Thin-ply CFRP for cryogenic fuel tank applications

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Roberts Joffe
To my mom, whose moral and financial support allowed me to live everything I lived through all of my university years.
Abstract

The present work studies the failure behaviour of thin-ply CFRP under different thermal and mechanical stresses, and a possibility of establishing limit from which the thin-ply effect takes place.

Six new layups were manufactured by manually assembling the thin-ply tapes and curing the plate in a heated press. Additionally, material from a previous project was tested, with a “previous” version of the resin used for the new plates.

The different samples were subjected to 3-point-bending, tensile tests, mechanical fatigue, and thermal cycling. To check for damage, these sample were polished and checked under an optical microscope, in some cases under applied tension, and a few samples were subjected to a CT Scan.

From the results, it was evident that thin-ply samples behavior is superior to traditional CFRP in regards to crack initiation under cyclic stresses. No damage was inflicted on the samples by the tests they were subjected to, however there is still a lot of room to increase the severity of the tests. Samples under bending stresses or DCB test delaminated quite easily, with the approximate value of G1c being 0.4kJ/m2. As an initial result, the material seems to be ideal for its intended applications.

Keywords: Thin-ply, Carbon Fiber, Cryogenic, Microcracking, Fatigue, Composite, Damage.
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Apart from my supervisor Roberts Joffe, this work was of course possible thanks to the help from the people at Oxeon AB, particularly Florence Rinn, who guided me through my days at Oxeon and also during the experimental phase back at LTU. The previous work directly related to this one was a very importante pillar in the current results and all of it was carried out hand in hand with my colleague Jorge Valilla, with a lot of help and training from soon-to-be PhD Zainab Al-Maqdasi. Thanks to the work of all these people the following project was able to take shape.

Luleå, May 2019
Bernardo Sandoval
Chapter 1

Introduction

“The exploration of space will go ahead, whether we join in it or not...”

John F. Kennedy

1.1 Introduction

Over the last few decades, resin composites have replaced conventional metallic structures in improving aircraft and spacecraft performance. [3] In modern times, composite materials play a key role in the development of new aircrafts and components. These materials generally provide a great performance to weight ratio, as well as a broader spectrum of design possibilities. As such, commercial planes currently on active duty, and future aircraft designs are increasing their use.

Figure 1.1: Concept design for the Spaceliner 7
The cost of access to space is the major deterrent in space exploration and space utilization. The idea is to assure multiple time use to reduce cost of payloads and possible substitution of sub sonic air crafts, as well as reduction in environmental impact[16]. A reusable launch vehicle is the unanimous solution to achieve low cost, reliable and on-demand space access[11]. Some of the promising concepts for the next generation of reusable launch vehicles rely on polymer composite fuel tanks. These applications can benefit from the key properties of these materials, such as their reduced weight through increased specific strength, reduced cost of production and in an ideal case a good behavior towards thermal stress. [17]

A reusable launch system (RLS, or reusable launch vehicle, RLV) is a space launch system intended to allow for recovery of all or part of the system for later reuse. To date, several fully reusable sub-orbital systems and partially reusable orbital systems have been flown. However, the design issues are extremely challenging and no fully reusable orbital launch system has yet been demonstrated. A wide variety of system concepts have been proposed, and several are represented in those which have actually flown.

One of the main concerns regarding current technologies, as well as future hypersonic flight research, is not only the reduction of weight because of its improvement towards performance, but also because of environmental reasons. The production of lighter planes requires a lower investment of energy during the whole process, as well as a reduction in fuel consumption. On this same line of thought, fossil fuels are slowly being pushed aside in all industries in favor of greener technologies, and in this particular sector the key alternative has mainly been cryogenic fuels such as LH2 or liquid LO2 [16]. These fuels require very specific conditions of storage to be able to maintain a liquid state and be used for propulsion.

Figure 1.2: NASA Cryogenic tank [1]
High pressure hydrogen storage is the most conventional type of hydrogen storage. As the storage pressure increases, the density of the hydrogen gas will also increase. The tradeoff with utilizing high density/high pressure storage is the increase in tank mass necessary to withstand the higher pressures. The wall thickness of the tank will increase with the increasing hoop stress due to the higher gas pressure.

To reduce tank mass and volume over high-pressure gas storage, cryogenic storage of hydrogen can be used. The properties of liquid hydrogen enable significant increases in density over high-pressure gas storage as well as reduced tank mass due to lower pressure operation. The tank is usually a thin walled pressure vessel surrounded by a liner material. The tank materials must be resistant to hydrogen embrittlement, impermeable to hydrogen gas and capable of structurally withstanding the temperatures of liquid hydrogen. Also, because of the great change in temperature when the tank is fueled or emptied, thermal expansion and contraction is a major concern. Because of this it is usually required that the tank be made out of one type of material [5].

The materials used for these applications must thus have the capability of being exposed to extreme fluctuations in temperature without damage, to the pressure applied by the fuel from inside the tank, and ideally would be made of one solid piece to avoid any possible leakage in joints.

1.2 Previous research

In the last years, thin ply composites have been present in a broad spectrum of applications. These materials provided an increase in design space and mechanical properties.

Previous studies have shown the advantages of the thin-ply composites in regards to their failure resistance to both mechanical fatigue and thermal cycling. In [17], results showed that micro crack generation was delayed in samples with thinner plies (samples tested between 0.13 and 0.21mm), with a reduction in thickness of 30% resulting in a delay in crack initiation of around 200 cycles. After a higher number of cycles, the crack density is not much higher, but there are plies with no damage shown, which is ideal for fluid containment applications.

According to [2], the initiation of micro cracks in the matrix was subdued at lower temperatures, and there was a slight increase in the strength of the matrix, with an increase in shear modulus and tensile modulus for the composite. This would speak in favor of the particular application for cryogenic fuels, assuming this behavior is not hindered by the temperature contrast with the outside atmosphere. This is supported in part by [10], which showed that interlaminar shear strength increased at cryogenic temperatures, attributing it to a possible higher compression stress due to thermal shrinkage.

Specific studies focused on the fuel tank application this project is aiming at have shown the same results of crack initiation delay, with the samples subjected to an average flight temperature variations not cracking after 10 cycles[13]. The study suggests that cracks might be too small for traditional optical microscopy observation, and proposes the use of SEM to possibly locate any undetected damage, which is one of the main hypotheses in the following work.
As an additional observation, [4] argues that for thermally induced damage, edge crack counts might not necessarily be good indicators of the internal state of the laminate, which opens the question of new evaluation methods to be used, if available, on this project. Among these options, [12] suggests X-ray microtomography, or Micro CT, can facilitate the characterization of internal damage to samples, including micro cracks.

To assess the damage induced during these project, a formula provided by [9] will be used, which relates the material's Moduli, thermal expansion coefficients and temperature variation to the thermal stress to which the layers are subjected, as follows:

\[
\sigma_{th} = \frac{fE_T(\alpha_T - \alpha_L)\Delta T}{(1 - f)E_T + E_L}
\]

*\(\sigma\) = Residual thermal stress in the 90-layer.
*\(E\) = Young Modulus
*\(\alpha\) = Thermal expansion coefficient.
*\(f\) = ratio between number of 90-layers and total number of layer in the laminate.
*\(\Delta T\) = Different between test temperature and curing temperature.
*Indexes T and L refer to transverse and longitudinal values.

Regarding failure under mechanical loading, most of the research shows that thinner plies translate to higher resistance to crack initiation, as well as improved mechanical properties.

In [8] the effects of ply thickness was studied, among other factors, on strength and damage tolerance, presenting that current epoxy based composites can benefit from a reduction in thickness down to 34\(\mu\)m, which significantly improved tensile performance. These results also showed the increase in the onset point of damage relative to the ultimate strain of the fiber, related to the ply thickness as shown in NAME FIGURE. This is supported by a comparative study [15] which subjected samples to strains up to 1.5% and resulted in not only delayed initiation of cracks but also lower crack density overall.

Once the cracks have been initiated,[7] presents that the growth of these preexisting cracks

![Figure 1.3: Onset of damage strain in plain tension [8]](image-url)
is hindered in thinner plies, showing significantly narrower crack openings in 40\textmu m layers than in 80\textmu m and 160\textmu m layers.

Figure 1.4: Damage from strain: a) 40\textmu m, b) 80\textmu m, c) 160\textmu m
1.3 Previous work from this project

During the project preceding this one, a series of plates manufactured from TeXtreme material were tested. The plates all consisted of the same fiber and resin, with 8 different lay-ups to be studied.

The samples were divided into two main tests: Mechanical fatigue and thermal cycling. Mechanical samples (200x30mm) were subjected to 0.5% strain for 10, 100, 1000 and in some cases 10,000 cycles at 5Hz frequency. The samples subjected to thermal cycling (100x30mm) were submerged in liquid nitrogen for 10 minutes and then at room temperature (between 20-22°C) up to 100 times. After every step in the mechanical fatigue process, and after every 25 thermal cycles, the samples were inspected under a microscope.

None of the samples showed any sign of damage, adding to the evidence that thinner plies in composites provide improvement in their onset of visible damage. From these conclusions some alternatives for future work were though of, among which more strenuous tests and more advanced observation methods were discussed, such as Micro CT or SEM.

1.4 Failure mechanisms

Composite materials are peculiar materials themselves, due to the microstructure and way of manufacturing, so defects are not as consistent and widely known as the ones in metal alloys, for example. Defects can appear due to the manufacturing process or during the service life of the part. Normally, if the defect comes from the manufacturing of the material, porosity will be the main issue. Porosity are voids inside the matrix caused by poor curing processing or because of foreign bodies (impurities) inside the matrix that may appear during the manufacturing or even when the material is in pre-preg form [6].
1.4. Failure mechanisms

If the defects appear during the service life, we will find different kind of damages, mostly based on fatigue or impact. One of the most common would be delamination, where the stress generated in the material creates separation between the fibers and the matrix, producing an adhesive failure between both counterparts. This produces layers of separated fibers, just like it’s shown in Figure 1.6.

The other kind of defect commonly found in these kinds of materials are the matrix cracks and the matrix/fiber debonding both which are caused by severe loading or poor manufacturing of the specimen. It is worth noticing, since the main objective of this project is to make test in different ranges of temperature, that the difference in CTE (coefficient of thermal expansion) can cause enough stresses within the material so that no severe loading is needed to create these defects.

It is also worth mentioning that cracks may not reduce in a noticeable manner the stiffness of the material (due to the stiff load bearing fibers) but other parameters like CTE and Poisson ratio due decrease.

Figure 1.6: Example image of hard delamination

Figure 1.7: Example of cracks converging into the interface to produce delamination
For standard composites, the general failure sequence under fatigue stress is as follows [14]:

\begin{figure}[h]
\centering
\includegraphics[width=\textwidth]{standard_damage_generation.png}
\caption{Standard damage generation process due to mechanical fatigue}
\end{figure}

The initial matrix cracks are generated because of the stress, and grow both in length and width until the edges of the layers. Once coupled on the layer borders, the damage is spread in the direction of the layer generating delamination and finally fiber breaking and complete failure.

\section{1.5 TeXtreme material}

The material used during the project was provided by the company Oxeon AB. This is a Swedish company founded in 2003 after Dr. Nandan Khokar developed the Tape Weaving technology at Chalmers University of Technology. Since then, they have produced high performance reinforcements under the brand name TeXtreme. In 2004, TeXtreme Spread Tow Fabrics were launched to the market.

Since then, Oxeon AB has grown into a leading company in the field, where their materials are being used in applications such as sporting goods, industrial development and aerospace.

The TeXtreme® products are based on three different patented technologies. One of these technologies is the Spread Tow Technology, which works by spreading the fibers into unidirectional tapes. Other technologies are weaving of these Spread Tow Tapes either in 0/90 or in +alpha/-beta orientation. The materials used during this research were thin-ply carbon fiber/epoxy composites manufactured with TeXtreme Spread Tow
1.5. TeXtreme material

Tapes, in different configurations depending on the application and test required. The samples will be explained later in detail, since each one may have different behavior towards the tests performed to them.
2.1 Previously Tested Material - DS18-102XX

As mentioned in the introduction, this project derives from previous work done on thin-ply CRFP from Oxeon. The following table presents the different lay-ups and dimensions of the plates used, out of which four plates (two from the mechanical test batch, and two from the thermal test batch) were selected for further tests on this project.

<table>
<thead>
<tr>
<th>Name</th>
<th>Layup</th>
<th>Size (mm)</th>
<th>FVF (%)</th>
<th>Test</th>
</tr>
</thead>
<tbody>
<tr>
<td>DS18-10211</td>
<td>[(0/90)]_5s</td>
<td>270x270x1.00</td>
<td>55.7</td>
<td>M</td>
</tr>
<tr>
<td><em>DS18-10212</em></td>
<td>[(0/90)]_8s</td>
<td>270x270x1.54</td>
<td>56.9</td>
<td>T</td>
</tr>
<tr>
<td><em>DS18-10213</em></td>
<td>[(0/90)/±45/(0/90)/±45/(0/90)]_S</td>
<td>270x270x1.08</td>
<td>51.6</td>
<td>M</td>
</tr>
<tr>
<td><em>DS18-10214</em></td>
<td>[0_4/90_3/0_2/90/0_2/90_2/0_2]_S</td>
<td>270x270x1.65</td>
<td>53.5</td>
<td>T</td>
</tr>
<tr>
<td><em>DS18-10215</em></td>
<td>[90_2/0_3/90/0_2/90_2]_S</td>
<td>270x270x1.04</td>
<td>53.3</td>
<td>M</td>
</tr>
<tr>
<td>DS18-10216</td>
<td>[0/90]_5s</td>
<td>270x270x1.04</td>
<td>53.7</td>
<td>M</td>
</tr>
<tr>
<td>DS18-10217</td>
<td>[(0/90)/±45]_4s</td>
<td>270x270x1.53</td>
<td>58.2</td>
<td>T</td>
</tr>
</tbody>
</table>

*M: Mechanical fatigue
**T: Thermal cycling

The four batches selected were chosen to provide a comparative view between samples previously subjected to mechanical or thermal damage, as well as between woven samples and UD samples. The following images show the polished surfaces which were observed throughout the many experiments to look for any presence of damage.

Despite the mechanical stresses produced due to testing, it seemed clear that the limit of the material was not yet reached. This has to be reached in order to measure and characterize the defect creation and evolution in a reliable manner. That meant that further testing had to be performed, both mechanical and thermal fatigue. Once the appearance of cracks begins, it will be possible to track their evolution.
2.2 Resin

The new batch of plates was manufactured with Textreme tapes pre-impregnated with a new version of the epoxy resin used for the DS18-102XX batch. The new version has the same chemical composition but has been slightly improved for better processing. The resin must be carefully handled and preferably in small quantities, since the curing process is highly exothermic and if uncontrolled can lead to a high amount of fumes.
2.3 Newly Manufactured Material - DS19-1027X

The new batch of samples was manufactured by tape hand-laying, as explained in the following section. Each different lay-up was chosen to study a specific aspect, with the number of total iterations limited due to the amount of material and time. As a result, the following seven configurations were considered:

- Sample 10272 was manufactured in one thick 90 degree layer to calculate the transverse modulus of the material.
- Samples 10273 and 10274 are meant to emulate the lay ups tested in the CHATT project and samples 10214 and 10215 from the previous batch
- Samples 10275 and 10276, UD and woven respectively were manufactured to study if there are any significant differences in behavior.
- Sample 10277, with a thick 90 degree layer was designed to help define the threshold of the thin-ply effect.
- Sample 10278, which has a release film inserted on half of the plate, was manufactured specifically for a Double Cantilever Beam test to study the material’s fracture toughness.

<table>
<thead>
<tr>
<th>Name</th>
<th>Layup</th>
<th>Size (mm)</th>
<th>RC (%)</th>
<th>Estimated void%</th>
</tr>
</thead>
<tbody>
<tr>
<td>DS19-10272</td>
<td>[90]_{21}</td>
<td>269x270x1.06</td>
<td>34.01</td>
<td>2.02</td>
</tr>
<tr>
<td>DS19-10273</td>
<td>[0_4/90_3/0_2/90/0_2/90_2/0_2]_s</td>
<td>271x271x1.82</td>
<td>41.52</td>
<td>0.00</td>
</tr>
<tr>
<td>DS19-10274</td>
<td>[90_2/0_3/90/0_2/90_2]_s</td>
<td>272x275x1.29</td>
<td>42.23</td>
<td>8.71</td>
</tr>
<tr>
<td>DS19-10275</td>
<td>[0/90]_{4S}</td>
<td>135x270x0.85</td>
<td>39.25</td>
<td>0.00</td>
</tr>
<tr>
<td>DS19-10276</td>
<td>[(0/90)]_{4S}</td>
<td>130x275x0.76</td>
<td>32.62</td>
<td>0.00</td>
</tr>
<tr>
<td>DS19-10277</td>
<td>[0_2/90_6]_s</td>
<td>270x275x0.77</td>
<td>34.84</td>
<td>0.00</td>
</tr>
<tr>
<td>DS19-10278</td>
<td>[0]_{20} (Film inserted)</td>
<td>274x274x1.01</td>
<td>32.55</td>
<td>4.24</td>
</tr>
</tbody>
</table>

The following image presents the different sample surfaces that will be studied, all images have been resized to fit all layers and be comparable, so they are not corresponding to their relative size.
As seen in the images, samples 10273 and 10274 present some voids, which could also be present in the other samples. However, since the samples are cut and polished in only one spot of the plate, it is only possible to assess the damage due to manufacture in those places.

Additionally, Sample 10273 presented a high variation in thickness in different points of the plate, possibly due to wrongly distributed pressure in the curing plates. Because of this, only a few samples were taken, from the area most stable in thickness.
3.1 Plate manufacturing

3.1.1 Manual Distribution and Curing cycle

The manufacturing of the new plates was done on-site at the Oxeon AB facilities in Borås. The UD lay-up of the plies was done manually, by cutting the pre-manufactured spread tape, peeling off the protective/anti-adhesive film and laying it in the corresponding direction. To avoid variations of thickness in the overlaps or gaps between the tapes, every consecutive layer was slightly shifted to the side.

Woven samples were manufactured using a traditional weaving pattern for the tapes, in which alternate rows of tape are folded up to allow for the perpendicular row to be placed. This woven layers are made with larger dimensions and then cut and stacked to fit the desired size and number of plies.

For samples 10272 and 10277, a tape winding process has been used to build laminate thickness more efficiently. The tapes were laid over a spinning paper core covered with release film, so some slight angling of the tapes might have happened. However, since not every individual fiber on the tape is perfectly parallel, the difference in orientation is negligible.
Methodology

The laid plates were then placed inside a heated metal press which operates with calibrated springs to provide the corresponding pressure depending on the step on the curing cycle, which is described in the following table. The press was covered with release film to assist on the removal of the sample, and the sample itself covered with a silicone mat in order to distribute pressure more evenly.

<table>
<thead>
<tr>
<th>Step</th>
<th>Initial t (°C)</th>
<th>Final t (°C)</th>
<th>Rate (°C/min)</th>
<th>Time (min)</th>
<th>Pressure (bar)</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>R.T</td>
<td>120</td>
<td>5/min</td>
<td>20</td>
<td>1</td>
</tr>
<tr>
<td>2</td>
<td>120</td>
<td>120</td>
<td>-</td>
<td>10</td>
<td>1</td>
</tr>
<tr>
<td>3</td>
<td>120</td>
<td>150</td>
<td>5/min</td>
<td>6</td>
<td>1</td>
</tr>
<tr>
<td>4</td>
<td>150</td>
<td>150</td>
<td>-</td>
<td>10</td>
<td>1</td>
</tr>
<tr>
<td>5</td>
<td>150</td>
<td>150</td>
<td>-</td>
<td>60</td>
<td>4</td>
</tr>
<tr>
<td>6</td>
<td>150</td>
<td>R.T</td>
<td>Open to R.T</td>
<td>≈30</td>
<td>1</td>
</tr>
</tbody>
</table>
3.2 Sample Preparation

3.2.1 Cutting

The samples were cut using a Discotom 100 semi-automatic wet cutting machine. Initial samples were of the dummy plate, with different widths referred to the “unit cell”: 1x, 1.5x, 2x. This parameter, however, is not relevant to the properties, since the layers are not exactly placed on top of one another. The wheel is made of diamond particles and a significant thickness which eats into the sample up to 2mm when it’s cut. For that reason, some adjustments were made prior to cutting the sample. The automatic process was done at a linear speed of 1mm/s, and 2750rpm for the disc rotation. The samples were marked, leaving some margin for the wheel to remove material.

3.2.2 Grinding-Polishing

In order to obtain a consistent and clear image of any defects using the microscope, the samples must be grinded and polished. After cutting the sample, the surface of the specimen is rough and heterogeneous, not ideal for testing and further characterization. For that reason, some material must be removed, as well as to level up and normalize the width of all the samples to have a more standardized size. The samples were grinded with SiC paper in 4 different steps and for increasing lengths of time: 600, 1200, 2500, 4000. Following the grinding, the polishing was also done in increasing steps of time and decreasing grain size: 6μm, 3μm, 1μm, 0.25μm.
## 3.3 Sample dimensions

<table>
<thead>
<tr>
<th>Name</th>
<th>Length</th>
<th>Width</th>
<th>Thickness</th>
</tr>
</thead>
<tbody>
<tr>
<td>10213</td>
<td>200</td>
<td>11.0</td>
<td>1.08</td>
</tr>
<tr>
<td></td>
<td>200</td>
<td>10.1</td>
<td>1.08</td>
</tr>
<tr>
<td>10215</td>
<td>200</td>
<td>8.0</td>
<td>1.04</td>
</tr>
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<td>8.0</td>
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<thead>
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<th>Width</th>
<th>Thickness</th>
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<tbody>
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<td>20</td>
<td>1.29</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>20</td>
<td>1.29</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>20</td>
<td>1.29</td>
</tr>
<tr>
<td>10275</td>
<td>119</td>
<td>20</td>
<td>0.85</td>
</tr>
<tr>
<td></td>
<td>118</td>
<td>20</td>
<td>0.85</td>
</tr>
<tr>
<td></td>
<td>119</td>
<td>20</td>
<td>0.85</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>20</td>
<td>0.85</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>20</td>
<td>0.85</td>
</tr>
<tr>
<td>10276</td>
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<td>0.76</td>
</tr>
<tr>
<td></td>
<td>117</td>
<td>8.0</td>
<td>0.76</td>
</tr>
<tr>
<td></td>
<td>118</td>
<td>8.1</td>
<td>0.76</td>
</tr>
<tr>
<td></td>
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<td>20</td>
<td>0.76</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>20</td>
<td>0.76</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>20</td>
<td>0.76</td>
</tr>
<tr>
<td>10277</td>
<td>135</td>
<td>8.0</td>
<td>0.77</td>
</tr>
<tr>
<td></td>
<td>135</td>
<td>7.9</td>
<td>0.77</td>
</tr>
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<tr>
<td></td>
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<td>20</td>
<td>0.77</td>
</tr>
<tr>
<td></td>
<td>100</td>
<td>20</td>
<td>0.77</td>
</tr>
</tbody>
</table>
3.4  Fatigue Test

3.4.1  Holding tabs fitting

The carbon fiber samples used in this project have a very smooth surface, which is likely to generate problems with the grip of the tensile testing machine. To prevent this, the samples were fitter with additional tabs on the area in contact with the machine. For the fitting of the tabs, the samples were sanded on both ends, followed by the application of a thin layer of epoxy resin, and placing the tabs over the samples, with an additional weight on top to maintain its position for the following 12 hours. Once the curing was done, the batches of samples were cut back to individual ones, and the tabs were grinded down to ensure that the lateral surface of the sample would be as close as possible to the microscope lens, in order to avoid any focusing problems.

3.4.2  Sample placing

The tensile testing machine used works with manual clamps, and 3 different levels of movement. To establish the complete range of movement, the machine must be placed on level 0, and the head which holds the top clamp moved to the position from which the experiment will be done. Once the head has been placed, level 1 is selected to adjust the height of the upper arm and place the sample. The sample is fastened using two manual bolts on each end, with consideration to apply the same amount of torque on all four bolts. For this test, the applied torque was approximately 25 Nxm. The machine will always be kept on level 1 to prevent any damage to the sample due to unintended movement. Once the method of testing has been chosen, the machine will have to be changed to level 2 before starting.

3.4.3  Procedure definition

The first procedure involved the rough evaluation of the Young Modulus of the samples. The test was defined in two blocks: a loading part up to 0.5% strain and its corresponding unloading. Through the results of the unloading curve, a value of the Young Modulus was extracted, which was then applied to the parameters of the fatigue test cycles. The next process defined was the cycling, where an initial step was established to strain the sample up to 0.5% in one minute and then release until 0.05% at the same rate before starting the first cycle. The process was done at a frequency of 5 Hz, from 0.05% to 0.5% strain 10, 100 and 1000 times depending on the test, with a final unloading step symmetric to the first one. This was repeated for the 0.9% strain tests. The parameters for stresses and amplitudes are presented in the following table. Before each of the two procedures is started, all measured parameters must be balanced either automatically by establishing the 0 value to the parameter or manually by adjusting the height of the clamps on the level 1 of the machine.
3.5 Tensile Test

The preparation procedure for the tensile testing of the samples is exactly the same as that followed for the fatigue testing. Once the samples are prepared and set in place, the test is defined on the software for a loading curve and unloading curve.

The samples were subjected to higher strains on each test, with the increase in strain being smaller as the value is near the established failure point of 1.46% strain.

The loading steps were the following: 1.000%, 1.100%, 1.200%, 1.300%, 1.350%, 1.375%, 1.400%, 1.425%, 1.450%, 1.475%.

3.6 Bending Test

Following the sample preparation procedure mentioned above, the samples were cut and sized according to the ASTM D790 standard. Similarly, the support span distance was established following the same standard.

The position and load readings on the machine must be calibrated before the first use, as well as the alignment of the loading nose, insuring that the values obtained are not affected by any previous mistakes.

Using markings on both the samples and the support span, the sample is placed centered
on top of both supports, and as centered as possible across the length of the supports. Following this, the nose is lowered until it sits close to the sample, without touching it.

Once the sample is in place, the test parameters are set on the software, and the experiment is initiated. The loading steps were the following: 150N, 175N, 200N, 225N, 240N.

<table>
<thead>
<tr>
<th>Samples</th>
<th>Load rate (mm/min)</th>
<th>Support span (mm)</th>
<th>Nose diameter (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10212</td>
<td>3.0</td>
<td>62</td>
<td>10</td>
</tr>
<tr>
<td>10214</td>
<td>3.0</td>
<td>64</td>
<td>10</td>
</tr>
</tbody>
</table>

### 3.7 Thermal Cycling

The current study has been focused exclusively on the effects of cryogenically induced stresses, leaving the evaluation of the effects of high temperatures for further investigations.

The samples were observed at intervals of approximately 20 to 30 cycles, with the intention of mapping any area where cracks or delamination appeared, and studying the growth in crack size, number of cracks and delaminated areas in relation to the established area.

In order to make sure that the effects of cycling were being observed in the same section of the sample, some markers were made on the side of the samples. This was followed by the identification by microscope of any areas in which the distribution of plies provided a distinguishable check point from which to find the spot again after re-cycling the samples.

The stresses induced in the samples due to the contrasts in temperature can be estimated by using the following equation mentioned in the introduction [9]:

$$\sigma_{th} = \frac{fE_L E_T (\alpha_T - \alpha_L) \Delta T}{(1 - f)E_T + E_L}$$  \hspace{1cm} (3.1)

For the cycling, a gadget was devised by attaching a metal chain to a polymer foam disc which acted as a lid for the double layered insulating cylinder. Under this lid, two open chain links were hoked for the quick mounting and dismounting of the sample sets.

The container was a double layered plastic cylinder, with an insulating foam between...
both layers. It had a capacity of approximately 1L and was filled completely every 4 to 5 cycles depending on the level of nitrogen left.

The samples were drilled on one end and fit in batches on a 10cm screw, which was held at both ends by 6 links of a chain to hook them into the ones on the foam lid. Each batch of samples was submerged from 50% to 100% in liquid nitrogen depending on the available level. Due to the size of the container, any volume not occupied by liquid nitrogen was easily filled by evaporated nitrogen, thus providing an almost even temperature distribution through the sample, despite them being fully or half-submerged in LN2.

Due to the geometry of the samples, very thin and relatively small, and the cooling rate provided by the nitrogen, the time needed for them to reach the desired temperature was relatively short, so the cycle times were as follows:

<table>
<thead>
<tr>
<th>Samples</th>
<th>Temp in (°C)</th>
<th>Time in (min)</th>
<th>Temp out (°C)</th>
<th>Time out (min)</th>
</tr>
</thead>
<tbody>
<tr>
<td>10273</td>
<td>-196</td>
<td>5</td>
<td>60</td>
<td>5</td>
</tr>
<tr>
<td>10275</td>
<td>-196</td>
<td>5</td>
<td>60</td>
<td>5</td>
</tr>
<tr>
<td>10276</td>
<td>-196</td>
<td>5</td>
<td>60</td>
<td>5</td>
</tr>
<tr>
<td>10277</td>
<td>-196</td>
<td>5</td>
<td>60</td>
<td>5</td>
</tr>
</tbody>
</table>

The temperature inside the container is -198.8°C, or the boiling point of nitrogen. The temperature marked as R.T. has been between 21 and 22°C. While the effects of this might be negligible, it is worth noting that the thermal cycles were not made in a continuous fashion. Some factors, such as the difference in the amount of nitrogen in the container, slight variations in cycle times due to the handling of the samples, interruptions of the number of cycles due to lack of time or the possible variation in the cooling rate between batches of different number of samples at the same time.

### 3.8 CT Scan

In order to study in closer detail the damage inflicted on the samples after the fatigue cycles and high strain tests, one of the pieces from a broken sample was subjected to a CT scan. The availability of the equipment allowed only for a 4h scan on lower resolution, which was taken as a preliminary review to check for the usefulness of this test when it comes to damage assessment. Only the results of this CT scan will be shown in this project, since the actual test was done by Professor Roberts Joffe, acting supervisor of the current project.

*Figure 3.4: 10215C sample broken after extreme tension. Sample used for CT Scan*
3.9 Microscope under tension

Following the results of the previous project, a hypothesis was brought up that cracks small enough to not be observed under the microscope were forming on the matrix, and closing after unloading.

In order to check this, a portable 3 point bending rig was built to keep the sample under tension while it’s being inspected on the microscope.

<table>
<thead>
<tr>
<th>Sample batch</th>
<th>DS-10213</th>
</tr>
</thead>
<tbody>
<tr>
<td>Length (mm)</td>
<td>120</td>
</tr>
<tr>
<td>Width (mm)</td>
<td>8.00</td>
</tr>
<tr>
<td>Thickness (mm)</td>
<td>1.05</td>
</tr>
<tr>
<td>Displacement (mm)</td>
<td>7.00</td>
</tr>
</tbody>
</table>

Figure 3.5: Constant tension rig for microscope / Tension distribution diagram
3.10 Sample inspection

After each significant step in testing, the samples were taken to an optical microscope to check for edge damage. Due to the size of the samples and the magnification required to check for cracks, both mechanical and thermal samples were checked throughout a length of approximately 50 around the center of the sample. If no sign of damage was found, a representative compound picture of each sample batch was taken by combining a grid of between 18 and 27 pictures at 500x magnification, with a 15% overlap between the pictures.

Figure 3.6: Sample observation surface explained

3.11 Double Cantilever Beam (DCB) Test

In order to study the resin’s fracture toughness, the sample batch number 10278 was cut and tested according to the ASTM D5528-13 standard, with additional polymeric tabs added on both top and bottom of the samples in a sandwich distribution in order to provide some rigidity to the sample during the test.

The samples cut were 20mm wide and 160mm long, with the pre-inserted film penetrating lengthwise up to 50mm inside the sample. In order to better observe the crack growth, white paint was applied on both sides of the sample and a magnifying glass was placed on the testing site. The samples are attached on the top and bottom to metal hinges which are then placed on the machine’s clamps, which will provide the tension load to the material.

The loading process is carried out by displacement control, at a rate of 2mm/min. The sample is loaded and carefully watched through a magnifying glass to determine when the crack has propagated approximately 5mm, at which point the sample is unloaded and the crack tip marked on the sample. This sequence was then repeated 4 different times to obtain the different G1 values.
Once the parameters are obtained, the following formula is used to calculate the value of $G_1$:

$$G_1 = -\frac{3P\delta}{2ba}$$  \hspace{1cm} (3.2)

*P = Load
*\(\delta\) = Displacement
*b = Sample width
*a = Crack length
4.1 Tests from DS18-102XX batch

4.1.1 Tensile to failure

As specified in the methodology section, the samples from the DS18-102XX batch were tested and different strains until failure, with the objective of finding the point of strain at which crack initiation occurred.

These different strain steps were done at decreasingly higher values, starting at 1% strain up to failure strain (around 1.41-1.47%). Between each step, the samples were checked under microscope for damage signs, and if none was present the test would continue to the following step. At values closer to the assumed breaking point of 1.46% strain, the increase in strain would be smaller, decreasing from steps of +0.1% to +0.025%.

Only one of the tested samples showed some sign of damage. Sample DS18-10213D showed fiber cracks after 1.45% strain. To check for further growth of the damage, this sample was then subjected to the same mechanical fatigue procedure that was used in the previous work, and will be used again with the new batches. The sample was subjected to 10, 100, 1000 and 10.000 cycles at 0.5% strain and 50Hz, and checked for growth in damage after each step of cycles was finished.

After all the fatigue tests, no growth of the cracks was detected, or initiation of new cracks. The sample was then strained at a higher value, following the procedure with an increase of +0.025% strain to continue evaluating the cracks, and finally broke at 1.475%.

No other sample showed any visible crack initiation before failure, which might suggest that there is a different damaging mechanism at play. The following image shows the previously mentioned cracks present on this one sample, these three cracked sites
were the only sign of damage found throughout the length of the studied surface.

![Figure 4.1: Side view of 10213 after 1.45% strain](image)

As well as in the bending tests, even by increasing the load in small steps (as low as 0.025% strain increase per step) and checking for damage after each step, the samples broke before showing any signs of damage. When cracks were visible, they appeared in the fibers and not in the matrix, these might have been only superficial. The cracks didn’t show any growth after mechanical fatigue tests. Samples broke at strains between 1.41% and 1.47%
4.1.2 3-Point-Bending to failure

Similar to the previous tests, the objective of the 3-point-bending tests was to force crack initiation sites on the samples tested on the previous project to study their behavior and density throughout the surface. The samples subjected to thermal cycling (from batches DS18-10212 and DS18-10214) were cut with that specific test in mind, so their approximate 30x100mm size was not appropriate for the tensile test machine.

Since the current aim of this project was to generate and study the damage, and not necessarily to characterize the samples mechanically, 3-point-bending was an ideal option to generate tension when the previously used tensile tests were not feasible.

Just as it happened with the tensile test samples, between the different steps described in the methodology, the sample didn’t show any initiation of cracks in any of the samples. All samples reached the breaking point without a previously visible initial point of damage, and only showed some matrix cracking once the specimen had failed. It is important to point out, as the methodology section states, that this samples were all observed from one on the edges, without an immediate possibility to check for internal damage at the same time.

There is, however, something important to point out from the failure of these samples. In all of the samples, the failure mode was mainly very pronounced delamination, which was not seen in the tensile test samples.

Figure 4.2: Side view of 10214 samples after failure
The samples were tested at gradually increasing loads (as low as +1N per step) and checked after each step, in order to look for any initiation of cracks before failure. All samples tested broke suddenly, before showing any previous signs of damage. The broken samples show delamination, with a clean separation of layers and almost no damage perpendicular to the layers. Sample broke at loads between 230N and 260N. 10212 samples broke in the compression layer, 10214 samples broke in the tension layer. On samples from 10214, there is a big difference between samples cut in one direction of the plate, and samples cut in the perpendicular direction (2x higher value of E).
4.1.3 CT Scan

In order to further analyze the possible damage within the samples, a part of the broken DS18-10215D sample was subjected to a CT Scan. While the resolution for the scan was not as high as desired, the results did show what could possibly be an interesting result.

Throughout the sample, the 90 layer showed some distributed “spots” which seem too small for conventional voids. In addition to this, the fact that these were present mostly on the 90 layer suggest that it might be some kind of damage. The resulting hypothesis has been the possibility of there being distributed damage in the sample as opposed to the traditionally expected crack initiation sites.

If this hypothesis is confirmed, it could explain the lack of cracks in the samples throughout this study, as well as in thin-ply laminates in general. It could also explain how the progress from crack initiation to sample failure occurs in such a violent manner, if the damage, once generated, can propagate easily through these "spots" along the 90 layer of the sample.

Figure 4.3: CT Scan pictures of DS18-10215D. Vertical Slice

In order to better evaluate this observed "spots", the sample was polished on the surface perpendicular to the strain (the one parallel to the CT images shown) and reviewed
under the microscope.

\textit{Figure 4.4: Surface view of CT scanned image. Surface perpendicular to tension. Standard clean}

As is shown on the image, the sample does show some kind of porosity. However, in order to make sure that there were no misleading results due to dust or dirt, the sample was subjected to cleaning under an ultrasonic bath at room temperature for 10 minutes, with the result being as follows:

\textit{Figure 4.5: Surface view of CT scanned image. Surface perpendicular to tension. Ultrasonic clean}

This porosity seen could correspond to the "spots" present on the CT scan. Were this to be the case, the hypothesis that this is spread damage would not be valid, since some samples have shown these kinds of pores before being subjected to stress of any kind.
4.1.4  Microscope under tension

The main idea behind this experiment was that very small cracks were being initiated within the material, and due to the elastic nature of the material these would close up, with the closed crack being too small to be detected via optical microscopy.

Due to the size of the rig used to hold the sample bent in place, the microscope used for this experience was a different one, not as powerful as the one normally used throughout the rest of this project. Nevertheless, the resolution should be sufficient to observe any damage, which was not the case.

The images below are representative of the whole surface studied, with no clear sign of damage or the hypothesized open crack under tension.

![Figure 4.6: Side view of 10213 sample under tension](image)
4.2 Transverse Modulus and Tensile Strength - 90 Layer

Two of the 10 samples tested failed while adjusting the clamps, which suggests that the tension induced by the grinding and polishing process is high enough to be taken into account in the future.

As for the remaining samples, the average value of $E_2=8.2\text{GPa}$ is well within the standard range of 8-12GPa. However, the values for tensile strength range from 36 to 50 MPa, which as will be shown in the thermal cycling section, is way below the theoretical value of stress induced by the thermal cycling.

These 3 samples broke without much delamination, as opposed to the previously mentioned 3-point-bending samples.
4.3 Fatigue Test

Adding upon the last project’s tests, the samples on this project were tested at higher strains of 0.9% to induce damage. In most of the cases observed, the stress induced, or more likely the low number of cycles, was not enough to generate crack initiation sites on the samples. The following images show representative examples of the surfaces studied, since there was no damage in any of them.

*Figure 4.8: Surface view of 10274/10275/10276/10277 samples after 1000 cycles 0.5% and 0.9% strain*

However, on sample DS19-10272A, as shown on the magnified image below, a small, approximately 10μm crack can be seen near the bottom edge of the 90 layer. Due to this crack being significantly small, and very close to an area that seems to have some manufacturing defect, it is likely that it is not in fact a result of the fatigue tests but some external factors.
Figure 4.9: Surface view of 10277 sample crack after 1000 cycles 0.5% and 0.9% strain
4.4 Thermal Cycling

To implement the formula 1.1 the following table of standard UD CFRP properties was used, due to lack of enough information on the current material.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>(E_L) (GPa)</td>
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</tr>
<tr>
<td>(E_T) (GPa)</td>
<td>10</td>
</tr>
<tr>
<td>(\alpha_L) ((\epsilon/K))</td>
<td>-0.3</td>
</tr>
<tr>
<td>(\alpha_T) ((\epsilon/K))</td>
<td>28</td>
</tr>
<tr>
<td>(T) (K)</td>
<td>77</td>
</tr>
<tr>
<td>(T_g) (K)</td>
<td>393</td>
</tr>
<tr>
<td>(\Delta T) (K)</td>
<td>316</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Batch</th>
<th>(f(N_{90}/N_{90+0}))</th>
<th>(\sigma(Pa))</th>
</tr>
</thead>
<tbody>
<tr>
<td>10272</td>
<td>1.000</td>
<td>71542</td>
</tr>
<tr>
<td>10273</td>
<td>0.375</td>
<td>65112</td>
</tr>
<tr>
<td>10274</td>
<td>0.500</td>
<td>67540</td>
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<tr>
<td>10275</td>
<td>0.500</td>
<td>67540</td>
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<td>10276</td>
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</tr>
<tr>
<td>10277</td>
<td>0.750</td>
<td>70157</td>
</tr>
</tbody>
</table>

\[
\sigma_{th} = \frac{f E_L E_T (\alpha_T - \alpha_L) \Delta T}{(1 - f) E_T + E_L} \quad (4.1)
\]

The average value is approximately -68MPa, which is 51\% higher than the average 45MPa Tensile strength value shown on the previous section. This alone would suggest that the thermal induced stress should be enough to generate some damage on the sample.

However, as can be seen in the following images from the samples after 25 and 100 cycles, there was no sign of crack initiation in any of the samples.

*Figure 4.10: Surface view of 10273/10275/10276/10277 samples after 25 thermal cycles*
Figure 4.11: Surface view of 10273/10275/10276/10277 samples after 100 thermal cycles
4.5 Double Cantilever Beam (DCB) Test

As was the case with the bending test, at seemingly random points during the tests the samples produced a "clinging" sound. This sound has not yet been identified as they appear very scattered throughout the tests.

The values obtained for $G_{IC}$ were calculated according to the previously mentioned standard, with the following equation:

$$G_{IC} = \frac{3P\delta}{2ba}$$  \hspace{1cm} (4.2)

An average value was obtained from only three samples, due to limitations in time. This value averaged at 0.35kJ/m² which is in line with other values obtained for aerospace-grade resins. And while some scattering was present on the results, it can be argued from this initial results that the lack of cracks observed in the samples from the other tests might not be related to the fracture toughness of the matrix material but to the distribution of the thin plies and their effect on the final properties.
This leaves further testing to be done with regards to the thickness threshold from which the thin ply effect is apparent, as well as assessing whether or not there has been some internal spread damage in the mechanical fatigue tests that is not present on this DCB tests.
The main objective of the current project was to assess the limit in stress and ply thickness after which crack initiation sites were generated. Since no crack generation was produced, there can’t be a definitive conclusion in regards to that specific aspect until more information is gathered. However, the results of all these tests have presented some information worth pointing out.

This being still an initial step in the process, the parameters selected for the tests were not very high, in order to make sure that every initial variable was covered. The material has shown clearly high mechanical properties under what would otherwise damage any conventional carbon fiber/epoxy composite.

The main advantage so far observed in relation to the application as material for cryogenic fuel tanks is that in the samples were failure was reached, no previous sign of cracking could be observed. If this is related to cryogenic liquid storage, the lack of cracking reduces, or perhaps eliminates, leakage of the fuel through the walls of the tank. This, of course, in addition to the material being able to withstand severe strain and extreme temperatures.

The thin-ply effect is clearly present in the range of thicknesses evaluated in the project, with the additional possibility of this taking place even when layers oriented in the same direction are layed up, which was initially intended to work as a thicker, unified layer. The samples showed no damage bellow 100 thermal cycles, or under 1000 cycles of 0.5% and 0.9% strain mechanical fatigue. The behavior of the samples on the DCB test showed that the values for G1c are standard, so the resistance of the material is hypothetically only due to the thin ply distribution.

There is, however, a problem with the mechanism through which the material fails under bending stresses, which is hard delamination on nearly every layer of the sample, that extends up to 1/5 of the sample length in some cases. Additionally, the small window between the hypothetical generation of cracks and the complete failure of the samples could provide a problem, with the material not providing any visible information that the object will fail until it has already happened.

One of the key points following this project is the possibility of this material having
a new failure mechanism, where the damage is spread throughout the matrix in the form of small pores that relieve tension evenly, so that it doesn’t concentrate on one specific crack. This, of course, will need to be confirmed by doing an additional CT scan on an untested sample, amongst other tests.

As a closing conclusion, the material could very well be suitable for the intended applications, where the thermal and mechanical stresses it would likely be subjected to would have to be very extreme in order to generate any kind of negative response.
Chapter 6

Further Work

One of the main points of interest for the further development of this project is to look further into the hypothesis of spread damage in the 90 layer, which if proven could mean a new subject of study when it comes to CFRP composites damage mechanisms. To fully consider this possibility the clear main option would be to study the material under CT Scans at higher resolutions. With a clearer image of a strained sample, as well as a scan of an untested sample, which was not possible to do for this project, the nature of the defects observed could be more easily assessed.

Apart from this, more strenuous fatigue tests would provide information to help give an approximate value range to the point of crack initiation, and therefore being able to describe the limitations of the material. Tests for higher number fatigue cycles (≥100,000) and higher number of thermal cycles (≥500) could, according to previous research work, finally show some damage initiation as well.

Some of the aspects of this project which were not able to be assessed remain on the table for any future work on this subject, such as finding the "limit thickness" from which the thin-ply effect stops taking place, to be able to produce material with as high thickness as possible without compromising the mechanical and thermal behavior, therefore reducing production costs. Additionally, the difference in behavior between Woven and UD samples, if any, which was the objective of the comparison between sample batches DS19-10275 and DS19-10276 should be looked further into, as well as the behavior of "thick plies" manufactured as one single ply as opposed to many thin plies together, to avoid thin-ply effect.

Regarding the tests themselves, some additions to the testing and observation procedures could improve efficiency and therefore allow for more thorough research. The use of fluorescent dyes to highlight possible cracks would drastically reduce the observation time since the crack check is done manually on a very homogeneous surface. The use of replicas on the analyzed surface, while not reducing the observation time, could allow for the full testing of the samples to be done without removing it from the equipment after every step. Finally, if the possibility is available, checking the samples under optical microscope while the loading tests are either in progress or under static tension, it could
prove a useful way of checking for previously ignored cracking initiation sites.

One additional point to consider is the constant appearance of a sound assumed to be related to fibre failure while the samples were loaded. This sound could be a point of interest if there is a way to quantify it or relate it to the internal damage of the samples.

Overall, there is still a lot to be done in regards to this project. However, the outline for the future work is fairly clear, and it could be resolved relatively fast.


