Figure 1.2 Fibre Strength Statistics between virgin and reclaimed carbon fibre	3
Figure 1.3 TGA curve	5
Figure 1.4 SEM at temperature (a) 500°C, (b) 600°C and(c) 700°C under nitrogen condition	6
Figure 2.1 Schematic representation of carbon fibers fabrication from PAN fibers	12
Figure 2.2 The process of recycled carbon fibre	13
Figure 2.3 Technologies of CFRP waste recycling	14
Figure 2.4 TGA curve	16
Figure 2.5 Higher TGA of the prepreg composite in inert atmosphere.....	17
Figure 2.6 Higher TGA of the prepreg composite in oxidative atmosphere.....	18
Figure 2.7 TG and DTG curves of composite material under nitrogen and inert atmosphere	19
Figure 2.8 Weight loss after dwelling time at maximum temperature	20
Figure 2.9 Mean strengths and their 95% confidence intervals obtained from single fibre tensile tests and bundle tensile tests	21
Figure 2.10 The “fluffy” physical condition of rCF	23
Figure 2.11 Schematic for single fiber tensile testing	23
Figure 2.12 Tensile strength of single carbon fibre.....	24
Figure 2.13 Fibre diameter image taken from advances optical microscope	25
Figure 2.14 Stress-strain curves of PAN and pitch based carbon fibres	26
Figure 2.15 Weibull plots for tensile strength of virgin and recycled carbon fibers.....	28
Figure 2.16 SEM images at different level of magnification of recycle carbon fibres show a clean and smooth surface with very limited resin or coating residue (a) x250 (b) & (c) x1000 (d) x5000	29
Figure 2.17 Fracture surfaces of virgin carbon fibre at (a) 2 µm (b) 1 µm	30
Figure 2.18 SEM under high tensile strength after single fiber tensile testing	30

Figure 3.1 Flowchart of the sample preparation.....	33
Figure 3.2 Flow diagram of the experiment	34
Figure 3.3 Condition of reclaimed carbon fibre (rCF) at dwelling time 3-hour after chemical treatment.....	35
Figure 3.5 Tensile testing machine.....	37
Figure 3.6 Schematic of rCF placed onto the template for single fiber tensile testing	38
Figure 4.1 SEM images of the (a) surface condition of rCF and (b) cross-sectional area of the rCF	42
(c).....	43
Figure 4.2 SEM images of the rCF at dwelling time 1-hour (a) before chemical treatment, (b) after chemical treatment (c) cross-sectional area.....	43
Figure 4.3 SEM images of the rCF at dwelling time 2-hour (a) before chemical treatment, (b) after chemical treatment (c) cross-sectional area.....	44
Figure 4.4 SEM images of the rCF at dwelling time 3-hour (a) before chemical treatment, (b) after chemical treatment (c) cross-sectional area.....	45
Figure 4.5 Diameter measurement of reclaimed carbon fibre	46
Figure 4.6 Typical stress-strain curves of reclaimed carbon fibre at gauge length of (a) 25 mm and (b) 40 mm	48
Figure 4.7 Tensile Strength of single carbon fibres	49
Figure 4.8 Weibull Plot of rCF at gauge length (a) 25 mm (b) 40 mm.....	50
Figure 4.9 SEM images of reclaimed carbon fibre after tensile testing	52

LIST OF ABBREVIATIONS

ASTM - American Society for Testing and Materials

PAN - polyacrylonitrile

CFRP - Carbon Fibre Reinforced Polymer

SEM - Scanning Electron Microscopy

TGA - Thermogravimetric analysis

DTG - Differential Thermogravimetric

rCF - Reclaimed Carbon Fibre

SFTT - Single Fibre Tensile Testing

ISO - International Organization for Standardization

LIST OF SYMBOLS

μ - micro

CO_2 - Carbon Dioxide

N_2 - Nitrogen

O_2 - Oxygen

Gpa – Giga Pascal

MPa – Mega Pascal

$^{\circ}\text{C}$ - degree Celsius

σ - Stress

P_f - Probability Failure Distribution

ε - Strain

ϵ - Modulus

π - pi

CHAPTER 1

INTRODUCTION

In this chapter, study on single fibre strength from reclaimed carbon fibre for secondary application will be briefed. The purpose of the introduction is to briefly explained on the background of study, state the problem statement, objectives, scope and significance of research work.

1.1 Background of Study

Carbon fibres can be categorized as the lightweight materials which mostly be used in any field. Its lightness and strength make it versatile and useful commercial product for a wide variety of markets. Carbon fibre commonly is used to reinforce composite materials in order to produce strong and lightweight materials such as carbon fibre reinforce polymer (CFRP). This carbon fibre composites mainly composed of two parts: a matrix and a reinforcement. The matrix usually a resin (example, epoxy) which use to bind the reinforcement together while carbon fibre act as reinforcement. The reinforcement gives the composite its strength and rigidity. In this case, the reinforcement is in fibre form. The fibre is the main component that carrying load within the composite.

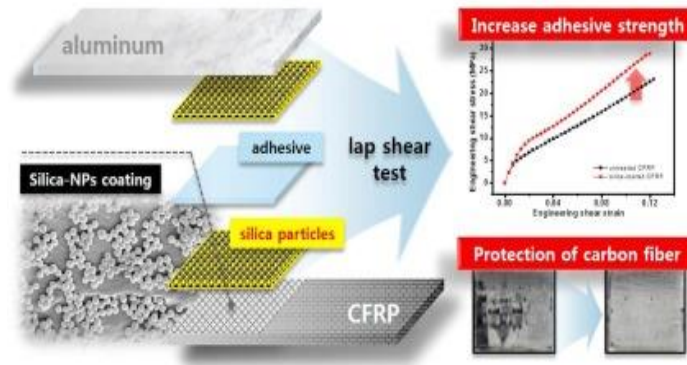


Figure 1.1 The part of composite with silica coating (Kyungtae et.al, 2018)

Properties of the carbon fibre composite are affected by the carbon fibre reinforcement characteristic which are the size, the arrangement, the distribution and etc. These characteristic significantly affect the mechanical properties especially strength of the carbon fibre. Carbon fibre is a unique and distinct appearance that is nearly impossible to replicate, excellent strength to weight ratio, works well with other material, high stiffness, high heat tolerances and resistance (TruDesign, 2015). The stiffness and modulus of elasticity of carbon fibers can be up to three times of steel. The carbon fibers promotes a very high strength of 7 GPa, axial compressive strength is 10-60 % of their tensile strength and transverse compressive strength is 12-20 % of their axial compressive strength (Prashanth S, 2017). However, the life cycle of the carbon fibre material is depend on the application used because the process and properties of the material could be change.

Recently many researchers have intensively studied on the process of reclaiming the strength of carbon fibre from CFRP. There are three main types of reclamation of CFRP; mechanical, thermal and chemical methods (Pickering, 2006). CFRP reclamation process can be divided into primary, secondary and tertiary (Morin et. al, 2012). Basically, primary and secondary processes are mechanical method that consist of various stages of scrap size reduction such as size reduction of large scale and followed by process of second size reduction. Tertiary processes typically thermal and chemical method since it recover more than just fibres, either it is organic components recovery and/or recovery of energy (Morin et.al, 2012 & Pickering, 2006). Among of the process that has been practiced to reclaimed the carbon fibre is pyrolysis. Pyrolysis process has been used widely in industry scale, since it has been reported that it can reclaimed the strength of carbon fibre (Pimenta, 2012).

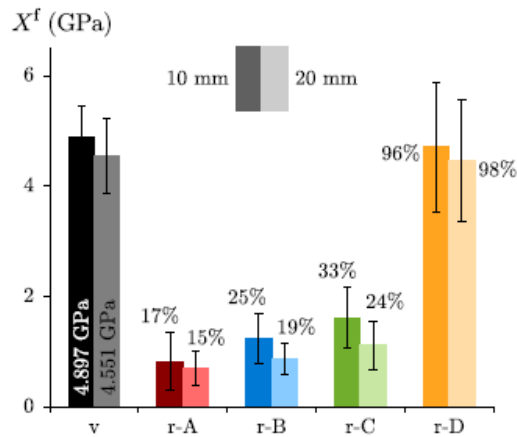


Figure 1.2 Fibre Strength Statistics between virgin and reclaimed carbon fibre (Pimenta, 2012)

The strength of reclaimed carbon fibre from pyrolysis process shown (Figure 1.2) that percentage strength of virgin carbon fibre higher than reclaimed carbon fibre but as the gauge length increasing, result in a reduction of strength retention (Pimenta, 2012). These results are obtained through single fibre testing at 10 mm and 20 mm of gauge length (Pimenta, 2012). However, this value is depends on the parameter processing of pyrolysis which most of the studied work focus on the industry scale. Daphiny (2007) states that at TUHH company , CFRP were pyrolysed at temperature up to 1300°C in a batch process with the atmosphere free of oxygen. Direct heating atmosphere was created with natural gas burners contains CO₂, N₂, and H₂O (Daphiny, 2007).

Industry scale uses a pyrolysis industry-scale which temperature and dwelling time are different with the pyrolysis lab-scale. The pyrolysis lab-scale use different parameter (temperature and dwelling time) since it depends on the quantity of carbon fibre waste which smaller compared to industry-scale. These parameters will affect the properties of the reclaimed carbon fibre especially the strength. As for the industry scale, the best mechanical properties were obtained at temperature 700°C (low temperature) compare to virgin fibres (Daphiny, 2007).

In most fibre testing, single fibre testing is a popular procedure to measuring the strength and modulus of reclaimed carbon fibre because these experiments are conducted on single filament (Ilankeeran et. al, 2012). The procedure of the single fibre testing follow ASTM C1557-03 standard the most. However, the variation of the mechanical properties can affect the final results which usually can be known from scanning electron microscope

(SEM). SEM detect the flaw on the fracture surface while the notch size (critical flaw size) estimated with Griffith relation (Tanaka et. al, 2013). Therefore, statistical data that is Weibull modulus are needed for the behaviour of these fibre. The analysis relies on the assumption that the failure of fibres as a function of applied load is controlled by the random distribution of a single type of defect (unimodal Weibull) along the length of the fibres (Thomason, 2013)

This study is way forward towards a basic characterization of single fibres for mechanical properties (strength and modulus) and damage properties evaluation which attributed for micromechanics of composites. Investigation on single fibre strength from reclaimed carbon fibre by using statistical analysis for secondary application was study for further research purpose. It is known that the single fibre testing employed high coefficient of variation and required variation of gauge length for test.

1.2 Problem Statement

It is known that different group of reclaimed carbon fibres with different fiber length have different mechanical properties to the original fibres. These mechanical properties is may different due to recycling processes involved. Pyrolysis is one of the recycling process of carbon fiber which have a strong influence on the quality of reclaimed carbon fibre. According to previous study (Daphiny, 2007) reported that the result of thermogravimetric analysis (TGA) (Figure 1.3) shows the pyrolysed of carbon fibre at temperature 550°C show the stabilization of the organics decomposition under inert atmosphere and no oxidation happen.

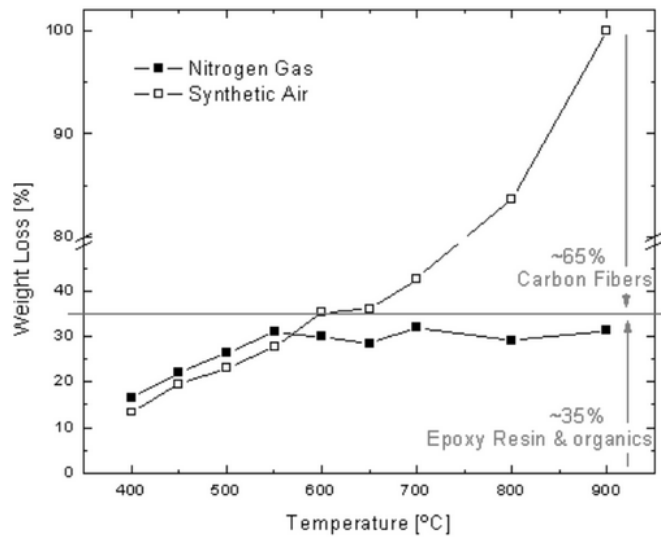


Figure 1.3 TGA curve (Daphiny, 2007).

The temperature of 550°C show the good surface morphology when observed under SEM. As reported by Daphiny (2007), material reclaimed at 550°C still presented organic on the surface (16.5% weight loss only) while compared to temperature 600°C and 700°C. At temperature 600°C and 700°C, SEM show the fibre seemed to have more brittle pyrolytic carbon and promotes the initial crack over some parts of fibres surface (Figure 1.4).

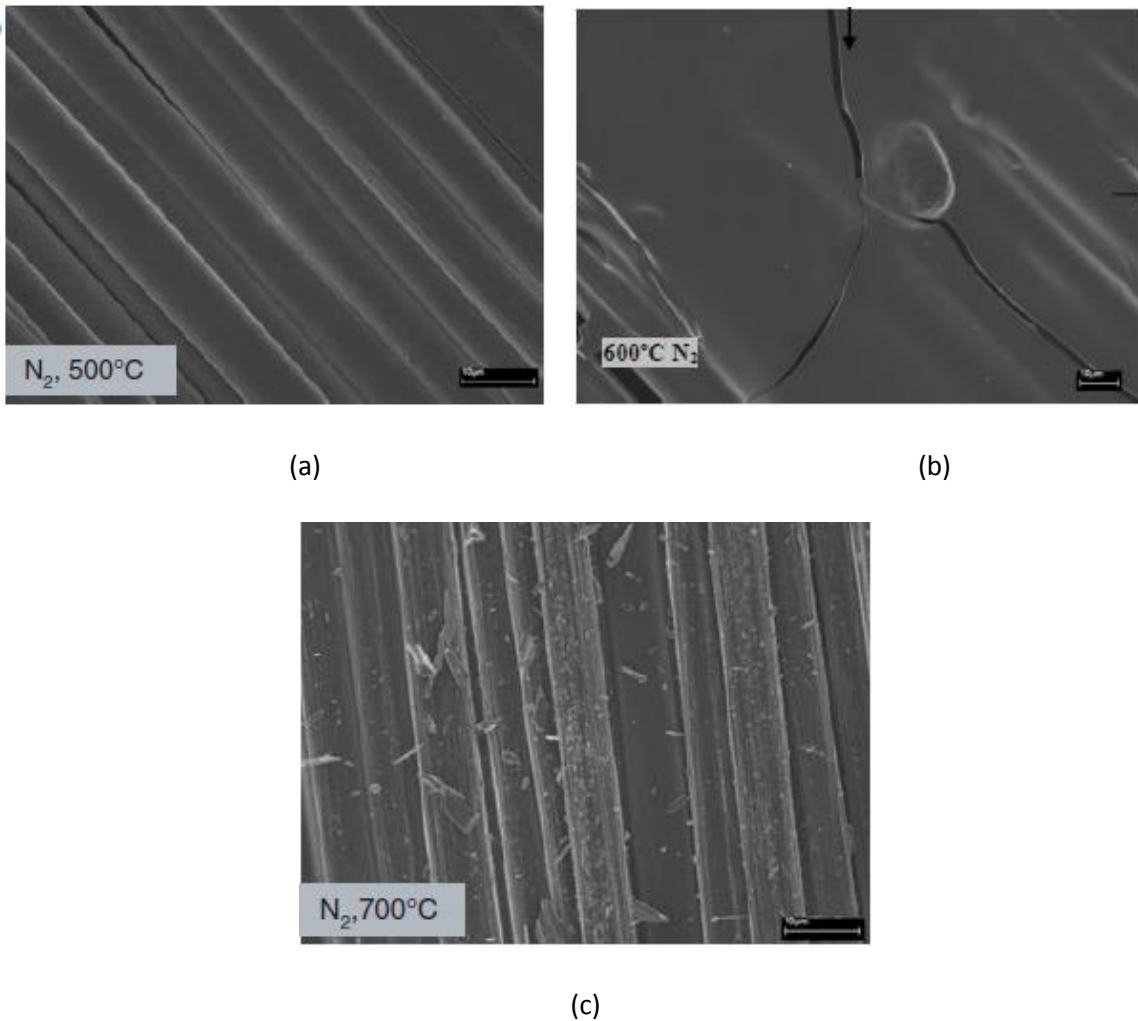


Figure 1.4 SEM at temperature (a) 500°C, (b) 600°C and (c) 700°C under nitrogen condition (Meyer, 2009 & Daphiny, 2007)

The fibre strength degrade which fibre stiffness is usually unaffected or presence of residual matrix. It is known that different dwelling time and temperature affect the quality of rCF. It is important to do a test in order to evaluate its strength and elastic modulus for the materials to be used as a secondary application. The sample of reclaimed carbon fibre were collected from the pyrolysis of previous study. In this study, single fibre tensile tests was carried out from different time taken of reclaimed carbon fibre (rCF) to observe its strength and elastic modulus. Statistical analysis such as Weibull approach was used to predict the distribution of reclaimed carbon fibre because the tensile strength distribution of individual rCF may differ for the ‘bundle batch’ of rCF. The strength of the fibre depends on the gauge length of single fibre testing. However, details of the variation gauge length on reclaimed carbon fibre pyrolysed using lab-scale are not much reported.

1.3 Objectives

- To determine the effect of dwelling time on strength and elastic modulus of reclaimed carbon fibre using single fibre testing.
- To analyze the probability distribution of rCF fractures using Weibull distribution at different gage length.
- To correlate the strength and modulus with damage behaviour using fractography method.

1.4 Scope of Research work

In this study, the reclaimed carbon fibre that has been pyrolysed at different temperature and dwelling time are used to observe its mechanical strength and elastic modulus. Chemical treatment was used to obtain the “fluffy” individual reclaimed carbon fibre. The single fibre tensile testing was carried out to observe its strength and modulus of elasticity. The different gauge length were used to observe the variation of mechanical properties. The strength and elastic modulus was compare between industry-scale reclaimed carbon fibre and lab-scale reclaimed carbon fibre (rCF). The Weibull analysis was used to predict the distribution of strength and modulus of the rCF and SEM was used to observe the morphology of surface damage.

1.5 Significant/ Important of Study

The important of this study is to achieve the objectives stated and problem statement. It is important to know the ability and strength of the reclaimed carbon fibre for the secondary application. Besides, the waste produce by carbon fibre can be reduce when it is been reuse again.

1.6 Organization of the report

This report consist of chapter 1, chapter 2, chapter 3, chapter 4 and chapter 5.

In chapter 1, the part that have been discussed are the background of the study, the problem statement, objectives and scope that narrows down the area of study. The contents are the idea of this research study and the aim to achieve the research study. In chapter 2, the content are focusing on the basic theories based on the research topic and the previous studies obtained from article, journal, conference proceeding, book and the internet. The process, procedure of experiment and method use by previous studies are explained briefly. In chapter 3, the method and the experimental design is been discussed. The content describe on the equipment used, statistical analysis used and the expecting result. In chapter 4, the results obtained from the experiment that has been carried out is discussed to confirm the findings from the previous research. In chapter 5, the content is on the recommendation that can be done in future for further research and improvement.

1.7 Summary

The importance of this chapter is able to understand the basic theories of the topic, state the objectives and problem statement. The objectives and problem statement is clearly been explained above. The scope of research work also been explained with theories and figures given. Next, as for the significance of study aim is to achieve the objectives and solve the problem statement. Lastly, the organization of the report part is described for every chapter.

CHAPTER 2

LITERATURE REVIEW

Several challenges and few parameter of carbon fibre may involve in measurement of mechanical properties such as strength and modulus of carbon fibre due to its different geometry. The experimental testing need to be carried out by carefully handling the single carbon fibre since the diameter of the carbon fibre and gauge length will be different. More research on the mechanical properties, process, testing, and analysis are needed. In this chapter, the issue that has been discussed is about the carbon fibre, pyrolysis, single fibre testing, the parameter of single fibre testing and the statistical analysis involved in this study.

2.1 Introduction

Outstanding mechanical properties of carbon fibre that is low density has make the material widely used at different applications. This is the reason that makes carbon fiber reinforced composites are frequently used in aerospace, aeronautics, sport and recreation goods. Besides, the applications range also can be extended to the automotive industry and become more cost-effective due to more recent development (Vautard, 2014).

Current levels of carbon fibre usage are in excess of 100,000 tonnes per annum with growth forecast to be between 10 % and 20 % per annum (Pickering et.al.,2016). This has shown that the use of carbon fibre increasing until now due to high demand in aeronautics, biomedical engineering field, transportation, sporting goods, construction and another industrial applications.