Titre: Title:	Recycling of CFRP by Pyrolysis under Ambient Air
Auteur: Author:	Ali Jadidinia
Date:	2023
Type:	Mémoire ou thèse / Dissertation or Thesis
Référence: Citation:	Jadidinia, A. (2023). Recycling of CFRP by Pyrolysis under Ambient Air [Mémoire de maîtrise, Polytechnique Montréal]. PolyPublie. https://publications.polymtl.ca/53363/

POLYTECHNIQUE MONTRÉAL

Affiliée à l'Université de Montréal

Recycling of CFRP by Pyrolysis under Ambient Air

ALI JADIDINIA

Département de génie mécanique

Mémoire Présenté en vue de l'obtention du diplôme de *Maîtrise* ès *sciences appliquées*

Génie mécanique

Avril 2023

POLYTECHNIQUE MONTRÉAL

Affiliée à l'Université de Montréal

Ce mémoire intitulé :

Recycling of CFRP by Pyrolysis under Ambient Air

présenté par **Ali JADIDINIA**

en vue de l'obtention du diplôme de *Maîtrise ès sciences appliquées*

a été dûment accepté par le jury d'examen constitué de :

Aurelian VADEAN, président
Rachid BOUKHILI, membre et directeur
Masoud MEHRABIAN, membre

DEDICATION

Dedicated to my family, namely:

My father, Mohammadreza, who has always been supporting and encouraging me, who has always inspired me;

My mother, Raziyeh, for her unconditional love and support, who has given me strength to keep going;

My sister, Samaneh, for always being there for me and supporting me;

And to my brother-in-law, Majid, who has been a guide in my life.

ACKNOWLEDGEMENTS

I would like to express my sincere gratitude to Professor Rachid Boukhili, my supervisor, for his endless support and trust. It was a great pleasure working with Professor Boukhili and learning not only academic lessons, but also lessons for life. Being a part of his research team was the greatest honor for me. This research could not be completed without his wise instructions, supports and patience.

I would like to extend my sincere thanks to Cynthie Dega for her genius helps and supports during the thesis. Cynthie paved the way for this project and her solutions played a key role in this project.

I am also grateful to CTT group and their professional team for providing the experimental equipment and helping with the mechanical tests.

As a final note, I also wish to thank my family for their unconditional support and love throughout my life.

RÉSUMÉ

L'utilisation de matériaux composites se développe rapidement et il existe une forte demande pour ces matériaux sur le marché. Il y a quelques obstacles à l'utilisation croissante : prix élevé des matières premières et difficultés dans la gestion de leurs déchets. Trouver une méthode de recyclage efficace peut surmonter ces difficultés.

L'objectif de cette étude est de trouver une méthode de récupération viable pour les préimprégnés carbone/époxy périmés à l'aide d'un four à usage général afin de minimiser le coût de recyclage et de maximiser la valeur du produit récupéré. Des expériences de pyrolyse dans des conditions d'air ambiant ont été réalisées à des températures allant de 450º C à 600º C. Pour chaque température de pyrolyse, le temps de pyrolyse variait de 1 à 7 heures. Les résultats obtenus montrent que les conditions optimales de pyrolyse étaient de 2,5 heures à 500 ˚C. Une température plus élevée induit une dégradation des fibres, et une température plus basse conduit à un temps de pyrolyse prolongé.

Après avoir trouvé les conditions optimales, des plaques composites ont été fabriquées à partir de fibres de carbone recyclées par la méthode d'infusion de résine assistée sous vide. D'autres caractérisations physiques et des tests mécaniques ont été effectués sur les plaques pour déterminer les propriétés du rCFRP (recycled Carbon Fiber Reinforced Plastics). Les résultats montrent que les plaques fabriquées à partir de fibres de carbone recyclées ont de bonnes propriétés physiques et mécaniques et peuvent être utilisées dans les applications où les propriétés complètes des fibres initiales ne sont pas requises. Les propriétés mécaniques des rCFRP fabriqués se comparent favorablement aux composites fabriqués avec de la fibre de carbone commerciale vierge à usage général utilisant la même résine époxy.

ABSTRACT

The use of composite materials is growing rapidly and there is a high demand for these materials in the market. There are some obstacles for the increasing usage: high price of raw materials and difficulties in their waste management. Finding an effective recycling method can overcome these difficulties.

The objective of this study is to find a viable recovery method for time expired carbon/epoxy prepregs using a general-purpose oven to minimize the cost of recycling and maximize the value of the recovered product. Pyrolysis experiments under ambient air conditions were carried out at temperatures ranging from 450 °C to 600 °C. For each pyrolysis temperature, the pyrolysis time varied from 1 to 7 hours. The obtained results show that the optimal pyrolysis conditions were 2.5 hours @ 500 ˚C. A higher temperature induces fibre degradation, and a lower temperature leads to a very prolonged pyrolysis time.

After finding the optimum conditions, composite plates were manufactured from recycled carbon fibers by Vacuum Assisted Resin Infusion method. Further physical characterizations and mechanical tests were performed on the plates to determine the properties of the rCFRP (recycled Carbon Fiber Reinforced Plastics). Results show that the manufactured plates from recycled carbon fibers have good physical and mechanical properties and can be used in the applications which full properties of initial fibers is not required. The mechanical properties of fabricated rCFRPs compare favorably to composites fabricated with general-purpose virgin commercial carbon fiber using the same epoxy resin.

TABLE OF CONTENTS

LIST OF TABLES

LIST OF FIGURES

LIST OF SYMBOLS AND ABBREVIATIONS

CFRP	Carbon Fiber Reinforced Plastic
PAN	Polyacrylonitrile
EoL	End-of-Life
CF	Carbon Fiber
V.A.R.I.	Vacuum Assisted Resin Infusion
TGA	Thermogravimetric Analysis
SEM	Scanning Electron Microscope
MAP	Microwave-Assisted Pyrolysis
GPM	Graphene Porous Material
LTP	Low Temperature and Pressure
TA	Tartaric Acid
T_g	Glass Transition Temperature
DMA	Dynamic Mechanical Analysis
R.R.T.S	Recovery Rate of Tensile Strength
R.R.T.M	Recovery Rate of Tensile Modulus of elasticity
R.R.C.S	Recovery Rate of Compressive Strength
R.R.C.M	Recovery Rate of Compressive Modulus

LIST OF APPENDICES

CHAPTER 1 INTRODUCTION

1.1 Overview

Nowadays human needs for manufacturing new parts and structures are increasing rapidly and this highlights the demand for new materials. Carbon Fiber Reinforced Plastics (CFRPs) provide numerous advantages that include but are not limited to high stiffness, high tensile strength, low weight to strength ratio, low thermal expansion, good chemical, abrasion and corrosion resistance, and these are the reasons for their extensive use. Reduced cost and time of manufacturing are among the other prominent properties of these materials.

Starting from the recent decades, composites consisting of carbonous fibers address the demands of different domains, from aerospace industries to wind, automobile, construction, sports facilities, etc. Since their emersion, they have played a key role in the market of high-quality products and they are replacing other materials that have been used for a long time, i.e., metals and alloys (steel, aluminum, etc.) [1-4]. According to [5], nearly 120,015 tons of carbon fibers have been used in 2020, indicating an 11% increase compared to 2019. Estimations show that the demand for CFRPs will be 194,000 tons in 2022 [6]. Figure 1.1 shows the global demands for carbon fibers and CFRPs in different continents.

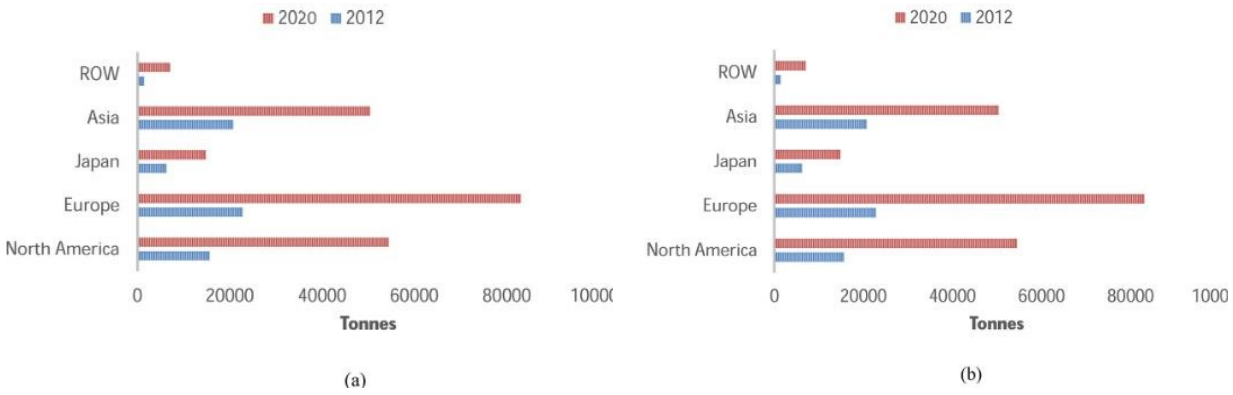


Figure 1.1: Global demands for (a) Carbon fiber and (b) CFRP in different regions of the world [7]

Despite the advantages that CFRPs provide, there are certain obstacles and challenges in using, or in other words, manufacturing composites. The main limitation in this way is the high price of virgin carbon fibers. The mechanical properties of carbon fibers depend on the type of precursor

and currently polyacrylonitrile (PAN) is the widely used precursor used for production of carbon fibers. PAN needs to undergo over 1500° C in order to change into carbon fibers. Clearly providing that high temperature requires big energy sources and money [5]. Because of these high expenses of production, the use of CFRPs in most cases is limited to high-value applications rather than high-volume productions. By the use of recycled carbon fibers and replacing the high production costs with lower recycling expenses, we can benefit more from the advantages of CFRPs in daily life.

The other restricting issue with the use of CFRPs is waste management. There are two main causes of composite waste. The first one is the scraps produced when manufacturing the bigger parts and the other source is the end-of-life (EoL) parts [8, 9]. As mentioned earlier, the most portion of composites used nowadays is devoted to aerospace, wind and automobile sectors. These are mostly critic parts and need to be replaced and occasionally changed. Consequently, there are massive amounts of composite wastes generated. It is predicted that the amount of CFRP wastes will be more than 263 kilotons in 2030 [10].

Currently, there are three traditional ways of treating composite wastes, disposing in landfills, incineration, and recycling. Great portions of the wastes are being disposed in landfills, as it is the cheapest way of getting rid of them. But there are certain environmental concerns and researchers are constantly trying to find alternative methods. When the organic material is put in the landfill and is decomposed, methane is released which is one of the greenhouse gases that is proven to have negative impacts on global warming and climate change. The development of landfills also endangers the lives of special species, known as the biodiversity impacts. Groundwater pollution and soil fertility effects are the other adverse effects of landfills [11, 12]. On the other hand, incineration is an expensive method to discard wastes. Despite the efforts to minimize the pollutants in the incineration process, some toxic and hazardous emissions are dangerous for human beings [13]. Therefore, finding a suitable and optimal way for their recycling is of great importance and is one of the hot topics among scientists.

Different methods of recycling composite wastes have been used so far. Among these methods, conventional pyrolysis is the most promising procedure for meeting the needs of recycling and recovering fibers from composite wastes [5, 10, 14-16]. Pyrolysis is defined as a process of thermal decomposition, which mainly takes place in an inert atmosphere at temperatures ranging from

400 C to 1000°C for different periods of time. During Pyrolysis, the polymeric resin is completely decomposed to volatile materials and the fibers are recovered. It is also possible to collect volatile as liquid by-products, such as tar, which can be used as oils and fuels. Other gaseous products like carbon dioxide and methane with lower calorific values can also be utilized as energy resources [1]. It has been shown that pyrolysis of CFRPs under an inert atmosphere, although preventing carbon fiber degradation does not yield clean carbon fibers due to incomplete decomposition of the polymer matrix and hence remaining resin residues. Therefore, pyrolysis under an inert atmosphere must be complemented by pyrolysis under an oxidizing atmosphere in order to obtain clean fibers [17]. The use of two stages (1-inert and 2-oxidative) pyrolysis for carbon fiber recovery is not cost-effective and makes the recyclability of CFRP less economically viable. It should be noted that oxidative pyrolysis could be carried out with ordinary furnaces. However, conventional pyrolysis requires a controlled atmosphere furnace that uses inert gases such as nitrogen. In order to keep the recyclability of CFRPs economically viable, this study proposes to use ordinary furnaces with an optimized temperature-time isothermal pyrolysis cycle.

1.2 Objective

The main objective of this research is to provide a viable recovery method for expired carbon/epoxy prepregs. The main objective can be achieved through these specific objectives:

Sub-objective 1: Determine the optimal time-temperature pyrolysis cycle to recover full carbon fiber (CF) rolls from expired composite prepregs using a general-purpose oven.

Sub-objective 2: Manufacture composite laminates with recovered carbon reinforcement fabrics.

Sub-objective 3: Characterization of the physical and mechanical properties of the composite laminates manufactured from the recovered carbon reinforcement fabrics.

1.3 Thesis Organization

This thesis includes 5 chapters, which are categorized as below:

- Chapter 1: Introduction
    - ✓ A short overview of the project domain and the importance of the research is presented.

- ✓ The main objective of the study is explained.

- Chapter 2: Literature review
  - ✓ A short summary of different recycling methods and some of the research done is presented. The focus of literature review is on pyrolysis.
- Chapter 3: Methodology
  - ✓ Explanation of pyrolysis process with a general-purpose furnace.
  - ✓ Description of different steps for manufacturing process vacuum-assisted resin infusion (V.A.R.I) used in the research.
  - ✓ Specifications of mechanical tests performed on manufactured composite plates made from recycled carbon fibers with their relevant standards.
- Chapter 4: Results and Discussion
  - ✓ Explanation of the optimum condition suggested for the material and summary of mechanical test results on the final samples.
- Chapter 5: Conclusion, Limitations and Recommendations
  - ✓ Summary of the results and explanation of the objective of the research obtained.
  - ✓ Explanation of limitations in the project.
  - ✓ Recommendations for the future Work on the subject.

CHAPTER 2 LITERATURE REVIEW

2.1 Methods for recycling composite materials

There are many methods for recycling composite materials. Although there are various classifications for recycling, but these methods mainly fall into three different categories: mechanical, thermal and chemical methods.

2.1.1 Mechanical Recycling Methods

Mechanical recycling is a practical procedure to manage the large volumes of composites and includes different steps to reduce the size of composite parts and put them in smaller pieces. Different steps of this method comprise shredding, crushing, grinding and milling of wastes. First, the waste material is shredded into parts of 50-100 mm. The shaft shredding makes uniform pieces by controlling the distance between the blades, the sieve size and the speed of the rotation [15, 18, 19]. These pieces are then put in a grinder so that they can be transformed into pieces (also called recyclates) which are collected in the bins. Finally, by the use of cyclones and screens, the recyclates are separated according to their sizes: coarse recyclates with higher fiber content, and finer pieces that are richer in resin content. Obviously, the complete separation of fibers and resins is not achievable by this method. The finer parts, which have higher content of resins, are used as fillers, but since the materials used as virgin fillers (Silica or Calcium carbonate) are cheap, there is no economic advantage in using them. It must also be considered that there is a deterioration in mechanical properties, but since they contain some amounts of light polymers, they have lower density compared to virgin fillers. Recyclates can also be used as partial reinforcement of composites. Because of the small fiber aspect ratio, the powders produced by this method are not highly demanded in the market and therefore are not very valuable [6].

Considering the nature of this method, the rate of abrasion and erosion in the machinery is relatively high, which results in high operational costs. All these reasons, along with the degradation of mechanical properties and their unstructured architecture, limits their use for remanufacturing composites [20, 21]. There have been research activities in recycling with mechanical method for both carbon fibers and glass fiber composites, but most of the studies are done on glass fiber composites. It might be a consequence of their low-value applications [22, 23].

Shuaib et.al [24] investigated the roles of different process parameters in mechanical recycling. They showed that the clearances between the blades and the sieve mesh size affects the recyclate' quality and the energy demand of the process. Smaller screen sizes lengthen the residence time of the material in cutting chamber that consequently increases the total energy consumption. They also studied the effect of material thickness in energy demand and the efficiency of the cut material. It was revealed that thick materials result in more useful and tidier recyclates. Although larger screen meshes decrease the power consumption, but the rate of coarse particles increases which requires some extra steps. Colucci et.al [25] studied final mechanical properties of artificially aged polyamide 66 composites that were made of shortened carbon fibers caused by mechanical recycling. At the beginning, the PA66 granules containing 30 wt.% carbon fibers of 0.3 mm length were melted and composites were manufactured in the forms of bone-like tensile tests. For simulating the aging process, they underwent an accelerated ageing process, which was under UV with different thermal and moisture conditions. Later on, a recycling process was performed in order to obtain recycled composites. A decrease in the elastic modulus and tensile strength was observed. The length of carbon fibers in manufactured parts was shorter, but they reported that it has an insignificant impact on properties of the manufactured parts.

2.1.2 Thermal Recycling Methods

Thermal recycling methods require high temperatures and fluids. The fundamental of this method is to degrade the polymers by heat and change them into other solid, liquid or gaseous products. In this method, fibers are released from the cross-linked matrices and are therefore recovered. Pyrolysis and fluidized bed methods are the two highly deployed procedures categorized in this method.

2.1.2.1 Pyrolysis

Pyrolysis is the most promising procedure for meeting the needs of recycling and recovering fibers from composite wastes [14, 26, 27]. Among the technologies for recycling, pyrolysis has been proven to be one of the very attractive and popular methods. The main reason for this popularity is the ease of parameter control and adjustment to ensure the best optimized results of the experiments, as well as relatively low cost of the method.

Pyrolysis is defined as a thermal decomposition process, which is mostly done in an inert

atmosphere, without the presence of oxygen. In this method, the composite material is gone under high temperature, mostly between 400 and 1000 °C for some hours. As a result, fibers are released and separated. The removed resin produces gas, tar or char; hence considering the high calorific value of heavy liquid by-products, like tar, they are used as oils and fuels, other gaseous products like carbon dioxide and methane with lower calorific values can also be utilized as energy resources [1, 28]. Normally, the temperature required for degradation of polyester resins is lower than the epoxy resins. Degree of conversion of the resins is the most influential factor in choosing the proper temperature for pyrolysis, there are vast improvements and studies conducted on this method, and nowadays it is used as an acceptable recycling method in industrial scale. Schematic of pyrolysis method is presented in figure 2.1.

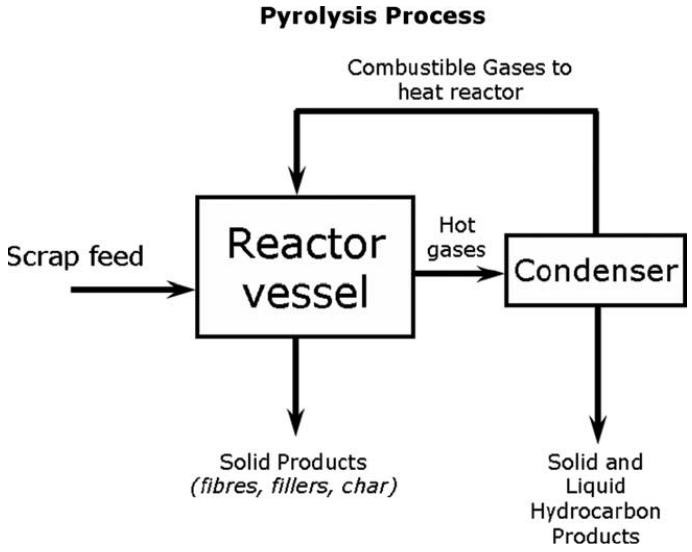


Figure 2.1: Schematic of Pyrolysis method [1]

There have been numerous efforts in optimizing the pyrolysis process by adjusting different elements and many articles have been published on this domain. In one of the research, L.O. Meyer et.al [14] tried to find the optimal parameters in pyrolysis process and produce recycled fibers with properties close to virgin fibers. The parameters studied in this study were pyrolysis temperature, dwelling time and oven temperature. The material was waste prepreg-type Hexply 913C/HTA that was generated during airplane production. Thermogravimetric analysis (TGA) and Scanning Electron Microscope (SEM) were used as the main tools to evaluate the quality of recycled fibers. The experiments were performed in two atmospheres, nitrogen and synthetic air. The rate of weight loss at different temperatures, and the effect of isothermal dwell time in weight loss of samples are shown in figure 2.2 and figure 2.3, respectively.

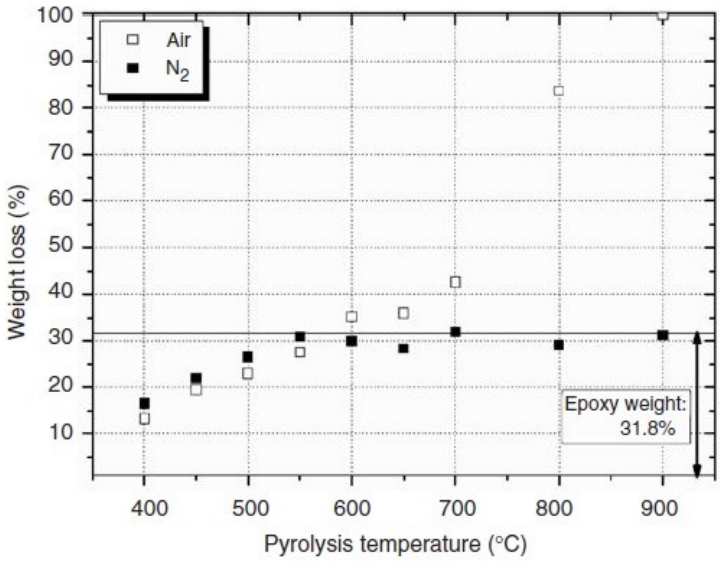


Figure 2.2: Weight Loss percentage of prepreg samples at different temperatures [14]

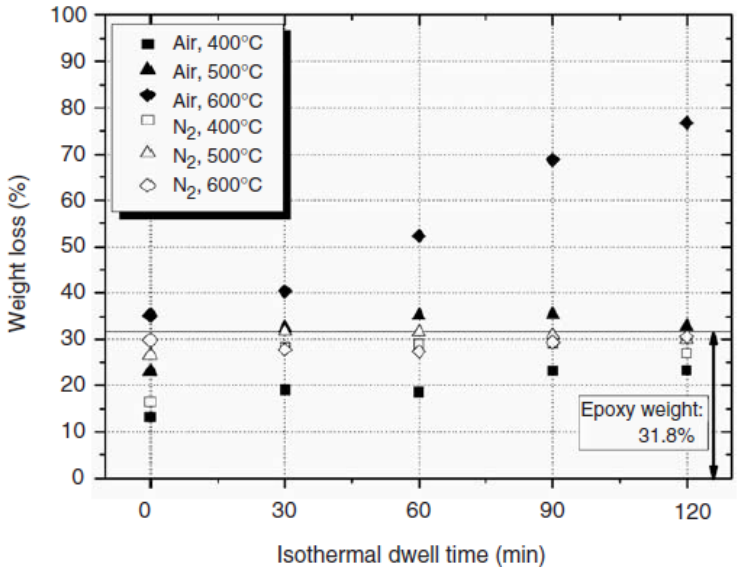


Figure 2.3: Weight Loss percentage of prepreg samples at different temperatures [14]

Finally, they did a pyrolysis test run, according to the optimal conditions, and performed fiber tensile test with at least 50 fibers. They reported that there is a reduction of 3.9% in fiber strength of pyrolyzed samples and 11.1% loss in modulus.

In another study, T.R. Abdou et.al [29] investigated recycling of HTS40 E13 3K carbon fibers mixed with polymeric matrix composed of epoxy resins. For this reason, they tested virgin carbon fibers and the polymeric composite. TGA and SEM were used for evaluating the properties and

surface of the recovered fibers. For finding the suitable pyrolysis atmosphere, they put virgin fibers inside the oven for temperatures ranging from 20° C to 700° C, under an inert atmosphere. They reported no great degradation of carbon fibers, the maximum mass loss was 9%, and there were no traces of water, CO, and CO_2 in the released gases. When the virgin fibers were put in a furnace in air, they reported mass losses equal to 14.5% and 70.7%, for 500° C and 600° C, respectively. It proves the degradation of carbon fibers in an oxidative atmosphere.

They presented the graph of mass loss vs. temperature for the composite in figure 2.4. They reported that the degradation happens in two steps, one at 268° C, and the other at 410° C. In the first step, chains and impurities that are not cured properly degrade, and in a second step, degradation is completed.

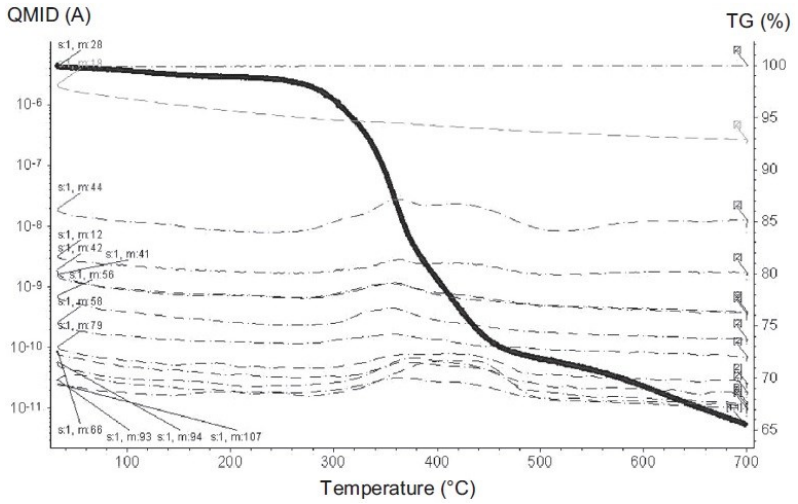


Figure 2.4: Mass loss vs. function of temperature in an inert atmosphere [25]

Afterwards, they did the pyrolysis test under argon atmosphere. They reported that carbon fibers were recycled at 550° C for 1 hour, and the higher temperatures cause degradation of carbon fibers.

S.A. Hadigheh et al. [10] tried to optimize the pyrolysis with studying the kinetic behaviors of the process in temperatures up to 800 °C. To study the quality of the process and fibers, they used TGA The reported that almost 55% of the matrices are removed in the first step which is up to 425° C and after that, an oxidation process, up to 550 °C is very effective in having pure recycled carbon fibers. According to their report, the second step of decomposition of the matrix was less effective in removing the residual matrices.

Some researchers have tried to make some modifications on the conventional pyrolysis method.

Jie Yang et al. [30] tried to investigate the effects of oxygen concentrations on recycling of carbon fiber reinforced epoxy resin composites along with the role of temperature. They used epoxy resin E-51 and PAN-based carbon fibers at a mass ration of 100:28 for manufacturing composites by vacuum bagging method. According to the digestion test, they did on the specimen; fiber content was reported wt56%. The schematic diagram of their deployed reactor is as figure 2.5.

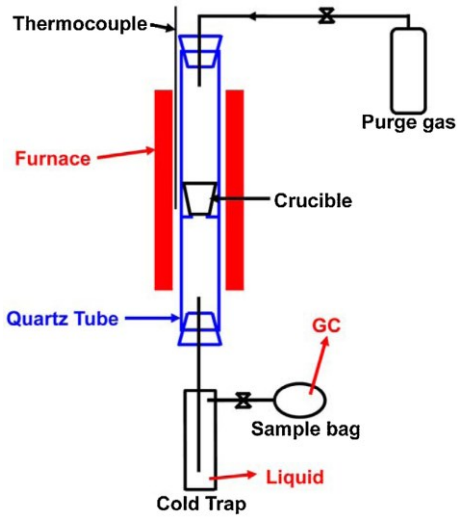


Figure 2.5: Schematic overview of the reactor used in [29]

The samples were put on the porcelain crucible, centered at the center of the Quartz tube. Different combinations of atmospheres, including N_2, air, and mixtures of N_2 and O_2 were used to study the effects. Liquid and gaseous products of the pyrolysis were gathered in cold trap and sample bag, respectively. Finally, and based on the TGA results conducted by them, they chose 550° C, 600° C and 650° C as the experiment temperatures and a mixture of 5% O_2 and 10% O_2 with Nitrogen were the reactant gases. Figure 2.6 is the summary of their results. According to the figure 2.6, the increase of oxygen concentrations results in a lower residue obtained and the residue rate in 550° C is closest to the real fiber content of the sample. They also reported that the effect of temperature in different atmospheres is different, and there is not a unique relation between them. They reported that in a good experiment condition, almost 80% of tensile strength and modulus could be recovered.

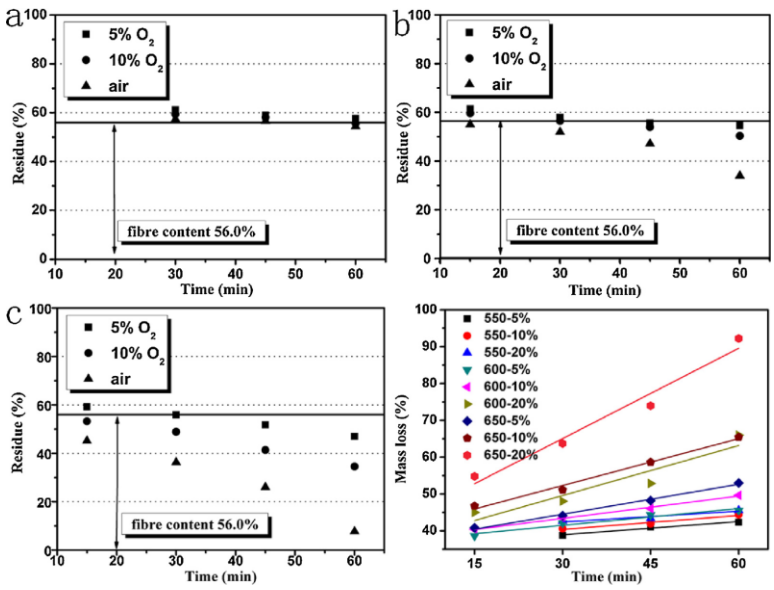


Figure 2.6: Residue percentage of samples at different temperatures: (a): 550° C, (b) 600° C and (c) 650° C [30]

Jin-Soo Jeong et al. [31] used superheated stream as an oxidant for recovering carbon fibers from composites in a short time. They manufactured their sample by hot pressing at 175° C, by mixing DGEBA YD-128 resins and polyacrylonitrile monofilament carbon fibers which resulted in a composite with resin content equal to 36%. They put their sample on an alumina container inside a furnace, and the superheated steam was purged at the rate of 1 ml per minute. The summary of recovery rates is as figure 2.7.

	600–60	650–60	700–60	750–60	800–60
Initial weight (g)	0.47	0.48	0.47	0.47	0.47
After pyrolysis (g)	0.32	0.32	0.30	0.24	0.08
Recovery rate (%)	68.09	66.67	63.83	51.06	17.02

Figure 2.7: Recovery rates for different experiment conditions [31]

Their results confirmed the decrease of the recovery rate with the increase in temperature, and according to their results and the initial resin content, 700-60 was an optimum condition for removing the resins completely and having clean and recycled fibers. They also tried to measure the diameter of the fibers to see the changes as a result of recycling process. Based on the SEM images, the smallest diameter was reported 6.69 ηm, compared to the 6.98 ηm initial diameter. They suggested that the diameter of the fibers might even be severely reduced with the increase of the temperature. They also tried to compare the mechanical properties of the recycled carbon fibers

with the virgin carbon fibers. The lowest measured values for tensile strength and modulus were 1.53 GPa and 159.04 GPa, respectively which occurred at 700˚ C, and the highest values were 2.68 GPa and 197.05 GPa, which were measured in 800˚ C. The original tensile strength and modulus of virgin fibers were 4.08 GPa and 195.17 GPa, respectively. Therefore, the minimum reduction in tensile strength was 34%, while for the modulus, the maximum recovered value is almost the same as the virgin ones. For explaining the maximum values happening at 800˚ C, they reported that as temperature increases, in spite of pyrolysis of the resins and etching of the fibers, all defects and amorphous parts of the carbon fibers also disappear, and that explains the high strength and modulus of the sample. They also did some tests to measure the interfacial shear strength, while the virgin carbon fibers were measured 28.74 GPa, while the highest measured value after pyrolysis was 34.20 GPa, which shows almost a 19% increase. The enhanced surface functionality and etching with the removal of the resins, help increase the shear strength as the process starts which also increases the surface roughness of the carbon fibers, but as temperature rises to 750-800˚ C, some functional groups of the surface are not able to withstand the high temperature, which results in a reduced interfacial shear strength. Changes of mechanical properties for their experiment is as figure 2.8.

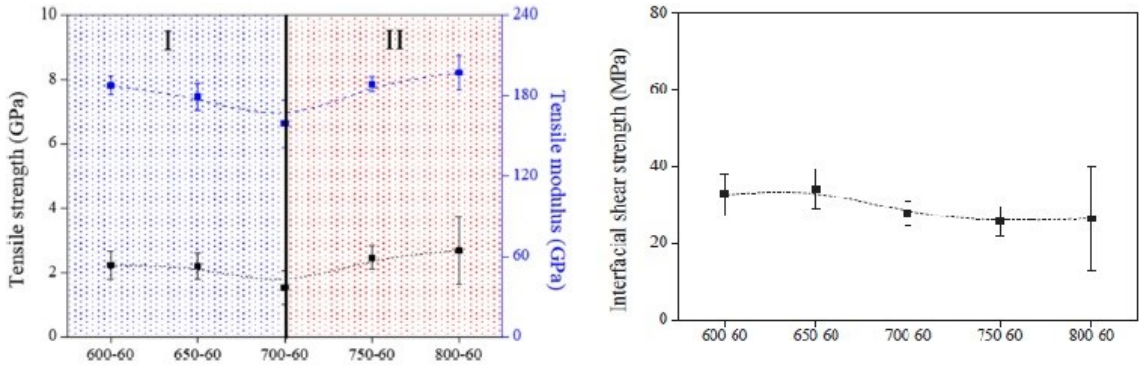


Figure 2.8: Changes of mechanical properties in a single recycled fiber in different experiment conditions [31]

2.1.2.2 Microwave-assisted Pyrolysis

In order to overcome the rather high energy demand and long processing time of conventional pyrolysis, using microwaves in pyrolysis method for degrading resins and recovering fibers has been of great interest for researchers in the recent years. In this method, electromagnetic energy is absorbed and is changed into heat throughout the volume of the material. The main superiority of

this method is the fast heat transfer, which results in reduced energy demands. Because of volumetric heating nature of MAP (microwave-assisted pyrolysis) method, it is considered as one of the methods with least energy consumption [32-35]. Compared to conventional pyrolysis, it produces a uniform heating, and is counted as a faster, more efficient and more environmentally friendly recycling method [36]. Besides the advantages of microwave heating, there are certain drawbacks of this method, including uneven energy distribution and inconsistency, arcing, tooling design and also the quality of the parts [37]. The same as the conventional pyrolysis, this procedure is also done in an inert atmosphere and gaseous and liquid products are collected after the experiment.

K. Obunai et al. [38] studied the effects of different atmospheres and field intensity on the microwave -assisted pyrolysis process. The atmospheres they studied were argon, nitrogen, and the air. They used a 700 W apparatus with the frequency of 2.45 GHz, and a fiber-type spectroradiometer for measuring the emission spectrum, as in figure 2.9. For better controlling the experiment atmosphere, the sample was put in a quartz glass chamber that its position was changed to see the effects of field intensity. They reported that resin elimination rate rises almost in a linear relation with respect to the irradiated field intensity, and the samples showed better results under argon and air atmospheres, rather than nitrogen.

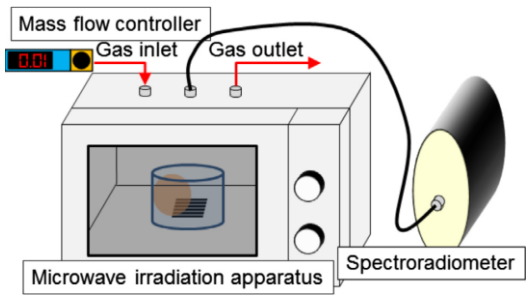


Figure 2.9: Schematic of the microwave apparatus used in [38]

Figure 2.10.a represents their results. Figure 2.10.b shows the resin elimination rate in different times and different environments. The results show a very high rate of resin elimination in the first 60 seconds, after that, and until 300 seconds, it shows an almost constant rate. According to their results, 100% of the resins are eliminated under air atmosphere in 300 seconds.

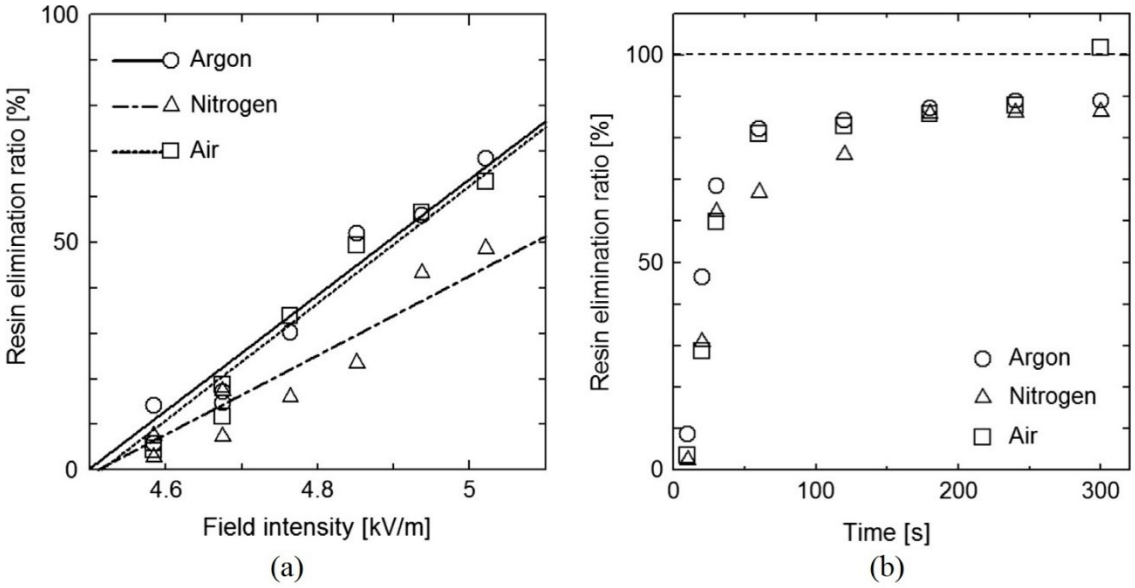


Figure 2.10: (a) Resin elimination ratio changes with respect to Field intensity, (b) Resin elimination ratio changes with respect to Time [38]

Deng et al. [36] compared the results of traditional pyrolysis and the microwave pyrolysis. They used a waste CFRP sample of thickness 3 mm, with the carbon fibers of TC33-3K, and E51 epoxy matrix. They used a 2.45 GHz microwave under an oxygen atmosphere. The sample was put inside a mullite crucible, which was insulated by a polycrystalline mullite fiber cotton-based chamber inside the reactor of the microwave. On the other side and for the comparison, they also studied the pyrolysis of the sample inside a furnace with oxygen atmosphere. The summary of the samples' weight loss in their work is as figure 2.11. Considering the initial 38wt% of resin content in the sample, they reported that the optimum temperature for their case is between 400˚ C and 450˚ C. Their work shows that the required time in microwave assisted method is reduced by 56.67%, which also has a better recovery ratio compared to the traditional pyrolysis.

Sample	Temperature/(°C)	Time/min	Average Power/W	Heating Method	Weight-Loss Ratio/%
1	400	30	850	Traditional	21.89
2	450	30	1060	Traditional	56.50
3	500	30	1300	Traditional	96.12
4	450	13	500	Microwave	47.03
5	450	30	500	Microwave	63.76

Figure 2.11. Weight loss ratios obtained in [36]

In another study, Siqi Hao et al. [39] after recovering carbon fibers by microwave in different temperatures, analyzed the gaseous and liquid products, and also compared mechanical properties of the virgin and recovered fibers. They used T700 carbon fibers and Bisphenol-A epoxy resin, with the resin content of 37 wt%. They used a 2.45 GHz microwave and nitrogen as the inert atmosphere. Based on their TGA results, they found that there is no chance of having clean fibers without any chars under a nitrogen atmosphere. Solid, liquid and gaseous products of the process and their changes with temperatures are listed in figure 2.12.

Temperature (°C)	Solid Residue (wt.%)	Char * (wt.%)	Oil (wt.%)	Gas (wt.%)
450	69.12	6.12	17.71	13.16
550	67.55	4.55	19.15	13.30
650	65.93	2.93	20.28	13.79

* Char is calculated by solid residue minus fibre weight fraction (63 wt.%).

Figure 2.12. Microwave products and their changes with temperature [39]

They reported that the increase in temperature yields in a decreased chat, consequently an increased in oil and gaseous products. After testing the mechanical properties of the samples, they reported that with increase in microwave temperature, there is a 13-20% drop in tensile strength, and for the tensile modulus, the reduction is almost 10%.

Zhang et al. [40] developed a new method for recycling CFRPs and could increase the temperature of the experiments by up to 1000 °C. They added graphene porous material (GPM) to the process which can produce high temperatures while being under microwave irradiation. GPM was prepared by dipping alumina fiberboard in graphene oxide aqueous solution. The reason for the high obtained temperature is the generation of delocalized electrodes by graphene on the surface of alumina fibers, these electrodes then ionized nitrogen between micropores of alumina fiber into plasma. CFRP specimens were put on GPM inside the Quartz tank under nitrogen. Their results show that the temperature of the GPM reached 1000 °C after 210 seconds in a 900 W microwave oven. Their results show 23% increase in the Young`s modulus and 12% decrease in Tensile strength.

2.1.2.3 Fluidized Bed

Fluidized beds have been used in different industries, like catalytic cracking, gasification, drying and transportation, and this is mainly because of its ability in heat transfer and the precise

controlling of the temperature. So, benefitting from these advantages, it has also been used as a thermal recycling method that for recovering carbon fibers and also glass fibers. In this technique, a bed of silica sand (with the size <1 mm) is fluidized by hot air, that is normally between 450˚ C and 550˚ C, under 10-25 kPa pressure and helps heat the samples in a fast way. The main drawbacks of this method are pollutant gases and the high-power demands [6]. As pyrolysis method, the temperature is chosen based on the experiment condition and the sample type, so that the resins are degraded completely, and the fibers are recycled and separated. The schematic of this process is illustrated in figure 2.13. The presence of oxygen in hot air is useful for decreasing the char products on recycled fibers, in other words, the pyrolytic char that is generated during degradation of the matrix is oxidized and therefore the fiber surfaces are clean and free of them [41].

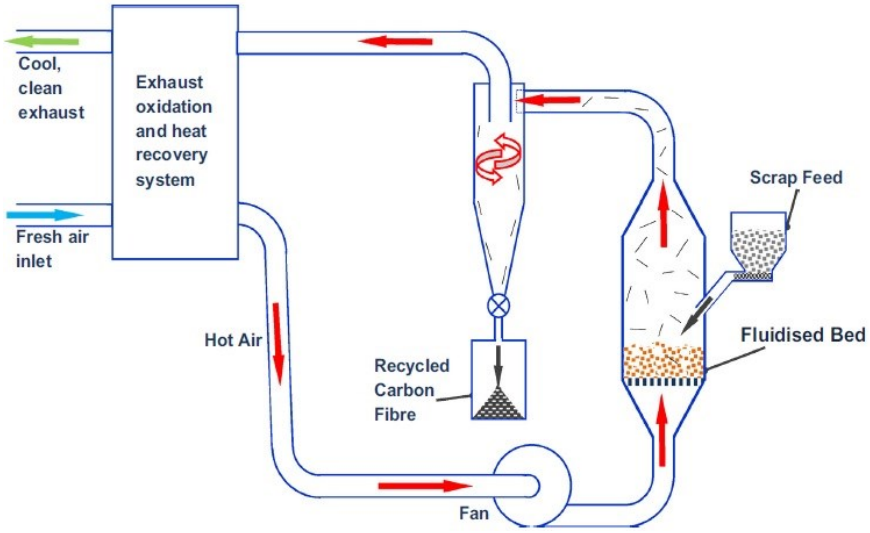


Figure 2.13: Schematic of fluidized bed process [42]

In the heat recovery unit, the gas stream is heated again so that all volatizing gases are oxidized, and the generated heat is used to raise the temperature of the incoming air.

A pilot plant at University of Nottingham for recovering fibers using fluidized bed method was run in 2015. Exhaust gas treatment and heat recovery units were also included in their plant. There were three size reduction steps in their unit, the first one was manual, and the second unit provided 100 mm samples by using a twin shaft shredder, and the third unit prepared samples for the process by using a hammer mill. The energy use for secondary size reduction was 0.04 MJ/kg, while for the third unit it was equal to 0.22 MJ/kg. The maximum input length of the fibers in their plant was 25 mm.

Their plant was designed for having yearly output of 50 to 800 tonnes, and the feeding rate was between 5 and 220 kg/h. figure 2.14 shows the summary of energy demand with respect to feeding rate. Recycled fibers in their units show a reduction of 20% in tensile strength.

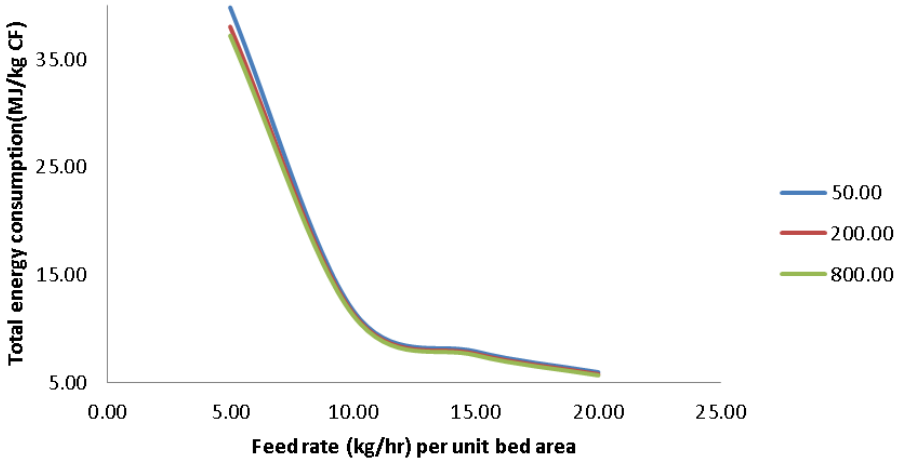


Figure 2.14: Total energy demand per feeding rate for pilot fluidized bed unit [43]

K. Pender and Liu Yang [41] studied the effects of metal catalysts in a fluidized bed system. They proposed that to help decomposition of the matrix and also lower energy demands, oxide catalyst can be added to the system. This modification can decrease the required temperature for the process, which consequently will decrease the energy and cost demands. For their experiment, they mixed PRIMEE 27 epoxy resins and PRIME 20LV hardener with CuO nano-powders with the size 50 nm. After mixing the nano-powders by hand, ultrasonic mixing was also utilized. For finding the effects of these nano-powders, their amount was chosen 0, 1.5 and 5wt%. Based on TGA reports by them, the second and the complete degradation temperatures are noticeably reduced with the presence of CuO nano-powders. Figures 2.15.a and 2.15.b are the result of their TGA test, while figure 2.15.c shows the effects of CUO content n the temperature. They also reported that adding 5wt% of CuO nano-powder does not make any changes on the tensile strength and tensile modulus of the epoxy and the composite but reduces the strain at break. They concluded that by adding 5wt% of CuO to the epoxy, the temperature required for complete decomposition of the epoxy was reduced by 60˚ C. By studying the kinetic energy, they also concluded that CuO reduces the activation energy requirements for the second phase of degradation.

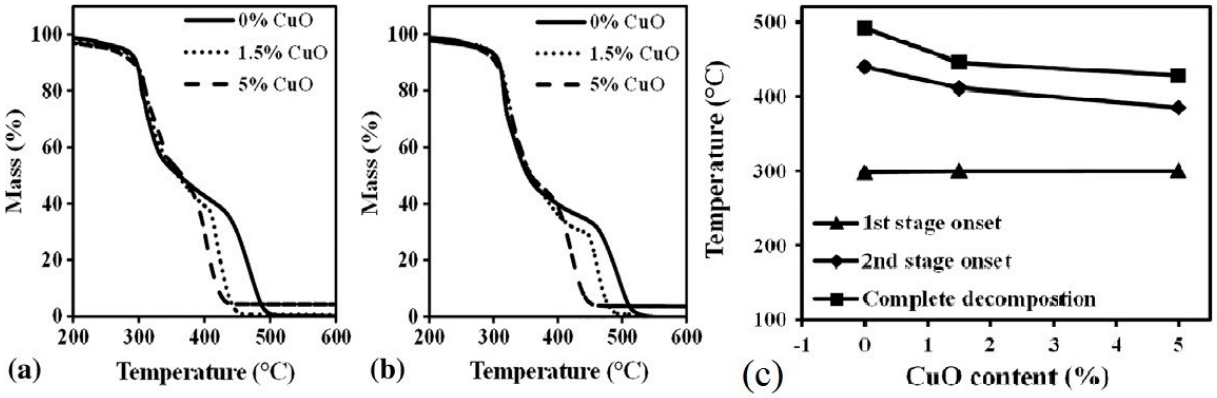


Figure 2.15: (a) TGA reports for epoxy degradation at heating rate of 1˚ C/min, (b) TGA reports for epoxy degradation at heating rate of 2˚ C/min, (c) The effect of CuO content on temperature requirements for heating rate of 1˚ C/min [41]

S.J Pickerling et al. [44] developed a combustion process according to the figure 2.16. They used a rotating sieve separator for removing the inorganic parts, like metal and have high value fibers collected separately. This separator allows the shorter fibers and fillers pass, therefore longer and valuable fibers are kept. These smaller parts are later collected in a cyclone. They also utilized a second combustion chamber in higher temperature (1000˚ C) for the complete degradation of the matrix. For preheating, they used a 43KW heater, bed diameter was 312 mm with the depth of 150 mm. For showing the ability of this method to recycle a wide range of products, they used three different scraps, SMC casing, pipe, and an E-glass/polyester sandwich panel. Mechanical testing of the recycled SMC casing revealed that at 450˚ C, the tensile strength was reduced by 50%, while for 550˚ and 650˚ C, the reduction ratio was 80% and 90%, respectively.

2.1.3 Chemical Methods

In chemical recycling method, during chemical reactions, polymers are converted into monomers and oligomers and the resin in composite wastes break by dissolving in a solvent. This method is mostly applicable for CFRPs and the type of solvent and chemicals are selected based on the composition of the polymers inside composites. Usually, the recycled fibers are washed after the process so that the small residues would be removed from their surface. The wise choice of solvent type, reaction time and its concentration are very important in properly depolymerizing the matrix.[45, 46] In some cases that water is used to dissolve the resin, the process is called hydrolysis. In supercritical conditions, the use of chemicals is harmful and has severe adverse environmental impacts; in this case, water and alcohol must be used instead of chemicals.

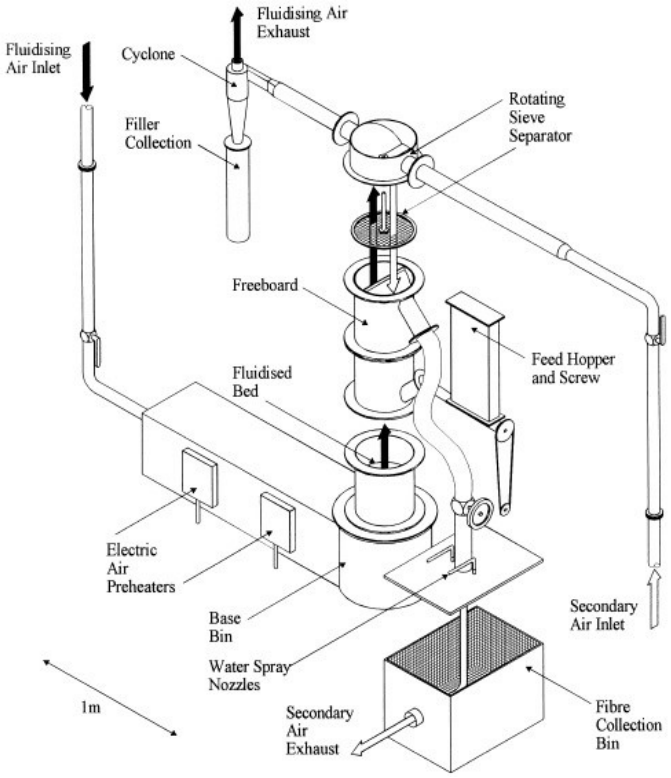


Figure 2.16. Experimental setup for fluidized bed recycling method used in [44]

Because of numerous available solvents, and a wide choice of temperature, pressure and catalysts, there are many possibilities and states of using this method. The main advantage of this procedure is the generally low temperature requirements for degrading the polymers, but on the other side when there is a supercritical situation, the costs of reactors for reaching high temperatures and pressures are high. Sometimes the solvent is mixed with other co-solvents, so they penetrate the composite waste and break the bonds, so this method avoids char products on the fibers. Generally, epoxy resins are more difficult to solvolyze compared to polyester resins, and therefore require higher temperature. So far, there have been numerous lab-scale studies done on this issue, but there are only a few industrial applications for this method until now.

Solvolysis is mostly classified into two different categories, based on the temperature and pressure requirements of the process, high temperatures (>200° C) and pressures also known as supercritical fluids, and low temperatures (<200° C) and pressures (LTP).

2.1.3.1 High temperature and pressure chemical method

Supercritical fluids show excellent solvolysis properties, and because of that, they have gained lots

of interests in recent years. Supercritical fluids have properties between liquid and gas and therefore benefit from some advantages of both. They have low viscosity like gases, density like liquids, excellent diffusivity and dissolving abilities that make them suitable for using in recycling composites [15, 47]. Therefore, they give many choices of solvent properties and the necessary time for reaction. Supercritical critical water in temperatures above 373° C and pressures above 22 bar is a good example of these categories of solvents. Sometimes to reduce the supercritical temperature of water, some catalysts are added to it.

Hernanz et al. [48] set up a supercritical water batch reactor, with the capacity of 10 mL for chemical recycling of CFRPs, which consisted of T600 carbon fibers and MTM 28-2 epoxy resins. They changed the temperature from 523 to 673 K, and the pressure range was 4.0-27 MPa. They considered different reaction times, from one to 30 minutes. According to their results, changes in temperature is the most important factor that affected the results of resin elimination by 80%. Reaction time was the other important factor, changing the results by 43%. For studying the effects of catalysts, they added alkali to the experiment, and found that it increases the resin elimination rate from 79.3wt% to 95.4 wt%. The comparison result is presented in figure 2.17. Mechanical properties of the recycled fibers were also studied, and they reported that tensile strength of the fibers was only decreased by 2-10%, compared to the virgin fibers.

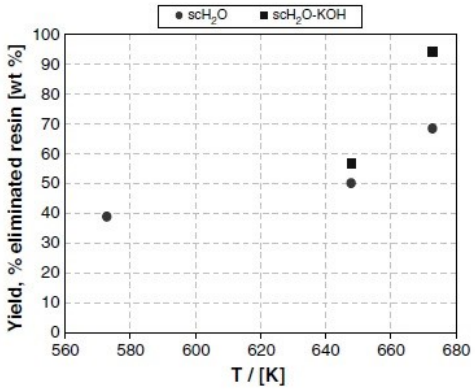


Figure 2.17- the effects of alkali catalyst on resin removal results [48]

J. Kieith et al. [49] studied a supercritical acetone/water solvent mixture with an 80:20 ratio for recovering waste CFRP samples with 35wt% epoxy resin. The experiments were done with 50 Ml of solvents inside a 100 mL tubular reactor. The temperature range was between 300-380° C and the reaction time was 0-150 minutes. The summary of the effects of temperature in different times on resin removal percentage is illustrated in figure 2.18. They also reported that the use of mains

water instead of distilled water, does not affect the results significantly. They reported that carbon fibers are recycled in temperatures above 32 C, and increasing the temperature increased the reaction time, so that in 340° C and 360° C, the resin was completely recycled in 45 and 15 minutes, respectively. However, increasing the temperature damages the fibers' architecture; hence, the optimum condition reported by them was 320-330° C, in 90-120 minutes.

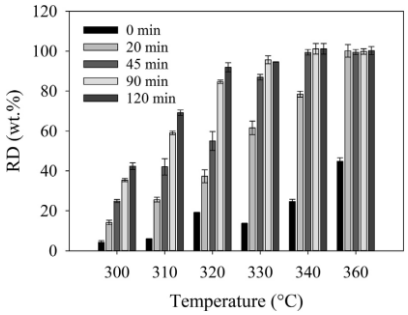


Figure 2.18: Resin decomposition in an acetone/water solvent [49]

Kim et al. [50] used supercritical water as a green solvent for recovering carbon fibers from CFRP composites. For having supercritical water, they put samples into 80 ml of water inside a batch type reactor. The temperature was increased to 405 °C, and the temperature was about 280 bars. The time considered for the experiment was up to 120 min. They could successfully recover 99% of fibers by this method.

Compared to water, supercritical and subcritical alcohols have lower critical temperature and pressure. Idzumi Okajima et al. [51] used supercritical methanol as the solvent for recovering CFRPs. They used two setups for their experiments, the batch-type reactor and the semi-flow-type reactor. For the batch-type reactor, they used supercritical methanol at 270°C and 8 MPa for 90 min, which resulted in 7% decrease in tensile strength compared to virgin fibers, and for the semi-flow-type reactor, they used supercritical methanol at 285°C and 8 MPa for 80 min which was showing 9% decrease in tensile strength of the carbon fiber.

2.1.3.2 Low Temperature and Pressure Chemical Method

In order to avoid the equipment required for supercritical solvents, some researchers have focused on low temperature and pressure chemical methods. For solvents to use in low temperatures (<200° C) and low pressures (<100 kPa), it is essential to use proper catalysts to have the appropriate rate of resin removal. Jiang et al. [52] tried to recycle carbon fiber composites with 40wt% epoxy. The samples were put inside nitric acid to decompose and delaminate initially, and later they were

exposed to ultrasonic cleaning in acetone solution for half an hour. Finally, the wastes were put inside solvents, consisting of macrogol 400, and potassium hydroxide as catalyst at 160° C for 200 minutes. A schematic view of their procedure is as figure 2.19.

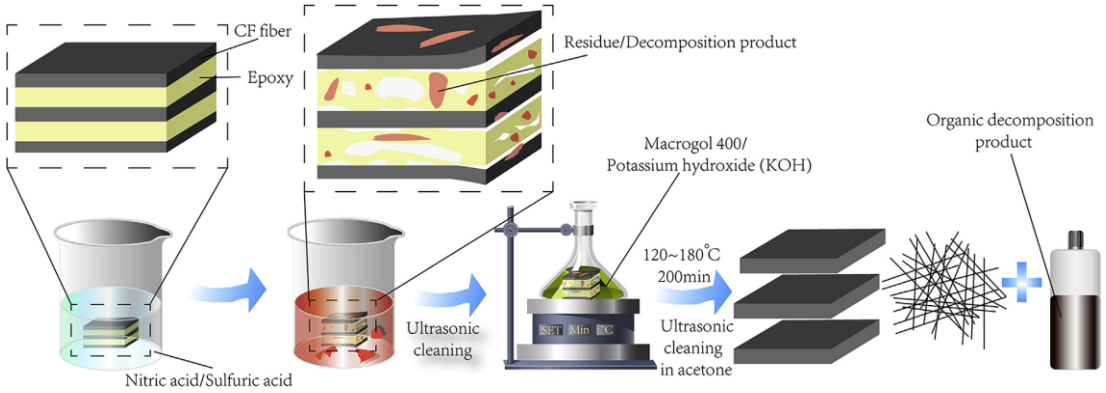


Figure 2.19: Schematic view of the experiment procedures in [52]

Based on their results, pre-treating the sample inside acid noticeably reduces the reaction time and energy demand of the process. The rate of resin removal in this step is reported 28wt%. They had a detailed study on the effects of different factors on resin removal efficiency, which is as figure 2.20.a. According to their report and mechanical tests done for obtaining mechanical properties, tensile strength of the recovered fibers was at least 90% of virgin carbon fibers. There are also some decreases in tensile strength (2-9%) when temperature increased from 120 to 180° C. These results are presented in figure 2.20.b.

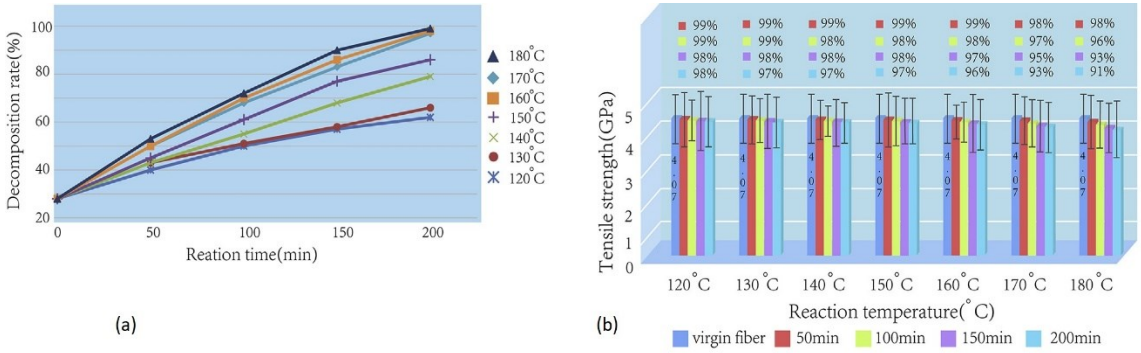


Figure 2.20: (a) The effects of temperature and reaction time on resin decomposition rate, (b) Tensile strength values obtained in different experiment conditions [52]

There have been numerous studies on recycling of composites using oxidants. Compared to supercritical and subcritical decomposition methods that use high pressure and temperature, using oxidants like nitric acid, hydrogen peroxide and potassium permanganate employs relatively low

pressure and temperature. Takuma Hanaoka et al. [53] tried to decompose the resin in a CFRP prepreg by using nitric acid as an oxidant, and sodium hydroxide and sodium sulfite solutions. They used T800 SC carbon fibers with epoxy resins by Toray Industries in two experiment setups: glass tube test, and reactor vessel scale tests. Despite the glass tube test that no resin was observed, The SEM results show some residual resins in reactor tests. According to the authors, it was a consequence of lower diffusion of the oxidant in large-scale test.

In order to have a safer and more energy efficient process, Omid Zabihi et al. [54] used hydrogen peroxide (H2O2) and tartaric acid (TA) in reclamation process with microwaves. Different ratios of these two materials were mixed to compare the results and tensile tests were performed as evaluation. The CFRP sample was put inside a mixture of these two solvents and was irradiated by microwave for 1 minute, while the temperature of the mixture was recorded 120°C. Finally, mechanical test results show that there is only 8% reduction in tensile strength, ne reduction in Young's modulus and only 6.3% reduction in strain-to-failure.

2.2 Life Cycle Assessment of recycling methods for composite materials

Life Cycle Assessment (LCA) is an approach to study the impacts of a method or product on ecosystems and the environment. This tool helps clarify all the negative and positive assessments of a product or service with considering different factor, including material production, manufacturing, the use phase, energy demand, global warning potential and etc. [55-59].

Y. Wei et al. [57] considered cumulative energy demand (CED) and global warning potential/CO_2. Their studies reveal that Pyrolysis and oxidation have lower CED, indicating the lower rate of energy consumption. Figure 2.21 shows CED for different waste treatment methods. According to the results, the energy demand for Solvolysis is approximately twice more than the demand for pyrolysis. The results for global warming potential suggests that landfilling and fluidised bed are among the worst methods, while the emission of CO_2 during pyrolysis is within the acceptable range.

Another study [56] finds that from an environmental point of view, pyrolysis can be implemented in large-scale, while using solvents for recycling make challenges for the environment and the

method cannot be applied in large industrial scale for now.

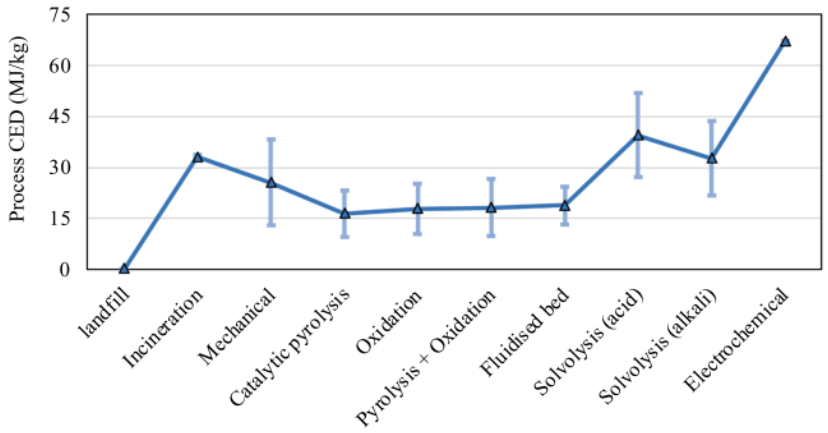


Figure 2.21: Cumulative energy demand for different recycling methods [57]

CHAPTER 3 METHODOLOGY

This chapter covers the three following steps for the project:

1) Using a general-purpose oven for recycling prepregs by conventional pyrolysis,

2) Manufacturing composite plates from recycled carbon fibers by V.A.R.I method,

3) Performing tests to measure the properties of the manufactured plates.

3.1 Experimental procedure for the recovery of carbon fibers by incineration of waste prepregs

For fulfilling sub-objective 1 one the research, pyrolysis was done. Pyrolysis, as defined by ASTM standard, is normally done under an inert atmosphere, but here in this study, oxidative pyrolysis was deployed, which is defined as pyrolysis under ambient air. For doing the pyrolysis, the Type 30400 ThermoLyne Furnace, manufactured by Geneq Inc available in the laboratory of Polytechnique Montreal was deployed, as figure 3.1.

Figure 3.1: Pyrolysis Oven used

The material used for the experiments was time expired prepreg roll from CYCOM (CYCOM 5320 8HS T6503k) provided by Bombardier Inc., which is illustrated in figure 3.2. The full properties of the prepreg are in appendix A.

Figure 3.2: Prepreg roll used in this study

In the first phase of the project, and for recycling the prepregs using general-purpose conventional oven, small 1" x 1" square specimens were cut from the roll and after measuring the weights, were put in the oven inside crucibles. Based on the literature review, preliminary experiments were performed on these samples at temperatures ranging from 450 °C to 600 °C. For each temperature, the experiment time was repeated from 1 to 7 hours. The weight of each specimen was measured after pyrolysis to measure the weight loss during pyrolysis. Figure 3.3 shows the placement of the crucibles inside the oven.

As a baseline to for the study of weight loss of each sample, standard chemical digestion test according to ASTM D3171 was performed which will be discussed in follow. The results of weight loss obtained from different conditions of pyrolysis were then compared to the results of aforementioned digestion test and the optimal condition for the pyrolysis was selected.

Figure 3.3: Placement of crucibles inside the furnace for pyrolysis

After having the optimal conditions from the preliminary experiments, secondary experiments were performed for evaluating to see if the conditions apply to bigger samples as well. In this phase, 10" x 10" samples were cut from the prepreg roll and put inside the oven on pizza plates according to the optimal conditions. The limitation for choosing the maximum size was imposed by the dimensions of the oven. Figure 3.4 demonstrates positioning of pizza trays inside the oven.

Figure 3.4: Positioning of 10" x 10" samples inside pyrolysis oven

Scanning Electron Microscope (SEM)

For observing the surface of the recycled samples deeper and more accurate, Scanning Electron Microscope (SEM) was utilized. As suggested by the name, SEM uses a stream of electrons to produce an image. The image well demonstrates topology and composition of the samples and provides many advantages over optical microscopes. In this study, Hitachi TM-1000, as shown in figure 3.5 was used.

Figure 3.5: HITACHI TM-1000 SEM

3.2 Manufacturing Process for the recycled plates

After pyrolyzing 10" x 10" plates, and for completing sub-objective 2 of the research, new composite plates were manufactured. For this step, vacuum-assisted resin infusion (V.A.R.I) manufacturing process was used in the lab B450.4.4 at Polytechnique Montreal. Recycled carbon fibers from previous steps were used as fibers and for the matrix, commercial Araldite epoxy resin and ARADUR 8602 US hardener were utilized. Resin Datasheet is presented in Appendix B. The epoxy and hardener were mixed in a pitcher for 5 minutes with the ratio of 4 to 1 (as suggested by the manufacturer) and then left in the ambient air for 15 minutes to degas. The weight of the matrix was chosen 3 times the weight of the fibers, considering the waste matrix inside inlet and outlet

tubes. For collecting the extra resin and providing a vacuumed environment, Catch Pot was added to the process. Resin, hardener, and the mixture of them used in the manufacturing process are shown in figure 3.6.

(a) (b) (c)

Figure 3.6: a) Resin, b) hardener and c) mixture of resin and hardener used for making matrix

The full description of V.A.R.I method is presented in appendix C.

The next step for the manufactured plates was post-curing inside the oven. Considering the glass transition temperature of the resin which is 73°C (according to the technical data sheet), they were post-cured at 80°C for 3 hours.

3.3 Physical Characterization

Primary step for evaluating the quality of the manufactured composite plates was inspecting the physical properties. It gives a good idea of the conditions of the final plates. A plate with an undesirable physical condition will end up with unfavorable mechanical properties at the end. Therefore, as a part of sub-objective 3 of the research and for measuring the physical properties of the manufactured composite plates, some experiments were done on the final manufactured plates following the relevant standards, as follows.

3.3.1 Determination of the density by ASTM D792

In this study, ASTM D792 standard test method is used for calculating the density of the specimens manufactured from recycled carbon fibers. Density is one of the crucial factors in composite materials. First, density is an important consideration in weight reduction. As mentioned earlier, one of the benefits of composite materials is their low weight and high strength to weight ratios. On the other hand, density is fundamental for determining other characteristics of composite materials. As it will be discussed in follow, for finding the volume percentage of constituents of the specimen, and eventually for calculating the void volume of the final product, knowledge of density value is vital.

ASTM D792 recommends various procedures for determining the density. The procedure that best matches the requirements of this study is test method B, which is testing solid plastics in liquids other than water. In this study, ethanol was used for density measurements. For calculations, the weight of the specimen is measured in the air, then the specimen is immersed inside ethanol. For measurement, first the special gravity is calculated in accordance with equation 3-1.

$$\text{Sp gr } 23/23°C = a/(a+w-b) \tag{3-1}$$

Where:

a = Apparent mass of specimen without wire or sinker in air,

b = Apparent mass of specimen, sinker and wire immersed in Ethanol, and

w = Apparent mass of sinker and wire.

Afterwards, density is calculated according to equation 3-2.

$$D^{23C}, \text{kg/m3} = \text{sp gr } 23/23°C \times d \tag{3-2}$$

Where:

d = Specific gravity 23/23°C of Ethanol, kg/m^3.

3.3.2 Determination of the constituent content and void content of composite materials by ASTM D3171

In this research, the ASTM D3171 standard test method is used for two purposes:

a- Determination of the fiber content by weight in the waste prepregs under study intended for the recovery of carbon fibers,

b- Determination of the void content by weight in composites made with recovered carbon fibers.

a- Determination of the fiber content by weight in the waste prepregs under study intended for the recovery of carbon fibers:

As indicated in the literature review, the disadvantage of the incineration method for the recovery of carbon fibers from composite waste is that the carbon fibers can be degraded by excessive temperature. The degradation in question is manifested by a loss of fibrous mass. Similarly, in the process for recovering carbon fibers from the incineration of prepreg waste, it is desired to get rid of the resin of the prepreg by incineration without degrading the fibers. However, there is a risk that the carbon fibers will also be degraded. Therefore, it is important to know the exact fiber content by weight after incineration to see whether or not there has been a loss in mass. The fiber content of the prepreg before incineration will serve as a baseline and can be accurately determined according to the ASTM D3171 method as detailed in the following.

ASTM D3171 proposes numerous procedures to determine the constituent content of composite materials. The procedure which best applies to carbon fiber reinforced epoxies is the removal of the matrix by digestion. Specifically, in this research and within ASTM D3171, we used the procedure entitled:

«A2. PROCEDURE B- MATRIX DIGESTION USING SULFURIC ACID/HYDROGEN PEROXIDE»

The matrix portion of a material specimen of known mass is removed in a hot liquid medium. When dissolving in a hot liquid medium, the remaining residue, containing the reinforcement, is then filtered washed, dried, cooled and weighted. The weight percent of the reinforcement is calculated, and from this value, and if densities of both the composite and the reinforcement are

known, the volume percent is calculated. An additional calculation for void volume may be made if the density of the matrix is known or determined.

b- Determination of the void content by weight in composites made with recovered carbon fibers.

In this study, after manufacturing composite plates from recovered carbon fibers, it is planned to evaluate their mechanical properties to estimate their structural value for potential applications. However, it is well known that the mechanical properties of composite materials can be significantly affected by the presence of voids introduced during the manufacturing process. Therefore, it is important to determine the void content of the manufactured plates to ensure that the manufacturing process is adequate. Indeed, as specified in the ASTM D3171 standard, knowledge of the void volume of a composite material is desirable as an indication of the quality of a composite part. ASTM D3171 outlines the details of the procedures which consist of the following steps:

1- Reinforcement content, weight percent

2- Reinforcement content, volume percent

3- Matrix content, weight percent

4- Matrix content, volume percent

5- Void volume

1- The reinforcement content, in weight percent is calculated in accordance with equation 3-3.

$$W_f = (M_f / M_i) \times 100 \qquad (3\text{-}3)$$

Where:

M_i = Initial mass of the composite specimen before digestion

M_f = Final mass of the composite specimen after digestion.

2- The reinforcement content, in volume percent is calculated in accordance with equation 3-4.

$$V_r=(M_f/M_i) \times 100 \times \rho_c/\rho_r \quad (3\text{-}4)$$

Where:

ρ_r= Density of the reinforcement, g/cm3 and

ρ_c= Density of the specimen, g/cm^3.

3- The matrix content, in weight percent, is calculated in accordance with equation 3-5.

$$W_m=(M_i-M_f)/M_i \times 100 \quad (3\text{-}5)$$

4- The matrix content, in volume percent, is calculated in accordance with equation 3-6.

$$V_m=(M_i - M_f)/ M_i \times 100 \times \rho_c/\rho_m \quad (3\text{-}6)$$

Where:

ρ_m= Density of the matrix, g/cm^3.

5- The void volume is calculated in percent, in accordance with equation 3-7.

$$V_v=100 - (V_r + V_m) \quad (3\text{-}7)$$

3.3.3 Determination of Glass Transition Temperature by ASTM D7028

In this study, ASTM D7028 is used to determine the glass transition temperature of the specimens. Glass transition temperature (T_g) is one of the crucial characteristics of polymers and plays an important role in most of mechanical, chemical and physical properties of the polymers. It is defined as the temperature that carbon chains start moving. At this point, the amorphous areas transfer from solid state to a flexible state. Polymers start losing their elastic modulus and transferring from glassy state to a rubbery state.

ASTM D7028 is a useful method for measuring the glass transition temperature of polymer matrix composites by Dynamic Mechanical Analysis (DMA). For measurements, the specimen inside the DMA machine is oscillated and heated at the range of 5°C/min. The temperature at which storage modulus decreases indicatively is referred to as T_g.

3.4 Mechanical Tests of the Manufactured Plates From Recycled Carbon Fibres

For measuring the mechanical properties and functionality of manufactured composite plates made from recycled carbon fibers, and as a part of sub-objective 3 of the research, different mechanical experiments were conducted on the specimens, following the relevant standards. Some of these tests and standards will be discussed in the following.

3.4.1 Determination of Tensile Properties by ASTM D3039

In this study, the ASTM D3039/D3039M-17 was followed to measure tensile properties of the manufactured composite plates from recovered carbon fibers. Tensile properties provide useful information about the ability of composite materials to withstand tension forces without being failed or permanently deformed. The information acquired by this test determine the reliability of manufactured plates in application where tensile stresses are applied to the composite.

As suggested by the standard, specimens with constant rectangular cross sections is placed in the grips of the testing machine and tension load is applied to it with a constant test speed. The force and displacements are recorded during the test and by the use of these data, following properties are obtained.

1- Tensile strength

2- Tensile strain

3- Tensile Chord Modulus of Elasticity

1- The ultimate tensile strength is calculated in accordance with equation 3-8.

$$F^{tu} = P^{max}/A \quad (3\text{-}8)$$

Where:

F^{tu} = Ultimate tensile strength, MPa,

P^{max} = Maximum force before failure, N. and

A = Average cross-sectional area, mm^2, which equals thickness times width of the specimen.

2- **The tensile strain is calculated according to equation 3-9 and from the data read by the extensometer**.

$$\varepsilon_i = \delta_i / L_g \quad (3\text{-}9)$$

Where:

ε_i = tensile strain at *i*th data point, με,

δ_i = Extensometer displacement at *i*th data point, mm , and

L_g = Extensometer gauge length, mm.

3- **The tensile Chord Modulus of Elasticity is calculated from the data acquired for stress and strain, in accordance with equation 3-10.**

$$E^{chord} = \Delta\sigma / \Delta\varepsilon \quad (3\text{-}10)$$

Where

E^{chord} = Tensile chord modulus of elasticity, GPa,

$\Delta\sigma$ = Difference in tensile stress between two measured points (1000-3000 με), MPa, and

$\Delta\varepsilon$ = Difference between the two strain points (2000 με).

3.4.2 Determination of Compressive properties by ASTM D6641

During this thesis, and after manufacturing plates, ASTM D6641 standard was employed to determine the compressive properties of the final plates. Compressive properties play a critical role in evaluating the performance of composite plates. These materials may undergo compressive loads in a variety of applications, hence compressive properties are important considerations in assessing the performance of CFRPs.

ASTM D6641 suggests two different procedures, procedure A where the specimen is not tabbed, and procedure B for the tabbed specimens. In this study, for 8-ply plates, procedure A is followed, while for 4-ply plates, because of smaller values of thickness, procedure B is used. In both procedures, the specimens are placed inside a proper fixture and are subjected to the combined end-and-shear loading, while the fixture is only subjected to compressive loading applied by the testing

machine. Following compressive properties are calculated from the load-strain data acquired by the testing machine and strain gauges bonded to the unsupported (gauge) length of the specimens.

1- Compressive Strength

2- Compressive Modulus

1- Compressive strength of the samples is calculated using equation 3-11.

$$F^{cu} = P_f/wh \quad (3\text{-}11)$$

Where

F^{cu} = Compressive strength, MPa,

P_f = Maximum load to failure, N,

w = Gage width of the specimen, mm, and

h = Gage thickness of the specimen, mm.

2- Compressive modulus of the specimens is calculated in accordance with equation 3-12.

$$E^c = \frac{P_2 - P_1}{(\varepsilon_{x_2} - \varepsilon_{x_1})wh} \quad (3\text{-}12)$$

Where:

E c = Compressive modulus, MPa,

P_1 = Load at ε_{x1}, N and

P_2 = Load at ε_{x2}, N.

ε_{x1} and ε_{x2} are the actual strain nearest lower and upper end of strain range used, and are considered 1000 and 3000 microstrain.

3.4.3 Determination of Flexural Properties by ASTM D790

ASTM D790 standard test method is used for determining the flexural properties of composite plates manufactured from recycled carbon fibers. Flexural properties are one of the influential properties of composite materials that play an important role while being under bending load or

deflection.

There are different procedures and methods offered by this standard to measure the flexural properties. For the purpose of this study, procedure A, test type I is followed. For testing, the rectangular specimen cut from the manufactured plates is placed on supports and the bending force is applied to the samples. For deflection measurements, crosshead position is utilized. Following data are obtained from ASTM D790 standard test.

1- Flexural Stress

2- Flexural Strain

3- Modulus of Elasticity.

1- Flexural stress is calculated in accordance with equation 3-13.

$$\sigma_f = 3\ PL/2bd^2 \qquad (3\text{-}13)$$

Where:

σ = Stress in the midpoint of outer fibers, MPa,

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

L = Support span, mm,

b = Width of the specimen, mm, and

d = Depth of the specimen, mm.

2- Flexural strain in mm/mm is calculated in accordance with equation 3-14.

$$\varepsilon_f = 6\ Dd/L^2 \qquad (3\text{-}14)$$

Where:

ε_f = Strain in the outer surface, mm/mm,

D = Maximum deflection in the center of the specimen, mm, and

d = Depth of the specimen, mm.

3- Modulus of elasticity of the samples, MPa, is measured in accordance with equation 3-15.

$$E_B = L^3m/4bd^3 \quad (3\text{-}15)$$

Where:

m = Slope of the tangent to the initial straight-line portion of the load-deflection curve, N/mm.

3.4.4 Determination of Shear properties by ASTM D5379

In this study, ASTM D5379 standard test method is used to measure shear properties of the plates manufactured from recycled carbon fibers. Composite materials may undergo shear loads in different applications, and therefore the knowledge of their shear properties is an important consideration. As indicated by the ASTM D5379, two v notches were cut in the center of the specimen, and were placed inside a standard fixture. Two sides of the fixture undergo compression force implied by the testing machine. Schematic of the fixture and the applied load are presented in figure 3-7. By the help of two strain gauges oriented at ±45° to the loading axis, and the load history of the machine, following properties are calculated.

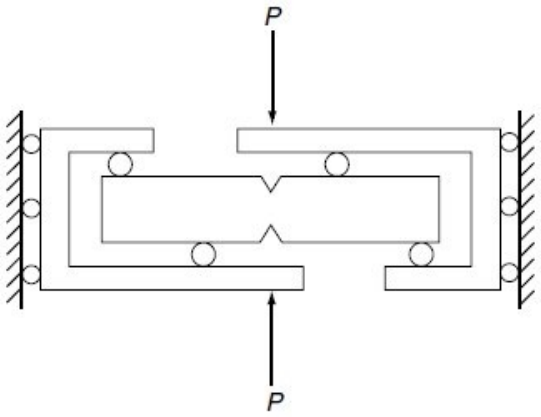


Figure 3-7: Schematic of the standard fixture for v-notch ASTM D5379 shear test and the applied loads [60]

1- Ultimate Shear Strength

2- Ultimate Shear Strain

3- Shear Chord Modulus of Elasticity

1- Ultimate Shear Strength is calculated in accordance with equation 3-16.

$$F_u = P_u/A \quad (3\text{-}16)$$

Where:

Fu = Ultimate Strength, MPa,

Pu = the lower of ultimate or force at 5% engineering shear strain, N, and

A = Cross-sectional area, which is calculated according to equation 3-17.

$$A = w \times h \tag{3-17}$$

Where:

w = Width of the specimen across the notch, mm, and

h = Thickness of the specimen at the notch, mm.

2- Shear Strain and Ultimate Shear Strain are calculated in accordance with equation 3-18 and 3-19.

$$\gamma_i = | \varepsilon_{+45} | + | \varepsilon_{-45} | \tag{3-18}$$

$$\gamma^a = \min \{ 5\% \text{ or } \gamma \text{ at ultimate load} \tag{3-19}$$

Where:

γ_i = Engineering Shear strain at ith data point, με,

ε_{+45} = +45° normal strain at ith data point, με,

ε_{-45} = -45° normal strain at ith data point, με, and

γ^a = Ultimate Shear Strain, με.

3- Shear Chord Modulus of Elasticity is calculated in accordance with equation 3-20.

$$G^{chord} = \Delta\tau / \Delta\gamma \tag{3-20}$$

Where:

G chord = Shear Chord Modulus of Elasticity, GPa,

$\Delta\tau$ = Difference in applied shear stress between two measured points, MPa, and

$\Delta\gamma$ = Difference between two strain points, which is nominally 0.004.

3.4.5 Determination of Short Beam Strength by the ASTM D2344

In this study, ASTM D2344 is used for determining short beam strength of the plates manufactured from recycled carbon fibers. Short beam strength is highlighted in the applications where the specimen is subjected to bending or twisting forces. For the test, the specimen is placed on two supports, and the load is applied to the midpoint of the specimen. The short beam strength is calculated in accordance with equation 3-21.

$$F^{sbs} = 0.75 \times \frac{Pm}{b\ x\ h} \tag{3-21}$$

Where:

F^{sbs} = Short-beam strength, MPa,

P_m = Maximum load obsereved, N,

b = Width of the specimen, mm, and

h = Thickness of the specimen, mm.

CHAPTER 4 RESULTS AND DISCUSSIONS

4.1 Determination of the optimum temperature-time cycle for pyrolysis in ambient air

Based on the literature review, three temperatures were chosen for the pyrolysis experiments: 450 °C, 500 °C and 600°C. For each round of experiments, 12 samples were pyrolyzed, and each 30-minute, one sample was taken out of the oven. The results for different experiments conditions performed in the first phase of this study, which is the main contribution of this study, are presented in figure 4.1.

As mentioned in the section 3.3.2, standard test according to ASTM D3171 was performed to determine the exact value of fiber content by weight percentage in the waste prepregs. The test results show that the experimental average value of weight fraction of fiber reinforcement in the time-expired prepreg roll is equal to 62.5% (W_f=62.5%). So, this ratio was chosen as the baseline for finding the optimal time and temperature for different pyrolysis experiments. Dotted red line in figure 4.1 corresponds to the optimum fiber weight fraction of the prepregs as determined by ASTM D3171 and the results are compared with this value.

The samples under 600°C (experiments #1 and #3 in figure 4.1) tend to lose the most portion of their weights after a short time. The part of losing weights up to the baseline (dotted red line) corresponds to the weight of resin, which is degraded at elevated temperature, but the weight loss below the dashed line indicates burning of the carbon fibers. This extreme temperature and the subsequent degradation of carbon fibers reveal that 600 °C is not a good option for the prepreg and causes the loss of fibers. At this temperature, even the geometry and shape of the fibers were changed. Some photos of the samples after pyrolysis at 600 °C are presented in figure 4.2. Apparently, samples are crumpled and damaged physically.

Results for experiment #4 which corresponds to this temperature shows the trend of recycling at 450 °C. The samples pyrolyzed at 450 °C eventually reach the dashed line in a gentle and slow process. It takes almost 6 hours for the samples to lose 37.5% of their initial mass. The pyrolysis experiments at 500°C were repeated in two sets of experiments, corresponding to experiments #2

and #5. The same as the experiments for 450 °C, the final loss in mass under this temperature reaches the desired dashed line, which indicates that all the matrix is removed from the samples and carbon fibers are left after the pyrolysis. It should be noted that the sudden jump in the data for experiment #2 at 300 minutes is related to the wrong reading of the data for mass measurements.

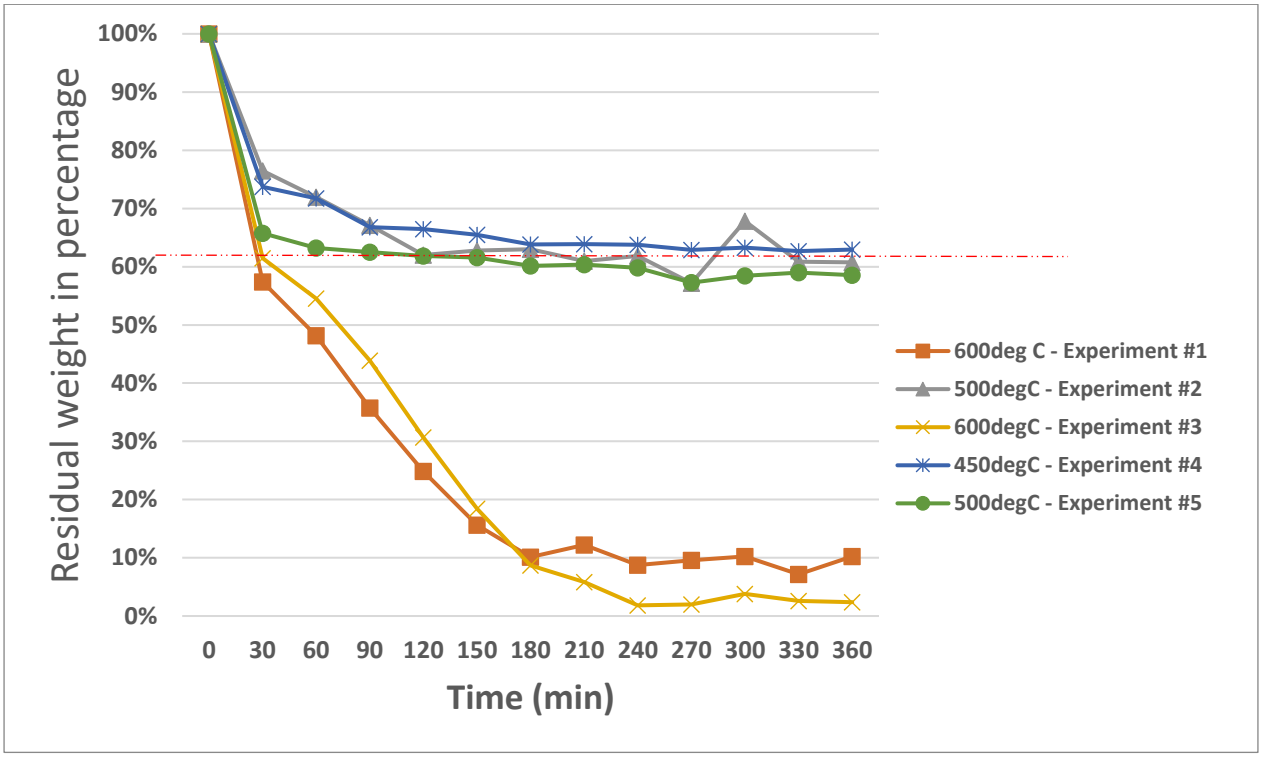


Figure 4.1: Comparative pyrolysis results

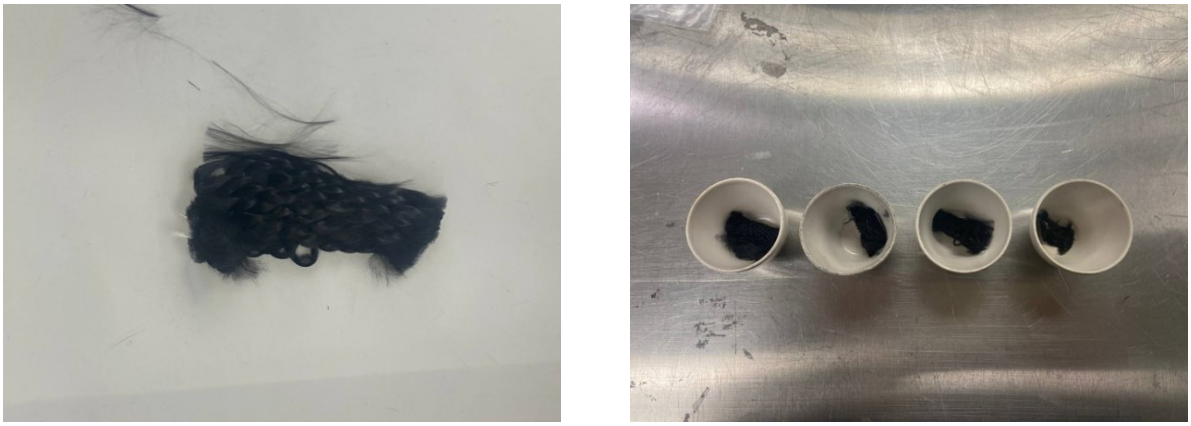


Figure 4.2- Samples after pyrolysis under 600°C

Supposedly, both 450 °C and 500 °C are good conditions for pyrolysis of time-expired prepreg rolls and removing all the matrix from carbon fibers. The only difference between the two is the time to converge. According to the data, it takes almost 7 hours for the samples to lose 37.5% of the initial mass at 450 °C, in other words, the sufficient time for the material to lose all the matrix under these conditions is 7 hours. On the other hand, and based on experimental data and figure 4.1, the time of convergence for pyrolysis at 500°C is lower. The effective time of recovery, i.e., removing all the matrix from prepreg samples, is only 2.5 hours at 500 °C. Hence, the most optimum condition for recycling prepreg samples of this study was selected as 2.5 hours at 500 °C. These results satisfactorily meet the needs of sub-objective-1for this research.

4.1.1 Scanning Electron Microscope (SEM) Results

After selecting the optimal conditions in terms of temperature and time for the pyrolysis, further SEM imaging was done to see the surface of recovered carbon fibers. Benefitting from SEM, different images with different magnifications were taken from random samples to see the surface of the recovered carbon fibers. Some of the SEM images taken from pyrolyzed samples at 500°C and after 2.5 h of pyrolysis are presented in figure 4.3.

According to SEM results for the optimal condition, the surface of the recovered carbon fibers is clean and almost free of any residual resin, which shows the efficiency of the recycling procedure. There are also no signs of fiber damage on the surface of the fibers, as well as no signs of any remaining resin.

SEM was also used to observe the surface of carbon fibers recovered from other pyrolysis conditions. As shown in figure 4.4a and 4.4b, pyrolysis at 500°C for 1 hour is not sufficient and there are some residual resins left on the surface of the fibers.

Figures 4.4c and 4.4d show SEM images of pyrolysis at 450°C after 3 hours. There are obvious residuals of resins on the fibers, therefore 3 hours of pyrolysis at 450°C is not sufficient to get rid of all matrices inside the initial prepreg samples

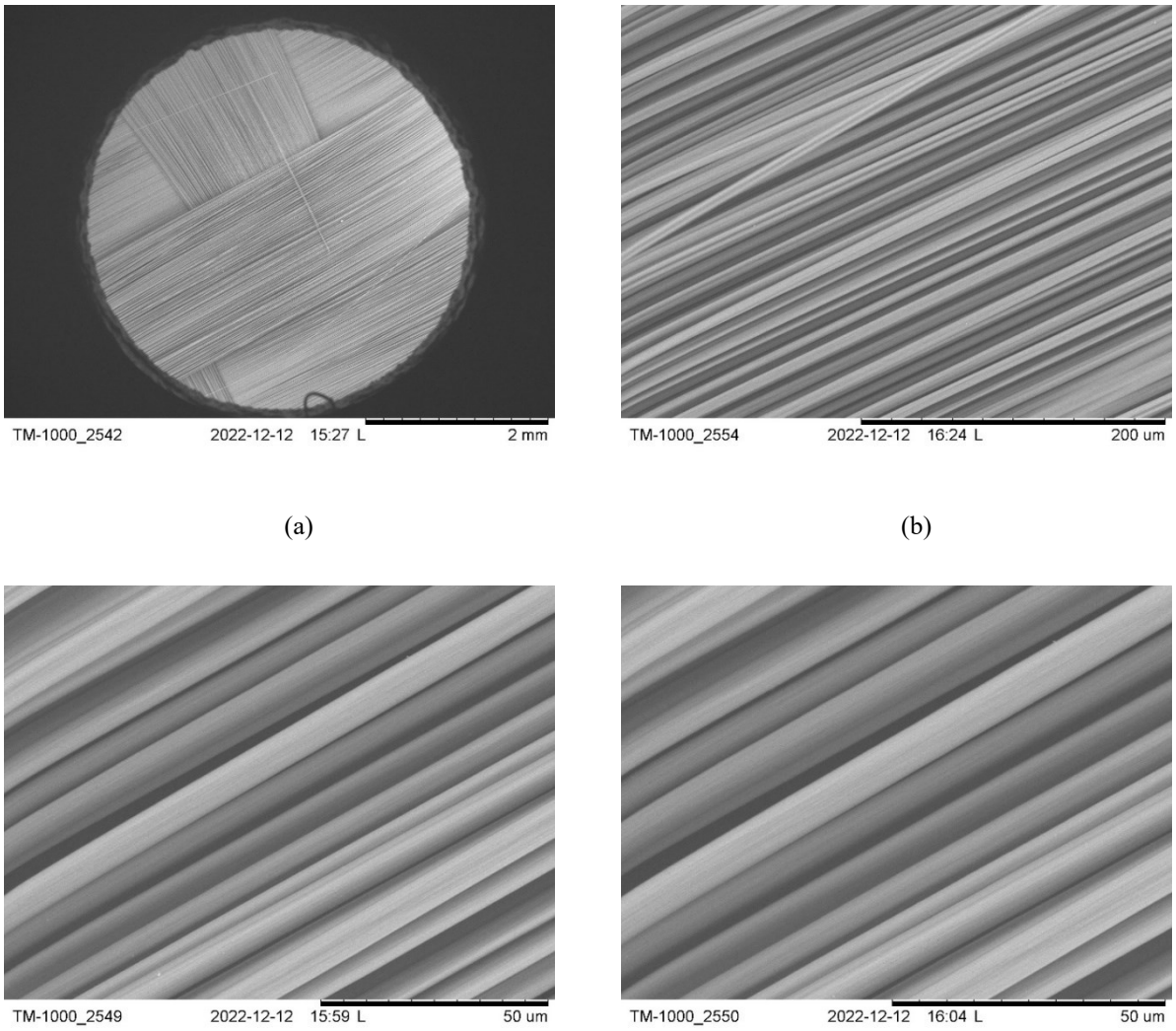


Figure 4.3: SEM images for samples pyrolyzed at 500°C for 2.5h- a) X30, b) X500, c) X1500, d) X1800

4.2 Confirmation of the optimal pyrolysis cycle by additional tests

After finding the optimal conditions for pyrolysis, pyrolysis was repeated on more than 70 samples of 10″×10″. The results for weight loss in all experiments were very close to the desired value. The small differences in W_f values in some tests could be a result of minor errors in weight measurements, or initial defects of the prepreg roll. Figure 4.5 shows the frequency of the final W_f results for 1″×1″ samples being pyrolysed at 500 °C for 2.5 h.

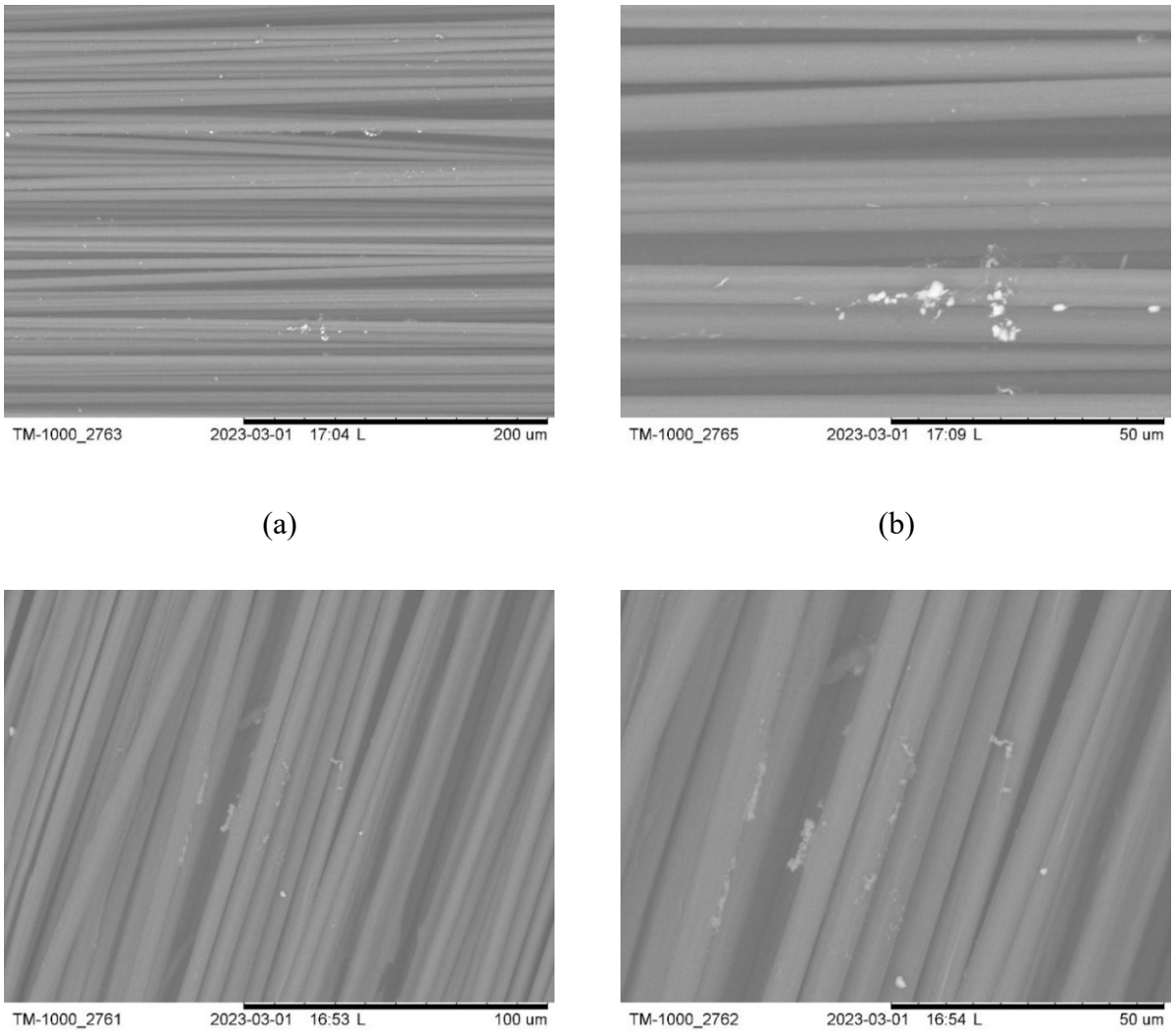


Figure 4.4: SEM images for pyrolyzed samples: a) 1h @500°C- X500, b) 1h @ 500°C- X1800, c)3h @450°C-X500 and d) 3h @450°C-X1800

The minimum value obtained for W_f was 61.46, while the maximum amount was observed as 63.83%. Other values for the average data and standard deviation are presented in table 4.1.

Table 4.1: Data analysis for W_f results for 1″×1″ samples

No. of data	Max. Value	Min. Value	Average Value	STDEV.P
70	63.83%	61.46	62.78%	0.44%

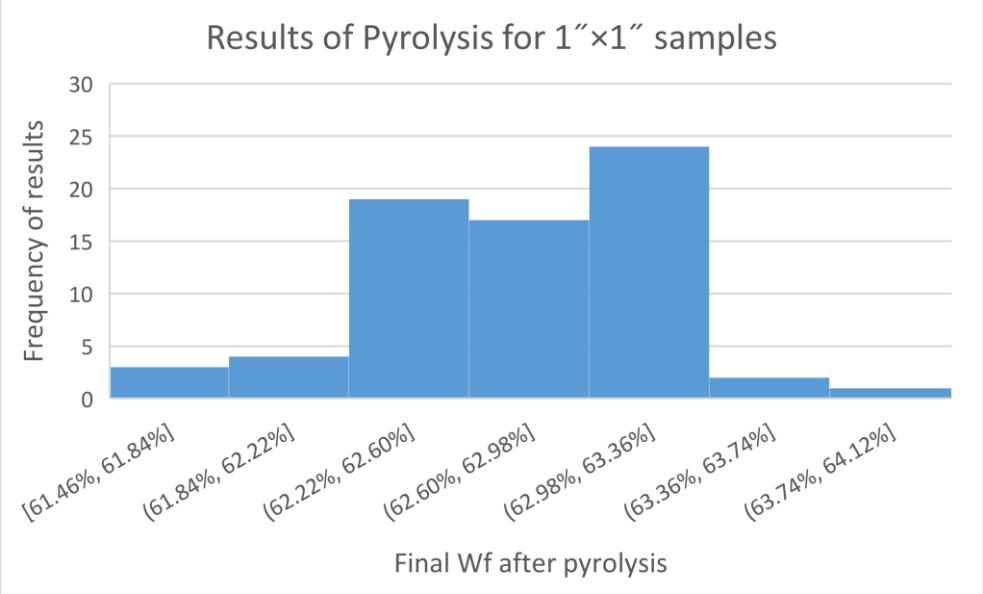


Figure 4.5: Frequency of obtained W_f results for pyrolysis at 500 °C for 2.5 h

4.3 Manufactured Plates

The pyrolysis of the expired prepregs made it possible to recover the carbon fiber satin fabrics in the form of 10" × 10" sheets. The vacuum-assisted resin infusion (V.A.R.I) process was used in order to manufacture composite plates to fulfill sub-objectives 2 and 3. The reinforcements plies were stacked to give cross-ply laminates [(0/90)n]s. the number of plies depends on the properties that will be measured. For some tests it is 4 plies and others it is 8 plies. A sample of 8-ply woven plate manufactured from recycled carbon fibers by V.A.R.I method is presented in figure 4.6.

4.4 Physical Characterization Results

4.4.1 Density

Density measurements were done in 4 different plates. From each plate, 3 specimens were cut and densities were measured according to ASTM D792-20 Method B. Ethanol at 23.5 °C was used as the immersion liquid. The summary of results is presented in Table 4.2, and the test reports are in appendix D.

The maximum and minimum values of density in 12 samples were reported 1.503 g/cm^3 and 1.474 g/cm^3 respectively, which shows a difference less than 2% between the results. The average value

of density for 12 plates is 1.486 g/cm^3, which can be considered as the density of the composite plates manufactured from recovered carbon fibers in this study.

Figure 4.6: Manufactured 8-ply plate from recycled carbon fibers by V.A.R.I. method

Table 4.2: Density measurements according to ASTM D792-20 Method B in plates manufactured from recycled carbon fibers

Plate Number	**No. of specimens**	**Min. Density (g/cm^3)**	**Max. Density (g/cm^3)**	**Avg. Density (g/cm^3)**
1	3	1.497	1.503	1.500
2	3	1.474	1.476	1.475
3	3	1.489	1.492	1.491
4	3	1.474	1.480	1.478

As early discussed, one of the advantages of composite materials, is their weight light and high strength to weight ratio. When compared to other materials, the density of manufactured plates is far lower than steel and aluminum alloys. Table 4.3 is the comparison of density between some metallic and composite materials where rCFRP refers to the CFRPs manufactured from recycled carbon fibers in this study.

Table 4.3: Density of some metallic and composite materials

Material	SAE 1010 Steel	6061-T6 aluminum alloy	High Strength CFRP (Unidirectional)	E-Glass fiber-epoxy matrix (Unidirectional)	rCFRP
Density (g/cm^3)	7.87*	2.7*	1.55*	1.85*	1.47

* The values are taken from [60].

Table 4.3 demonstrates well the low density value of rCFRP compared to other materials, while comparison of density between unidirectional CFRP and the rCFRP shows that there is only a 5% difference. The 5% difference can be an indicator of a slightly higher percentage of resin inside the rCFRP.

4.4.2 Void Content

As a baseline for evaluating the quality of the plates, void contents were measured in the plates. Void content test was performed in 4 different plates, with 3 samples from each plate, which gives a total of 12 experiments. Table 4.3 summarizes the results for the void content. Test reports are attached in appendix E.

As observed from table 4.4, values obtained for the percentage of void are not the same in different plates. The reason is the process used for manufacturing plates (V.A.R.I. method) which is not considered as an automatic method, and therefore the results vary. The average of void content in manufactured plates is 1.55%, while the maximum void content reported in the plates is 5.5%.

Table 4.4: Percent of Void Content in plates manufactured from recycled carbon fibers measured according to ASTM D3171-15, Proc . B

Plate Number	No. of specimens	Max. Void Content (%)	Average Void Content (%)
1	3	1.2	1.1
2	3	0.4	0.2
3	3	0.5	0.5
4	3	5.5	4.4

4.4.3 Glass Transition Temperature

There have been six experiments according to ASTM D7028-07 for finding the glass transition temperature of the manufactured plates. The TA Instruments Q800 Dynamic Mechanical Analyzer (DMA) according to figure 4.7 was used.

Figure 4.7: Q800 DMA used for determining T_g of the manufactured plates

According to table 4.5, results suggest that the average glass transition temperature of the manufactured plate is 59.6 °C, while the data sheet of the initial prepreg suggests that the dry T_g is 190°C. T_g is one of the properties that is highly dependent on the resin, and fibers don't have an impact on its value, therefore the difference between calculated T_g and the value of the data sheet originates from the different resins that have been used in prepreg and the manufactured plates.

Table 4.5: Glass Transition Temperature of plates manufactured from recycled carbon fibers measured according to ASTM D7028-07

Plate Number	**Number of plies**	**Number of specimens**	**Max. T_g (°C)**	**Min. T_g (°C)**	**Avg. T_g (°C)**
1	8	2	64.1	58.4	61.3
2	8	2	62.5	62.1	62.3
3	4	2	56	54.6	55.3

All test reports for Glass transition properties are presented in appendix F.

4.5 Mechanical Testing Results

4.5.1 Tensile Properties

To determine the tensile properties of the manufactured composite plates, the tensile tests were carried out on a ZWICK Z250 machine according to the ASTM D3039-17 standard. A total of 8 tests were performed from two different plates and tensile strength, tensile chord modulus of elasticity, elongation at break and rupture time were measured. For securely holding the specimen in the grips and removing any slacks, a pre-load of 50 N was applied to the machine. Test speed was 0.5 mm/min and the size of the samples was 1″×10″. Figure 4.8 shows the positioning of two extensometers mounted for measuring the strains and placement of the specimen inside the testing machine.

4.5.1.1 Tensile Strength

Table 4.6 summarizes the results of tensile strength. Plate number 1 consisted of 8 plies, and the average thickness was 2.99 mm, while plate number 2 was 4 plies and the average thickness was 1.41 mm. The average ply thickness of the plates, which corresponds to the thickness of the plate divided by the number of plies, is higher for the 8-ply plate by 6%. This means that the fiber volume content V_f is 6% higher in 4-ply plates. Therefore, 4-ply plates are expected to have higher tensile strength.

The maximum and minimum values for Tensile strength in the plates were 745 MPa and 676 MPa, respectively, which shows a difference of 10%. The average value for tensile strength in 8 different tests is equal to 697 MPa (the average of tensile strength for 8 specimens) that can be considered as the average tensile strength of the plates manufactured from the carbon fibers recovered in this study.

Referring to the data sheet of the initial prepreg, tensile strength is 131 ksi (903 MPa). Hence, the average recovery rate of tensile strength is calculated in accordance with equation 4-1.

$$\text{R.R.T.S}=\frac{Average\ value\ of\ tensile\ strength\ of\ manufactured\ plates}{Initial\ value\ of\ tensile\ strength\ for\ prepreg}\times 100 \quad (4\text{-}1)$$

$$\text{R.R.T.S}=\frac{697}{903}\times 100=77.2\%,$$

Where R.R.T.S is the recovery rate of tensile strength.

Table 4.6: Tensile Strength of plates manufactured from recycled carbon fibers measured according to ASTM D3039

Plate Number	Number of plies	Number of specimens	Max. Tensile Strength (MPa)	Min. Tensile Strength (MPa)	Avg. Tensile Strength (MPa)
1	8	3	702	676	688
2	4	5	745	684	703

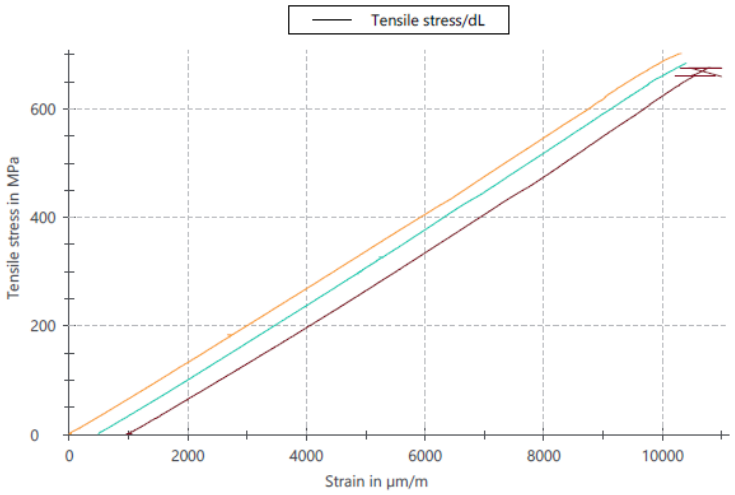
(a) (b)

Figure 4.8: a) Positioning of two extensometers, b) placement of the sample for ASTM D3039 tensile test

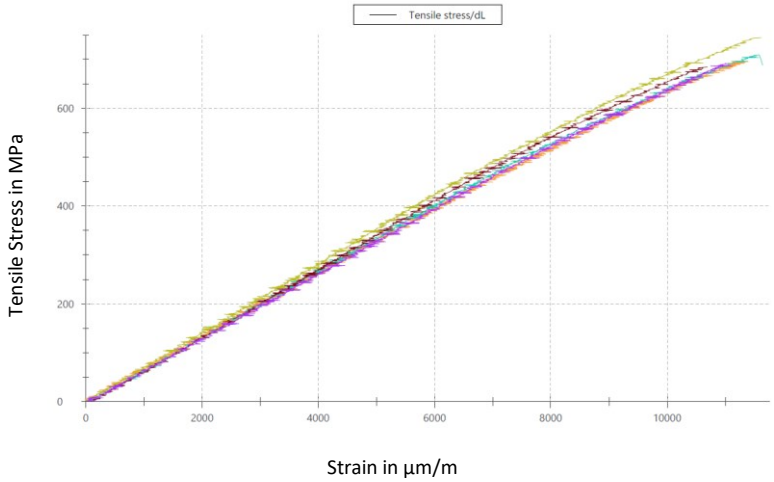
Considering the maximum and minimum values for obtained tensile strengths, the maximum and minimum recovery rate of tensile strength would be 82.5% and 74.9%.

Considering the R.R.T.E, we can expect a loss of 22.8% in tensile strength. There are different factors that lead to this loss. The main reasons can be expressed as 1: the manufacturing process, that is a low-cost process, and 2: the low-cost resin used for manufacturing. Regarding the data sheet of the initial prepreg, data are acquired by using a high-quality resin, suitable for aerospace applications.

Tensile Stress-Strain curves for the two manufactured plates are presented in figure 4.9a and 4.9b. The stress-strain response for two plates is almost a linear line, which indicates brittle behavior of the plates. Compared to ductile materials like steel and aluminum, there is not much plastic deformation area in the curves. So, the plates are expected to break without noticeable deformation. The slope of the curve, which indicates the modulus of elasticity and will be discussed in the following, is almost the same for each plate.



(a)



(b)

Figure 4.9: Tensile Stress-strain curves for a) 8-ply composite samples and b) 4-ply composite samples manufactured from recycled carbon fibers

Despite 22.8% loss in tensile strength compared to the initial fibers, the obtained tensile strength is still competitive with other metallic and composite materials. Table 4.7 compares well the values of tensile strength for different materials. Tensile strength of rCFRPs is almost double the strength of Steel and 6061 aluminum alloy, so for the applications where tensile strength is the most favorable property, the recycled composite plates can replace conventional materials.

Table 4.7: Comparison of Tensile Strength of some metallic and composite materials

Material	SAE 1010 Steel	6061-T6 aluminum alloy	Carbon fiber epoxy matrix (quasi-isotropic)	Sheet molding compound (SMC) composite (isotropic)	rCFRP
Tensile Strength (MPa)	365*	310*	579*	164*	697

* The values are taken from [60].

In terms of ratio of tensile strength to weight, which is obtained by dividing the tensile strength to gravity multiplied with the acceleration due to gravity, rCFRPs show superior performance compared to metallic and composite materials listed in table 4.8. The relatively high value of this ratio makes it suitable for the purposes where weight is a key factor. Manufacturing of numerous light-weighted and high strengthen structures is possible using rCFRPs.

Table 4.8: Comparison between ratios of tensile strength to weight

Material	SAE 1010 Steel	6061-T6 aluminum alloy	Carbon fiber epoxy matrix (quasi-isotropic)	Sheet molding compound (SMC) composite (isotropic)	rCFRP
Ratio of tensile strength to weight (10^3 m)	4.72	11.7	38	8.9	48.3

4.5.1.2 Tensile Modulus of Elasticity

Tensile Modulus of elasticity results are reported in table 4.9. The same as tensile strength, 1st plate was 8 plies, while the 2nd plate was 4 plies. The average value for Tensile Modulus is 67.26 GPa, while all the results are within the range of 64.54 GPa and 69.64 GPa. Referring to the prepreg data sheet, the Modulus is reported 69.08 GPa (10.02 Msi), therefore the recovery rate of Modulus is calculated in accordance with equation 4-2.

Table 4.9: Tensile Modulus of Elasticity of plates manufactured from recycled carbon fibers measured according to ASTM D3039

Plate Number	Number of plies	Number of specimens	Max. Modulus (GPa)	Min. Modulus (GPa)	Avg. Modulus (GPa)
1	8	3	68.3	65.8	67.2
2	4	5	69.64	64.54	67.3

$$\text{R.R.T.M}=\frac{Average\ value\ of\ tensile\ Modulus\ of\ manufactured\ plates}{Initial\ value\ of\ tensile\ Modulus\ for\ prepreg}\times 100 \quad (4\text{-}2)$$

$$\text{R.R.T.M}=\frac{67.26}{69.08}\times 100=97.4\%,$$

Where R.R.T.M represents recovery rate of tensile modulus of elasticity. Apparently, the method utilized for recovering carbon fibers in this study well preserves the Modulus of the carbon fibers and the composite plate.

When compared with other composite materials, despite losing 2.6% of tensile modulus, the value is still better than most used composites in industry, and is almost the same as the modulus of 6061-T6 aluminum alloy. The results are summarized in table 4.10.

Table 4.10: Comparison between ratios of tensile strength to weight

Material	6061-T6 aluminum alloy	E-Glass fiber-epoxy matrix (Unidirectional)	Carbon fiber epoxy matrix (quasi-isotropic)	Sheet molding compound (SMC) composite (isotropic)	rCFRP
Modulus (GPa)	68.9*	39.3*	45.5*	15.8*	67.26

* The values are taken from [60].

4.5.1.3 Elongation at break

Deformation percentages at the break in the manufactured samples were measured in 8 different specimens from two plates and the results are shown in table 4.11. Based on the results, the average deformation was 1.06%, while the maximum and minimum values were measured 1.2% and 1%,

respectively. The relatively low value of elongation at break indicates the brittle behavior of the manufactured plates. These materials cannot undergo remarkable plastic deformations and cannot deform significantly before failure.

Table 4.11- Deformation at break of plates manufactured from recycled carbon fibers measured according to ASTM D3039

Plate Number	Number of plies	Number of specimens	Max. Deformation (%)	Min. Deformation (%)	Avg. Deformation (%)
1	8	3	1	1	1
2	4	5	1.2	1.1	1.1

4.5.1.4 Failure time

Failure time for 8 samples was measured during the tests. Results show that the average failure time was 6.16 minutes. According to ASTM D3039 standard, the failure should occur within the first 10 minutes of the test. The times obtained by all the experiments well comply the requirements. Table 4.12 summarizes the results.

Table 4.12- Failure time of plates manufactured from recycled carbon fibers measured according to ASTM D3039

Plate Number	Number of plies	Number of specimens	Max. Failure time (min)	Min. Failure time (min)	Avg. Failure time (min)
1	8	3	6.7	6.4	6.6
2	4	5	7.7	5.1	5.9

4.5.1.5 Tensile Test Failure Modes

Tensile Failure modes of the 8 samples almost follow the same modes. According to the ASTM D3039 standard failure codes, and as indicated in figure 4.10, all the failure areas in the samples are within a close distance to the grips and follow the LWV code, where L refers to the Lateral type, W indicates <1 W from grip, and v stands for various locations.

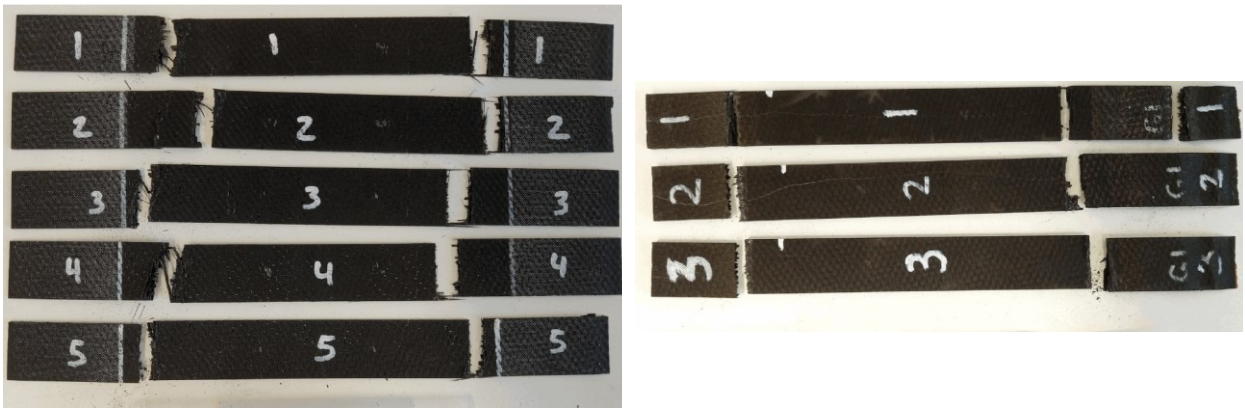


Figure 4.10: Tensile test failure modes of the manufactured plates

Appendix G shows test results for tensile properties.

4.5.2 Tensile properties of burned plates

During the post-curing process, two plates were post-cured at 180 °C for 3 hours. Considering the properties of the resin that is used for manufacturing the composite plates, the plates were burned. For finding the properties and comparing with other plates, standard ASTM D3039 was also performed on these plates. All testing conditions, including the sample sizing, extensometers used, and the test speed were the same as previous tests.

4.5.2.1 Tensile Strength of Burned Plates

The values of tensile strength for the burned plates are presented in table 4.9. The average tensile strength is 632.7 MPa. Comparison of this value with the average tensile strength obtained in section 4.4.3.1 reveals that over burning the plates reduce the tensile strength by 9.2%. It is worth mentioning that tensile strength in burned plates is reduced by 30% compared to the initial prepreg tensile strength.

Although most of the tensile load is carried by the fibers, but the results suggest that over burning the plates, which affects the matrix, has a negative impact on the tensile strength of the composite plates.

Figure 4.13 presents the tensile stress-strain diagram for one of the burned plates. The tensile behavior of burned plates is the same as the plates manufactured from recycled carbon fibers. The material tends to behave like a brittle material and there is not considerable plastic deformation for

these plates and the slope of the curve which represents the modulus of elasticity is almost the same for each plate. Compared to the unburned plates, the maximum tensile strength is lower, and there is a slight decrease in the slope of the curves.

Table 4.13- Tensile Strength of burned plates manufactured from recycled carbon fibers measured according to ASTM D3039

Plate Number	Number of plies	No. of specimens	Max. Tensile Strength (MPa)	Min. Tensile Strength (MPa)	Avg. Tensile Strength (MPa)
1	8	3	665	593	631
2	4	4	639	631	634

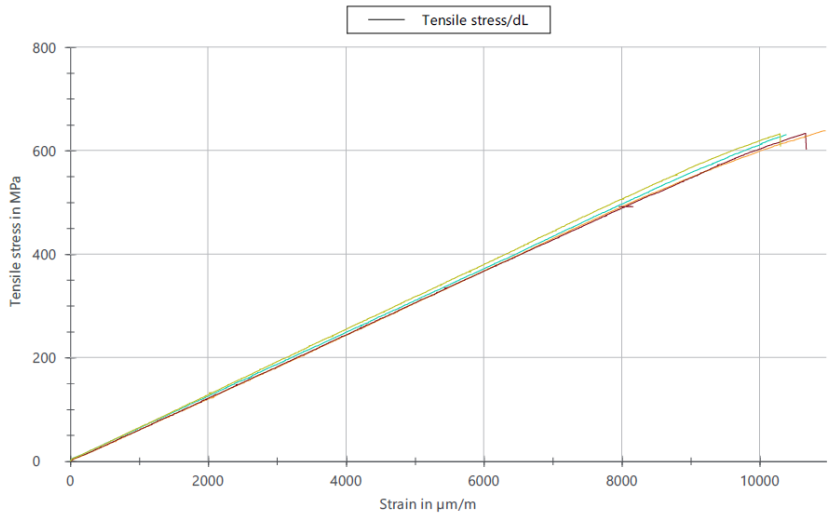


Figure 4.11: Tensile stress-strain curve for the burned manufactured plate

4.5.2.2 Tensile Modulus of elasticity for burned plates

Based on the results of the modulus of elasticity in table 4.14, the average Modulus for burned plates is equal to 61.9 GPa. Compared to the results in 4.4.3.2, over burning the plates reduces Modulus of Elasticity by 8%, and compared to the prepreg data sheet, Modulus is reduced by 10.04%. The impact of over burning in Modulus of Elasticity is less severe compared to the results of Tensile Strength.

Table 4.14: Tensile Modulus of Elasticity of burned plates manufactured from recycled carbon fibers measured according to ASTM D3039

Plate Number	Number of plies	Number of specimens	Max. Modulus (GPa)	Min. Modulus (GPa)	Avg. Modulus (GPa)
1	8	3	63.6	60.6	61.87
2	4	4	62.8	61.5	61.92

4.5.2.3 Effects of over burning the plates on elongation at Break and Failure Time

The results for elongation at the break and the failure time of over burned plates suggest that the over-burning does not affect these properties noticeably. Table 4.15 is the comparative table for these properties. The elongation at break for the two burned and normally cured plates are exactly the same, indicating that over burning does not change the brittle properties of the composites. While for the failure time, there is a10% decrease. Regarding the same test speed in both conditions, the rather smaller failure time for burned plates shows the weakness of the plates under these conditions. These results are in accordance with the tensile strength results, which there is a decrease in tensile strength.

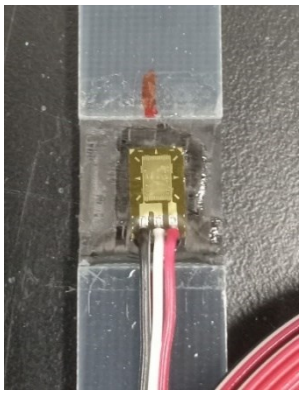
Table 4.15: Comparison of average force at failure, deformation at break and failure time between burned plates and normally cured plates measured according to ASTM D3039

Plate Characteristics	No. of plies	Avg. elongation at break (%)	Avg. Failure time
Burned Plates	8	1.0	6.6
	4	1.1	5.2
Normally cured Plates	8	1.0	6.6
	4	1.1	5.9
Percentage of Changes	8	0	0
	4	0	10.8%

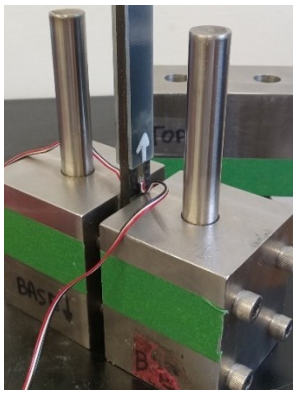
Test results for tensile properties of burned plates are found in appendix H.

4.5.3 Compressive properties of the manufactured plates

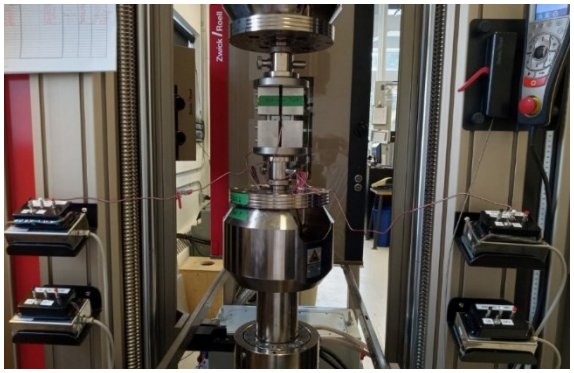
Compressive tests according to ASTM D6641 were done on 10 samples. 0.5″×5 1/2″ samples were cut from two plates and compressive strength and compressive modulus of elasticity were measured. Plate number #1 consisted of 4 plies, while plate number #2 was 8 plies. Test speeds were 1 mm/min and 1.3 mm/min, respectively. For plate #1, because of the thinner thickness, fiberglass tabs were bonded, while plate #2 was tested without tabbing. Figure 4.12 represents the test procedure.



(a)



(b)



(c)

Figure 4.12: a) Bonding of strain gauge, b) placement of specimen inside fixture and c) testing machine for compression test

Full test reports of compressive properties are presented in appendix I.

4.5.3.1 Compressive Strength

Table 4.16 is a summary of compressive strength measurements. The average compressive strength of the manufactured plates is therefore calculated as 378 MPa, while the maximum and minimum compressive strengths are 428 MPa and 324 MPa, respectively. The possible misalignments in 8-ply plates might be the reason for having lower value of compressive strength.

Table 4.16: Compressive Strength of plates manufactured from recycled carbon fibers measured according to ASTM D6641

Plate Number	Number of plies	Number of specimens	Max. Compressive Strength (MPa)	Min. Compressive Strength (MPa)	Avg. Compressive Strength (MPa)
1	4	5	428	379	403
2	8	5	387	324	353

Following technical data sheet, compressive strength of the prepreg is 750 MPa. So, recovery rate of compressive strength is calculated in accordance with equation 4-3.

$$\text{R.R.C.S}=\frac{Average\ value\ of\ compressive\ strength\ of\ manufactured\ plates}{Initial\ value\ of\ compressive\ strength\ for\ prepreg}\times 100 \quad (4\text{-}3)$$

$$\text{R.R.C.S}=\frac{378}{750}\times 100=50.4\%,$$

Where R.R.C.S represents recovery rate of compressive strength. As calculated, there is a 49.6% loss in compressive strength. Resin and hardener play an important role in compressive strength of the composite materials. As mentioned before, a low-cost general-purpose resin is used for manufacturing the plates from recovered fibers, while the initial resin impregnated in the prepreg is a high-quality resin with high aerospace standards. The mechanical properties of the matrix and the strength of the fiber/matrix interface can affect considerably the compression strength of CFRPs [61-63]. So, considering the type of resin used, it was expected to have a rather great difference between compressive strength of the manufactured plate with the initial prepreg. On the other hand, the manufacturing process is not the same, and there are several factors during infusion process that can affect the results.

Compressive-stress strain responses of two tested plates are presented in figure 4.13. The curves follow the pattern of brittle materials. Despite the ductile materials, like steel and aluminum that

normally have high ranges of plastic deformations, the rCFRPs fail suddenly after reaching the ultimate load. The slope of the curve for samples from the 4-ply plate is almost the same, implying the same value for compressive modulus. However, there are some variations in the slope of 8-ply plates. Referring to the test reports, the coefficient of variation (CV) for thickness between 5 specimens is 1.5%. Compared to the thickness CV results of other plates, this value is considerable. There are some possible explanations for the variations in thickness within the same plate, but the manufacturing process contributes most to this phenomenon. Sometimes during the manufacturing process, the side of the plate that is close to the inlet pipe is thicker compared to the other side. The reason is the accumulation of more resins during the manufacturing process. The higher thickness results in higher area which eventually will cause lower stress.

4.5.3.2 Compressive Modulus

Compressive modulus for 10 samples were measured according to ASTMD 6641. According to table 4.17, the average compressive modulus of the manufactured plates is calculated as 45.4 GPa. The initial compressive modulus of the prepreg was 63.2 GPa, so the recovery rate of compressive modulus is calculated in accordance with equation 4-4. The higher possibility of misalignments in 8-ply plate can be the reason for the lower modulus in these plates.

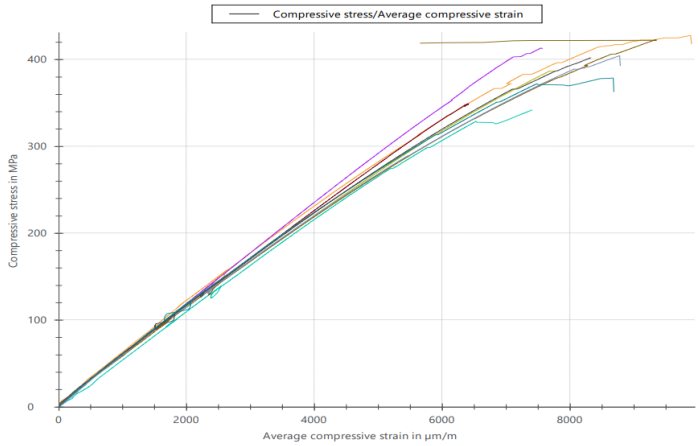
$$\text{R.R.C.M}= \frac{Average\ value\ of\ compressive\ modulus\ of\ manufactured\ plates}{Initial\ value\ of\ compressive\ modulus\ for\ prepreg}\times 100 \qquad (4\text{-}4)$$

$$\text{R.R.C.M}=\frac{45.4}{63.2}\times 100=71.8\%,$$

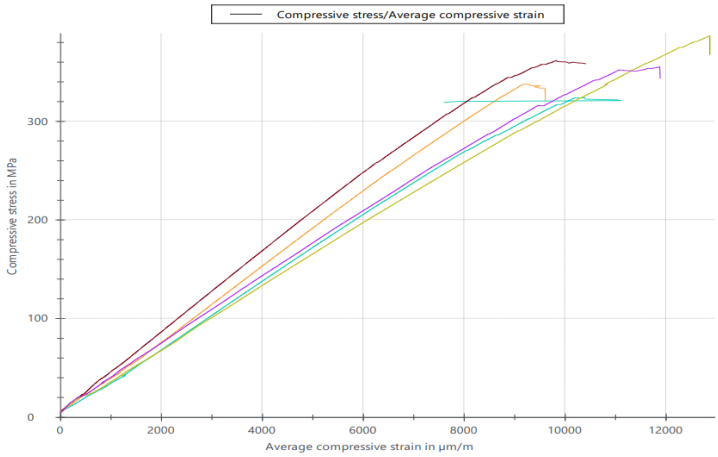
Where R.R.C.M is the recovery rate of compressive modulus. The same as compressive strength, the type of the resin has a key role in decrease in compressive modulus and can be improved by using a better kind of resin and hardener.

Table 4.17: Compressive Modulus of Elasticity of plates manufactured from recycled carbon fibers measured according to ASTM D6641

Plate Number	Number of plies	Number of specimens	Max. Modulus (GPa)	Min. Modulus (GPa)	Avg. Modulus (GPa)
1	4	5	58.5	52.2	54.4
2	8	5	41.2	33	36.4



(a)



(b)

Figure 4.13: Compressive stress-strain curves for a) 4-ply and b) 8-ply plates manufactured from recycled carbon fibers

Comparison of compressive strength and modulus between manufactured plates and some composite materials are summarized in table 4.18. As observed, despite a 49.6% decrease in compressive strength, the manufactured plates from recycled carbon fibers have still better compressive strength compared SMC, and almost the same strength as glass/epoxy. From Modulus point of view, the values obtained are 51% higher than glass/epoxy. Therefore, compressive properties of the manufactured plates are comparable to other common composite materials, and

they can be considered as a potential substitute in applications where high compressive properties are not required.

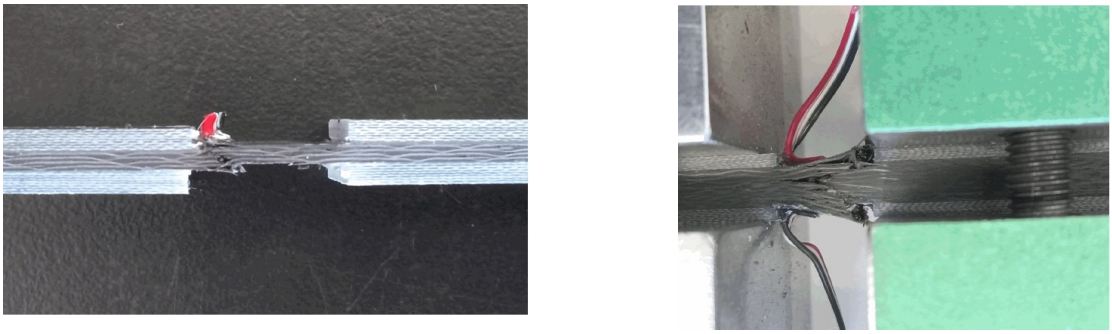
Table 4.18: Comparison of compressive properties between some composite materials

Material	Glass/Epoxy plain wave reinforcement	Sheet molding compound (SMC) SMC-R25	rCFRP
Modulus (GPa)	22[a]	N.A	45.4
Compressive Strength (MPa)	380[a]	183[b]	378

[a] Taken from [64] [b] Taken from [60].

4.5.3.3 Compression Test Failure Modes

Failure modes for two plates were different. For plate number #1, consisting of 4 plies, two failure modes of TGM, two modes of TGB and one failure mode of TGT were reported. According to ASTM D6641, T represents transverse shear (failure mode), and G indicates gauge (failure area). The third character shows the location of the failure, where M, B and T represent middle, bottom and top, respectively. Therefore, all the samples failed because of transverse shear in the gauge. For plate number #2, two modes of HGB, two modes of HGT, and one mode of HGM were reported where the first character, H, indicates failure through thickness. Therefore, the failure mode for specimens from the second plate was different location in gauge, caused by failure through thickness. Figure 4.14 represents the failure mode in two plates.



(a) (b)

Figure 4.14: a) Transverse shear failure in 4-plied samples, b) failure through the thickness in 8-plied samples

4.5.4 Flexural Properties

For finding flexural properties of the manufactured plate, 8 samples made from two different plates were tested following ASTM D790-17. Test speed was between 1.31 mm/min and 1.35 mm/min and flexural strength, maximum elongation, and modulus were measured. It is worth noting that although both samples are 8-plies, but they are cut from different plates.

4.5.4.1 Flexural Strength

Flexural strength is calculated based on the failure load and the dimensions of the samples according to equation 3-13. Table 4.19 summarizes the results of flexural strength for the plates. Maximum and minimum values for flexural strengths are 747.5 MPa and 620.9 MPa, and the average value is 692.9 MPa.

Table 4.19: Flexural strength of plates manufactured from recycled carbon fibers measured according to ASTM D790-17

Plate Number	Number of plies	Number of specimens	Max. Flexural Strength (MPa)	Min. Flexural Strength (MPa)	Avg. Flexural Strength (MPa)
1	8	3	681.4	620.9	648.6
2	8	5	747.5	700.9	719.5

4.5.4.2 Flexural Modulus of Elasticity

The average value for flexural modulus of elasticity for the manufactured plates is 38.26 GPa. Values for bending modulus have been measured in 8 samples, and the results are presented in table 4.20.

Table 4.20: Flexural Modulus of Elasticity of plates manufactured from recycled carbon fibers measured according to ASTM D790-17

Plate Number	Number of plies	Number of specimens	Max. Modulus (GPa)	Min. Modulus (GPa)	Avg. Modulus (GPa)
1	8	3	43.9	36.69	39.89
2	8	5	41.74	33.12	37.28

4.5.4.3 Maximum flexural deformation

Results show that the average maximum flexural deformation for the samples being tested is 1.6%. The maximum measured value is 1.7%, while the minimum is 1.5%. Table 4.21 summarizes the results.

Table 4.21: Maximum deformation of plates manufactured from recycled carbon fibers measured according to ASTM D790-17

Plate Number	Number of plies	Number of specimens	Max. Deformation (%)	Min. Deformation (%)	Avg. Deformation (%)
1	8	3	1.5	1.7	1.6
2	8	5	1.7	1.6	1.6

Appendix J is the test report for flexural properties of the composite plates.

Figure 4.15 represents flexural force-deformations curves of the samples. For the elastic region, the force-deformation determine that all the samples have almost the same slope. After reaching the maximum force, there is a drop in force value caused by the failure. The fluctuations in the force value are repeated several times after the first decrease. These fluctuations are due to the failure of internal plies. As already mentioned, the plates that were tested, consisted of 8 plies, and failure of each ply induce a fluctuation and decrease in force.

Comparison of flexural properties of the manufactured plates with other composite materials are summarized in table 4.22. Despite the cheap resin used for manufacturing the plates, the flexural properties of rCFRPs are competitive. The modulus is higher than unidirectional E-glass fibers and SMC-R25, while the flexural strength is better than Kevlar-49 and SMC-R25.

Table 4.22: Comparison of flexural properties between some composite materials

Material	E-Glass fiber-epoxy matrix (UD)	Kevlar-49 epoxy (UD)	Sheet molding compound (SMC) SMC-R25	rCFRP
Modulus (GPa)	36.5*	76*	4.48*	38.26
Flexural Strength (MPa)	1137*	621*	220*	692.9

* taken from [60].

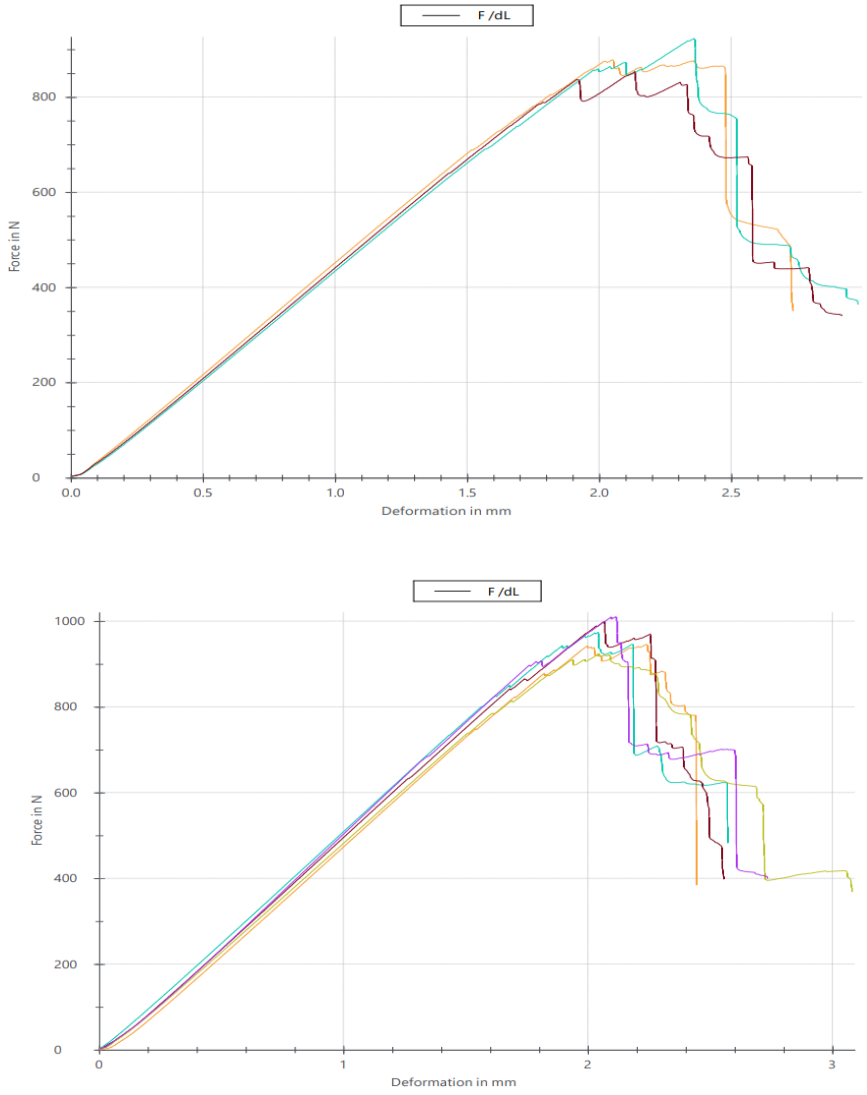


Figure 4.15: Flexural force-deformation curves for plates manufactured from recycled carbon fibers

4.5.5 Shear Properties

For determination of shear properties by ASTM D5379, 5 samples were cut from an 8-ply plate. The size of the samples was 3″ × 0.75 ″ and a v-notch was cut on the samples. Two strain gauges were bonded to each sample to measure strain at ±45° to the loading axis, according to figure 4.16. The strain gauge for samples 1 and 2 consisted of two gauges perpendicular to each other, measuring strain in two directions. The test was done with Zwick Z250 testing machine, with a 0.8 mm/min test speed. The special fixture in accordance with the ASTM D5379 standard was used

for the tests. Figure 4.17 shows the placement of specimens inside the fixture and its positioning in the testing machine.

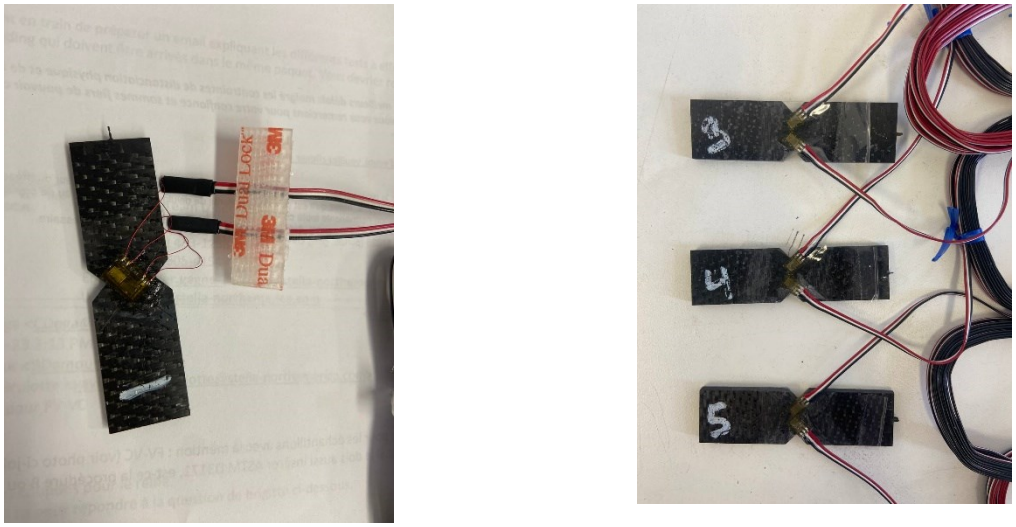
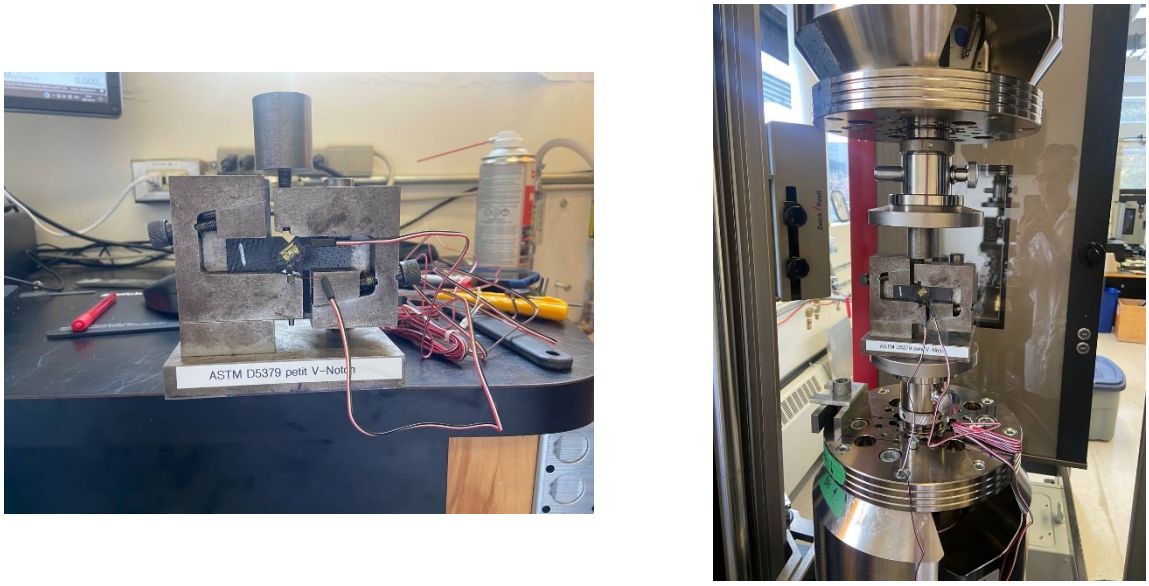


Figure 4.16: Bonding of strain gauges to specimens for ASTM D5379 test



(a) (b)

Figure 4.17: a) fixture and b) test machine for determining the shear properties according to ASTM D5379

The results of shear test are summarized in table 4.23. Shear properties are dominated by the matrix, so using a higher-grade matrix will improve the shear properties. Test reports for shear test are

presented in appendix K.

Table 4.23: Shear properties of plates manufactured from recycled carbon fibers measured according to ASTM D5379

Property	Number of plies	No. of specimens	Max. Value	Min. Value	Avg. Value
Shear Strength (MPa)	8	5	48.7	40.7	45
Deformation at Break (%)	8	5	5	5	5
Modulus of Elasticity (MPa)	8	5	3060	2140	2580

Corresponding stress-strain curve is presented in figure 4.18. A blue line showing constant stress whit a sharp increase in shear strain is observed. The reason is slipping of one of the strain gauges which has caused an increase in strain.

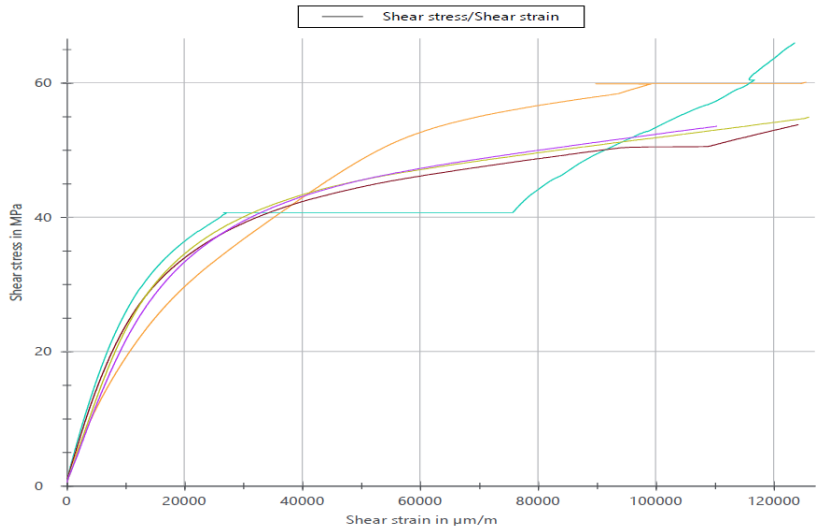


Figure 4.18: Stress-Strain curve for ASTMD5379

4.5.6 Short-Beam Strength

Short-Beam strength of manufactured plates was determined by ASTM D2344 test method. 5 samples were cut from a 4-ply plate and were tested with a 1 mm/min speed. The values determined

for short-beam strength are summarized in table 4.24. Short-beam strength of composites highly depend on the manufacturing process and the type of resin used in the composite. Hence, by improving these two factors, higher values for short-beam strength can be expected from recycled carbon fibers. Figure 4.19 shows force-deformation curve for the short-beam test. Appendix L shows test report for short-beam test.

Table 4.24: Short-Beam Strength of plates manufactured from recycled carbon fibers measured according to ASTM D2344

Property	Number of plies	No. of specimens	Max. Value	Min. Value	Avg. Value
Short-beam Strength (MPa)	4	5	35.2	33.6	34.4

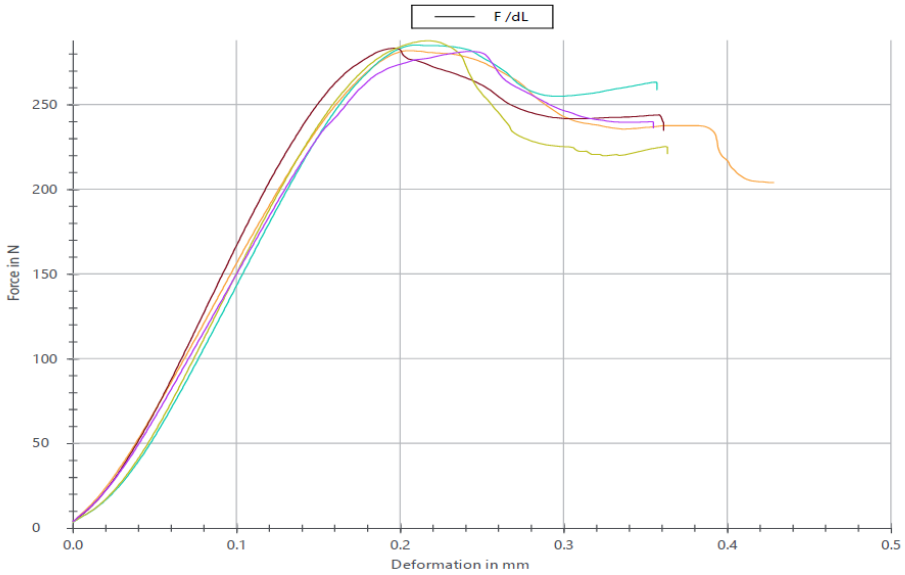


Figure 4.19: Force-deformation curve for short-beam test according to ASTM D2344

4.5.7 Summary of Results for Manufactured Plates

Considering all the above-mentioned results, the final properties of composite plates manufactured from time-expired prepreg rolls can be summarized as table 4.25. It should be noted that these are average values for different properties that have been measured in different experiments with

different plates. Therefore, table 4.25 can be a good representation of the properties of CFRPs made by V.A.R.I method from recycled carbon fibers with a general-purpose resin.

Table 4.25: Properties of the manufactured plates from recovered CFs

Properties	Unit	Value
Density	g/cm^3	1.486
Void Content	%	1.55
Glass Transition Temperature	℃	59.6
Tensile Strength	MPa	697
Tensile Modulus	GPa	67.26
Tensile Elongation at break	%	1.06
Compressive Strength	MPa	378
Compressive Modulus	GPa	45.4
Flexural Strength	MPa	692.9
Flexural Modulus	GPa	38.26
Maximum Flexural Deformation	%	1.6
Shear Strength	MPa	45
Maximum Shear Deformation	%	5
Shear Modulus	MPa	2580
Short-Beam Strength	MPa	34.4

CHAPTER 5 CONCLUSION, LIMITATIONS AND FUTURE WORK

5.1 Conclusion

In this study, pyrolysis experiments of time-expired prepreg roll were performed under ambient air. Later, composite plates were manufactured from recovered carbon fibers and several tests according to ASTM standards were done to find properties of the composite plates. The following conclusions can be drawn from this study.

- Optimal pyrolysis condition for the time-expired prepreg roll investigated in this study is 2.5 hours @ 500°C. Although there are other scenarios and conditions that satisfactorily meet the needs of this study, but considering the required time and temperature, 2.5 h @ 500°C was the best choice for pyrolysis. According to the percentage of resin in initial prepreg according to chemical digestion results, all the resins are decomposed under these conditions.
- SEM results suggest there are no resins and chars left on the surface of the recovered fibers, confirming post oxidation that is followed pyrolysis under an inert atmosphere, is not necessary for pyrolysis under ambient air.
- The reclamation process results in 22% reduction in Tensile Strength and 2.5% reduction in tensile Modulus. On the other hand, the results for compressive tests show there is 50% decrease in Compressive Strength and 28% reduction in Compressive Modulus. The most notable reason for the higher rate of loss in compressive properties is the type of resin that is used for manufacturing plates. In the original prepreg, a higher-grade resin with better properties was used, which results in better compressive properties.
- The method utilized in this study is a cost-effective way of recycling composites compared to pyrolysis under inert air. All the experiments were done with a general-purpose oven, while for pyrolysis under an inert atmosphere, a more complicated and high-priced furnace, with a flow of an inert gas is required. Besides, as mentioned above, after pyrolysis under

inert air, another round of post-oxidation must be performed, which is eliminated in the method used in this study.

- This method is suitable for the applications where recovery of 100% of virgin CF mechanical properties is not critical.
- Over-burning the manufactured plates during post-curing process can decrease the tensile strength and tensile modulus by 9.2% and 8%, respectively.

5.2 Limitations

The main limitation of the project was the lack of an oven with capability of injecting inert or ambient gas inside. In the case of being able to measure the flow of air inside the oven, characteristics of CFs recovered from various flows could be compared and could lead to better and comparable results.

5.3 Future Work

As a recommendation for future work, it might be interesting to manufacture plates directly from time-expired prepreg rolls and compare the properties with original carbon fibers and also the properties of plates manufactured from recovered carbon fibers. For better comparison of compressive properties, it might be a good idea to reinforce the same resin system used in initial prepreg with recycled carbon fibers. Cost analysis of the recycled prepregs can also be interesting as a future work.

REFERENCES

1. Rani, M., et al., *A review on recycling and reuse methods for carbon fiber/glass fiber composites waste from wind turbine blades.* Composites Part B: Engineering, 2021. **215**: p. 108768.
2. Zhao, Q., et al., *Efficient recycling of carbon fibers from amine-cured CFRP composites under facile condition.* Polymer Degradation and Stability, 2020. **179**: p. 109268.
3. Das, M., R. Chacko, and S. Varughese, *An Efficient Method of Recycling of CFRP Waste Using Peracetic Acid.* ACS Sustainable Chemistry & Engineering, 2018. **6**(2): p. 1564-1571.
4. Wu, J., et al. *Recycling Carbon Fiber from Carbon Fiber-Reinforced Polymer and Its Reuse in Photocatalysis: A Review*. Polymers, 2023. **15**, DOI: 10.3390/polym15010170.
5. He, D., et al., *Comparative life cycle energy analysis of carbon fibre pre-processing, processing and post-processing recycling methods.* Resources, Conservation and Recycling, 2020. **158**: p. 104794.
6. Zhang, J., et al., *Current status of carbon fibre and carbon fibre composites recycling.* Composites Part B: Engineering, 2020. **193**: p. 108053.
7. Naqvi, S.R., et al., *A critical review on recycling of end-of-life carbon fibre/glass fibre reinforced composites waste using pyrolysis towards a circular economy.* Resources, Conservation and Recycling, 2018. **136**: p. 118-129.
8. Meng, F., et al., *Comparing Life Cycle Energy and Global Warming Potential of Carbon Fiber Composite Recycling Technologies and Waste Management Options.* ACS Sustainable Chemistry & Engineering, 2018. **6**(8): p. 9854-9865.
9. Vo Dong, P.A., C. Azzaro-Pantel, and A.-L. Cadene, *Economic and environmental assessment of recovery and disposal pathways for CFRP waste management.* Resources, Conservation and Recycling, 2018. **133**: p. 63-75.
10. Hadigheh, S.A., Y. Wei, and S. Kashi, *Optimisation of CFRP composite recycling process based on energy consumption, kinetic behaviour and thermal degradation mechanism of recycled carbon fibre.* Journal of Cleaner Production, 2021. **292**: p. 125994.
11. https://sciencing.com/effects-landfills-environment-8662463.html.
12. https://www.unisanuk.com/what-is-a-landfill-why-are-landfills-bad-for-the-environment/.
13. https://greentumble.com/waste-incineration-advantages-and-disadvantages/.
14. Meyer, L.O., K. Schulte, and E. Grove-Nielsen, *CFRP-Recycling Following a Pyrolysis Route: Process Optimization and Potentials.* Journal of Composite Materials, 2009. **43**(9): p. 1121-1132.
15. Pakdel, E., et al., *Recent progress in recycling carbon fibre reinforced composites and dry carbon fibre wastes.* Resources, Conservation and Recycling, 2021. **166**: p. 105340.

16. Guo, L., et al., *Research on a two-step pyrolysis-oxidation process of carbon fiber-reinforced epoxy resin-based composites and analysis of product properties.* Journal of Environmental Chemical Engineering, 2022. **10**(3): p. 107510.

17. Blazsó, M., *5 - Pyrolysis for recycling waste composites*, in *Management, Recycling and Reuse of Waste Composites*, V. Goodship, Editor 2010, Woodhead Publishing. p. 102-121.

18. Pimenta, S. and S.T. Pinho, *Recycling carbon fibre reinforced polymers for structural applications: Technology review and market outlook.* Waste Management, 2011. **31**(2): p. 378-392.

19. Nayak, K.C., et al., *Microstructural, physical and mechanical characterization of grinding sludge based aluminium metal matrix composite.* Materials Science and Engineering: A, 2020. **773**: p. 138895.

20. Borjan, D., Ž. Knez, and M. Knez *Recycling of Carbon Fiber-Reinforced Composites—Difficulties and Future Perspectives*. Materials, 2021. **14**, DOI: 10.3390/ma14154191.

21. Khalid, M.Y., et al., *Recent trends in recycling and reusing techniques of different plastic polymers and their composite materials.* Sustainable Materials and Technologies, 2022. **31**: p. e00382.

22. Krauklis, A.E., et al. *Composite Material Recycling Technology—State-of-the-Art and Sustainable Development for the 2020s*. Journal of Composites Science, 2021. **5**, DOI: 10.3390/jcs5010028.

23. Pickering, S.J., *Recycling technologies for thermoset composite materials—current status.* Composites Part A: Applied Science and Manufacturing, 2006. **37**(8): p. 1206-1215.

24. Shuaib, N.A. and P.T. Mativenga, *Effect of Process Parameters on Mechanical Recycling of Glass Fibre Thermoset Composites.* Procedia CIRP, 2016. **48**: p. 134-139.

25. Colucci, G., et al., *The effect of mechanical recycling on the microstructure and properties of PA66 composites reinforced with carbon fibers.* Journal of Applied Polymer Science, 2015. **132**(29).

26. Lopez-Urionabarrenechea, A., et al., *Reclamation of carbon fibers and added-value gases in a pyrolysis-based composites recycling process.* Journal of Cleaner Production, 2020. **273**: p. 123173.

27. Onwudili, J.A., et al., *Recovery of glass fibre and carbon fibres from reinforced thermosets by batch pyrolysis and investigation of fibre re-using as reinforcement in LDPE matrix.* Composites Part B: Engineering, 2016. **91**: p. 154-161.

28. Mazzocchetti, L., et al., *Validation of carbon fibers recycling by pyro-gasification: The influence of oxidation conditions to obtain clean fibers and promote fiber/matrix adhesion in epoxy composites.* Composites Part A: Applied Science and Manufacturing, 2018. **112**: p. 504-514.

29. Abdou, T.R., et al., *Recycling of polymeric composites from industrial waste by pyrolysis: Deep evaluation for carbon fibers reuse.* Waste Management, 2021. **120**: p. 1-9.

30. Yang, J., et al., *Recycling of carbon fibre reinforced epoxy resin composites under various oxygen concentrations in nitrogen–oxygen atmosphere.* Journal of Analytical and Applied Pyrolysis, 2015. **112**: p. 253-261.

31. Jeong, J.-S., et al., *Fast recovery process of carbon fibers from waste carbon fibers-reinforced thermoset plastics.* Journal of Environmental Management, 2019. **247**: p. 816-821.

32. Bartoli, M., et al., *An Overview of Temperature Issues in Microwave-Assisted Pyrolysis.* Processes, 2019. **7**(10): p. 658.

33. Jiang, L., et al., *Recycling carbon fiber composites using microwave irradiation: Reinforcement study of the recycled fiber in new composites.* Journal of Applied Polymer Science, 2015. **132**(41).

34. Li, Y.-F., et al. *An Experimental Study on Mechanical Behaviors of Carbon Fiber and Microwave-Assisted Pyrolysis Recycled Carbon Fiber-Reinforced Concrete*. Sustainability, 2021. **13**, DOI: 10.3390/su13126829.

35. Seiler, E., H. Urban, and U. Teipel, *Microwave Pyrolysis of Carbon-Fiber-Reinforced Plastics as Recycling Option.* Chemie Ingenieur Technik, 2020. **92**(4): p. 469-75.

36. Deng, J., et al., *Recycling of Carbon Fibers From CFRP Waste by Microwave Thermolysis.* Processes, 2019. **7**(4): p. 207.

37. Kwak, M., et al., *Microwave curing of carbon–epoxy composites: Penetration depth and material characterisation.* Composites Part A: Applied Science and Manufacturing, 2015. **75**: p. 18-27.

38. Obunai, K., T. Fukuta, and K. Ozaki, *Carbon fiber extraction from waste CFRP by microwave irradiation.* Composites Part A: Applied Science and Manufacturing, 2015. **78**: p. 160-165.

39. Hao, S., et al., *Recovery of Carbon Fibre From Waste Prepreg via Microwave Pyrolysis.* Polymers (Basel), 2021. **13**(8).

40. Zhang, L., et al., *Upcycling of carbon fiber-reinforced polymer composites.* Composites Science and Technology, 2023. **231**: p. 109824.

41. Pender, K. and L. Yang, *Investigation of catalyzed thermal recycling for glass fiber-reinforced epoxy using fluidized bed process.* Polymer Composites, 2019. **40**(9): p. 3510-3519.

42. Meng, F., et al., *Energy and environmental assessment and reuse of fluidised bed recycled carbon fibres.* Composites Part A: Applied Science and Manufacturing, 2017. **100**: p. 206-214.

43. Turner, T.A., S.J. Pickering, and N.A. Warrior, *Development of Recycled Carbon fibre moulding compounds – Preparation of waste composites.* Composites Part B: Engineering, 2011. **42**(3): p. 517-525.

44. Pickering, S.J., et al., *A fluidised-bed process for the recovery of glass fibres from scrap thermoset composites.* Composites Science and Technology, 2000. **60**(4): p. 509-523.

45. Chaabani, C., E. Weiss-Hortala, and Y. Soudais, *Impact of Solvolysis Process on Both Depolymerization Kinetics of Nylon 6 and Recycling Carbon Fibers From Waste Composite.* Waste and Biomass Valorization, 2017. **8**(8): p. 2853-2865.

46. Lebedeva, E.A., et al., *Application of Low-Temperature Solvolysis for Processing of Reinforced Carbon Plastics.* Russian Journal of Applied Chemistry, 2020. **93**(6): p. 845-853.

47. Oliveux, G., L.O. Dandy, and G.A. Leeke, *Current status of recycling of fibre reinforced polymers: Review of technologies, reuse and resulting properties.* Progress in Materials Science, 2015. **72**: p. 61-99.

48. Piñero-Hernanz, R., et al., *Chemical recycling of carbon fibre reinforced composites in nearcritical and supercritical water.* Composites Part A: Applied Science and Manufacturing, 2008. **39**(3): p. 454-461.

49. Keith, M.J., et al., *Recycling a carbon fibre reinforced polymer with a supercritical acetone/water solvent mixture: Comprehensive analysis of reaction kinetics.* Polymer Degradation and Stability, 2019. **161**: p. 225-234.

50. Kim, Y.N., et al., *Application of supercritical water for green recycling of epoxy-based carbon fiber reinforced plastic.* Composites Science and Technology, 2019. **173**: p. 66-72.

51. Okajima, I., et al., *Chemical recycling of carbon fiber reinforced plastic using supercritical methanol.* The Journal of Supercritical Fluids, 2014. **91**: p. 68-76.

52. Jiang, J., et al., *On the successful chemical recycling of carbon fiber/epoxy resin composites under the mild condition.* Composites Science and Technology, 2017. **151**: p. 243-251.

53. Hanaoka, T., et al., *Recovery of carbon fiber from prepreg using nitric acid and evaluation of recycled CFRP.* Composites Part B: Engineering, 2022. **231**: p. 109560.

54. Zabihi, O., et al., *Development of a low cost and green microwave assisted approach towards the circular carbon fibre composites.* Composites Part B: Engineering, 2020. **184**: p. 107750.

55. Das, S., *Life cycle assessment of carbon fiber-reinforced polymer composites.* The International Journal of Life Cycle Assessment, 2011. **16**(3): p. 268-282.

56. Karuppannan Gopalraj, S. and T. Kärki, *A review on the recycling of waste carbon fibre/glass fibre-reinforced composites: fibre recovery, properties and life-cycle analysis.* SN Applied Sciences, 2020. **2**(3): p. 433.

57. Wei, Y. and S.A. Hadigheh, *Cost benefit and life cycle analysis of CFRP and GFRP waste treatment methods.* Construction and Building Materials, 2022. **348**: p. 128654.

58. Pillain, B., et al., *Positioning supercritical solvolysis among innovative recycling and current waste management scenarios for carbon fiber reinforced plastics thanks to comparative life cycle assessment.* The Journal of Supercritical Fluids, 2019. **154**: p. 104607.

59. Petrakli, F., et al. *End-of-Life Recycling Options of (Nano)Enhanced CFRP Composite Prototypes Waste—A Life Cycle Perspective*. Polymers, 2020. **12**, DOI: 10.3390/polym12092129.

60. Mallick, P.K., *Fiber-Reinforced Composites: Materials, Manufacturing, and Design*. Vol. Third Edition. 2007, Boca Raton: CRC Press. 638

61. Opelt, C.V., G.M. Cândido, and M.C. Rezende, *Compressive failure of fiber reinforced polymer composites – A fractographic study of the compression failure modes.* Materials Today Communications, 2018. **15**: p. 218-227.

62. Camarena, E., R.J. Clarke, and B.L. Ennis, *Development of a compressive failure model for carbon fiber composites and associated uncertainties.* Composites Science and Technology, 2021. **211**: p. 108855.

63. Sharma, M., et al., *Carbon fiber surfaces and composite interphases.* Composites Science and Technology, 2014. **102**: p. 35-50.

64. Yang, B., et al., *Bending, compression, and shear behavior of woven glass fiber–epoxy composites.* Composites Part B: Engineering, 2000. **31**(8): p. 715-721.

APPENDIX A PREPREG TECHNICAL DATASHEET

TECHNICAL DATA SHEET

CYCOM® 5320-1

PREPREG

Table 4 | Lamina Level Properties – T650-35 8 Harness Satin Fabric

Material: Solvay Aerospace Materials CYCOM® 5320-1 T650-35 3K 8HS Fabric 36%			**Solvay – CYCOM 5320-1/8HS T650/35 3k 8-Harness Satin Fabric Lamina Mechanical Properties Summary**
Prepreg: CYCOM® 5320-1 T650-35 3K 8HS 36%			
Fiber: Solvay T650/35 3k	**Resin:** CYCOM® 5320-1		
Tg (dry): 374°F	**Tg (wet[1]):** 316°F	**Tg Method:** DMA (ASTM D7028, 35mm dual cantilever)	

Processing: full vacuum, vacuum bag only room temperature for 16 hours plus integrated 200°F/350°F cure/post cure cycle with a 200°F cure for 10 hours followed immediately under the same vacuum bag by a post cure at 350°F for two hours.

Data reported as normalized (except where noted by parenthesis) by 0.0145 in. (56.7% nominal fiber volume)

Condition	-100°F Dry	75°F Dry	250°F Dry	300°F Dry	180°F Wet[1]	250°F Wet[1]
Property	Mean	Mean	Mean	Mean	Mean	Mean
0° Tensile ASTM D3039						
Strength (ksi)	117.4	131.3	128.8		138.0	136.4
Modulus (Msi)	10.20	10.02	10.06		10.20	9.97
Poisson's Ratio	0.056	0.048	0.042		0.041	0.041
90° Tensile ASTM D3039						
Strength (ksi)	119.3	129.9	133.8	137.0	138.4	134.2
Modulus (Msi)	10.14	9.90	10.00	10.00	10.02	9.94
0° Compression ASTM D6641						
Strength (ksi)	121.1	108.8	93.9		77.6	66.0
Modulus (Msi)	10.01	9.18	9.24		9.23	9.18
90° Compression ASTM D6641						
Strength (ksi)	122.3	110.6	95.4	87.8	87.9	66.7
Modulus (Msi)	9.42	9.18	9.20	9.30	9.36	9.15
±45° Tensile In-Plane Shear (500-3,000 µstrain chord) ASTM D3518						
0.2% Offset Strength (ksi)	(12.13)	(8.29)	(5.67)	(4.72)	(5.23)	(3.31)
Shear Modulus (Msi)	(0.95)	(0.81)	(0.72)	(0.67)	(0.71)	(0.53)
0° Short Beam ASTM D2344						
Strength (ksi)	(12.39)	(11.93)	(9.68)	(8.51)	(8.66)	(6.45)

[1] Wet conditioning to equilibrium at 85%RW and 160°F

APPENDIX B RESIN TECHNICAL DATASHEET

HUNTSMAN
Enriching lives through innovation

Advanced Materials

Araldite®LY 8601 / Aradur® 8602 System

Freeman 360° Account Become a member!

EPOXY RESIN SYSTEM

DESCRIPTION:

Araldite®LY 8601 (Resin) / Aradur® 8602 (Hardener) is a two-component, low-viscosity epoxy system developed for use in the production of advanced composites using vacuum-assisted resin transfer molding (VARTM), resin transfer molding (RTM), Seemans Composite Resin Injection Molding Process (SCRIMP℠), or other infusion processes. The low-mixed viscosity and wet-out potential of Araldite® LY 8601 / Aradur® 8602 enhance processability parameters.

Araldite® LY 8601 / Aradur® 8602 has excellent toughness, and the increased reactivity allows for a faster ambient temperature cure and reduced de-mold time.

MIX RATIO:

By weight: 100 to 25 Resin to Hardener

Mixing Instructions: Measure each component accurately (± 5%) into clean containers. Thoroughly mix resin and hardener together (minimum 2 minutes) scraping container sidewalls, bottom and mixing stick several times to assure a uniform mix.

TYPICAL HANDLING PROPERTIES:

Tested @ 77 °F (25 °C) unless otherwise noted.

Property	Criteria	ASTM Test Method	Test Value
Color	Mixed		Transparent
Viscosity, cP	Resin	D-2393	580
	Hardener		25
	Mixed		175
Gel Time, minutes	14 fl. oz.	D-2471	70

NOTE : Typical Properties – These physical properties are reported as typical test values obtained by our test laboratory. If assistance is needed establishing product specifications, please consult with our Quality Control Department.

RECOMMENDED CURE SCHEDULE:

Temperature	Demold Time	Full Cure Time
77 °F (25 °C)	24 Hours	3 Days

HUNTSMAN
Enriching lives through innovation

NEAT SYSTEM

TYPICAL CURED PROPERTIES:

Tested @ 77 °F (25 °C) and cured 7 days @ 77°F (25°C) unless otherwise noted.

Property	ASTM Test Method	Test Values[1]
Specific Gravity	D-792	1.12
Cubic inch per Pound	--	24.6
Hardness (Shore D)	D-2240	82
Ultimate Flexural Strength, psi	D-790	11,013
Flexural Modulus, psi	D-790	322,560
Ultimate Tensile Strength, psi	D-638	7,871
Tg by DMA, °F (°C)	D-4065	164 (73)
Linear Shrinkage, Mold 0 in/in	D-2566	0.001
Ultimate Compressive Strength, psi	D-695	15,410
Compressive Modulus, psi	--	305,432
% Elongation	D-638	6
Coefficient of Thermal Expansion, in/in/°F -22 to 86 °F (-30° to 30 °C)	D-3386	42 x 10^{-6}
Izod Impact, Notched, ft.lb./in.	D-256	0.4116

NOTE : All properties are of neat product form (non-composite)

INFUSION PROCESS

TYPICAL CURED PROPERTIES:

Tested @ 77 °F (25 °C) unless otherwise noted.

Property	ASTM Test Method	Test Values[1]	Test Values[2]
Hardness (Shore D)	D-2240	88	92
Ultimate Flexural Strength, psi	D-790	47,600	102,165
Flexural Modulus, psi	D-790	2.5 x 10^6	6.9 x 10^6
Ultimate Tensile Strength, psi	D-638	44,545	72,851
Ultimate Compressive Strength, psi	D-695	30,023	45,387
Compressive Modulus	D-695	3.6 x 10^6	9.7 x 10^6
% Elongation	D-638	1.6	1.0

LAY-UP PROCESS:

	Glass Laminate[1]	Graphite Laminate[2]
Panel Type :	Approximately 2 ft. x 2 ft. flat panel	Approximately 2 ft. x 2 ft. flat panel
Cloth Type :	8 layer, Volan A 7500, 10oz	8 layer, 3K, 70P
Cloth Rotation :	0 degrees	0 degrees
Procedure :	VARTM, flat panel	VARTM, flat panel
Cure Schedule:	7 days @ 77°F	7days @ 77°F

CONDITIONING:

Stir well before use. This material will separate.

APPENDIX C V.A.R.I METHOD

Different steps of the VARI method used in manufacturing composite plates are as follows:

1- The glass table used as the mold for the process was first cleaned thoroughly with acetone and WypAll wipers. This step is necessary for having a clean and glassy surface, and for removing any extra material from the mold, which results in better adherence of the sealant tape to the mold and a better vacuumed mold.

2- All around the mold and the inlet and outlet for resin were separated by Scotch tape. This ensures the prevention of penetration of release agent to adhesive sealants.

3- Release agent was applied to the mold area (area between Scotch tape) in 4 steps, with a 10-15

Minute period between each step. Release agent prevents bonding of the material to the surface of the table.

4- Scotch tape was removed, and adhesive sealants were put instead.

5- After measuring the weight, recovered fibers were put in the middle of the mold, the peel ply and mesh were placed over them. Peel ply is used for easily removing the extra materials from finished composite plate. Mesh is placed over the fibers to ensure the smooth and even flow of the resin over the surface of the fibers. Positioning of CFs, peel ply and mesh is demonstrated in figure A.1.

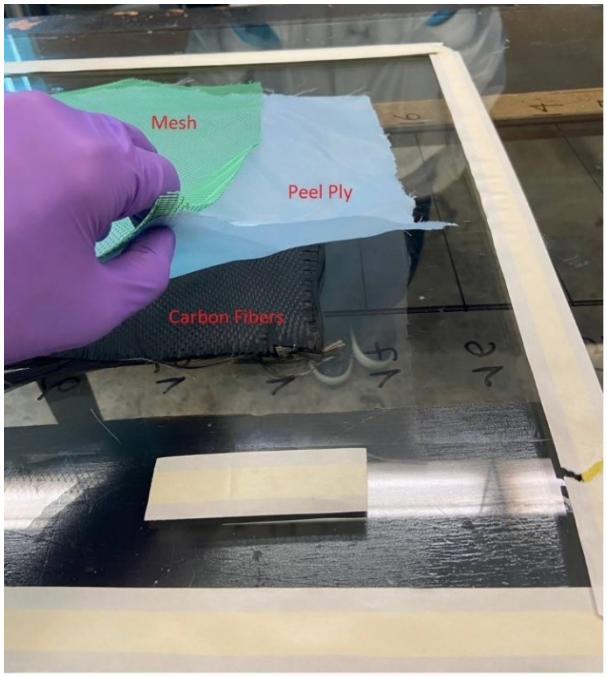


Figure A.1: Carbon Fibers covered by Peel ply and mesh

6- Two sets (inlet and outlet) of spirals connected to the pipes were prepared and attached to the mold.

7- Vacuum bagging film was attached to the sealants around the mold, making a uniform vacuum environment for the mold.

After this step, the inlet pipe was blocked by the clamps, and the outlet was connected to the catch pot. To inspect the mold and to figure out if there is any leakage, Central vacuum was connected to the catch pot, and a negative vacuum below -25 mmHg was applied to the system. Once the desired pressure was approached, the central vacuum was disconnected from the catch pot, and the system was left for one hour to see if there would be any pressure drops, which could be a sign of a leakage in the system. As indicated by figure A.2, the vacuum gauge mounted on the catch pot shows excellent vacuum conditions, without any leakage in the mold and process.

Figure A.2: Vacuum gauge and the pressure on it before doing the infusion process

8- After ensuring that there is no leakage and pressure drop in the system, the inlet pipe was put inside the mixture of resin and hardener, and clamp was removed, so that the mixture could easily flow into the mold and the infusion process started.

9- Once the resin approached the outlet pipe and all the fibers were wet enough, the inlet and outlet were clamped again, and the system was isolated for at least one day.

Figure A.3 illustrates the mold during the infusion process. The inlet pipe on the right is inside the matrix, while the outlet pipe on the left is connected to the catch pot.

Illustration of the whole system used in V.A.R.I. with all the parts is shown in figure A.4.

It should be noted that the excessive length of inlet and outlet pipes will cause loss of resin inside the pipes.

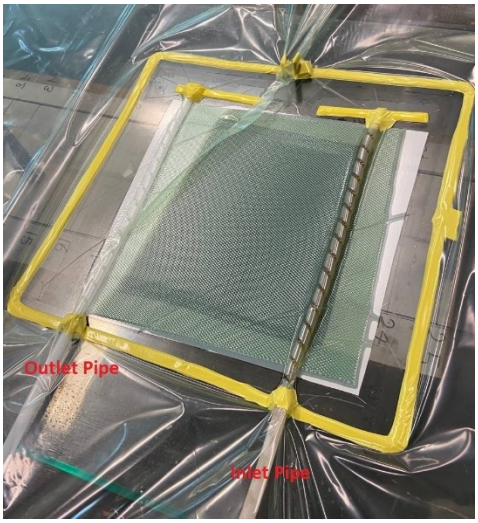


Figure A.3: Infusion of resin into the mold

Figure A.4: V.A.R.I. method and the parts used in the process

APPENDIX D DENSITY TEST REPORTS

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 26 février 2023
Rapport: 6391-001S-1A-fr

IDENTIFICATION:	Plaque de fibre de carbone recyclée (500°C, 2.5 hours): #1 Réception: 31 mai 2022	
NORME:		
ESSAI:	"Density and Specific Gravity (Relative Density) of Plastics by Displacement"	ASTM D792-13 Méthode B
CONDITIONS D'ESSAI:	Méthode d'essai B; Atmosphère de conditionnement: minimum 40 heures à 23°C, 50% H.R. ; Liquide d'immersion: Éthanol Température du liquide d'immersion (°C): 23.5 Date de l'essai: 27 juin 2022	

RÉSULTATS:	Résultats individuels			Moy.	E.-T.	CV %
Densité (g/cm³):	1.492	1.489	1.491	**1.491**	0.002	**0.1**

Préparé par:
Stacy Lanéuville
Stacy Lanéuville-Frost, Tech.
Technicien(ne)

Approuvé par:
Cynthie Dega
Cynthie Dega, M.Ing.
Directeur innovation-Composites

Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens — Date: 26 février 2023
Centre des technologies Textiles-PART-4 (CTT-084) — Rapport: 6391-001S-2A-fr

IDENTIFICATION:	Plaque de fibre de carbone recyclée (500°C, 2.5 hours): #2 Réception: 31 mai 2022	
NORME:		
ESSAI:	"Density and Specific Gravity (Relative Density) of Plastics by Displacement"	ASTM D792-20 Méthode B
CONDITIONS D'ESSAI:	Méthode d'essai B; Atmosphère de conditionnement: minimum 40 heures à 23°C, 50% H.R. ; Liquide d'immersion: Éthanol Température du liquide d'immersion (°C): 23.5 Date de l'essai: 27 juin 2022	

RÉSULTATS:	Résultats individuels			Moy.	E.-T.	CV %
Densité (g/cm³):	1.475	1.474	1.476	**1.475**	0.001	**0.1**

Préparé par:
Stacy Laneuville-Frost, Tech.
Technicien(ne)

Approuvé par:
Cynthie Dega, M.Ing.
Directeur innovation-Composites
Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 26 février 2023
Rapport: 6391-001S-5A-fr

IDENTIFICATION:	Plaque de fibre de carbone recyclée: Sample #1, 500°C, 2,5h Réception: 2 août 2022	
NORME:		
ESSAI:	"Density and Specific Gravity (Relative Density) of Plastics by Displacement"	ASTM D792-20 Méthode B
CONDITIONS D'ESSAI:	Méthode d'essai B; Liquide d'immersion: Ethanol Température du liquide d'immersion (°C): 23.1 Date de l'essai: 12 septembre 2022	

RESULTATS:	Résultats individuels			Moy.	E.-T.	CV %
Densité (g/cm³):	1.480	1.480	1.474	**1.478**	0.003	**0.2**

Préparé par:
Stacy Laneuville-Frost, Tech.
Technicien(ne)

Approuvé par:
Cynthie Dega, M.Ing.
Directeur innovation-Composites

Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 26 février 2023
Rapport: 6391-001S-6A-fr

IDENTIFICATION:	Plaque de fibre de carbone recyclée: Sample #2, 500°C, 2,5h Réception: 2 août 2022	
NORME:		
ESSAI:	"Density and Specific Gravity (Relative Density) of Plastics by Displacement"	ASTM D792-20 Méthode B
CONDITIONS D'ESSAI:	Méthode d'essai B; Atmosphère de conditionnement: minimum 40 heures à 23°C, 50% H.R. ; Liquide d'immersion: Ethanol Température du liquide d'immersion (°C): 22.9 Date de l'essai: le 12 septembre 2022	

RESULTATS:	Résultats individuels			Moy.	E.-T.	CV %
Densité (g/cm³):	1.500	1.497	1.503	**1.500**	0.003	**0.2**

Préparé par:
Donaldo Lopez-Martelo, M.Sc.A.
Technicien(ne)

Approuvé par:
Cynthie Dega, M.Ing.
Directeur innovation-Composites

Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

APPENDIX E VOID CONTENT TEST RESULTS

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 26 février 2023
Rapport: 6391-001S-1A-fr

IDENTIFICATION: Plaque de fibre de carbone recyclée (500°C, 2.5 hours): #1
Réception: 31 mai 2022

NORME:

ESSAI: "Constituent Content of Composite Materials - Void Content" — ASTM D3171-15, Proc. B

CONDITIONS D'ESSAI:
Méthode utilisée: Méthode I;
Procédure utilisée: B—Digestion de la matrice avec Acide sulfurique/Peroxyde d'hydrogène;
Densité des fibres fournie par le client (g/cm³): 1.770
Densité de la résine fournie par le client (g/cm³): 1.12
Temps de séchage (h): Minimum 2
Température du four (°C): 105 ± 3°C
Date de l'essai: 27 juillet 2022

RÉSULTATS:	Résultats individuels			Moy.	E.-T.	CV %
Masse du spécimen (g):	2.3648	2.3926	2.3630	**2.3735**	0.0166	**0.7**
Densité du spécimen (g/cm³):	1.492	1.489	1.491	**1.491**	0.002	**0.1**
Pourcentage de poids du renfort (%):	67.8	68.4	66.7	**67.6**	0.9	**1.3**
Pourcentage volumique de renfort (%):	57.2	57.6	56.2	**57.0**	0.7	**1.3**
Pourcentage de poids de la matrice (%):	32.2	31.6	33.3	**32.4**	0.9	**2.7**
Pourcentage volumique de matrice (%):	42.9	42.0	44.3	**43.1**	1.2	**2.7**
Pourcentage volumique de vide (%):	0.0	0.5	-0.5	**0.0**	0.5	**0.0**

Préparé par: Stacy Laneuville
Stacy Laneuville-Frost, Tech.
Technicien(ne)

Approuvé par: Cynthie Dega
Cynthie Dega, M.Ing.
Directeur innovation-Composites
Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 26 février 2023
Rapport: 6391-001S-2A-fr

IDENTIFICATION: Plaque de fibre de carbone recyclée (500°C, 2.5 hours): #2
Réception: 31 mai 2022

NORME:

ESSAI: "Constituent Content of Composite Materials - Void Content" — ASTM D3171-15, Proc. B

CONDITIONS D'ESSAI:
Méthode utilisée: Méthode I;
Procédure utilisée: B—Digestion de la matrice avec Acide sulfurique/Peroxyde d'hydrogène;
Densité des fibres fournie par le client (g/cm³): 1.770
Densité de la résine fournie par le client (g/cm³): 1.12
Temps de séchage (h): Minimum 2
Température du four (°C): 105 ± 3°C
Date de l'essai: 27 juillet 2022

RÉSULTATS:	Résultats individuels			Moy.	E.-T.	CV %
Masse du spécimen (g):	2.3695	2.3752	2.4148	**2.3865**	0.0247	**1.0**
Densité du spécimen (g/cm³):	1.475	1.474	1.476	**1.475**	0.001	**0.1**
Pourcentage de poids du renfort (%):	65.5	66.4	65.9	**65.9**	0.5	**0.7**
Pourcentage volumique de renfort (%):	54.6	55.3	54.9	**54.9**	0.4	**0.6**
Pourcentage de poids de la matrice (%):	34.5	33.6	34.1	**34.1**	0.5	**1.3**
Pourcentage volumique de matrice (%):	45.4	44.3	45.0	**44.9**	0.6	**1.2**
Pourcentage volumique de vide (%):	0.0	0.4	0.1	**0.2**	0.2	**124.9**

Préparé par:
Stacy Laneuville
Stacy Laneuville-Frost, Tech.
Technicien(ne)

Approuvé par:
Cynthie Dega
Cynthie Dega, M.Ing.
Directeur innovation-Composites

Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 26 février 2023
Rapport: 6391-001S-5A-fr

IDENTIFICATION: Plaque de fibre de carbone recyclée: Sample #1, 500°C, 2,5h
Réception: 2 août 2022

NORME:

ESSAI: "Constituent Content of Composite Materials - Void Content" — ASTM D3171-15, Proc. B

CONDITIONS D'ESSAI: Méthode utilisée: Méthode I;
Procédure utilisée: B—Digestion de la matrice avec Acide sulfurique/Peroxyde d'hydrogène;
Densité des fibres fournie par le client (g/cm³): 1.770
Densité de la résine fournie par le client (g/cm³): 1.12
Temps de séchage (h): Minimum 2
Température du four (°C): 105 ± 3°C
Date de l'essai: 13 septembre 2022

RÉSULTATS:	Résultats individuels			Moy.	E.-T.	CV %
Masse du spécimen (g):	2.6354	2.6084	2.6279	**2.6239**	0.0139	**0.5**
Densité du spécimen (g/cm³):	1.481	1.481	1.474	**1.479**	0.004	**0.3**
Pourcentage de poids du renfort (%):	77.7	74.5	73.2	**75.1**	2.3	**3.1**
Pourcentage volumique de renfort (%):	65.0	62.3	61.0	**62.8**	2.0	**3.3**
Pourcentage de poids de la matrice (%):	22.3	25.5	26.8	**24.9**	2.3	**9.3**
Pourcentage volumique de matrice (%):	29.5	33.7	35.2	**32.8**	3.0	**9.0**
Pourcentage volumique de vide (%):	5.5	4.0	3.8	**4.4**	0.9	**21.0**

Préparé par:
Stacy Lanéville
Stacy Lanéville-Frost, Tech.
Technicien(ne)

Approuvé par:
Cynthie Dega
Cynthie Dega, M.Ing.
Directeur innovation-Composites

Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens — Date: 26 février 2023
Centre des technologies Textiles-PART-4 (CTT-084) — Rapport: 6391-001S-6A-fr

IDENTIFICATION: Plaque de fibre de carbone recyclée: Sample #2, 500°C, 2,5h
Réception: 2 août 2022

NORME:

ESSAI: "Constituent Content of Composite Materials - Void Content" — ASTM D3171-15, Proc. B

CONDITIONS D'ESSAI: Méthode utilisée: Méthode I;
Procédure utilisée: B—Digestion de la matrice avec Acide sulfurique/Peroxyde d'hydrogène;
Densité des fibres fournie par le client (g/cm³): 1.770
Densité de la résine fournie par le client (g/cm³): 1.12
Temps de séchage (h): Minimum 2
Température du four (°C): 105 ± 3°C
Date de l'essai: 13 septembre 2022

RÉSULTATS:	Résultats individuels			Moy.	E.-T.	CV %
Masse du spécimen (g):	2.6733	2.6926	2.6208	**2.6622**	0.0372	**1.4**
Densité du spécimen (g/cm³):	1.500	1.497	1.503	**1.500**	0.003	**0.2**
Pourcentage de poids du renfort (%):	71.1	70.8	71.9	**71.3**	0.6	**0.8**
Pourcentage volumique de renfort (%):	60.3	59.9	61.1	**60.4**	0.6	**1.0**
Pourcentage de poids de la matrice (%):	28.9	29.2	28.1	**28.7**	0.6	**2.0**
Pourcentage volumique de matrice (%):	38.7	39.0	37.7	**38.5**	0.7	**1.8**
Pourcentage volumique de vide (%):	1.0	1.1	1.2	**1.1**	0.1	**9.1**

Préparé par: Stacy Laneuville
Stacy Laneuville-Frost, Tech.
Technicien(ne)

Approuvé par: Cynthie Dega
Cynthie Dega, M.Ing.
Directeur innovation-Composites — Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

APPENDIX F GLASS TRANSITION TEMPERATURE TEST RESULTS

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens — Date: 26 février 2023
Centre des technologies Textiles-PART-4 (CTT-084) — Rapport: 6391-001S-1A-fr

IDENTIFICATION: Plaque de fibre de carbone recyclée (500°C, 2.5 hours): #1
Réception: 31 mai 2022

NORME:

ESSAI: "Glass Transition Temperature (DMA Tg) of Polymer Matrix Composites by Dynamic Mechanical Analysis" — ASTM D7028-07(2015)

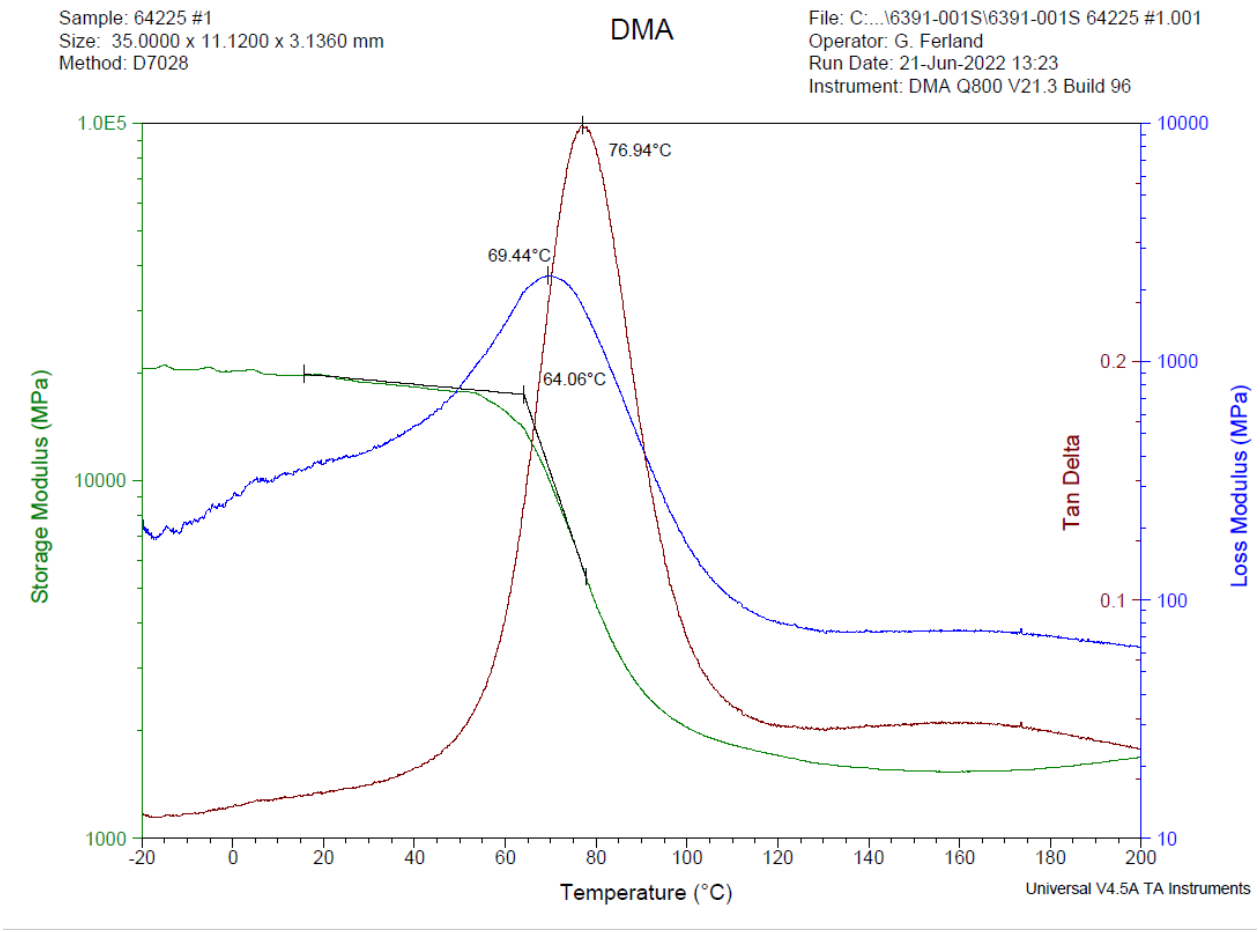
CONDITIONS D'ESSAI:
Appareil utilisé: TA Instruments Q800;
Procédure de calibration utilisée: électronique, force, dynamique, attache (masse, décalage, conformité), température;
Géométrie des spécimens d'essai: rectangulaire
Type d'attache: Dual Cantilever
Mode de chargement du DMA: Multi-Frequency-Strain
Fréquence (Hz): 1
Amplitude (µm): 20
Taux de chauffe (°C/min): 5
Gaz de purge: air
La méthode tangente est utilisée pour déterminer la température de transition vitreuse ;
Date de l'essai: le 21 et 22 juin 2022

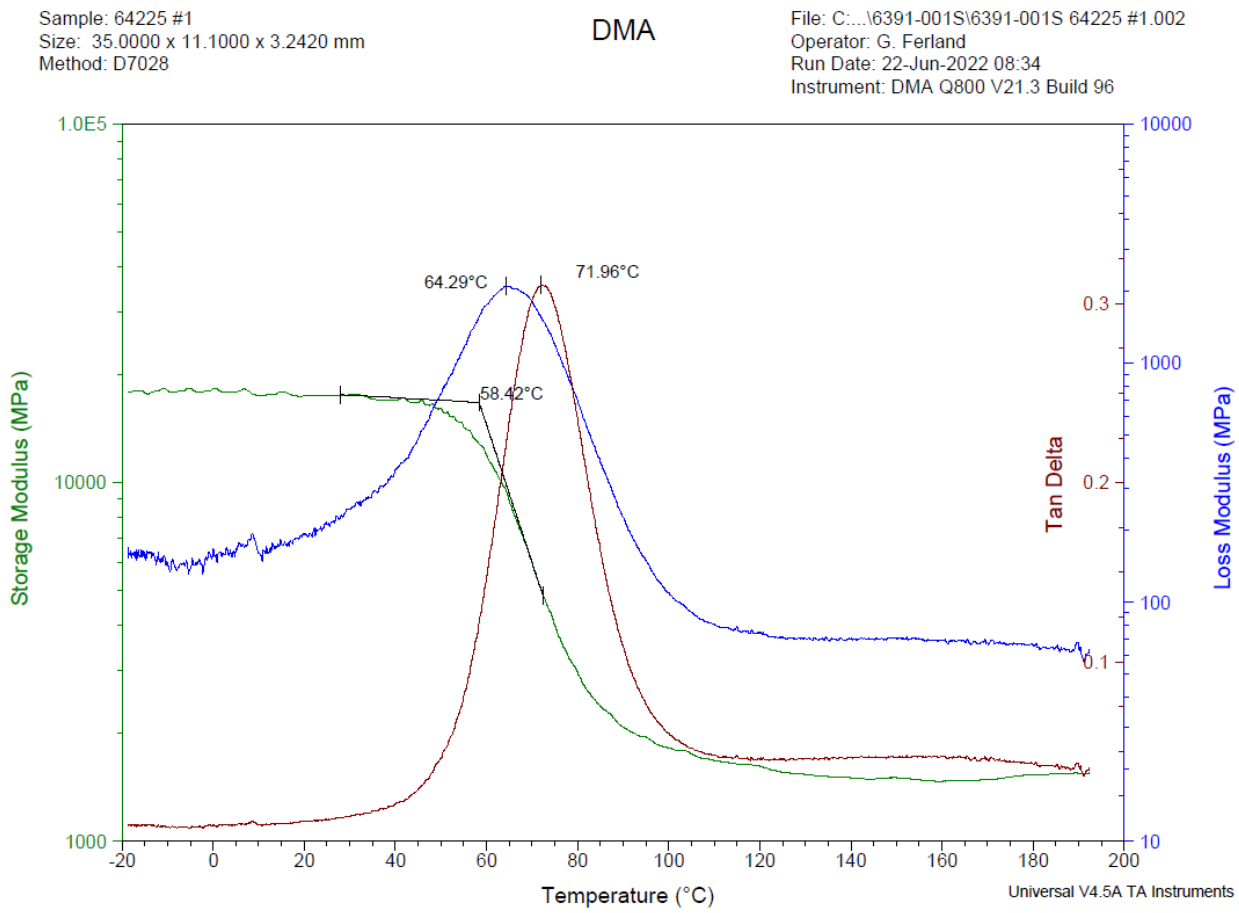
RÉSULTATS:	Résultats individuels		Moy.	E.-T.	CV %
Longueur entre les appuis (mm):	35.0				
Largeur du spécimen (mm):	11.12	11.10	**11.11**	0.01	**0.1**
Épaisseur du spécimen (mm):	3.136	3.242	**3.189**	0.075	**2.4**
Poids de l'échantillon avant DMA Tg (g):	3.064	3.076	**3.070**	0.008	**0.3**
Poids de l'échantillon après DMA Tg (g):	3.034	3.073	**3.053**	0.028	**0.9**
Perde de poids après DMA Tg (%):	0.979	0.0975	**0.5382**	0.6233	**115.8**
Température de transition vitreuse, DMA Tg (°C):	64.1	58.4	**61.3**	4.0	**6.6**
Valeur du pic delta tangent, Tt (°C):	76.9	72.0	**74.5**	3.5	**4.7**

Préparé par: Gabriel Ferland, Tech.
Technicien(ne)

Approuvé par: Cynthie Dega, M.Ing.
Directeur innovation-Composites — Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.


Sample: 64225 #1
Size: 35.0000 x 11.1200 x 3.1360 mm
Method: D7028
DMA
File: C:...\6391-001S\6391-001S 64225 #1.001
Operator: G. Ferland
Run Date: 21-Jun-2022 13:23
Instrument: DMA Q800 V21.3 Build 96
76.94°C
69.44°C
64.06°C
Storage Modulus (MPa)
Tan Delta
Loss Modulus (MPa)
Temperature (°C)
Universal V4.5A TA Instruments


Sample: 64225 #1
Size: 35.0000 x 11.1000 x 3.2420 mm
Method: D7028
DMA
File: C:...\6391-001S\6391-001S 64225 #1.002
Operator: G. Ferland
Run Date: 22-Jun-2022 08:34
Instrument: DMA Q800 V21.3 Build 96
64.29°C
71.96°C
58.42°C
Storage Modulus (MPa)
Tan Delta
Loss Modulus (MPa)
Temperature (°C)
Universal V4.5A TA Instruments

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens — Date: 26 février 2023
Centre des technologies Textiles-PART-4 (CTT-084) — Rapport: 6391-001S-5A-fr

IDENTIFICATION: Plaque de fibre de carbone recyclée: Sample #1, 500°C, 2,5h
Réception: 2 août 2022

NORME:

ESSAI: "Glass Transition Temperature (DMA Tg) of Polymer Matrix Composites by Dynamic Mechanical Analysis" — ASTM D7028-07(2015)

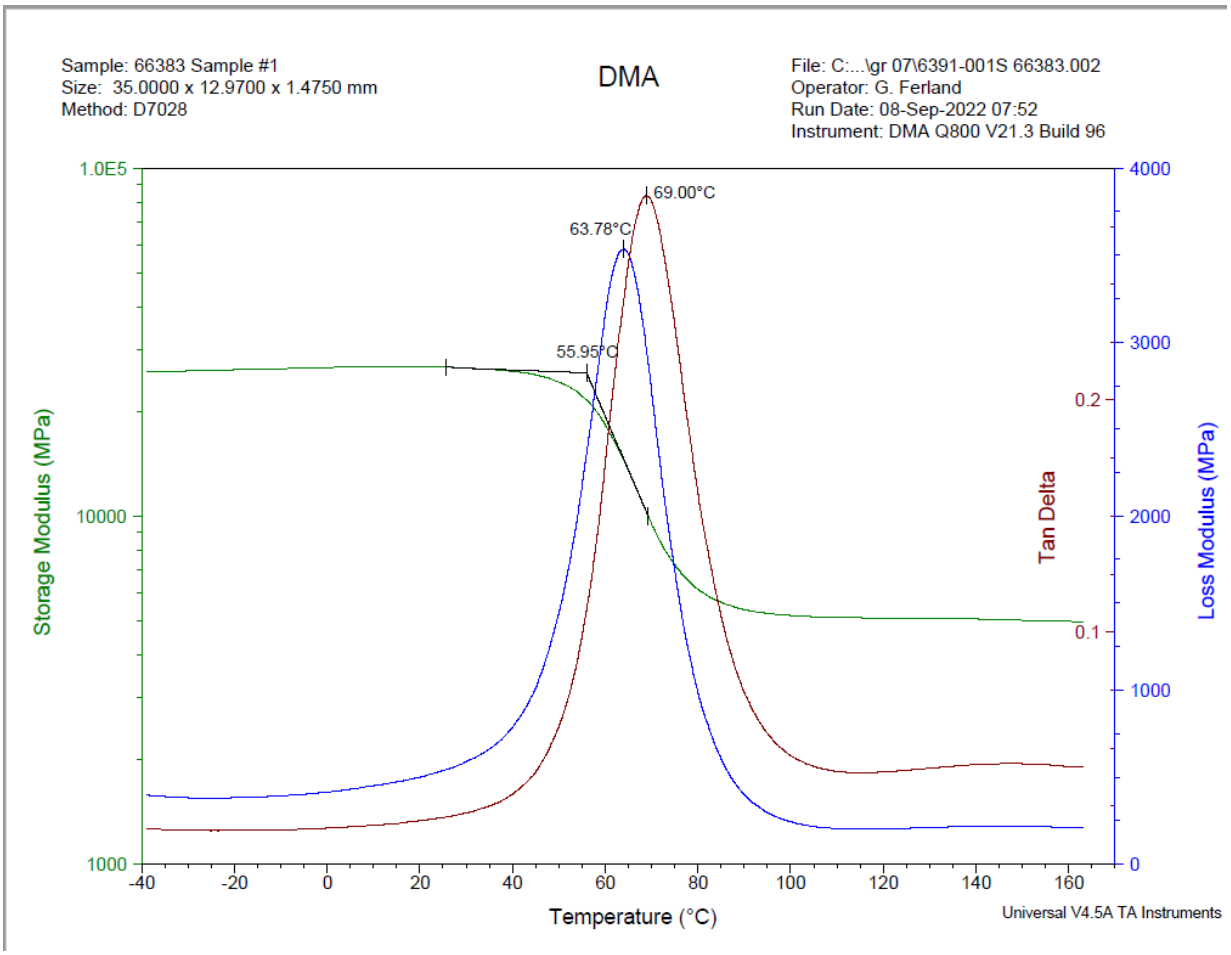
CONDITIONS D'ESSAI: Appareil utilisé: TA Instruments Q800;
Procédure de calibration utilisée: électronique, force, dynamique, attache (masse, décalage, conformité), température;
Géométrie des spécimens d'essai: rectangulaire
Type d'attache: Dual Cantilever
Mode de chargement du DMA: Multi-Frequency-Strain
Fréquence (Hz): 1
Déformation (%): 0.1
Taux de chauffe (°C/min): 5
Gaz de purge: air
La méthode tangente est utilisée pour déterminer la température de transition vitreuse ;
Date de l'essai: le 8 septembre 2022

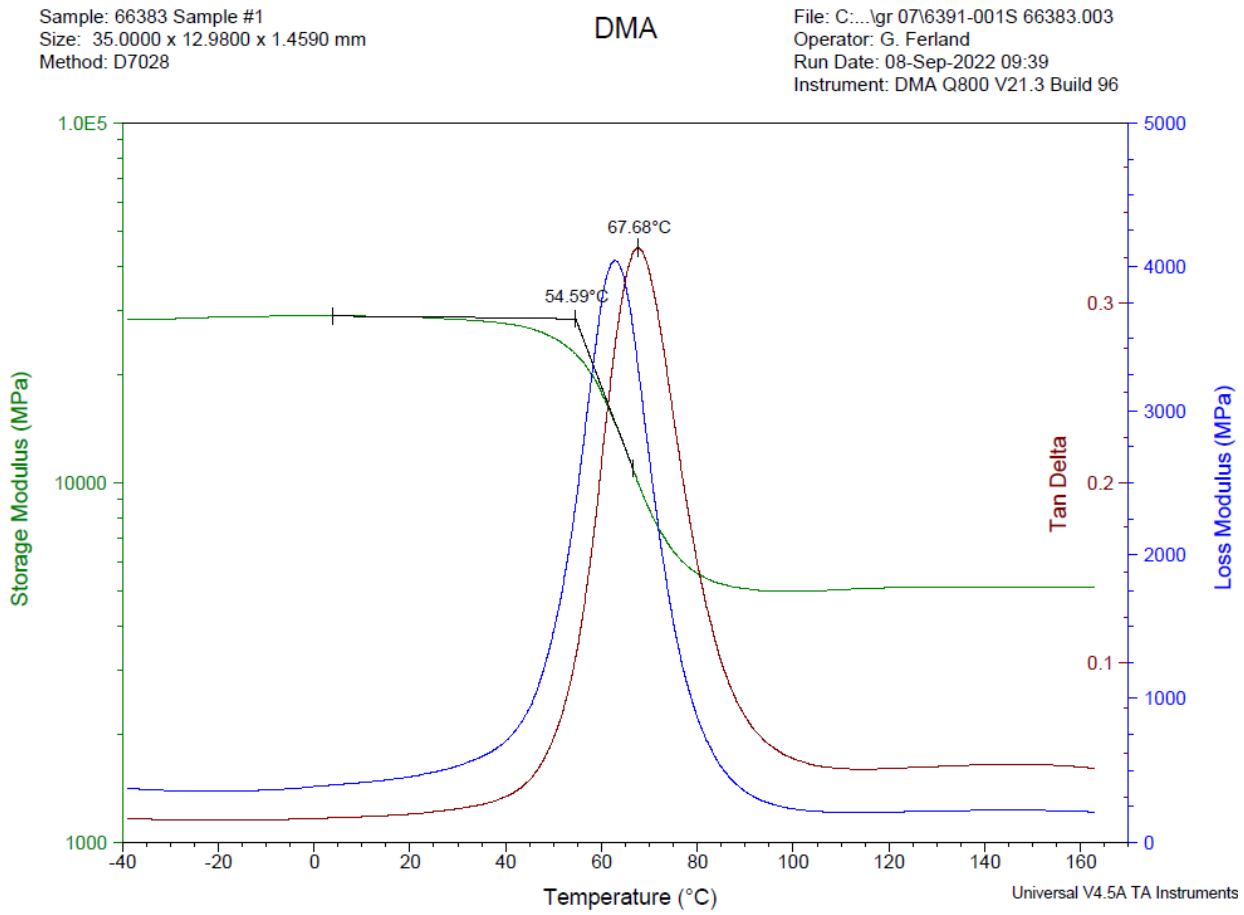
RÉSULTATS:	Résultats individuels		Moy.	E.-T.	CV %
Longueur entre les appuis (mm):	35.0				
Largeur du spécimen (mm):	12.97	12.98	**12.98**	0.01	**0.1**
Épaisseur du spécimen (mm):	1.475	1.459	**1.467**	0.011	**0.8**
Poids de l'échantillon avant DMA Tg (g):	1.495	1.500	**1.498**	0.004	**0.2**
Poids de l'échantillon après DMA Tg (g):	1.491	1.496	**1.494**	0.004	**0.2**
Perde de poids après DMA Tg (%):	0.268	0.267	**0.268**	0.001	**0.3**
Température de transition vitreuse, DMA Tg (°C):	56.0	54.6	**55.3**	1.0	**1.8**
Valeur du pic delta tangent, Tt (°C):	69.0	67.7	**68.4**	0.9	**1.3**

Préparé par:
Gabriel Ferland, Tech.
Technicien(ne)

Approuvé par:
Cynthie Dega, M.Ing.
Directeur innovation-Composites
Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.


Sample: 66383 Sample #1
Size: 35.0000 x 12.9700 x 1.4750 mm
Method: D7028
DMA
File: C:...\gr 07\6391-001S 66383.002
Operator: G. Ferland
Run Date: 08-Sep-2022 07:52
Instrument: DMA Q800 V21.3 Build 96
69.00°C
63.78°C
55.95°C
Storage Modulus (MPa)
Tan Delta
Loss Modulus (MPa)
Temperature (°C)
Universal V4.5A TA Instruments


Sample: 66383 Sample #1
Size: 35.0000 x 12.9800 x 1.4590 mm
Method: D7028
DMA
File: C:...\gr 07\6391-001S 66383.003
Operator: G. Ferland
Run Date: 08-Sep-2022 09:39
Instrument: DMA Q800 V21.3 Build 96
67.68°C
54.59°C
Storage Modulus (MPa)
Tan Delta
Loss Modulus (MPa)
Temperature (°C)
Universal V4.5A TA Instruments

APPENDIX G TENSILE PROPERTIES TEST RESULTS

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 26 février 2023
Rapport: 6391-001S-1A-fr

IDENTIFICATION:	Plaque de fibre de carbone recyclée (500°C, 2.5 hours): #1 Réception: 31 mai 2022
NORME:	
ESSAI:	"Tensile Properties of Polymer Matrix Composite Materials" ASTM D3039/D3039M-17
CONDITIONS D'ESSAI:	Atmosphère de conditionnement: 23±2°C, 50±5% H.R.; Appareil utilisé: Dynamomètre à Taux Constant d'Extension (TCE); Dimensions des spécimens d'essai: 1 po x 10 po (coupe à l'eau); Mâchoires utilisées: "Wedge action"; Distance entre les mâchoires: 6 po; Appareil utilisé: Zwick Z250 (#787299) Extensomètre utilisé: MakroXtens Zone de déformation utilisée pour le module et le Coefficient de Poisson: 1000-3000 microstrain : Vitesse de l'essai (mm/min): 0.5 Vitesse de l'essai (po/min): 0.02 Date de l'essai: le 9 juin 2022

RESULTATS:	Résultats individuels			Moy.	E.-T.	CV %
Épaisseur (mm):	3.00	3.00	2.97	**2.99**	0.02	**0.6**
Largeur (mm):	23.8	23.8	23.8	**23.8**	0.0	**0.0**
Force à la rupture (N):	50200	48900	47800	**48 967**	1 201	**2.5**
Force à la rupture (lbf):	11300	11000	10800	**11 033**	252	**2.3**
Résistance à la rupture (MPa):	702	685	676	**688**	13	**1.9**
Résistance à la rupture (ksi):	102	99.3	98.1	**99.8**	2.0	**2.0**
Déformation à la rupture (%):	1.0	1.0	1.0	**1.0**	0.0	**0.0**
Module d'élasticité "chord" (MPa):	67500	68300	65800	**67 200**	1 277	**1.9**
Module d'élasticité "chord" (ksi):	9800	9900	9540	**9 747**	186	**1.9**
Module d'élasticité "chord" (Mpsi):	9.80	9.90	9.54	**9.75**	0.19	**1.9**
Temps à la rupture (min):	6.7	6.6	6.4	**6.6**	0.2	**2.3**
Mode de rupture:	LWV	LWV	LWV			

Préparé par:
Patrick Dubois,
Technicien(ne)

Approuvé par:
Cynthie Dega, M.Ing.
Directeur innovation-Composites
Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 26 février 2023
Rapport: 6391-001S-5A-fr

IDENTIFICATION: Plaque de fibre de carbone recyclée: Sample #1, 500°C, 2,5h
Réception: 2 août 2022

NORME:

ESSAI: "Tensile Properties of Polymer Matrix Composite Materials" ASTM D3039/D3039M-17

CONDITIONS D'ESSAI:
Atmosphère de conditionnement: 23±2°C, 50±5% H.R.;
Appareil utilisé: Dynamomètre à Taux Constant d'Extension (TCE);
Dimensions des spécimens d'essai: 1 po x 10 po (coupe à l'eau);
Mâchoires utilisées: "Wedge action";
Distance entre les mâchoires: 6 po;
Appareil utilisé: ZWICK Z250 (#787299)
Extensomètre utilisé: MakroXtens
Zone de déformation utilisée pour le module et le Coefficient de Poisson: 1000-3000 microstrain ;
Vitesse de l'essai (po/min): 0.02
Date de l'essai: le 9 aout 2022

RÉSULTATS:	Résultats individuels					Moy.	E.-T.	CV %
Épaisseur (po):	0.0555	0.0563	0.0547	0.0539	0.0567	**0.0554**	0.0011	**2.1**
Largeur (po):	1.00	1.00	1.00	0.998	1.00	**1.00**	0.00	**0.1**
Force à la rupture (lbf):	5600	5790	5430	5810	5700	**5 666**	156	**2.8**
Résistance à la rupture (ksi):	101	103	99.2	108	100	**102**	4	**3.4**
Déformation à la rupture (%):	1.1	1.2	1.1	1.2	1.1	**1.1**	0.1	**4.8**
Module d'élasticité "chord" (ksi):	9360	9950	10000	10100	9360	**9 754**	364	**3.7**
Module d'élasticité "chord" (Mpsi):	9.36	9.95	10.0	10.1	9.36	**9.75**	0.36	**3.7**
Temps à la rupture (min):	7.7	5.7	5.1	5.7	5.3	**5.9**	1.0	**17.6**
Mode de rupture:	LWV	LWV	LWV	LWV	LWV			

Préparé par:
Patrick Dubois,
Technicien(ne)

Approuvé par:
Cynthie Dega, M.Ing.
Directeur innovation-Composites

Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

3000, avenue Boulé, Saint-Hyacinthe, QC, Canada, J2S 1H9
info@gcttg.com - T.:(+1) 877 288-6378 - gcttg.com

SAGEOS GROUPE CTT

APPENDIX H TENSILE PROPERTIES OF BURNED PLATES TEST RESULTS

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 26 février 2023
Rapport: 6391-001S-2A-fr

IDENTIFICATION: Plaque de fibre de carbone recyclée (500°C, 2.5 hours): #2
Réception: 31 mai 2022

NORME:

ESSAI: "Tensile Properties of Polymer Matrix Composite Materials" ASTM D3039/D3039M-17

CONDITIONS D'ESSAI:
Atmosphère de conditionnement: 23±2°C, 50±5% H.R.;
Appareil utilisé: Dynamomètre à Taux Constant d'Extension (TCE);
Dimensions des spécimens d'essai: 1 po x 10 po (coupe à l'eau);
Mâchoires utilisées: "Wedge action";
Distance entre les mâchoires: 6 po;
Appareil utilisé: Zwick Z250 (#787299)
Extensomètre utilisé: MakroXtens
Zone de déformation utilisée pour le module et le Coefficient de Poisson: 1000-3000 microstrain :
Vitesse de l'essai (mm/min): 0.5
Vitesse de l'essai (po/min): 0.02
Date de l'essai: le 8 juin 2022

RÉSULTATS:	Résultats individuels			Moy.	E.-T.	CV %
Épaisseur (mm):	3.08	3.15	3.18	**3.14**	0.05	**1.6**
Largeur (mm):	23.8	23.8	23.8	**23.8**	0.0	**0.0**
Force à la rupture (N):	46600	49900	44900	**47 133**	2 542	**5.4**
Force à la rupture (lbf):	10500	11200	10100	**10 600**	557	**5.3**
Résistance à la rupture (MPa):	636	665	593	**631**	36	**5.7**
Résistance à la rupture (ksi):	92.2	96.4	85.9	**91.5**	5.3	**5.8**
Déformation à la rupture (%):	1.0	1.1	1.0	**1.0**	0.1	**5.6**
Module d'élasticité "chord" (MPa):	63600	61400	60600	**61 867**	1 553	**2.5**
Module d'élasticité "chord" (ksi):	9220	8910	8790	**8 973**	222	**2.5**
Module d'élasticité "chord" (Mpsi):	9.22	8.91	8.79	**8.97**	0.22	**2.5**
Temps à la rupture (min):	7.3	6.7	5.9	**6.6**	0.7	**10.6**
Mode de rupture:	LGV	LWB	LWB			

Préparé par:
Patrick Dubois,
Technicien(ne)

Approuvé par:
Cynthie Dega, M.Ing.
Directeur innovation-Composites
Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 27 février 2023
Rapport: 6391-001S-20A-fr

IDENTIFICATION: Recycled carbon fiber plaque, 4 plys: 500 C, 2.5h
Réception: 13 décembre 2022

NORME:

ESSAI: "Tensile Properties of Polymer Matrix Composite Materials" ASTM D3039/D3039M-17

CONDITIONS D'ESSAI:
Atmosphère de conditionnement: 23±2°C, 50±5% H.R.;
Appareil utilisé: Dynamomètre à Taux Constant d'Extension (TCE);
Dimensions des spécimens d'essai: 1 po x 10 po (coupe à l'eau);
Mâchoires utilisées: "Wedge action";
Distance entre les mâchoires: 6 po;
Appareil utilisé: ZWICK Z250 (#787299)
Extensomètre utilisé: MakroXtens
Zone de déformation utilisée pour le Module et le Coefficient de Poisson (microstrain): 2500-5000
Vitesse de l'essai (mm/min): 0.5
Date de l'essai: le 22 decembre 2022

RESULTATS:	Résultats individuels				Moy.	E.-T.	CV %
Épaisseur (mm):	1.47	1.49	1.48	1.48	**1.48**	0.01	**0.6**
Largeur (mm):	25.3	25.3	25.3	25.3	**25.3**	0.0	**0.0**
Force à la rupture (N):	23700	23700	23800	23700	**23 725**	50	**0.2**
Force à la rupture (lbf):	5340	5330	5350	5330	**5 338**	10	**0.2**
Résistance à la rupture (MPa):	639	631	634	633	**634**	3	**0.5**
Résistance à la rupture (ksi):	92.7	91.6	91.9	91.8	**92.0**	0.5	**0.5**
Déformation à la rupture (%):	1.1	1.0	1.1	1.0	**1.1**	0.1	**5.5**
Module d'élasticité "chord" (MPa):	61700	61500	61700	62800	**61 925**	591	**1.0**
Module d'élasticité "chord" (ksi):	8950	8930	8950	9100	**8 983**	79	**0.9**
Module d'élasticité "chord" (Mpsi):	8.95	8.93	8.95	9.10	**8.98**	0.08	**0.9**
Temps à la rupture (min):	5.1	5.2	5.3	5.1	**5.2**	0.1	**1.9**
Mode de rupture:	LAV	LAV	LGM	LAV			

Préparé par:
Patrick Dubois,
Technicien(ne)

Approuvé par:
Sylvie Dalpé, Tech.
Directrice de laboratoires

Date: 27 février 2023

Pour toute information concernant ce rapport, veuillez contacter Sylvie Dalpé.

APPENDIX I COMPRESSIVE PROPERTIES TEST RESULTS

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens — Date: 26 février 2023
Centre des technologies Textiles-PART-4 (CTT-084) — Rapport: 6391-001S-6A-fr

IDENTIFICATION: Plaque de fibre de carbone recyclée: Sample #2, 500°C, 2,5h
Réception: 2 août 2022

NORME:

ESSAI: "Compressive Properties of Polymer...Materials Using a Combined Loading Compression Test Fixture" — ASTM D6641/D6641M-16e2

CONDITIONS D'ESSAI:
Atmosphère de conditionnement: 23±2°C / 50±5% H.R.
Appareil utilisé: Dynamomètre à taux constant d'extension (TCE);
Dispositif de test utilisé: Tel que décrit dans la méthode d'essai;
Force de serrage des boulons: 3N.m;
Grandeur des spécimens: 1/2 x 5 1/2 po;
Longueur d'essai: 1/2 po;
Jauges de déformation utilisées: C4A-06-125SL-350-39P
Facteur de la jauge: 2.09
Zone de déformation utilisée pour calculer le module: 1000-3000 microstrain 2000-4000
Vitesse de l'essai (mm/min): 1
Date de l'essai: le 13 septembre 2022

RÉSULTATS:

	Résultats individuels					Moy.	E.-T.	CV %
Identification du spécimen:	#6	#9	#10	#12	#13			
Épaisseur (mm):	1.40	1.43	1.41	1.41	1.42	**1.41**	0.01	**0.8**
Largeur (mm):	12.6	12.6	12.6	12.6	12.5	**12.6**	0.0	**0.4**
Charge à la rupture (N):	7520	6970	7330	7170	6740	**7 146**	304	**4.3**
Charge à la rupture (lbf):	1690	1570	1650	1610	1520	**1 608**	66	**4.1**
Résistance à la compression (MPa):	428	388	413	405	379	**403**	20	**4.9**
Résistance à la compression (ksi):	62.1	56.3	59.9	58.7	55.0	**58.4**	2.8	**4.8**
Module de compression (MPa):	55200	52200	58500	52700	53400	**54 400**	2 558	**4.7**
Module de compression (ksi):	8010	7570	8480	7640	7750	**7 890**	370	**4.7**
Temps de rupture (min):	1.5	1.6	1.6	1.7	1.5	**1.6**	0.1	**5.3**
Type de rupture:	TGM	TGB	TGB	TGT	TGM			

Préparé par: Patrick Dubois, Technicien(ne)

Approuvé par: Cynthie Dega, M.Ing. Directeur innovation-Composites — Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens — Date: 27 février 2023
Centre des technologies Textiles-PART-4 (CTT-084) — Rapport: 6391-001S-21A-fr

IDENTIFICATION:	Recycled carbon fiber plaque, 8 plys: 500 C, 2.5h Réception: 13 décembre 2022	
NORME:		
ESSAI:	"Compressive Properties of Polymer...Materials Using a Combined Loading Compression Test Fixture"	ASTM D6641/D6641M-16e2
CONDITIONS D'ESSAI:	Atmosphère de conditionnement: 23±2°C / 50±5% H.R. Appareil utilisé: Dynamomètre à taux constant d'extension (TCE); Dispositif de test utilisé: Tel que décrit dans la méthode d'essai; Force de serrage des boulons: 3N.m; Grandeur des spécimens: 1/2 x 5 1/2 po; Longueur d'essai: 1/2 po; Jauges de déformation utilisées: C4A-06-125SL-350-39P Facteur de la jauge: 2.09 Zone de déformation utilisée pour calculer le module: 1000-3000 microstrain 2000-4000 Vitesse de l'essai (mm/min): 1.3 Vitesse de l'essai (po/min): Date de l'essai: le 23 janvier 2023	

RESULTATS:	Résultats individuels					Moy.	E.-T.	CV %
Identification du spécimen:	6	7	8	9	10			
Épaisseur (mm):	4.04	4.09	4.19	4.09	4.17	**4.12**	0.06	**1.5**
Épaisseur (po):	0.159	0.161	0.165	0.161	0.164	**0.162**	0.002	**1.5**
Largeur (mm):	12.8	12.8	12.8	12.9	12.8	**12.8**	0.0	**0.3**
Largeur (po):	0.505	0.505	0.506	0.506	0.504	**0.505**	0.001	**0.2**
Charge à la rupture (N):	17500	17000	19400	20300	18900	**18 620**	1 359	**7.3**
Charge à la rupture (lbf):	3930	3820	4360	4560	4250	**4 184**	306	**7.3**
Résistance à la compression (MPa):	338	324	362	387	355	**353**	24	**6.8**
Résistance à la compression (ksi):	49.0	47.0	52.5	56.1	51.5	**51.2**	3.5	**6.8**
Module de compression (MPa):	38800	34900	41200	33000	34100	**36 400**	3 460	**9.5**
Module de compression (ksi):	5630	5060	5980	4790	4950	**5 282**	502	**9.5**
Module de compression (Mpsi):	5.63	5.06	5.98	4.79	4.95	**5.28**	0.50	**9.5**
Type de rupture:	HGM	HGT	HGB	HGT	HGB			

Préparé par: Patrick Dubois, Technicien(ne)

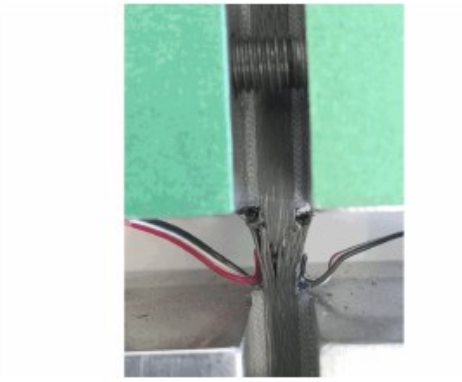
Approuvé par: Sylvie Dalpé, Tech., Directrice de laboratoires — Date: 27 février 2023

Pour toute information concernant ce rapport, veuillez contacter Sylvie Dalpé.

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 27 février 2023
Rapport: 6391-001S-21A-fr



Approuvé par:

Sylvie Dalpé, Tech.
Directrice de laboratoires

Date: 27 février 2023

Pour toute information concernant ce rapport, veuillez contacter Sylvie Dalpé.

Les rapports sont identifiés par un code alphanumérique, la lettre précédant "-fr" fait référence au numéro de révision, émis en ordre croissant. La version électronique reçue du Groupe CTT est la version officielle du rapport. L'identification rapportée est basée sur ce qui a été observé sur l'échantillon reçu et/ou l'information fournie par le client. Les échantillons en lien avec ce rapport sont conservés pendant une période de 30 jours à partir de la date de transmission du rapport. Les résultats ci-haut mentionnés ne se rapportent qu'aux échantillons soumis à l'ess ai. Ce rapport ne doit pas être reproduit, sinon en entier, sans l'autorisation écrite du Groupe CTT. ‡ La portée d'accréditation ISO/CEI 17025 du Groupe CTT est disponible à www.gcttg.c om. Dans ce rapport, les essais dont le numéro est suivi du symbole ◊ ne sont pas couverts par cette accréditation. Pour l'adresse complète du client, veuillez consulter le courriel.

APPENDIX J FLEXURAL PROPERTIES TEST RESULTS

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens — Date: 26 février 2023
Centre des technologies Textiles-PART-4 (CTT-084) — Rapport: 6391-001S-1A-fr

IDENTIFICATION: Plaque de fibre de carbone recyclée (500°C, 2.5 hours): #1
Réception: 31 mai 2022

NORME:

ESSAI: "Flexural Properties of Unreinforced Plastics and Electrical Insulating Materials" — ASTM D790-17

CONDITIONS D'ESSAI: Appareil utilisé: Dynamomètre avec un Taux Constant d'Extension (TCE);
Déflexion mesurée en utilisant la position de la traverse (Test Type I);
Atmosphère de conditionnement: 40h à 23 ± 2°C, 50 ± 10% H.R. :
Procédure utilisée: A
Nombre de spécimens d'essai testés "Flatwise" par produit: 5
Rapport de distance entre les appuis vs épaisseur: 16:1
Vitesse d'essai (mm/min): 1.31
Date de l'essai: le 30 juin 2022

RÉSULTATS:	Résultats individuels					Moy.	E.-T.	CV %
Distance entre les appuis (mm):	49							
Épaisseur des spécimens (mm) :	3.07	3.08	3.07	3.05	3.14	**3.08**	0.03	**1.1**
Largeur des spécimens (mm) :	10.47	10.45	10.46	10.46	10.45	**10.46**	0.01	**0.1**
Force maximale (N):	947.0	974.9	1000.1	924.8	1011.0	**971.6**	36.0	**3.7**
Force maximale (lbf):	212.9	219.2	224.8	207.9	227.3	**218.4**	8.1	**3.7**
Contrainte maximale (MPa):	703.8	725.5	747.5	700.9	719.9	**719.5**	18.8	**2.6**
Contrainte maximale (psi):	102075	105225	108421	101661	104414	**104 359**	2 727	**2.6**
Déformation à la contrainte maximale (%):	1.7	1.6	1.6	1.6	1.7	**1.6**	0.1	**3.3**
Module d'élasticité (MPa):	37008	40369	33125	41744	34136	**37 276**	3 765	**10.1**
Module d'élasticité (ksi):	5367.6	5855.1	4804.4	6054.5	4951.0	**5 406.5**	546.0	**10.1**
Module sécant à 1% de déformation (MPa):	48341.0	49965.0	49515.0	49594.0	47157.0	**48 914.4**	1 155.8	**2.4**

Préparé par: Patrick Dubois, Technicien(ne)

Approuvé par: Cynthie Dega, M.Ing. Directeur innovation-Composites — Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

3000, avenue Boulé, Saint-Hyacinthe, QC, Canada, J2S 1H9
Info@gcttg.com - T.:(+1) 877 288-8378 - gcttg.com

SAGEOS GROUPE CTT

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 26 février 2023
Rapport: 6391-001S-2A-fr

IDENTIFICATION: Plaque de fibre de carbone recyclée (500°C, 2.5 hours): #2
Réception: 31 mai 2022

NORME:

ESSAI: "Flexural Properties of Unreinforced Plastics and Electrical Insulating Materials" — ASTM D790-17

CONDITIONS D'ESSAI:
Appareil utilisé: Dynamomètre avec un Taux Constant d'Extension (TCE);
Déflexion mesurée en utilisant la position de la traverse (Test Type I);
Atmosphère de conditionnement: 40h à 23 ± 2°C, 50 ± 10% H.R. :
Procédure utilisée: A
Nombre de spécimens d'essai testés "Flatwise" par produit: 3
Rapport de distance entre les appuis vs épaisseur: 16:1
Vitesse d'essai (mm/min): 1.35
Date de l'essai: le 30 juin 2022

RESULTATS:	Résultats individuels			Moy.	E.-T.	CV %
Distance entre les appuis (mm):	51					
Épaisseur des spécimens (mm) :	3.16	3.15	3.17	**3.16**	0.01	**0.3**
Largeur des spécimens (mm) :	10.47	10.48	10.48	**10.48**	0.01	**0.1**
Force maximale (N):	879.0	923.7	853.9	**885.5**	35.4	**4.0**
Force maximale (lbf):	197.6	207.6	192.0	**199.1**	7.9	**4.0**
Contrainte maximale (MPa):	643.5	681.4	620.9	**648.6**	30.6	**4.7**
Contrainte maximale (psi):	93325	98833	90052	**94 070**	4 438	**4.7**
Déformation à la contrainte maximale (%):	1.5	1.7	1.6	**1.6**	0.1	**6.3**
Module d'élasticité (MPa):	43904	36691	39078	**39 891**	3 675	**9.2**
Module d'élasticité (ksi):	6367.7	5321.5	5667.8	**5 785.7**	533.0	**9.2**
Module sécant à 1% de déformation (MPa):	46547.0	45855.0	45369.0	**45 923.7**	592.0	**1.3**

Préparé par:
Patrick Dubois,
Technicien(ne)

Approuvé par:
Cynthie Dega, M.Ing.
Directeur innovation-Composites

Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.

APPENDIX K SHEAR TEST RESULTS

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 20 mars 2023
Rapport: 6391-001S_27_FR

IDENTIFICATION: Tissu recyclé - pyrolyse conventionnelle: 500degC, 2.5h
Réception: 9 février 2023

NORME:

ESSAI: "Shear Properties of Composite Materials by the V-Notched Beam Method" — ASTM D5379/D5379M-19e1≈

CONDITIONS D'ESSAI: Atmosphère de conditionnement: 23±2°C, 50±5% H.R.;
Appareil utilisé: Dynamomètre à Taux Constant d'Extension (TCE);
Mâchoires utilisées: Appareil de cisaillement de rail à entailles en V (comme indiqué dans la méthode d'essai);
Dimensions des spécimens d'essai: 3.0 po x 0.75 po avec un entaille de 0.15 po;
Appareil utilisé: Zwick Z250 (#7872990 + fixture D5379
Jauge de déformation utilisée: C4A-06-125SL-350-39P
Facteur de la jauge: 2,09
Zone de déformation utilisée pour le module et le Coefficient de Poisson: 1000-3000 usn 1500-2500
Vitesse de l'essai (mm/min): 0.8
Date de l'essai: le 2 mars 2023

RÉSULTATS:	Résultats individuels					Moy.	E.-T.	CV %
Épaisseur (mm):	3.73	3.81	3.77	3.75	3.66	**3.74**	0.06	**1.5**
Épaisseur (po):	0.147	0.150	0.148	0.148	0.144	**0.147**	0.002	**1.5**
Largeur (mm):	12.0	11.8	12.1	11.9	11.9	**11.9**	0.1	**1.0**
Largeur (po):	0.470	0.465	0.476	0.469	0.470	**0.470**	0.004	**0.8**
Force à la rupture (N):	2680	2970	2450	2460	2340	**2 580**	250	**9.7**
Force à la rupture (lbf):	602	668	551	553	526	**580**	56	**9.7**
Résistance au cisaillement (MPa):	48.7	40.7	44.6	45.6	45.6	**45.0**	2.9	**6.4**
Résistance au cisaillement (ksi):	7.06	5.90	6.47	6.61	6.61	**6.53**	0.42	**6.4**
Déformation au cisaillement (%):	>5.0	>5.0	>5.0	>5.0	>5.0			
Module d'élasticité "chord" (MPa):	2140	3060	2780	2570	2350	**2 580**	360	**13.9**
Module d'élasticité "chord" (ksi):	310	444	403	373	341	**374**	52	**14.0**
Mode de rupture:	MGN	MGN	MGN	MGN	MGN			

Préparé par:
Patrick Dubois,
Technicien(ne)

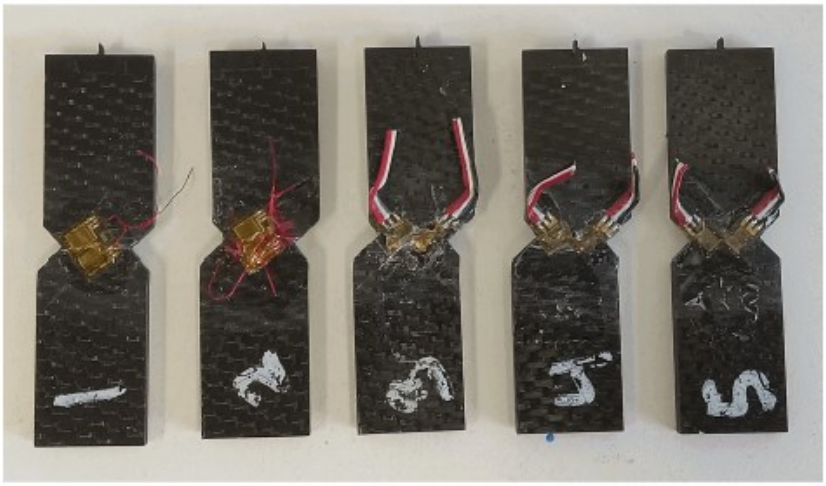
Approuvé par:

Date: 20 mars 2023

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 20 mars 2023
Rapport: 6391-001S_27_FR



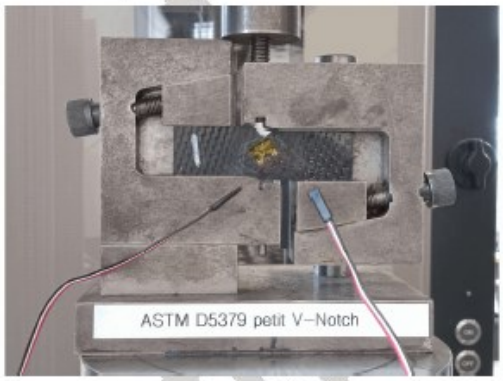
Approuvé par:

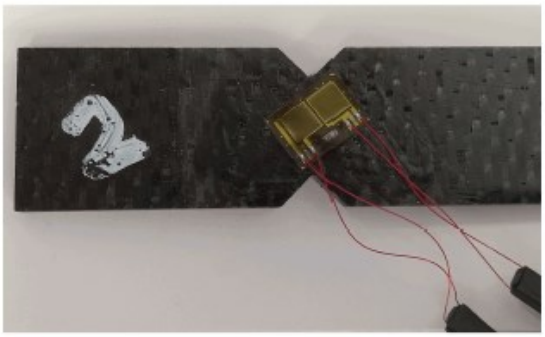
Date: 20 mars 2023

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens
Centre des technologies Textiles-PART-4 (CTT-084)

Date: 20 mars 2023
Rapport: 6391-001S_27_FR





Approuvé par:

Date: 20 mars 2023

APPENDIX L SHORT BEAM TEST RESULTS

RAPPORT D'ANALYSES
No. d'accréditation du CCN: 40‡

Mme Justine Decaens — Date: 26 février 2023
Centre des technologies Textiles-PART-4 (CTT-084) — Rapport: 6391-001S-5A-fr

IDENTIFICATION: Plaque de fibre de carbone recyclée: Sample #1, 500°C, 2,5h
Réception: 2 août 2022

NORME:

ESSAI: "Short-Beam Strength of Polymer Matrix Composite Materials and Their Laminates" — ASTM D2344/D2344M-16

CONDITIONS D'ESSAI:
Atmosphère de conditionnement: 23 ± 3°C, 50 ± 10% H.R.;
Appareil utilisé: Dynamomètre à taux constant d'extension (TCE);
Diamètre de la tête de chargement: 6 mm;
Diamètre des supports: 3 mm;
Nombre de spécimens testés: 6
Vitesse (po/min): 0.04
Date de l'essai: le 14 septembre 2022

RESULTATS:	Résultats individuels					Moy.	E.-T.	CV %
Écart entre les appuis (in):	0.315							
Épaisseur du spécimen (mm):	1.46	1.50	1.48	1.47	1.50	**1.48**	0.02	**1.2**
Épaisseur du spécimen (po):	0.0575	0.0589	0.0582	0.0578	0.0592	**0.0583**	0.0007	**1.2**
Largeur du spécimen (mm):	4.17	4.18	4.19	4.18	4.18	**4.18**	0.01	**0.2**
Largeur du spécimen (po):	0.164	0.165	0.165	0.165	0.164	**0.165**	0.001	**0.3**
Force maximum (N):	282	285	283	288	282	**284**	3	**0.9**
Force maximum (lbf):	63.4	64.1	63.6	64.7	63.4	**63.8**	0.6	**0.9**
Résistance 'short-beam' (MPa):	34.7	34.3	34.3	35.2	33.6	**34.4**	0.6	**1.7**
Résistance 'short-beam' (ksi):	5.03	4.97	4.98	5.10	4.88	**4.99**	0.08	**1.6**
Mode de rupture:	ILS	ILS	ILS	ILS	ILS			

Préparé par: Patrick Dubois, Technicien(ne)

Approuvé par: Cynthie Dega, M.Ing., Directeur innovation-Composites — Date: 26 février 2023

Pour toute information concernant ce rapport, veuillez contacter Cynthie Dega.