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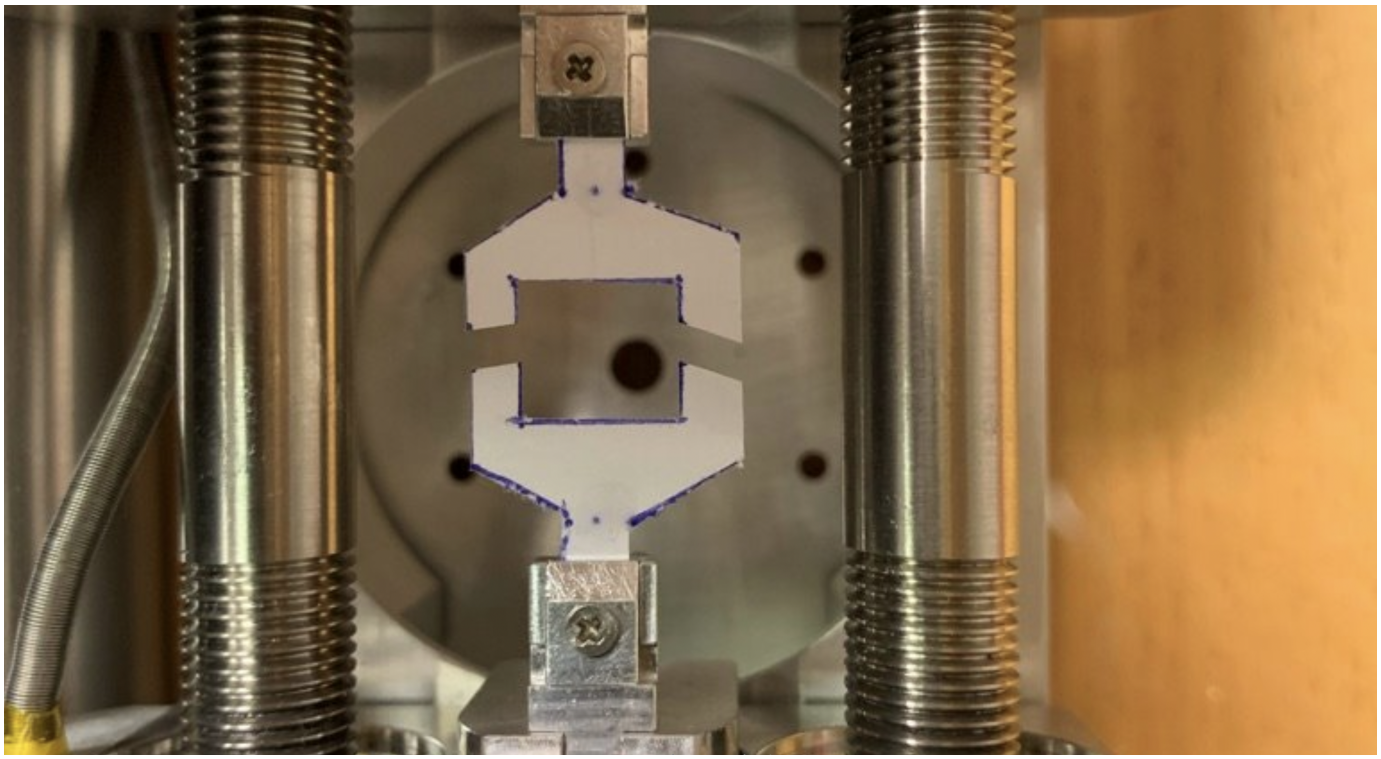

Micromechanical Testing of Pristine and Delithiated Single Carbon Fibres for Structural Battery Composite

Master's thesis in Materials Engineering

Sreehari Batni Ravindranath

DEPARTMENT OF Industrial and Materials Science

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Master's Thesis 2021

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Abstract

Reduction of mass of the battery system in electric vehicles (EV) is one of the major requirements of the use of EV in place of internal combustion (IC) engines. A novel solution to reduce the mass of the system is by replacing the present battery material with multifunctional material. These are the materials that can simultaneously carry the load and act as an electrode for the battery system.

In this study, carbon fibre is used as a candidate multifunctional material. This project aims to examine the tensile strength, modulus, and Weibull distribution of pristine and delithiated single carbon fibre. The tensile properties of 25 mm and 15 mm gauge length fibres were examined using a microtester. The fractured surface was investigated with SEM and EDX.

The delithiation doesn't affect the tensile modulus of the fibre. A considerable amount of difference was detected in the Weibull modulus. The Weibull value 'm' is high for pristine fibre and a bit less for delithiated fibres. The SEM and EDX results showed that there is a slight change in the surface morphology of fibre and an increase in the content of oxygen in delithiated fibres. This can be due to the remnants of lithium from lithiation and delithiation which were oxidized once they were exposed to the atmosphere.

Keywords: Structural battery composite, lithiation/delithiation, fractography, Weibull modulus, microtester.

Acknowledgements

I would gladly thank my supervisors Marcus Johansen and Johanna Xu at Chalmers University of Technology for their invaluable encouragement and support during my thesis work. They have always motivated and encouraged me throughout my thesis at Chalmers University of Technology. I would also like to express my sincere gratitude to my examiner, Fang Liu, at the Department of Industrial and Materials Science, Chalmers University of Technology for endorsing and providing significant inputs during the thesis work and for providing his extended support related to the administration tasks at University.
To my family and friends – Thank you for being there for me.

Sreehari Batni Ravindranath, Gothenburg, August 2021.

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1
Introduction

This chapter gives a background to structural battery composites and the motivation behind this Master's thesis project. Furthermore, this chapter formulates the projects aim, scope and specific issues under investigation. This is followed by the contribution and the outline of the thesis.

1.1 Background

In today's fast-growing technology we are striving for solutions to reduce greenhouse gas emissions. The emissions from the transportation sector are one of the prime contributors to the environmental crisis. One of the prominent ways to combat the situation is by replacing internal combustion (IC) engines with electrification and battery technology which allows cleaner and more sustainable transportation.

Even though adapting to electric vehicles can lead to cleaner transport, the limited range of these vehicles represents a significant drawback. Minimizing the weight of the battery has become a priority to make electric vehicles a more viable option and match the range of vehicles reliant on IC engines. A significant way to decrease the weight of the system is by implementing multiple functions in the same component i.e., a multifunctional device such as structural battery composites (SBC). The SBC can simultaneously store electrochemical energy and act as a mechanical load bearer[1] [2].

A research team from the US Army research laboratory (ARL) made the first attempt to develop a structural battery composite by using carbon fiber as a negative electrodes [3]. The results from those attempts showed that the fibers were mechanically strong but they observed self-discharge because of poor electrical conductivity. Another approach towards SBC was done by Neudecker et al. [4] where they incorporated energy storage function to the thin film battery layers called "power fibers". The main advantage of these fibers over flat film batteries is the larger surface area of power fibers. Investigations on structural battery composites were conducted, where carbon fibres were used as negative electrode, $LiFePO_4$ mixed acetylene black as a positive electrode and glass fibers as a separator [5]. The mechanical and electrochemical properties were investigated where metallic cathode showed higher stiffness than carbon fiber because of weak matrix and sample dimensions [5]. In recent work by Asp et.al, an SBC with good electrochemical and mechanical performance was presented. The results from their work concluded that the combined electrochemical

and mechanical properties outperform all other SBC materials [6]

A composite material refers to two or more materials that are mixed in different proportions to get desired properties. Composites consist of matrix and reinforcement where the main function of the matrix is to hold the reinforcement in a particular direction and prevent it from the hazardous environment. The majority of the loads are carried by reinforcement. The use of composites for structural battery composites depends on load-bearing capability of carbon fibres and their ability to host Lithium (Li)- ions in their microstructure [7].

An SBC consists of a Li-ion source performing as a positive electrode; glass fiber performing as a separator; a bicontinuous polymer liquid electrolyte matrix performing as load-transferer and ion conductor; carbon fibers performing as load-carriers and the negative electrode (Figure 1.1) [8].

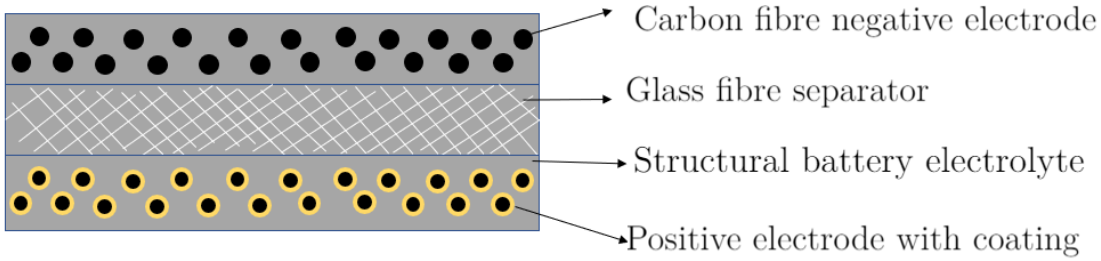


Figure 1.1: Schematic representation of structural battery cell.

The ability of carbon fiber to host Li-ions in its microstructure, high strength, and high modulus makes carbon fibers one of the candidate materials for SBC application. The properties of the carbon fibers vary with the precursor material. The most commonly used carbon fiber is manufactured from Polyacrylonitrile (PAN) and pitch-based precursors. Pitch-based fibers exhibit high stiffness, high thermal conductivity, medium strength with the modulus ranging from 170-980 GPa. The PAN-based fibers are the most commonly used because of their low cost and good properties. Among these, the fibers manufactured from PAN precursor material are well suited for SBC applications. The microstructure of fibers can be described as a micro composite structure, which consists of turbostratic folded and interlinked carbon layers [9]. The Li-ion insertion into the carbon fiber depends on the microstructure. By tailoring the microstructure of carbon fiber the structural performance of SBC can be improved [10].

It is still not fully understood to what extent electrochemical loading influences the structural performance of carbon fibers. This study focuses on evaluating the tensile properties of pristine and delithiated carbon fibers and investigating the impact of Li-ion insertion on the tensile properties of a single carbon fiber.

1.2 Aim

This thesis aims to study the effect of lithium-ion insertion on the mechanical properties of carbon fibres by performing single filament tensile tests on pristine and delithiated PAN-based carbon fibres.

1.3 Scope

This study is focused on evaluating the mechanical properties of pristine and delithiated PAN-based carbon fibers for energy storage applications. This study will be limited to T800 carbon fibres from Troy. The material characterisation will be limited to single filament tensile tests using micromechanical tester and fractography using scanning electron microscopy (SEM). All the specimens for the tensile test are prepared according to ASTM C-1557-20 standards.

1.4 Specific issue under investigation

The following issues are adressed in this thesis:

- How to perform tensile tests on pristine single carbon fibres?
- How to lithiate/delithiate carbon fibres?
- How to perform tensile tests on delithiated single carbon fibres?

1.5 Thesis Contribution

This study aims to investigate the effect of Li-ion in the graphitic microstructure of single carbon fibers. In contrast to the study presented in [8], the research focus on the Li-ion intsertion into the bundle of T800H and IMS65 carbon fibres. The difference is that the goal of this research is to inspect the Li-ion insertion on single fibers.

This research builds on the lithiation and de-lithiation of the T800 carbon fiber bundle by using the half cell method. Single fibers are extracted from the bundle and tested with a micromechanical tester of a 1N load cell. The second purpose is to carry out the mechanical testing with the help of a tester that is compatible with a scanning electron microscope to analyze by carrying the test inside the SEM. Due to the time limitations the second purpose is not covered in this study. The results from the tester are analyzed with the Weibull distribution to check whether there is a significant decrease or increase in the proprieties due to lithiation.

1.6 Thesis outline

This thesis is divided into seven chapters. After the introduction, Chapter 2 serves as a theory. This includes deeper information about the Li-ion battery, tensile modulus of fibres and Weibull modulus. Chapter 3 presents the methodology followed by a

results, discussion chapter, and conclusion chapter. The report is wrapped up with a future work as a final chapter.

2

Theory

This chapter gives a brief overview of the tensile strength and tensile modulus, fractography of the fiber followed by the Li-ion battery and the Weibull modulus.

2.1 Lithium-ion battery

Storing electrical energy in the form of batteries can be considered as one of the potential alternatives to reduce the use of fossil fuels. Demand for rechargeable batteries for storing energy in various electrical devices such as mobiles, laptops, power banks is increasing. Among various elements, Lithium has the highest oxidation potential and it is the third lightest element which makes stand out as a potential battery material because of its various properties such as high energy density, long life, low self-discharge properties, and high-performance [16]. Li-ion batteries satisfy the energy requirements for several applications such as electric vehicles, but challenges to be addressed such as avoiding overcharge or discharge, increasing in charge rate [17].

The present-day Li-ion battery system (Figure 2.1) consists of a positive and negative electrode where the Li-ions travel back and forth between the electrodes during charging and discharging. The most commonly metal oxides such as $LiCoO_2$, $LiFePO_4$, $LiMn_2O_4$ are used as positive electrode. The negative electrode is made of graphitic carbon. A porous separator is used to electrochemically separate the two electrodes.

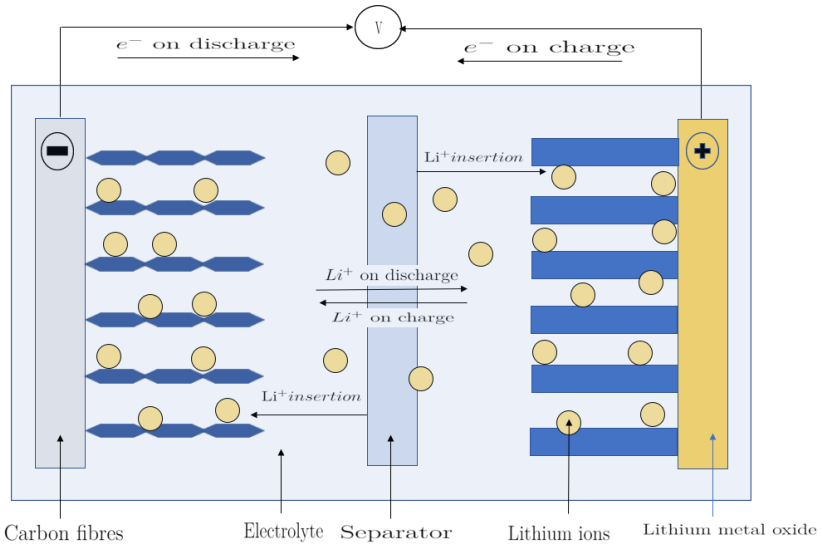


Figure 2.1: Schematic representation of Li-ion battery

The Li-ions travel between the electrodes leading to converting chemical energy into electrical energy through an oxidation and a reduction reactions. During the charging process in the Li-ion battery, the positive electrode undergoes oxidation reaction, and the negative electrode undergoes reduction process. In the electrochemical cycling process Li-ions are extracted from the positive electrode and inserted into the negative electrode as shown in cell reactions below [18]:

$$LiMO_2 \underset{\text{discharge}}{\overset{\text{charge}}{\rightleftharpoons}} Li_{1-x}MO_2 + xLi^+ + xe^- \tag{2.1}$$

$$C + xLi^+ + xe^- \underset{\text{discharge}}{\overset{\text{charge}}{\rightleftharpoons}} Li_xC \tag{2.2}$$

$$\mathrm{LiMO_2} + C \underset{\text{discharge}}{\overset{\text{charge}}{\rightleftharpoons}} Li_xC + Li_{1-x}MO_2 \tag{2.3}$$

In the present study, carbon fibers are considered as negative electrodes to improve the capacity and decrease the weight of the battery system. In this study, the half-cell method is used to insert the Li-ions into the carbon fiber (Figure 2.1). The samples for half cell technique are prepared in section 3.2.1

2.2 Tensile strength and tensile modulus

The tensile strength of the material quantifies the maximum amount of stress a material can withstand before fracture. Fracture in carbon fibers can occur due to internal flaws or the flaws present on the surface [11]. Young's modulus is a material property that can be derived from the stress-strain diagram, which quantifies the material stiffness. The ideal stress-strain diagram for brittle material shows a small amount of plastic behaviour before failure. The theoretical Young's modulus of a

defect-free carbon fiber lies around 1000 GPa, theoretical strength around 100 GPa but in practical applications, the fibers fail way below the theoretical value this can be accounted for by the defects present in the fiber. The Young's modulus of the material is calculated by calculating the slope of then the stress-strain curve. With the help of an experimental approach, the stress of the material can be obtained by

$$\sigma = \frac{\mathrm{F}}{\mathrm{A}} \tag{2.4}$$

where σ is the stress, F is the force acting on the object and A is the cross-section area of the material [12]. Brittle materials like ceramics and carbon fibers show a size effect (strength decreases with increase in length) Weibull distribution is used to interpret the modulus of such material [13]. Tensile strength σ of the samples can be calculated:

$$\sigma = \frac{P_{max}}{\left(\frac{\pi d_f^2}{4}\right)} \tag{2.5}$$

Where P_{max} is the load at which the specimen failed, d_f is the diameter of single fibre.

2.3 Weibull modulus

The Weibull distribution is a parameter that is used to define the variation of material strength in brittle materials. The samples of carbon fibers are tested.
Even though brittle materials like ceramics, carbon fiber was tested in ideal conditions different samples fail at different stress this can be explained in terms of Weibull modulus [19]. The Weibull modulus is a dimensionless parameter that can be related to the physical flaws and distribution of flaws. The flaws in the carbon fiber may arise due to various reasons these include defects that may occur during manufacturing, fiber misalignment, etc., Hence the Weibull modulus cannot be considered as a material constant as it varies depending on the type, a distance of flaws [20]. The Weibull modulus of stress σ is given by

$$P_F = 1 - \exp\left[-L\left(\frac{\sigma_f}{\sigma_0}\right)^m\right] \tag{2.6}$$

Where P_F is the summation of the probability of fiber failure, L is the length of the fiber at applied stress σ_f, m is the Weibull modulus, σ_o is the scale parameter or normalizing parameter. The summation of the probability of failure is calculated by:

$$P_F = \frac{i}{n+1} \tag{2.7}$$

Where i is the total number of specimens that break below the stress limit, n is the total number of tested specimens. By substituting 2.7 in 2.6 and rearranging equations gives:

$$\ln\left(\ln\left[\frac{1}{1-P_F}\right]\right) = m\ln\left(\sigma_f\right) - m\ln\left(\sigma_0 L^{1/m}\right) \tag{2.8}$$

The Weibull modulus m can be calculated by the slope of the line plotted by using equation 2.8. The Weibull modulus m can be used to characterize the distribution of flaws in the carbon fiber. High values of m represent that the flaws are distributed evenly, and low values indicate that the flaws are distributed less evenly [21].

2.4 Fractography

Materials can undergo fracture due to various reasons and it is always necessary to conduct failure analysis of materials before implementing it in real-time applications. Once the materials fail, they leave marks behind which provides important information and helps in identifying the cause of failures. Various research has been conducted by several authors to identify the features on the fractured surface. Masa et. al conducted various tests on fractography of carbon fiber and concluded that chevron marks were visible when conducted macrofractography and radials were observed in micro-fractography [14].

This study mainly focuses on the single fiber specimens which failed under tension load. The fiber diameter of single filament fibers is around 5 µm, it's difficult to identify the chevron marks on the fibers through macro-fractography. The present study uses micro-fractography to analyze the features on the fractured surface. The fractured surfaces are studied under the SEM machine for identifying the features on the surface of the cracked fiber. A field emission gun is used as an electron source to analyze the fractured surface and the reflecting electrons are detected with the help of a backscattered electron detector and secondary electron detector and processed into images. Some of the features that can be observed on the fibers are such as radials, crack direction, remnants of Li-ion in the delithiated fibers, increase in the fiber diameter after lithiation, crack initiation points.

Energy Dispersive X-Ray Spectroscopy (EDX) is used to analyze the elemental composition in fibers. EDX analysis is carried out by detecting the characteristic X-ray signals with the help of a lithium drifted EDX detector and the spectrum of different elements are analyzed in the point of interest [15].

3

Methodology

This chapter presents the experiments that have been performed to answer the research questions stated in section 1.4. This chapter is divided into materials, specimen preparation, mechanical testing and fractography.

3.1 Materials

This study focuses on the T800 PAN-based type of carbon fiber with a density of 1.81 g/cm^3, tensile modulus of 294 GPa, strain at failure 1.9 % with the fiber diameter of 5µm [22]. According to the results from [23], T800 fibers showed the cycle capacity of 243 mAh/g after the 10th cycle. The T800 fibers were supplied from Toray industries.

3.2 Specimen preparation

To evaluate the mechanical properties of the carbon fibers, specimens for tensile testing were produced. The specimen preparation includes electrochemical cycling (for delithiated carbon fibers), isolation of single filament, and mounting of single filament to a tab.

3.2.1 Electrochemical cycling

To insert lithium into the carbon fibers, half cells were constructed (Figure 3.1). The whole process was carried out inside the glovebox (oxygen-free and water vapour free environment). The carbon fibers were placed on the copper strip. A porous separator was placed between lithium metal and carbon fiber. Liquid electrolyte was applied, and the pouch was vacuum sealed.

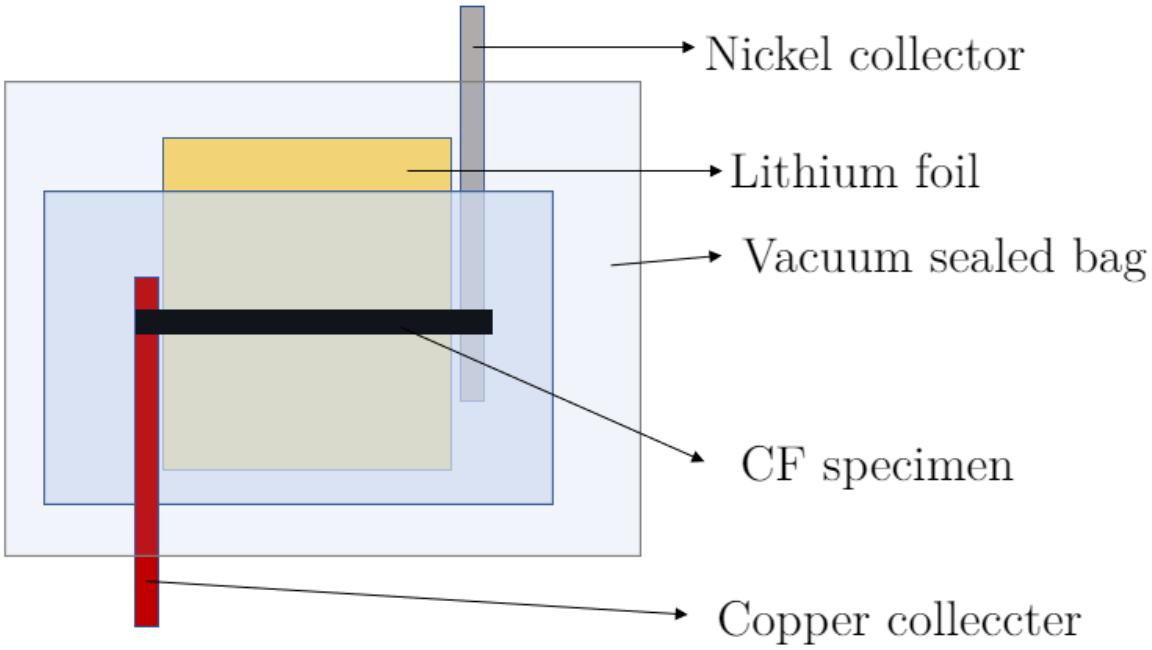


Figure 3.1: Schematic representation of electrochemical cell.

The prepared pouch cells were electrochemically cycled for 3 cycles with a voltage between 0.01-1.5 V and a constant current of 0.24 mA. Each cycle had four steps where the first step was the lithiation of fibers in which the cycle time was about 30 h. In the second step, the fibers were allowed to rest for 1 h. The third step was the delithiation process followed by a rest time of 1 h which completes 1 cycle.

3.2.2 Tabbing of single carbon fiber filament

Pristine and delithiated fibers were tabbed outside a glove box. The specimen preparation follows the sequence (Figure 3.2) where Single fibers were isolated from a dry single carbon fiber bundle. The isolated fibers were adhered to the paper holder with the help of Loctite epoxy glue to reduce the stress concentrations at the ends of fiber [21]. Paper coupons were prepared into desired shape (Figure 3.3). Gauge lengths of 25mm and 15mm were marked on the paper and Loctite glue was placed to hold the fiber in place (Figure 3.3) [24].

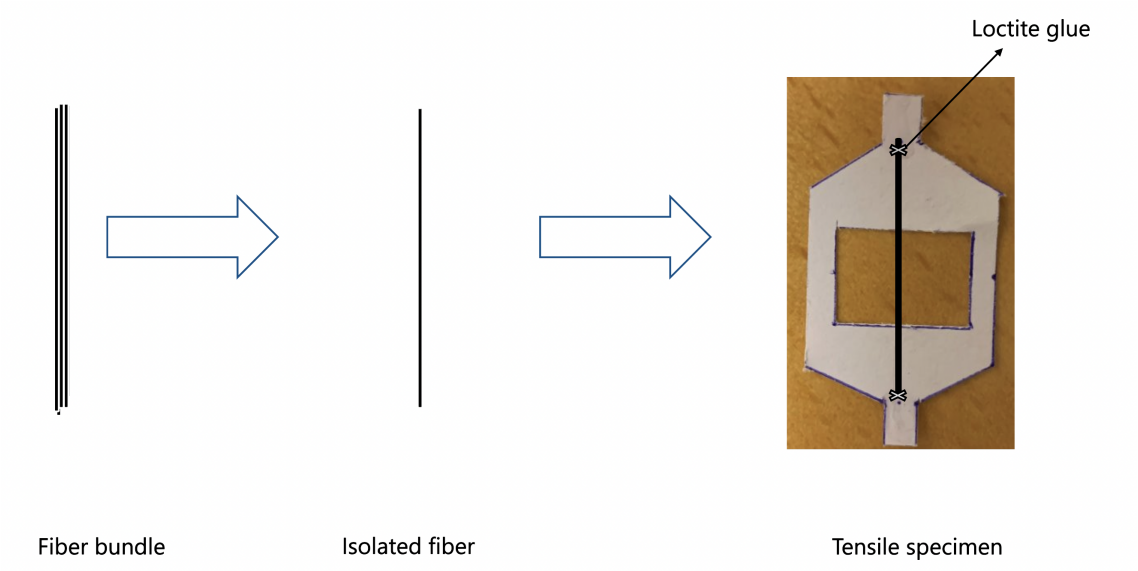


Figure 3.2: Manufacturing sequence of tensile specimen of pristine carbon fiber.

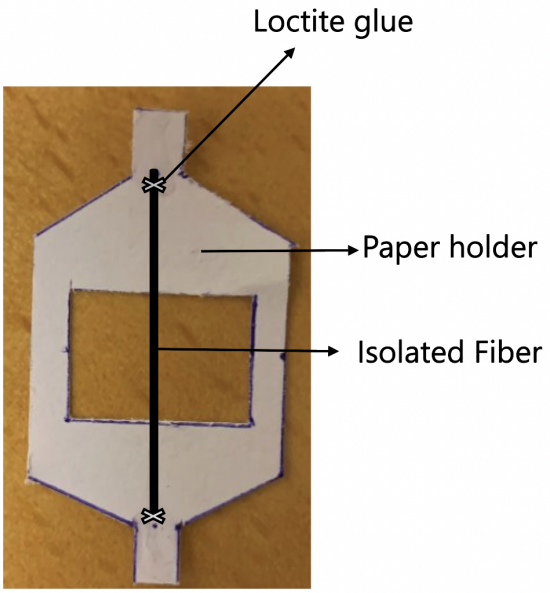


Figure 3.3: Single carbon fibre mounted on a tab and adhered with Loctite glue.

3.3 Mechanical testing

To analyze the variation in the mechanical properties of carbon fiber tensile test and fractography of single carbon fibers were carried out as described in the below subsections.

3.3.1 Tensile test

Tensile tests of single fibers were performed using a microtester from Kammrath & Weiss with a load cell of 1 N. The samples were tested with a low displacement speed of 1µm/S. The prepared specimen was mounted onto the tester and the sides of the paper were cut free before the test (Figure 3.4). The tests of pristine and delithiated fibers were conducted under the laboratory atmosphere. The MDS software from Kammrath & Weiss was used to acquire the test data. The results from the test were analyzed using the Weibull distribution.

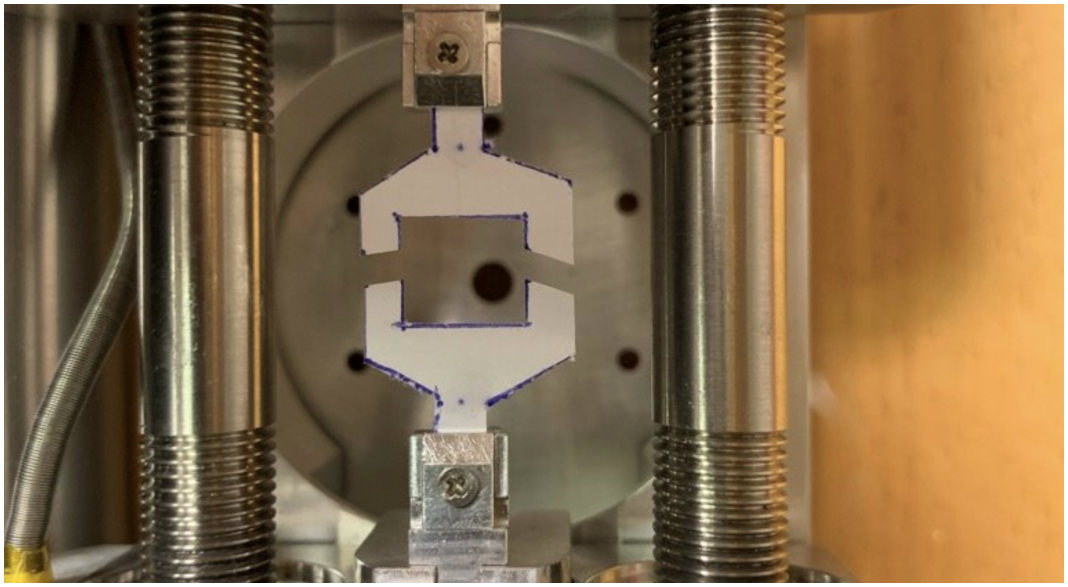


Figure 3.4: Mounted sample in microtester.

3.4 SEM imaging of fibers

The changes in the surface morphology of the T800 fibers due to lithiation and delithiation were analyzed using ZEISS SEM with a voltage of 5 kV. The samples of pristine and delithiated carbon fibers were prepared on an aluminium stub adhered with carbon tape as shown in Figure 3.5.

Figure 3.5: Single fibre mounted on aluminium stub.

The prepared specimens were mounted inside the sample chamber. The specimen were tilted to a desired angle so that the electron emitted from electron gun hits the fractured surface. Secondary electrons were used to image the fractured surface of the fiber. The information analyzed with the help of an EDX surface detector for elemental information.

4

Results

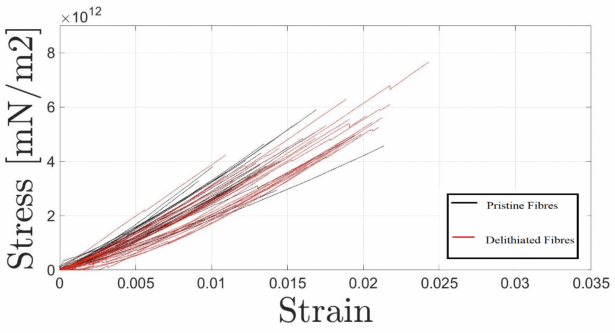
The chapter presents the results from the experiments conducted as mentioned in the above section. This chapter is divided into mechanical properties, SEM, and EDX analysis.

4.1 Mechanical Properties

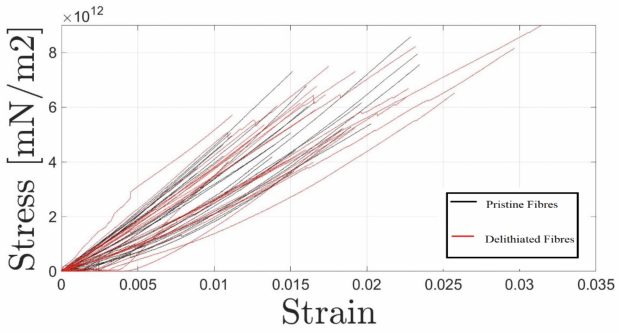
This section discusses the mechanical performance of single pristine and de-lithiated fibres which are analyzed by using a microtester with 1 N load cell. The results from the test are listed below in subsections.

4.1.1 Stress-strain plots

In this study, the results from the tensile test of 25 mm and 15 mm gauge length are presented. Figure 4.1 represents the stress-strain curve for 25 mm and 15 mm gauge length of 24 fibres. From the figures, the pristine and delithiated fibres failed in a brittle manner and are having almost similar young's modulus (slope) value.



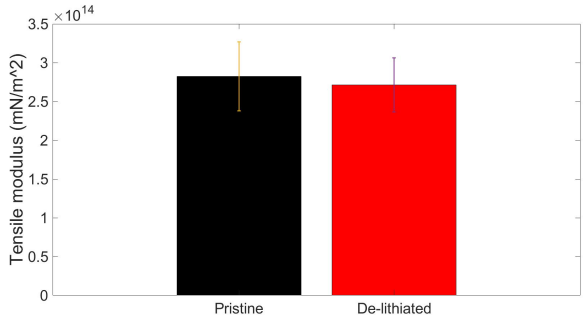
Stress strain curve : 25 mm gauge length



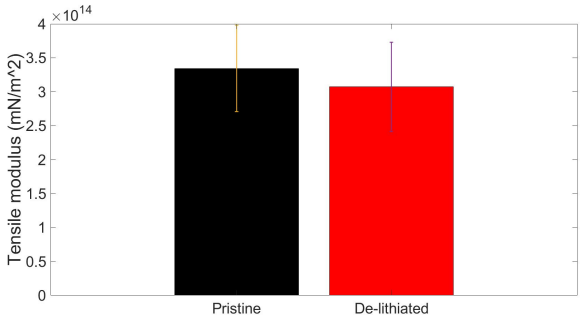
Stress strain curve : 15 mm gauge length

Figure 4.1: Stress-strain diagram of 25 mm and 15 mm gauge length of pristine and delithiated T800 carbon fibres.

From Figure 4.2 it can be seen that the difference in the tensile modulus of pristine and delithiated are not large and the average tensile modulus of the fibre comes to around 290 GPa for 25 mm gauge length and 310 GPa for 15 mm gauge length.



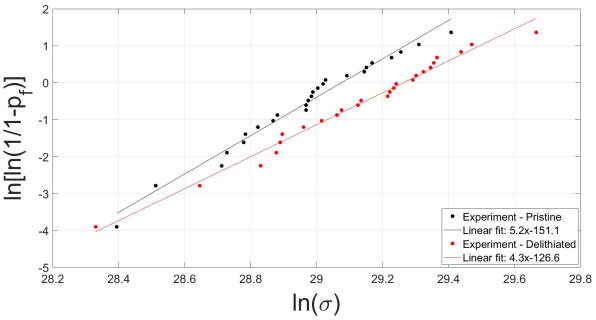
Tensile modulus : 25 mm gauge length



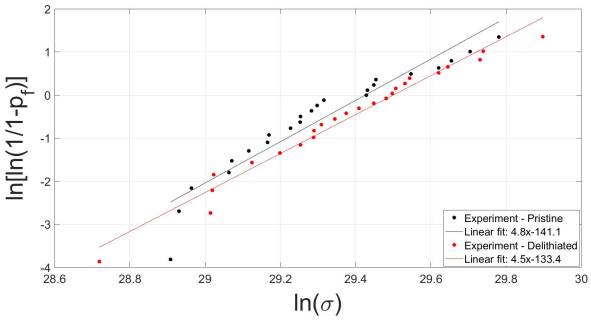
Tensile modulus : 15 mm gauge length

Figure 4.2: Tensile modulus of pristine and delithiated fibres of 25mm and 15 mm gauge length.

The scatter in the strength of the material is determined by the Weibull modulus. Figure 4.3 represents the Weibull plots for the 25 mm and 15 mm gauge lengths. The Weibull modulus 'm' value of 25 mm gauge length pristine fibres is 5.2 and for delithiated fibres is 4.3. The Weibull modulus 'm' value of 15 mm gauge length pristine fibres is 4.8 and for delithiated fibres is 4.5.



Weibull modulus : 25 mm gauge length

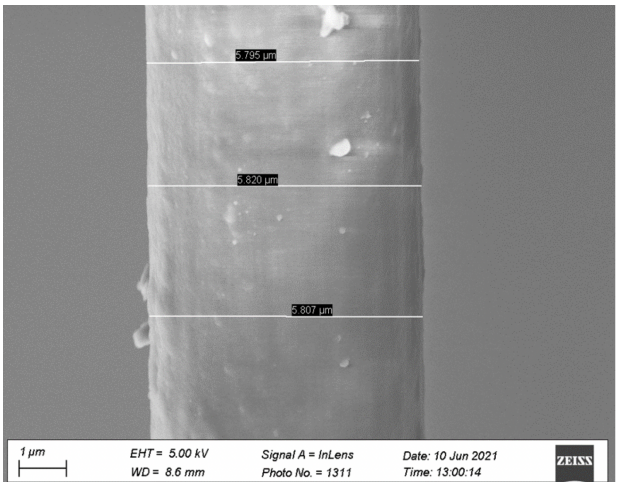


Weibull modulus : 15 mm gauge length

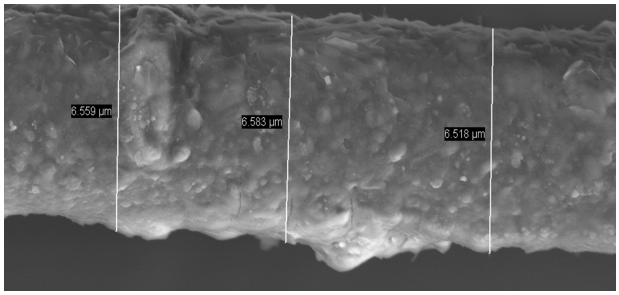
Figure 4.3: Weibull diagram of pristine and delithiated T800 carbon fibres with a gauge length of 25 mm and 15 mm.

4.2 SEM and EDX analysis

The surface of the fibre was analyzed in SEM Figure 4.4 shows the surface morphology of the pristine fibres and delithiated fibre. From the figure, the average fibre diameter of pristine fibres are 5.8 µm which is almost near to the values specified in the supplier datasheet. The average diameter of the delithiated fibre is around 6.5 µm which is almost 1.5 µm increase from the original diameter



Pristine fibres



Delithiated fibres

Figure 4.4: Surface morphology of pristine and delithiated single carbon fibres.

The SEM micrographs of pristine fibre are shown in Figure 4.5. From the micrograph, it can be seen that the pristine fibres failed under tension. The pristine fibre has granular morphology with a rough texture surface structure. The failure in the fibre is initiated near the surface of the fibre and the crack propagates through the fibre creating the rough morphology on the fibre. This can be identified through the radial marks which are created because of the crack propagation [25].

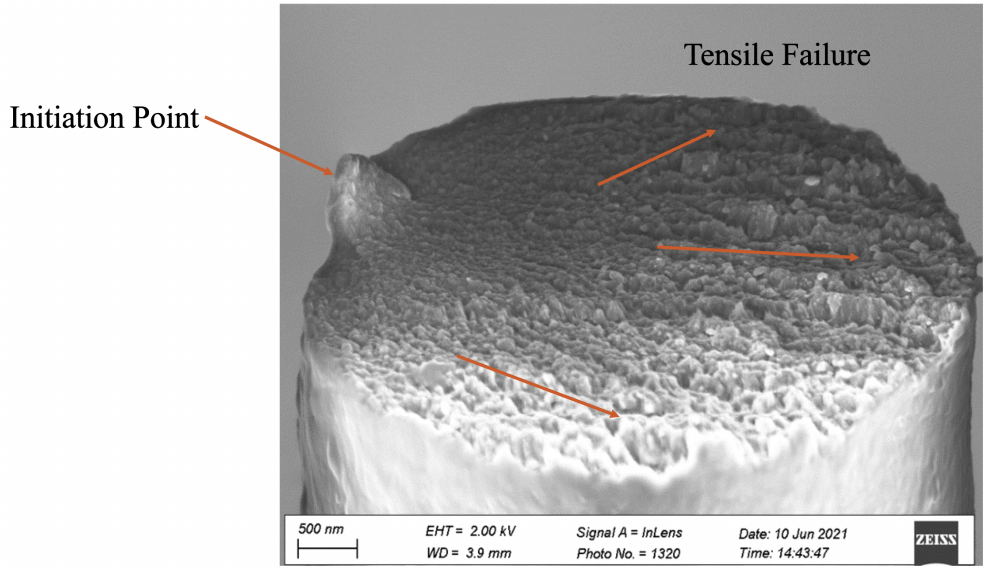
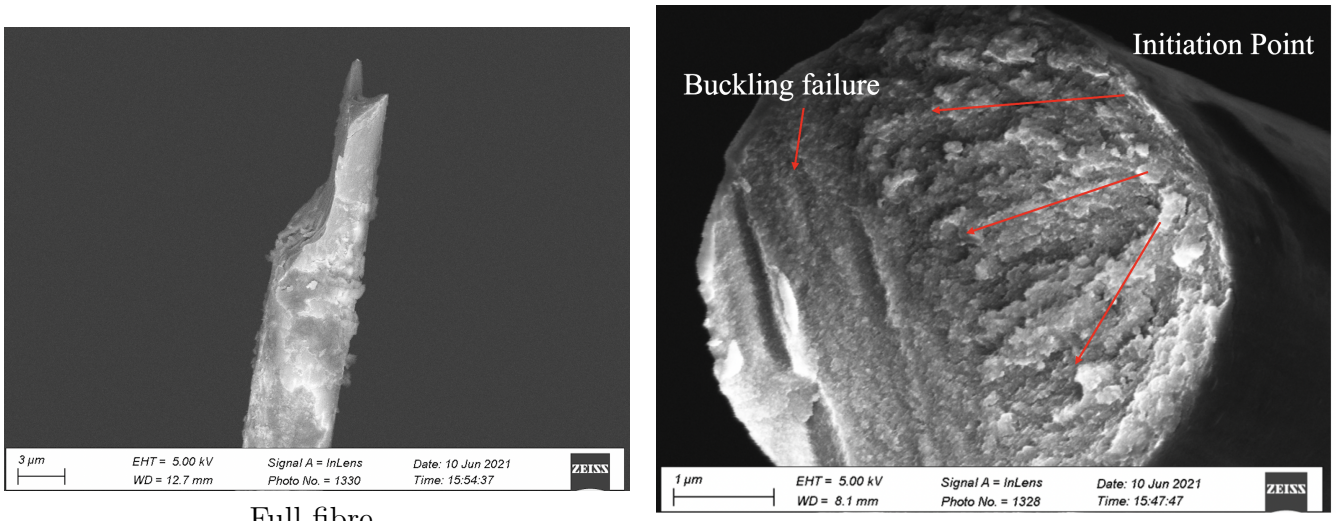


Figure 4.5: SEM micrograph showing fractured surface of pristine fibres

The fractured surface of delithiated fibre is shown in Figure 4.6. As the information from the micrographs is less it is difficult to conclude the type of fracture. The fibres have rougher surface morphology than pristine fibres. The micrograph implies that half of the fibres are failing in tension and another half in compression as shown in Figure 4.6. From the other SEM micrograph, it can be observed that only half of the fibre failed the other half remained in the fibre [25].



Full fibre

Fractured fibre

Figure 4.6: SEM micrograph showing fractured surface of delithiated fibres.

The EDX analysis was used to detect the elemental composition in the fibres. Figure 4.7 shows the comparison of elemental composition between pristine and delithiated fibres. The red spectrum indicates the pristine fibre and the yellow spectrum indicates the delithiated fibre. From the figure, it can be concluded that the pristine fibres have the maximum amount of carbon and a trace amount of oxygen in the fibre but the elemental composition in delithiated fibre is different from the pristine fibre. The delithiated fibre has more oxygen and a bit less carbon content when it is compared with pristine fibres.

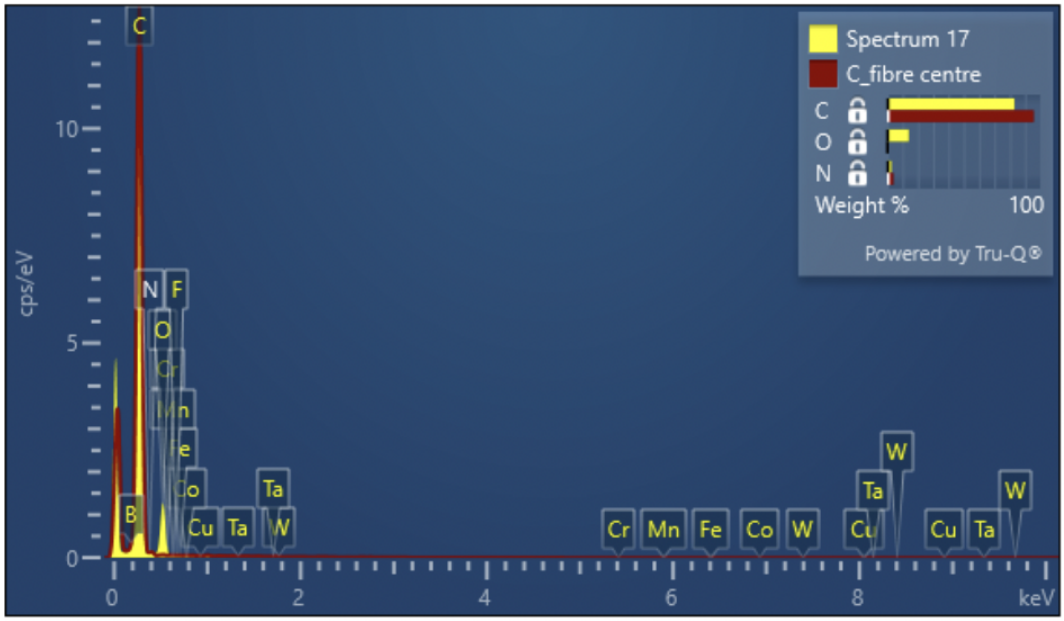


Figure 4.7: Comparison of EDX spectrum of pristine and delithiated fibres.

5

Discussion

5.1 Mechanical properties

Section 4.1 presents the stress-strain plots, tensile modulus, and Weibull modulus of pristine and delithiated fibre. Figure 4.1 shows the stress-strain plots of pristine and delithiated fibres. Even though all the fibres were tested in ambient room atmosphere scatter in the plots were observed. The scatter in the data sets can occur due to various reasons. Some of the differences in the data sets can be attributed to the microstructure of the fibre. As the fibres were lithiated and delithiated due to the insertion of Li-ions into the graphetic microstructure it might have disturbed the graphene layer which affects the stiffness of the material.

A constant value of 25 mm and 15 mm were assumed as the gauge length. The strain in the fibres was calculated by assuming the original length of fibres. Even though the gauge length of fibres was assumed as the constant value during the sample preparation they were not exact values there was a slight deviation from the original values. These deviations in the fibres can affect the modulus value as they are assumed for the calculation of strain.

The SEM results from surface morphology show that the diameter of the fibre is not constant over the length. The fibre diameter was considered constant in this project. As the diameter of each fibre was not calculated they had a varying crossection which can affect the modulus of the fibre as stress is considered for modulus calculation.

From the previous work, it was concluded that the stiffness will not be affected by the lithiation and delithiation, but it can be seen from the Figure 4.2 there is a small difference in the stiffness of pristine and delithiated fibres. This can be due to the lithiation and delithiation process which might have affected the crystalline structure of the fibres, as the difference is very small it cannot be concluded.

The Figure 4.3 represents the Weibull modulus of pristine and delithiated fibres. The Weibull modulus value for pristine is higher than the delithiated fibres. The 25 mm gauge length pristine fibres are having higher Weibull modulus than 15 mm gauge length, this can be accounted to the number of flaws and the distribution of flaws in the fibre increases as the length of fibre increases. The higher value of 'm' signifies that the defect in the fibre is more evenly distributed over the length of

fibre. The lower values signify that the defects are randomly distributed.

5.2 SEM and EDX analysis

Section 4.2 presents the results from the SEM and EDX analysis. Figure 4.4 represents the surface morphology of the pristine and delithiated fibres. The surface of the pristine fibre is smoother compared with delithiated fibre. This can be due to the remnants of Li-ion after the delithiation process. After delithiation, the fibres were washed in acetone for 30 minutes and vacuum dried. From Figure 4.5 it can be seen that the fibres failed due to tension. It can be clearly seen that the crack initiated and propagates through the surface. Figure 4.6 represents fractured surface from the delithiated fibre. From the fracture morphology, it is difficult to derive the information of the type of fracture. From the micrograph of fractured fibre it can be seen that half of the fibre is under tension and another half of the fibre is in compression. The surface texture of the fractured surface is rougher than pristine fibres. As there are no clear signs of the flow of cracks it is hard to deduce information from the fractured surface.

The results from EDX analysis are presented in Figure 4.7. The spectrum presents the comparison between pristine and delithiated fibres. The oxygen content in the delithiated fibres is higher than the pristine fibres. Once the fibres are taken out from the glovebox the remnants of lithium reacted with the atmosphere leading to formation lithium oxide. As lithium is one of the light elements it cannot be detected through EDX. The oxygen acts as an indirect indicator of residual lithium which shows that some lithium remained in the fibre.

6
Conclusion

This schapter summarizes the results from the experimental analysis of a single carbon fibre and concludes the influence of lithiation and delithiation on the stiffness and strength of the fibre.

The scatter in the stress-strain plot and difference in the tensile modulus of pristine and delithiated fibers can arise from various factors such as disturbance in the microstructure due to lithiation and delithiation, varying gauge length, and varying diameter of fibres. The difference in the tensile modulus is still relatively small.

The results from the Weibull modulus suggest that after delithiation some fibres are more prone to fail easily and strength degrades. The fibres with higher gauge length are having higher slope values compared with the fibres with lower gauge length, as the length of the fibre increases the fibre will have more defects and the higher Weibull value represents that the defects are evenly distributed in the fibre.

From the SEM micrographs, it can be concluded that there is an increase in the diameter of the fibre as a electrochemical layer deposited on the fibre. From the weibull modulus results it was seen that the fibres after delithiation are more prone to strength degradation and failure. They are still load bearers the matrix is too weak. The EDX results suggest that the delithiated fibres were having remnants of lithium which got oxidized when they were exposed to the atmosphere. The SEM micrographs of pristine suggested that the fibres failed under tension but the delithiated showed both tension and compression failure. As it is a difficult task to track back the information it is unclear whether it is the secondary failure or primary failure.

7
Future work

- In order to improve the performance of the SBC, various research can be conducted to have a better performing battery. Preparing samples on the better tabbing material instead of paper which will have less wetting when a Loctite glue is placed on the fibre.
- The fibres can be lithiated for different charge rate such as 25%, 50%, 75% and evaluate the effect of lithation on the mechanical properties of fibres. In the present study the speed of the test was kept constant to 1 µm/s, this speed can be increased and results can be analysed on how the properties are varying with respect to the test speed.Carrying the tests om different type of fibre samples other than T800 and comparing the results will help in having better material for SBC.
- Testing the lithiated single fiber for evaluating the effect of lithiation on the tensile modulus of the fiber.
- Conducting in-situ test on lithiated fibers in SEM to investigate failure process the fractured surface in higher magnification. Analysing the expansion in the diameter of fiber due to insertion Li-ions in the microstructure.
- Finding a feasible method to retain the fractured surface on the tabs to analyse the primary fracture surface for the better understanding fracture morphology
- Development of a feasible method for extracting lithiated single fiber from the bundle of lithiated fiber inside the glovebox and mounting it on the paper coupon for tensile testing of lithiated fibers.
- Method development is needed for transferring the lithiated fiber mounted on microtester from the glovebox to SEM for in-situ investigation of the failure of lithiated carbon fiber.

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