Bachelorarbeit

Nadine Tscheu

Macro- and Micro-Study of Carbon-Composite

Fakultät Technik und Informatik

Department Maschinenbau und Produktion

Nadine Tscheu

Macro- and Micro-Study of Carbon-Composite

Bachelorarbeit eingereicht im Rahmen der Bachelorprüfung
im Studiengang Maschinenbau/Entwicklung und Konstruktion am Department Maschinenbau und Produktion der Fakultät Technik und Informatik der Hochschule für Angewandte Wissenschaften Hamburg
in Zusammenarbeit mit:

University of Portsmouth
School of Engineering
Course: BEng Mechanical Engineering

Erstprüfer: Prof. Dr.-Ing. Friedrich Ohlendorf
Zweitprüferin: Dr. Sarinova Simandjuntak

Abgabedatum: 21. Juni 2013

Zusammenfassung

Nadine Tscheu

Thema der Bachelorarbeit
Makroskopische und mikroskopische Untersuchung von Kohlenstoff-Faserverbundwerkstoffen

Stichworte
Faservstärkte Kunststoffe, Rasterelektronenmikroskop, Versagensmechanismen

Kurzsusammenfassung
Faserverstärkte Kunststoffe werden in vielen technischen Anwendungen wegen ihrer excellenten Eigenschaften verwendet.
Diese Arbeit beschäftigt sich mit der Untersuchung der möglichen Schadensmechanismen und den Pamameter, die das Verhalten der Faserverbundstruktur bei unterschiedlichen Temperaturen unter Biegung beeinflussen.
Die Untersuchung und Beurteilung der unbehandelten Materialien behinhaltet Härteprüfung, Differenzkalorimetrie und optische Untersuchung mittels Mikroskop. Das Rasterelektronenmikroskop wir genutzt um die Faser-Matrix-Haftung, Faserausrichtung und Fehlstellen sowie Unregelmäßigkeiten fest zu stellen.
Zwei verschiedene Proben, aus einem Pultrusionsprozess und einem Prepreg-Auflegverfahren, werden auf ihr Versagensverhalten und die auftretenden Schadensmechamismen untersucht. Die Biegung der Proben wird durch Dreipunkt-Biegung verursacht.
Die Analyse zeigt, dass der Herstellungsprozess als auch die Anwendungtemperatur einen Einfluss auf das Materialverhalten und deren Eigenschaften hat.

Nadine Tscheu

Bachelor Thesis title
Macro- and Micro- Study of Carbon-Composite

Keywords
Composite, scanninc electron microscope, failure mechanism

Abstract
Composite materials are used in many technical applications because of their excellent properties.
This study investigates the potential damage mechanisms and the parameters that affect the composite structure at different temperature levels under flexural loading conditions.
The characterisation of the as-received material includes hardness testing, differential scanning calorimety, to determine the glass transition temperature (Tg), and the optical microscopy analysis. The scanning electron microscope is used to identify the fibre-matrix interface, fibre arrangement, and defects / discontinuity in the composite.
Two types of samples made from pultruded and roll-wrapped processes are examined and compared with respect to their failure behaviour and damage mechanism.
Flexural failure is introduced on the composite sample by conducting the three point bending test at different temperatures.
The study indicates that the manufacturing process, as well as the temperature, has an impact on the material behaviour and properties of the composite.

Contents

List of Figures

List of Tables

1 Introduction

The growing population and the increasing need for limited resources such as oil and gas is driving of the offshore industry to operate in deeper water.

Ten years ago, the offshore industry operated around 500m deep, but now they have to go deeper. Some extend up to 2000m and 3000m depth. This creates a specific need for materials to withstand the extreme cold such as Arctic sea and the deep subsea pressures.

Currently, for moderate depth, steel is used for most subsea components such as riser, pipe-lines and jumpers. However, with the demand for greater fatigue and corrosion resistance, alternative materials are being considered.

One of the materials that offers the above properties would be composite. In general, composites offer good strength-to-weight ratio, chemical resistance, wear resistance and ability to perform within extreme temperature ranges.

As a composite, the structure consists of fibre and matrix. The existence of discontinuities or defects between fibre and matrix or between the unidirectional layers reduces the properties and is a starting point for fracture. It is very important to understand the parameters that might affect the integrity of the composite structure, in particular, temperature and humidity. The integrity can relate to mechanical, thermal and tribological behaviours.

Characterising the as-received and fractured composites in order to identify the fibre-matrix interface, matrix morphology, fibre arrangement and defects or discontinuity to define the potential damage mechanisms depending on the influence of temperature and humidity will help to understand these parameters. This can be achieved through macro- and microstructural studies of the thermoset material, for example epoxy with carbon reinforcement.

Therefore, the objective of the project is to study the fibre-matrix interface, in particular how the presence of defects or discontinuity between layers affects the material and its mechanical behaviours, and to identify the potential damage mechanisms of the components.

2. Literature Review

2.1 Composite

[Ref. 1-2]

The materials studied in this project are composite materials. A composite is a combination of two or more materials, usually fibre and matrix made of various materials.

Through mutual interaction of these constituents the composite gains higher effective properties overall than both, the fibre and the matrix alone. The advantage of composite material is the high strength and high stiffness in combination with low weight.

2.1.1 Fibre

[Ref. 2 - 5]

The reinforcing elements of composites are fibres. Fibres could be classified in two aspects, the form and the material.

Related to their form the fibres could be subdivided into continuous fibres, and discontinuous fibres shown in Figure 2.1.1.

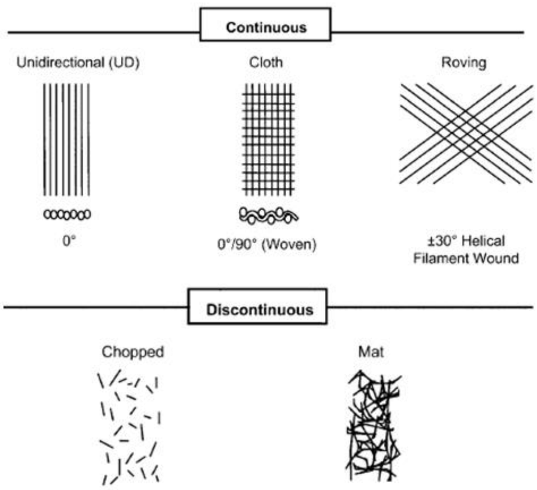


Figure 2.1.1-1 Reinforcement types

The continuous fibres are filamentary materials in a finite length of 100 times its diameter. They are normally orientated in one direction and could be used in unidirectional layer (Chapter 2.4), woven fabrics called cloth, or rowings contain the fibres wrapped with a specifc angle to each other. The discontinuous fibres have a short length and are random orientated. They could be used as chopped fibres and random mat.

Considering the material that fibres could be made of, there are categories such as natural fibres, oxide glass fibres, aramid fibres, carbon and graphite fibres, and metallic fibres.

Schürmann [Ref. 2] explains the superior properties of fibres by four effects:

- The size effect: as the volume of an object decreases so does the potential number of defects. It is related to the weakest link theory where the strength of the whole object is dependent on the weakest link.
- A consequence of the manufacturing process of the fibres is that the atomic bondings are orientated in the direction of the fibre and therefore provide higher properties in the length direction of the fibres with a simultaneous reduction of the properties normal to the length.
- Notches significantly reduce the strength of an object by forming areas of stress concentration. To ensure the absence of notches at the fibre surface a suitable manufacturing process and surface treatment should be chosen.
- The insertion of internal stresses due to the cooling process has a positive influence of the fibre surface.

2.1.2 Matrix

[Ref 1 - 2, 5 - 6]

The matrix is the constituent where the fibres or reinforced materials are embedded. It is a homogenous material made of thermoplastic, or thermoset resin as well as metals, ceramics, or glasses.

The matrix fixes the highly damageable fibre in the required position, structure, and orientation and provides a solid form which can be formed and manufactured.

A further gain is that the matrix binds the fibre together and transfers external load between fibres which reduces the stress concentration in the composite. In case of a failing fibre the load that was previously carried by this fibre is redistributed equally to the fibres close-by.

In addition, the matrix protects the fibre surface from mechanical abrasion and the environment. While the fibres have very good strength in their direction the matrix carries the loads normal to the fibre direction and compression stresses.

2.1.3 Interface

[Ref 1 - 2]

The fibre-matrix adhesion is a value that varies depending on the properties of the composite. It is influenced by both, the fibre and the matrix. The coupling between them should be as good as possible to reach the fibres superior mechanical properties and also transfer the load from fibre to fibre. The mechanism of adhesion contains chemical and physical bounds which forms the interface.

Another aspect is the roughness of the fibre surface which improves the coupling with matrix because a rough surface provides a major area than a flat surface.

It is desirable to achieve an ideal fibre-matrix-interface without defects or discontinuous. Fibres which are not completely covered by the matrix cannot transmit the mechanical load.

2.2 Used constituents

[Ref 1- 2, 4, 7 – 8]

The materials used in this project are epoxy pipes reinforced with carbon fibres.

Carbon fibres have a high performance and useful mechanical and physical properties. The carbon atoms are held together by van der Waals-forces and strong covalent bonds. The fatigue properties of carbon fibre composites are as good as any metal. In addition they can be used in high temperature environments because they do not melt or soften.

Glass fibres consist of silica and oxygen with high covalent bonds. They have an amorphous, structure without a specific orientation. The main properties of glass fibres are their excellent strength, and low humidity absorption. The disadvantage of glass fibres are a low Young's modulus, and extremely brittle breaking behaviour without any elastic or plastic deformation.

The matrix is made of a thermoset material. Thermosets are irreversible chemical highly cross-linked polymers. The high cross-linking reduces the creep ability and increases the dimensional stability at high temperatures.

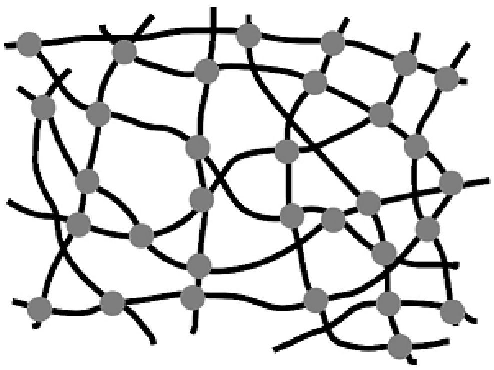


Figure 2.2- 1 Thermoset structure

The samples are made of an epoxy resin which is an organic liquid resin containing one or more epoxide groups. These groups are rings made of one oxygen atom and two carbon atoms.

Epoxy resins have good properties such as low shrinkage, good coupling with nearly every material, high chemical resistance, good deformation resistance at extreme temperatures, and good electrical properties. The viscosity of epoxy resins can vary depending on the softening agent and fillers. The curing process can be influenced by the curing agent that determines the speed and the temperature of the curing process.

2.3 Process of manufacture

[Ref 1 - 2, 9]

The manufacture process of composite parts is different to manufacturing of metal parts. In composite manufacturing the material and the component are usual manufactured at the same time. This forms the final shape in one manufacturing process without later machining.

There are three main manufacturing processes. The first is the manufacture with polymer pellets which contains short or chopped fibres. The second one is about thermoplastic impregnated fibres. And the third one, which is relevant for this project, is the manufacturing process where fibres are impregnated with thermoset resin.

Because of the low viscosity of thermoset resin the impregnation of aligned or woven continuous fibres is easy compared to thermoplastic resin with its high viscosity. Impregnation means the saturation of a fibre network with the matrix resin.

Thermosets are manufactured in a two-stage process. The first on is the position of not cross-linked polymer into a final shape and the second is the cross-linking reaction driven by heat catalysts or UV radiators.

After the thermoset resin is cured, it is irreversibly cross-linked in a three dimensional structure which makes it impractical to change the form of the composite or remelt the material.

In general the process of manufacturing composite materials is highly dependent on the applied pressure and temperature during the process and the process itself.

2.3.1 Pregpreg

[Ref 1, 3, 7]

Prepregs are semi-finished products made of continuous fibres aligned unidirectional and saturated with a certain amount of semi-solid matrix resin. As a semi-finished product they are uncured and ready to use for the subsequent process.

Prepregs could be divided into three types:

- Unidirectional prepreg tape with a tape that hold the fibres in their position.
- Woven prepreg which contains woven fibres arranged in different directions.
- Prepreg tows consist of preimpregnated fibres in the shape of a bundle as the fibres themselves.

2.3.2 Roll-wrapping

[Ref 7, 9 – 11]

In the roll-wrapping process unidirectional prepreg tapes are wrapped around a mandrel in one or more directions. The directions of the laid-up layers are characterised by declaring the angle between the layers. While 0° indicates fibres in the main direction and 90° a layer with fibres rotated in 90° to the main direction.

After the laying-up, a heat shrink tape is wrapped around the prepregs and the pipe is cured in an oven for a defined time. While the curing processes the shrink tape shrinks and at the same time the mandrel tries to expand due to high temperatures in the oven. The expansion of the mandrel and the shrinkage of the tape induce a pressure to the pipe surfaces. This pressure is necessary for a good fibre matrix coupling and prevents the formation of defects.

Figure 2.3.2-1 Roll-wrapped pipe

The ribbed surface of the pipe visible in figure 2.3.2-1 is cause by the shrinking behaviour of the shrink tape.

2.3.3 Pultrusion

[Ref 3, 7, 9 – 11]

Pultrusion is the only manufacturing process with a continuous length of a composite.

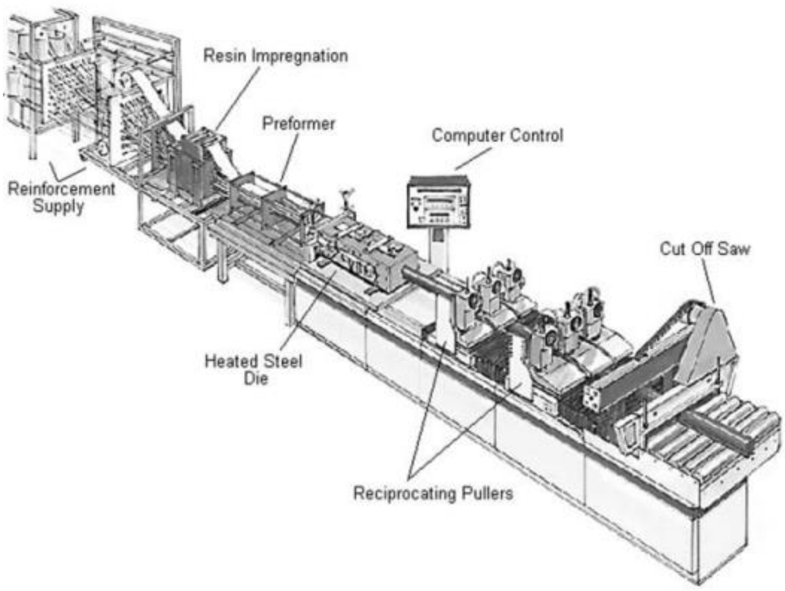


Figure 2.3.3- 1 The pultrusion process

The process is shown in figure 2.3.3-1 where the fibres come from the reinforcement supplier and are pulled into the resin impregnation wet bath. After the impregnation the desired shape is formed and the following heated die presses the fibres together and cures the resin. Behind the die is a pulling system which pulls the endless composite with a defined force or speed. Finally the composite parts are cut into the desired length at the sawing system.

Figure 2.3.3-2 Pultruded pipe

The pultruded pipe presented in figure 2.3.3-2 contains fibres that are all orientated parallel and longitudinal to the length of the pipe. That provides very good properties in the direction of the length and poor resistance to torsional or brushing loads.

2.4 Unidirectional fibre composite

[Ref. 1, 12]

Unidirectional fibre composite (UDC) is a thin walled layer without thickness which is used for better understanding of composite behaviour.

One layer contains parallel aligned continuous fibres embedded in a matrix. The fibres are circular and equally distributed over the cross-section. There is an ideal bonding of fibre and matrix which means that there are no movements under load. Caused by the alignment of the fibre the UDC-layer is clearly anisotropic more precisely transversely isotropic. Anisotropic means the properties such as Young's modulus, strength, stiffness, thermal behaviour are orientation dependent.

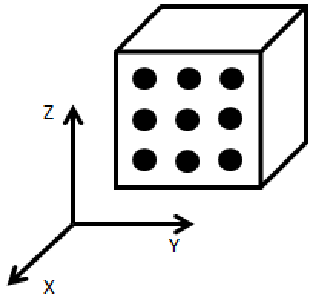


Figure 2.4-1 transversely isotropic X ≠(Y=Z)

A transversely isotropic material is shown in Figure 2.4-1 where the properties in the x direction are different to the y and z directions but y and z have the same properties. The opposite of anisotropic is isotropic where all properties are the same in all directions.

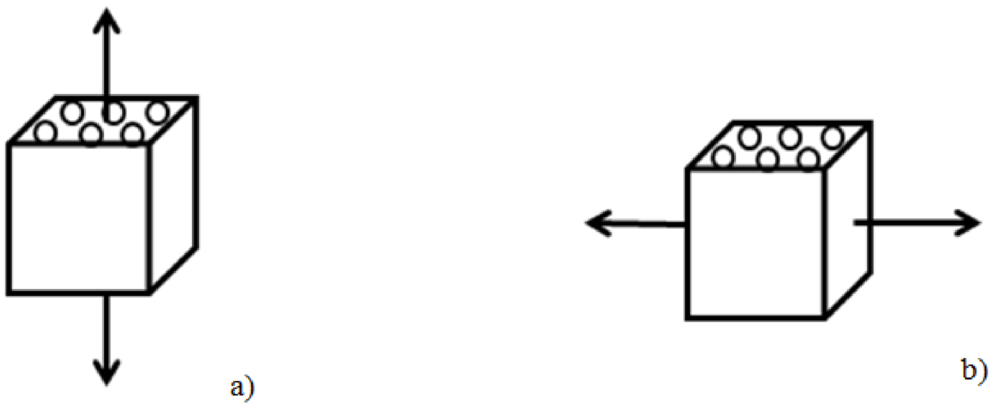


Figure 2.4-2 Potential loading case of anisotropy materials
a) Load in fibre direction
b) Load normal to the fibre direction

Figure 2.4-2 a) shows the load applied in the fibre direction. The matrix is transferring the load and stress form fibre to fibre with only small deformation of the matrix while the fibres are carrying the load.

Figure 2.4-2 b) shows the load applied normal to the fibre direction. Here the matrix has to carry the load and the fibre moves within the deforming matrix.

As the fibres usually have their higher properties in the direction of their length and can withstand higher load and stress in this direction it is desirable to apply load in fibre direction.

The following equations point out that the properties of a UDC-layer depend on the percentage content of fibre and matrix in a composite:

Tensile strength in fibre direction shown in figure 2.4-2 a) produces an equal strain of fibre and matrix called ε_1

$$\varepsilon_{f1} = \varepsilon_{m1} = \varepsilon_1$$

In this equation the index 1 is the load applied in fibre direction, f is the fibre, and m is the matrix.

The stress is shared between the fibre and the matrix related to their volume percentage

$$\sigma_1 = V_f \cdot \sigma_{f1} + \left(1 - V_f\right) \cdot \sigma_{m1}$$

The Hook's law says that stress is equal to the Young's modulus times the strain. Inserting this into the equation and canceling the strain gives the following equation:

$$E_1 = V_f \cdot E_f + (1 - V_f) \cdot E_m$$

The Young's modulus of the fibre is up to two times higher than the Young's modulus of the matrix, so the equation can be simplified

$$E_1 = V_f \cdot E_f$$

The final equation shows that the Young's modulus in fibre direction is only dependent on the volume of the fibre and its Young's modulus.

Is the load applied normal to the fibre direction illustrated in 2.4.2 b) it is assumed that the stresses of the fibre and matrix are the same.

$$\sigma_{f2} = \sigma_{m2} = \sigma_2$$

The strain is shared between the fibre and the matrix related to their volume percentage

$$\varepsilon_2 = V_f \cdot \varepsilon_{f2} + \left(1 - V_f\right) \cdot \varepsilon_{m2}$$

Insert the Hook's law and cancel the stress out the equation for the Young's modulus becomes

$$E_2 = \frac{E_f \cdot E_m}{V_f \cdot E_m + (1 - V_f) \cdot E_f}$$

As in the first calculation the influence of the Young's modulus of the matrix related to its volume could be neglected and the equation could be simplified.

$$E_2 = \frac{E_m}{(1 - V_f)}$$

This shows that the Young's modulus normal to the fibre direction is dependent on the Young's modulus of the matrix. The fibre only influences the deformation of the matrix.

Figure 2.4-3 shows the Young's modulus of both kind of load application related to the volume percentage of the fibre.

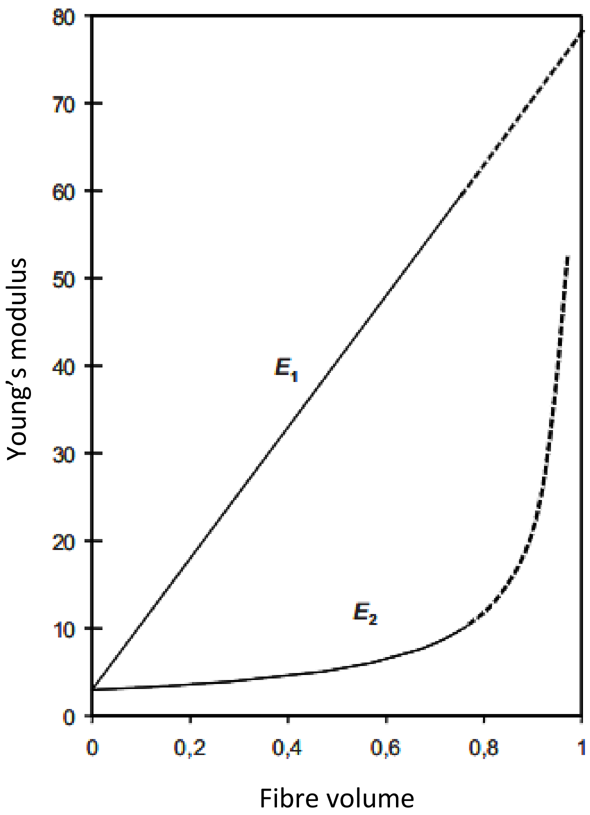


Figure 2.4-3 Young's modulus dependent on the fibre volume:
E1 for load in fibre direction
E2 for load normal to the fibre direction

It is clearly visible that the Young's modulus of the fibre in fibre direction is linear increasing with the content of fibres in the composite. The Young's modulus normal to the fibre direction is insignificantly increasing with the fibre content up to 50% fibres and very low compared to the Young's modulus in fibre direction. Considering the fibre direction an anisotropic material has different properties in fibre direction than in the direction normal to the fibres.

2.5 Laminate theory of composite

[Ref.1, 13 – 14]

Unidirectional fibre composite materials are transversely isotropic which means very good properties in the direction of the reinforcing fibre and inferior properties transverse to the fibres.

A compromise with good properties in both directions is a multilayer made of UDC-layer with different fibre orientation. Because this multilayer with different fibre directions does not provide the same properties a calculation method is needed to determine the properties of the multilayer.

A widely recognised and appropriate method is the laminate theory of composite materials.

To apply this theory some assumptions need to be done:

The calculated multilayer consists of unidirectional layer with transversely isotropic behaviour and the thickness of the layers is much smaller than the in-plane dimensions which means plane stress state.

To achieve an easier understanding of the laminate theory for anisotropic material it is explained first for isotropic materials (figure 2.5-1).

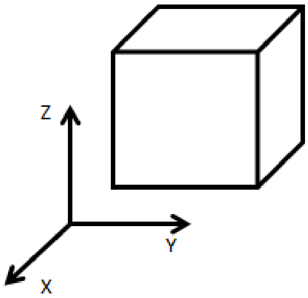


Figure 2.5-1 Isotropic model

For thin plates plane stress is assumed and therefore deformation and stress x- and y- direction and their shear stress are detected.

Written in matrix notation it gives the following equation:

$$\begin{Bmatrix} \varepsilon_x \\ \varepsilon_y \\ \gamma_{xy} \end{Bmatrix} = \begin{bmatrix} \frac{1}{E} & \frac{-\nu}{E} & 0 \\ \frac{-\nu}{E} & \frac{1}{E} & 0 \\ 0 & 0 & \frac{1}{G} \end{bmatrix} \begin{Bmatrix} \sigma_x \\ \sigma_y \\ \tau_{xy} \end{Bmatrix}$$

With $G = \frac{E}{2(1+\nu)}$

The compliance matrix is a symmetric 3x3 matrix which means that the material properties are the same in x- and y- direction.

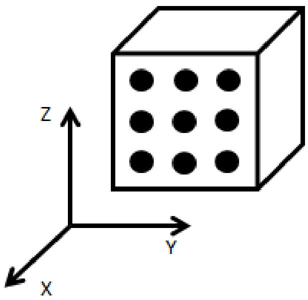


Figure 2.5-2 Transversely isotropic model

The laminate theory for transversely isotropic materials shown in figure 2.5-2 is also applied for thin plates with plane stress. But as the material is transversely isotropic the material behaviour in x- and y/z-direction is not the same.

Written in matrix notation it gives the following equation:

$$\begin{Bmatrix} \varepsilon_x \\ \varepsilon_y \\ \gamma_{xy} \end{Bmatrix} = \begin{bmatrix} \frac{1}{E_x} & \frac{-\nu_{yx}}{E_y} & 0 \\ \frac{-\nu_{xy}}{E_x} & \frac{1}{E_y} & 0 \\ 0 & 0 & \frac{1}{G_{xy}} \end{bmatrix} \begin{Bmatrix} \sigma_x \\ \sigma_y \\ \tau_{xy} \end{Bmatrix}$$

with

ν_{xy} - Ratio of strain induces the y-direction by a strain applied in the x-direction

ν_{yx} - Ratio of strain induces the x-direction by a strain applied in the y-direction

The 3x3 matrix for transversely isotropic material is not symmetric because the material behaviour is dependent on the properties in different axis-directions.

With respect to the layer direction the properties of the multilayer in x- and y- direction the composite is treated as a combination of single layers with different properties and can be calculated by assembling the compliance matrices for the single layer.

2.6 Failure analysis

[Ref. 1, 15-17]

Failure analysis is a systematic examination and documentation to detect and analyse the cause and consequences of existing failure. Therefore it is important to understand the different aspects of a failure analysis. The first thing to do is to identify indicators of failure and the area where the failure occurs which should be analysed and documented. To explain the existing failure and interpret the indicators different damage mechanism and failure modes of composite material must be understood and applied.

Another aspect of failure analysis is the identification of the cause which means to formulate a reason why failure had appeared.

The next step is to compare the existing failure with known damage mechanisms form textbooks and ascertain if they are consistent and comprehensive.

2.6.1 Damage mechanism

[Ref 1- 6, 16]

A damage mechanism describes the way a component fails on the microscale level. The microscale level is related to the material behaviour at the matrix-fibre level which means that this approach respect both matrix and fibre as a part of the material.

The other approach is on the marcoscale level where the composite is tread as a single layer continuum.

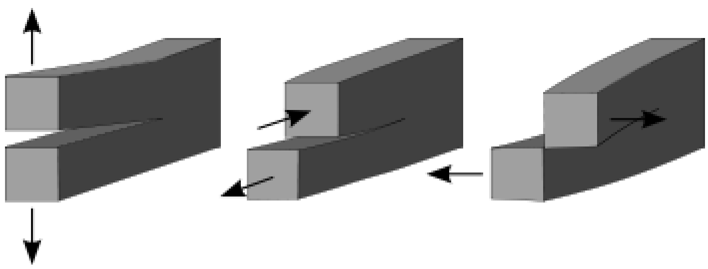


Figure 2.6.1- 1 Basic failure modes: opening mode, in plane-shear mode, and tearing mode

Failure mechanisms lead to failure modes shown in figure 2.6.1-1. These three failure modes could appear separately or simultaneous as a combination that results in complex fracture even under simple loading conditions.

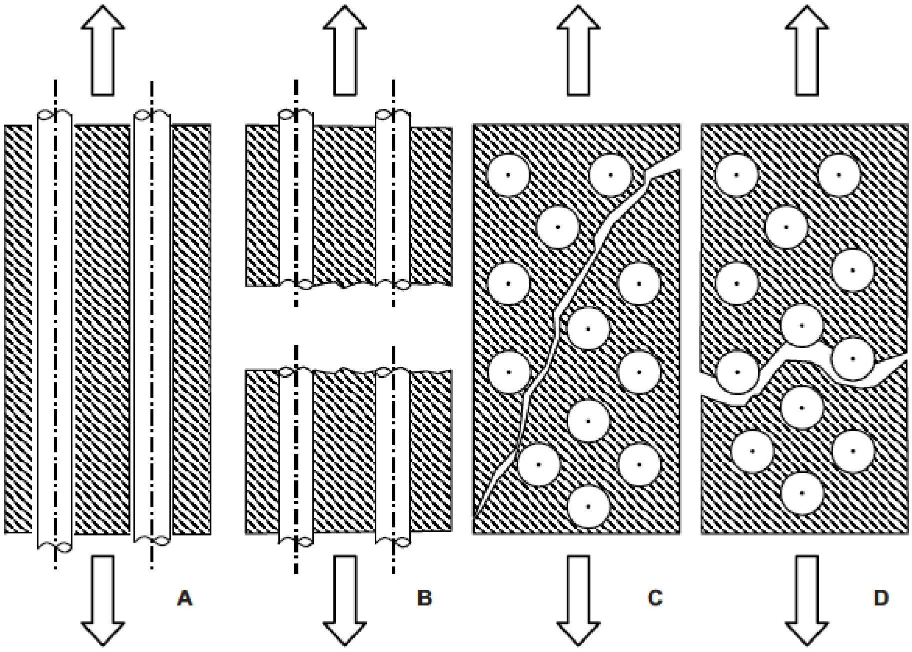


Figure 2.6.1-2 Potential damage mechanisms: A) Delamination, B) Fibre cracking, C) Matrix cracking, D) Cracking at the fibre-matrix- interface

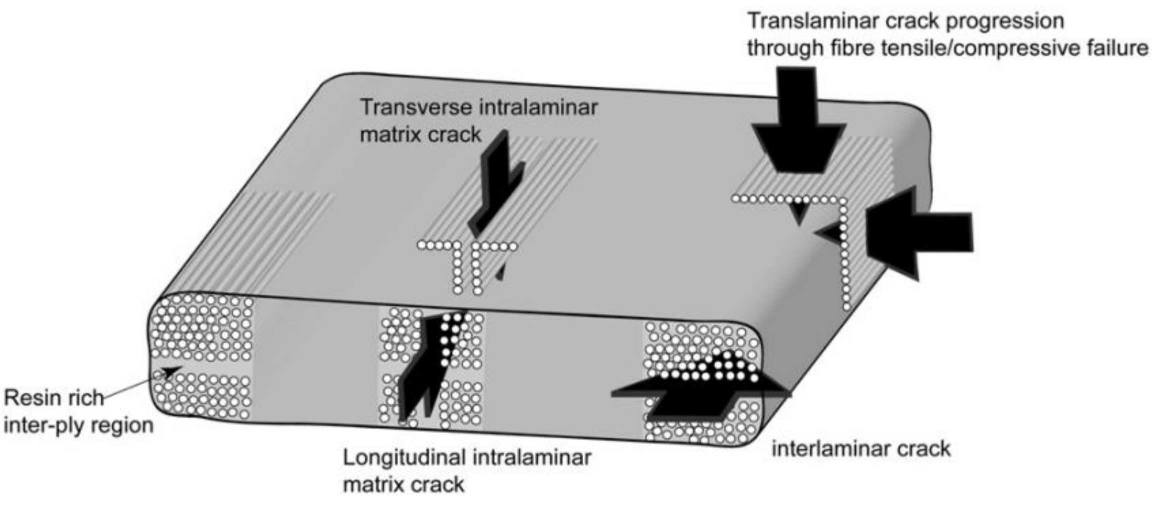


Figure 2.6.1-3 Potential damage mechanisms and their directions

Figure 2.6.1-2 and 2.6.1-3 present potential damage mechanisms which are explained in the following sections.

2.6.1.1 Fibre cracking

[Ref. 1-2, 15, 17-18]

Fibre cracking shown in figure 2.6.1-2 B is a break of many fibres at the same time which results in a degradation of the composite properties.

Figure 2.6.1-3 point out two kinds of fibre cracking of a composite shown in the right corner. Fibre cracking is always translaminar and depends if the applied load is tension or compression and in which direction the load was applied that forms the onset of fibre failure that could appear at different areas of the component.

2.6.1.2 Fibre pull-out

[Ref.1, 18-19]

Fibre pull out is a damage mechanism where fibres are pulled out of intact matrix because the interface between fibre and matrix is not bounded strong enough and separates under loading. As the applied load is not distributed over all fibres the single fibres cannot withstand the load anymore and fail. The breaking of single fibres does not have to appear at the same area where other fibres break.

2.6.1.3 Matrix cracking

[Ref. 1-2, 15]

Matrix cracking often occurs when the load is applied normal to the fibre and have to be carried by the matrix. Matrix failure is more common than fibre failure because the strength properties of the matrix are lower than of the fibres. Cracks form between the fibres shown in figure 2.6.1-2 C and 2.6.1-3. Matrix cracks could appear transverse or longitudinal intralaminar as well as interlaminar.

2.6.1.4 Delamination

[Ref 1-4, 12, 15, 18]

Delamination is the most critical failure and occurs in composite materials with many layers. It is a separation along the fibre-matrix interfaces of layers and leads to interlaminar stresses. Interlaminar stresses are tensile stresses normal to layers and shear stresses between layers.

Delamination reduces the stiffness of a composite and therefore the life of a component. The onset of delamination is caused by different factors such as free edge effects, structural discontinuous, manufacture errors, moisture and temperature variations, and internal failure mechanisms such as matrix failure.

Free edge interlaminar stresses are caused by different orientation of adjacent anisotropic plies. Moisture and temperature also induces internal stresses as the plies extend in different directions related to their orientation.

Three failure modes (figure 2.6.1-1): opening mode (I), in plane-shear mode (II) and tearing mode (III) are used to describe delamination of composite because every delamination failure is one or a combination of these modes.

2.6.2 Factors supporting the onset of failure

[Ref. 1-5, 12, 21]

Failure could occur due improper fibre or matrix, manufacturing defects, unexpected loads, environmental exposure, and in-service damage.

Manufacturing defects could be for example matrix resin which is not mixed or cured the way it should be. Over cured resin is brittle and tends to crack in contrast to under cured resin which could be not hard or strong enough to withstand and transfer the applied load.

A further manufacturing defect is improper placement of fibres. If the fibres are not separated and bounded together or they are placed in the resin with different distances to each other the strength is reduced.

Especially the manufacture of cylindrical pipes includes errors. If the resin and the fibres are not distributed equally over the cross section the strength is not equal and therefore the distribution of stresses is unequal.

Another important factor that could cause failure is the geometry of the component. For cylindrical components the manufacturing process and positioning of fibre into the matrix have an influence on the material properties. It can happen that fibres are not distributed completely equal over the whole cross-section. Due to this there might be areas with more reinforcing fibres and areas with more matrix resin. An improper fibre distribution relates to an imbalance of the strength properties over the cross-section which leads to a potential damage when external load is applied.

In addition voids, areas without material and entrapped water or air, can create stress concentration and reduce the strength. They could form during mixing the resin when air bubbles develop and were not removed before curing. They also occur during the curing process when moisture resin absorbs water under pressure and temperature influence. Another source is air or water which is entrapped during the lay-up process of prepregs. The existence of voids depends often on the thickness of the component. If the thickness is high there might be not enough pressure in deeper layer during curing. Figure 2.6.2-1 presents voids in deeper layer.

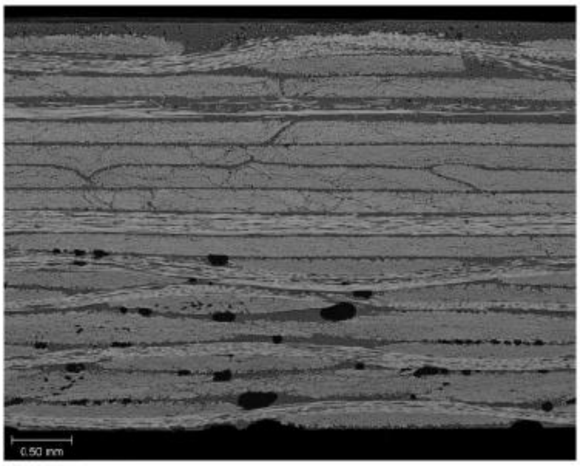


Figure 2.6.2-1 Voids at the bottom of the composite

Components with prepreg plies contain often areas where air is entrapped at ply-drops shown in figure 2.6.2-2. The edge of a ply-drop is beneficial for voids because of lower pressure at this area.

Figure 2.6.2- 2 Voids in the interlayer region at the ply-drop

2.6.3 Factors of environment

Factors of environment are factors influencing the material properties of a component. To prevent failure or even improve these properties these factors and the way they influence the material behaviour should be known. Two of these factors are temperature and moisture.

2.6.3.1 Temperature

[Ref. 1-2, 22]

Many materials change their behaviour at different temperature levels. The normal testing temperature is ambient temperature at 20°C. When the temperature increase the stiffness und strength of polymers decrease and the ability of deformation increase. At lower temperatures the polymer becomes more brittle and more sensitive to impact.

For composite a temperature influence is more an influence on matrix than fibre, because the fibres behaviour is not so much related to the temperature compared to the matrix.

At a higher temperature creep and relaxation processes becomes faster and internal stresses decrease and reduces stress concentration.

After curing a thermoset material it have no thermal elastic or thermal plastic region. By increasing the temperature thermoset changes from solid to thermal decomposition. This behaviour is cause by closed meshed cross-linked chains after curing.

2.6.3.2 Moisture

[Ref. 1-2]

Many materials are also sensitive to moisture that gets into the material through diffusion processes. Moisture influence has a bigger impact on the matrix than the fibres. Moisture can appear in a composite between molecular chains of the polymer resin or in voids, micro flaws and along fibre-matrix-interfaces. This leads to a chance of properties when the matrix is swelling and absorbs water. A positive properties change is that water molecules reduces the stiffness of the polymer and makes it more ductile which also leads to better relaxation behaviour.

But there are also disadvantages of the water absorption such as reduction of the glass transition temperature and decreasing strength properties due to reduced stiffness.

3 Experimental Methodology

[Ref. 1, 11]

The manufacturer (Easycomposite, UK) stated the pipe properties as listed in Table 3-1. There is no information given about the volume fraction of the fibres or the glass transition temperature.

Table 3- 1 Material data provided by the manufacturer

	Pultruded pipe	**Roll wrapped pipe**
Outer diameter [mm]	10	10
Inner diameter [mm]	8	8
Compressive strength in length direction [MPa]	200-320	570
Young's modulus in length direction [GPa]	10-20	70
Flexural strength in length direction [MPa]	250-400	----

Samples are cut and tested at ambient temperature and higher temperature by using the three point bending test.

For understanding composite failure behaviour, fracture analysis using SEM is performed before and after mechanical testing to identify the fibre and the resin, the size, type, number, location, and orientation of all plies as well as cracks, their origin, their direction and their failure mode.

The documentation of this investigation is necessary to analyse the failure and the cause of failure related to the damage mechanism.

3.1 Experimental sequences and sample preparation

[Ref. 1, 21]

Before the samples were analysed the following sequences are done to provide a testing environment which allows reproducing the test and get comparable results.

- The testing samples were cut out of the pipes in a length of 130mm
- The inner and outer diameter and the thickness of each sample were measured with a slide gauge
- The samples were numbered for identification
- The material information provided by the manufacturer were registered for later comparison with the testing results
- Photos were taken of the samples before and after the tests

For the verification of the fibre orientation, the volume of the fibre and potential defects it is important to determine how to section the samples to achieve the best results.

For cylindrical samples three sections are needed. The first one A is normal to the radial directions, B normal to the axial direction and C is normal to the circumference shown in figure.

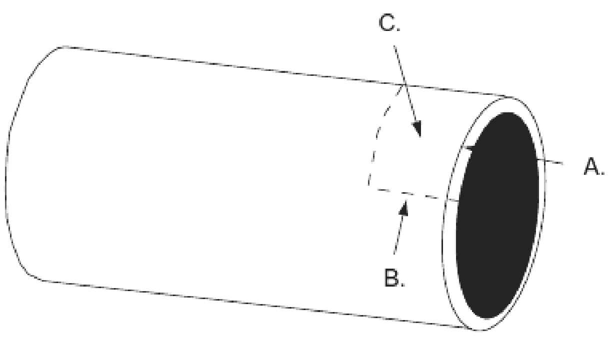


Figure 3.1-1 Sections of a cylindrical sample

3.2 Characterisation of the as-received material

For the characterisation of the as received material two different methods are utilised to achieve a better understanding of the material properties und behaviour.

3.2.1 Differential scanning calorimetry

[Ref. 1, 4, 17]

The glass transition temperature (Tg) is the critical temperature at which a non-crystalline material changes from solid with a brittle behaviour to rubbery with a more elastic behaviour.

To compare the material behaviour at ambient temperature and at 60% of its glass transition temperature differential scanning calorimetry (DSC) method is used to find the exact glass transition temperature of the sample as the manufacturer provided no further details about the used epoxy resin.

DSC is a thermal analysis method where a sample and a reference are heated up at the same program to detect a temperature range where the sample undergoes a thermal reaction and requires or releases heat.

To analyse thermal properties of the pipe material these sample must be prepared first. Therefore a powder of their polymer matrix must be produced which should contain as less fibres as possible. This powder is placed in small metal capsule and laid with a reference capsule into the machine. After the machine is cooled down and the two capsules placed the temperature is slowly increased to a defined temperature.

By reaching Tg the temperature of the sample differ from the temperature of the reference and the machine detects an endotherm or exothermic reaction. These temperature differences are shown in a diagram with the heat flow against the temperature.

The experimental procedure is done by the following steps:

- Define the aim: Comparing the two materials related to their glass transition temperature
- Preparation and documentation of the samples for the testing
- Guess the range of possible glass transition temperature related to textbooks. Afterwards determination of the start and end temperature and the increase in temperature
- Preparation of the testing machine: cooling down to start temperature, positioning of the capsule which contains the sample
- Start testing
- Documentation of the test results, understanding of the curve behaviour and determination of the glass transition temperature

3.2.2 Optical microscope

[Ref. 1, 21]

For the study with an optical microscope light with a wave-length between 350 and 780nm is necessary. Wave-length is the factor which determines the maximal resolution for an optical microscope.

Reflected light produces a picture of the surface while the contrast of different constituents is caused due to different reflectivity of the constituents.

3.2.3 Scanning electron microscope

[Ref. 1, 21]

The scanning electron microscope (SEM) is used to examine the microstructure of materials with a high magnification. SEM uses a thin focused electron beam which scans the surface of the sample.

To see the reflection of backscattering and emission electrons that produce a picture of the surface topography the samples must be coated with gold under vacuum.

3.3 Mechanical testing

Mechanical testing is used to generate a fracture of the sample which offers information about the properties and fracture behaviour of a component.

3.3.1 Hardness test Rockwell

[Ref. 4]

To determine the hardness of a solid material Rockwell could be used.

After applying a preload the specimen undergoes the total test load which is removed after a short time. The remaining depth of the impression is transformed into Rockwell hardness and gives information about the hardness of a specimen which is determined as mechanical resistance against another testing specimen.

Rockwell hardness test provide different test specimens for soft and hard materials. In case of testing a soft polymer material a hard metal ball with a diameter of 1/8” is used.

3.3.2 Tensile testing

[Ref. 1, 5, 20-22, 24]

The common aim of a tensile test is to figure out the mechanical properties under homogenous and uniaxial tensile stress.

The experimental procedure of the tensile test follows these steps:

- Define the aim: Comparing two different kinds of tubes at different temperatures
- Preparation of the testing machine: grip the samples at the right place and length
- Input data of the testing parameters such as the material, geometry, and testing speed

The tensile strength results from the maximum force over the starting cross-section of the specimen. The starting length related to the change of the length after break is the breaking elongation. The Young's modulus is stress divided by strain for a linear part of the stress-strain curve.

For composite which are anisotropic the Young's modulus and the Poisson's ratio are related to the fibre orientation. For a laminate with different layer orientations the laminate theory is applied shown in chapter 2.6.

As the pipe slipped in the grip when the load was applied tensile testing is not suitable to generate a fracture. Slipping in the grip leads to high material strength and the way to try to grip a tube with a plain surface. This induces only a line contact between the grip and the tube.

Generating a fracture of the pipe three point bending is used to have a testing method with simple handling and gripping.

3.3.3 Three point bending test

[Ref. 1, 5, 23-25]

Bending tests are short term uniaxial flexural testing methods and are used in particular for brittle materials. The most common is three point bending test where the sample is places on two supports in a universal testing machine and a load is applied in the middle of the sample shown in figure 3.3.3-1 and figure 3.3.3-2.

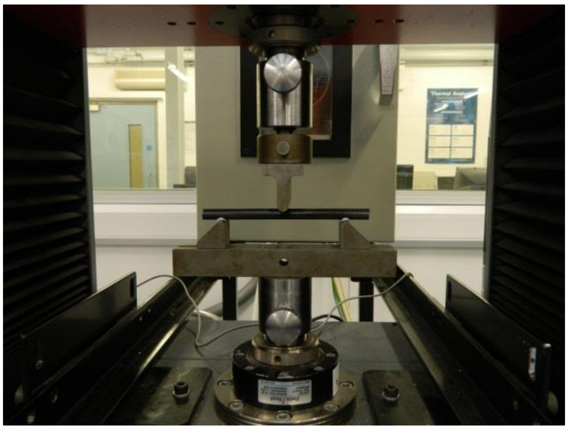


Figure 3.3.3- 1 Three point bending test machine

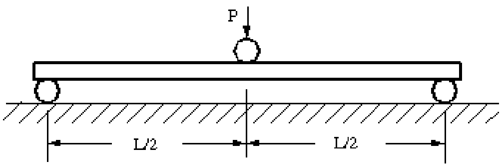


Figure 3.3.3- 2 Schematic representation of thee point bending

Three point bending induces compression at a section where the load is applied and tension on the other side of the specimen. Shear stresses could be neglected because of a long distance between the supports compared to the thickness of the sample.

Figure 3.3.3-3 provides a stress-distribution for a solid specimen with compression on the upper area and tension on the lower. These areas are bisected by the neutral axis.

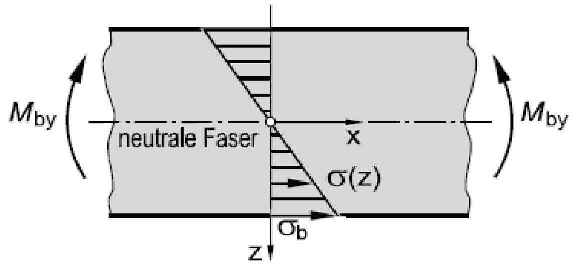


Figure 3.3.3- 3 Neutral axis

The experimental procedure of the three point bending test follows these steps:

- Define the aim: Comparing two different kinds of tubes at different temperatures
- Preparation of the testing machine: adjusting the supports that the load is exactly applied in the middle of the specimen, definition of the testing speed, positioning of the tube and for the higher temperature it is necessary to heat up the oven to the desired temperature
- Input data of the testing parameters such as the material, geometry, and testing speed
- Start the test
- Receive the recorded test results from the testing machine for analysing

A static test is performed at constant speed of 2mm/min and it was stopped before the sample breaks completely.

Advantages of the three point bending test are that there are no gripping, buckling, or end-tapping problems.

4 Results

4.1 Differential scanning calorimetry analysis

Thermal analysis of the two epoxy pipes is done twice to ascertain the glass transition temperature. The glass transition temperature of the pultruded sample is 139° and 121°C for the roll-wrapped sample as shown in figure 4.1-1 and 4.1-2.

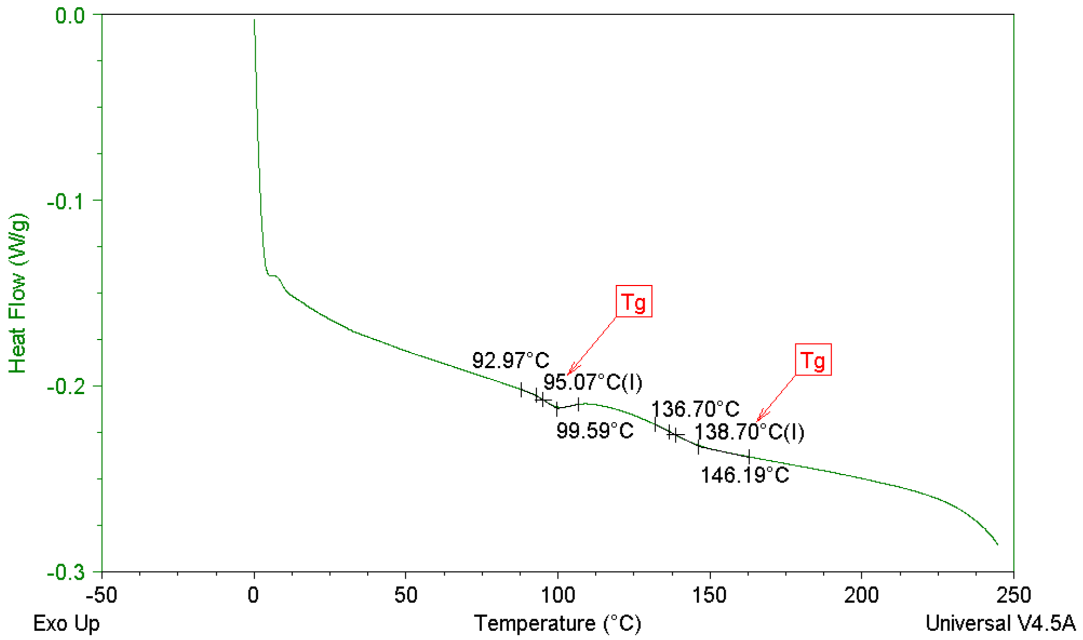


Figure 4.1-1 DSC analysis of the pultruded sample

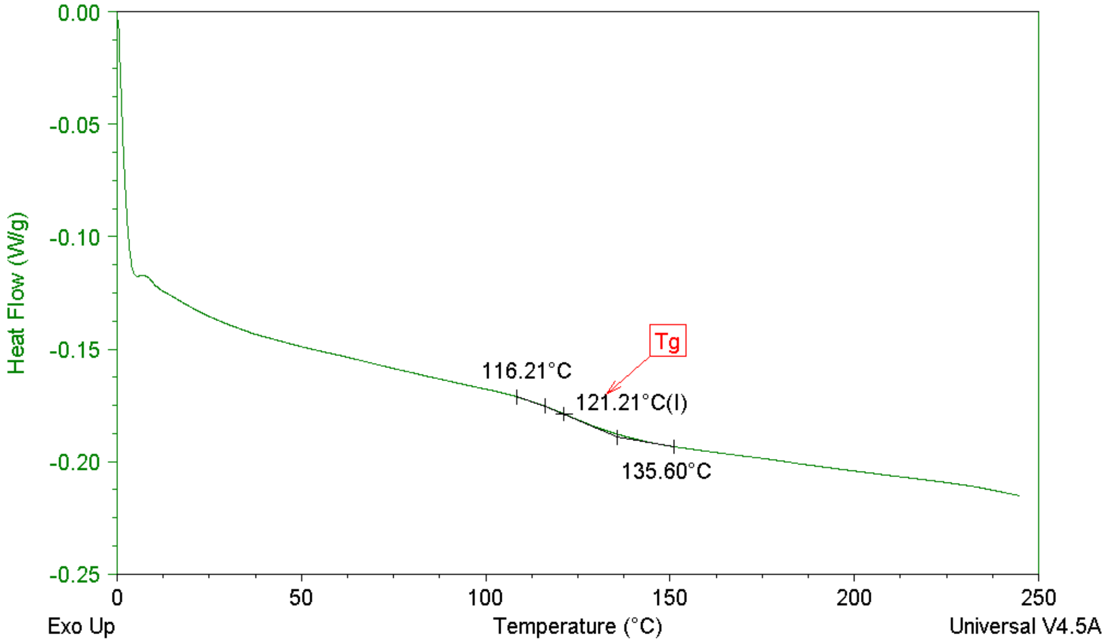


Figure 4.1- 2 DSC analysis of the roll-wrapped sample

4.2 Visual investigation

Pre-test observations were done of the as-received material before testing to identify the fibre matrix interface, fibre arrangement, and find any defects due the manufacturing process.

For the post test examination, photographs were taken of the damaged samples to compare them in order to define the fracture behaviour of different pipes. Afterwards the damaged pipes were examined using SEM to achieve a better insight of the fibre alignment, the crack direction, and the matrix and fibre morphology.

4.2.1 Photographs and optical microscopy analysis

Pre-test observations

Photographs taken with a camera and an optical microscope before the testing are presented for the pultruded pipe in figure 4.2.1-1 and for the roll-wrapped pipe in figure 4.2.1-2.

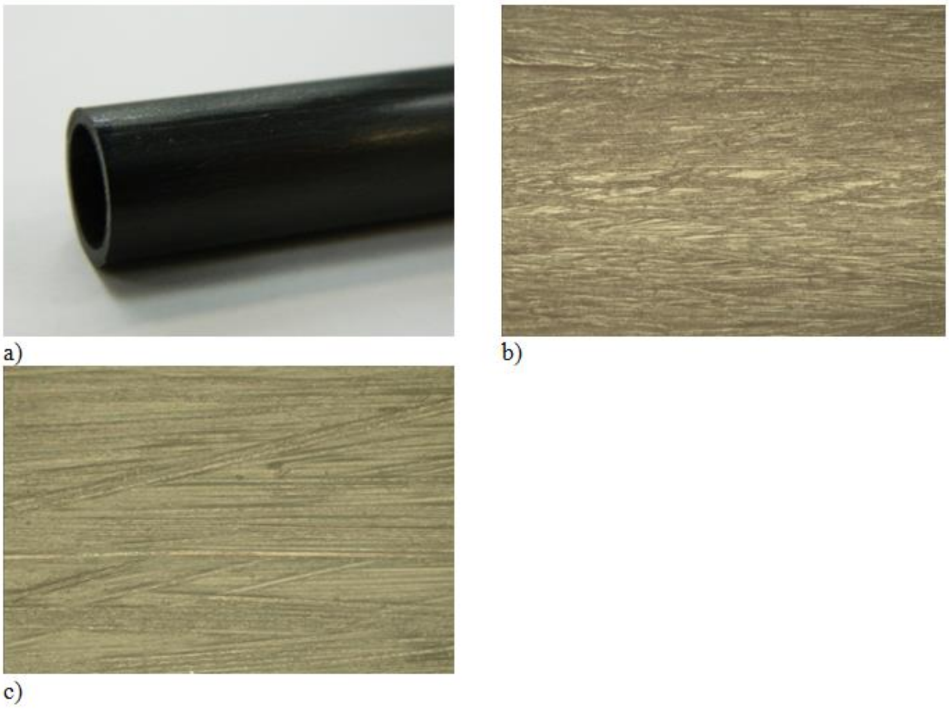


Figure 4.2.1- 1 As-received pultruded pipe a) Photograph
b) Microscopic photograph at x5 magnification
c) Microscopic photograph at x20 magnification

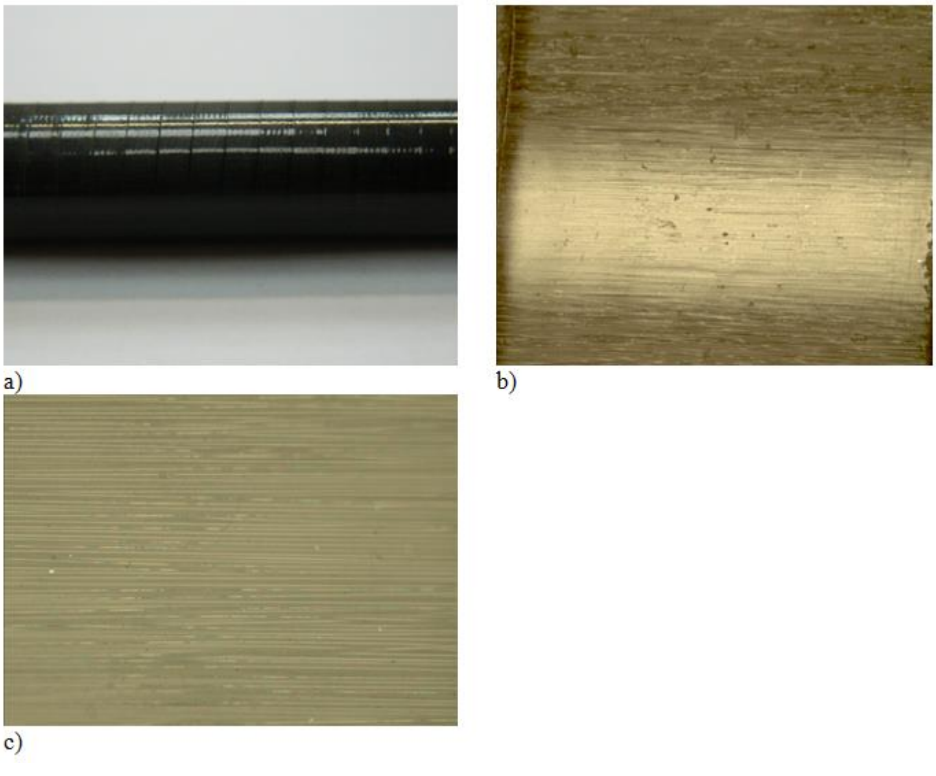


Figure 4.2.1- 2 As-received roll-wrapped pipe: a) Photograph
b) Microscopic photograph at x5 magnification
c) Microscopic photograph at x20 magnification

Comparing the fibre direction in a composite, figure 4.2.1-1 and 4.2.1-2 approve it being lengthways of the pipe for both manufacturing processes.

To examine the actual fibre alignment for the roll-wrapped pipe figure 4.2.1-2 c) present the fibres as ideal parallel with the same distance between the fibres while the pultruded pipe contains fibres aligned disordered over the circumference without any specific distance between fibres shown in figure 4.2.1-1 c).

Another noticeable fact is that the surface of the pultruded pipe looks dull and rough while the roll-wrapped surface is smooth and shiny.

Post-test examination

Photographs presented in figure 4.2.1-3 are taken of the damaged pipes to compare their fracture behaviour and crack direction related to different manufacture processes at ambient temperature and 60% of Tg.

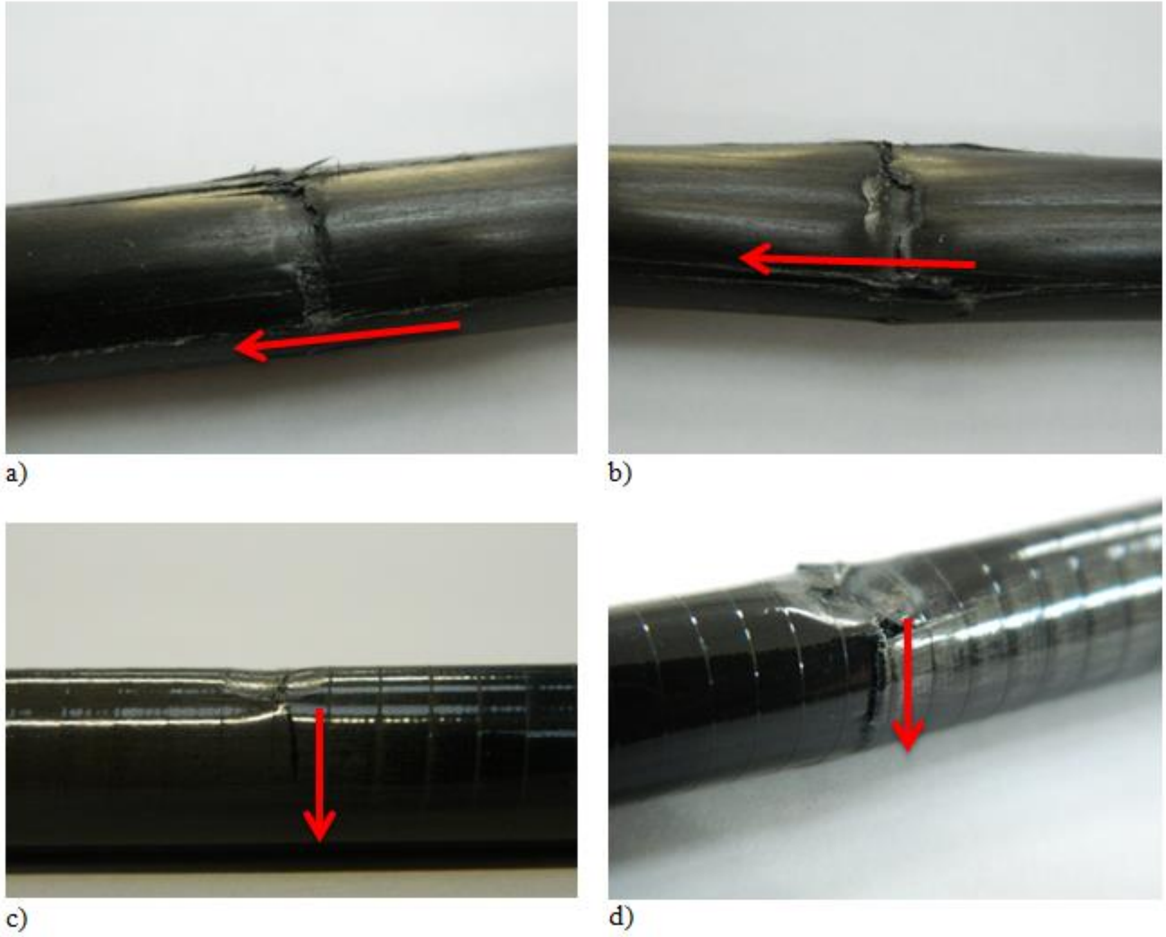
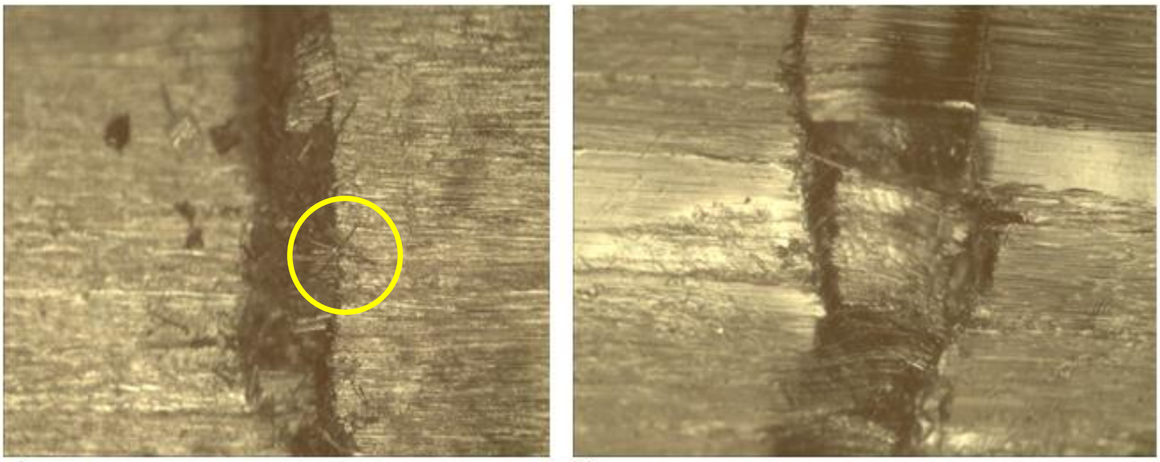


Figure 4.2.1-3 Photographs of fractured pipes a) Pultruded at ambient temperature
b) Pultruded at 60% of Tg
c) Roll-wrapped at ambient temperature
d) Roll-wrapped at 60% of Tg

For all pipes independent on the temperature or manufacturing process there is a visible plastic deformation of the area where the load was applied. However the crack directions are different for the pultruded and roll-wrapped sample.

Red arrows show the crack direction on the side of the pipe and point out that the crack of the pultruded pipe a) and b) is lengthways while the roll-wrapped pipe c) and d) breaks normal to the length.

Pictures provided in figure 4.2.1-4 were taken with an optical microscope after three point bending test at ambient temperature.



a) b)

Figure 4.2.1-4: Optical microscope photographs of fractured pipes at ambient temperature
a) Pultruded
b) Roll-wrapped

The pultruded pipe a) has more separated fibres near the crack opening which are highlighted with a yellow circle. Figure 4.2.1-4 b) illustrates breaking of fibre-matrix-pieces at the damage area instead of a separation of single fibres.

Further photographs taken with optical microscope are included in appendix 9. They are not presented in the study because of the optical microscopes poor depth of focus.

4.2.2 Scanning electron microscopy analysis

The examination of the microstructure of the damaged pipes is presented in the following sections where different samples are compared related to a certain aspect. Important conspicuities are highlighted with coloured lines, arrows and circles.

1. SEM Examination: Area of crack opening

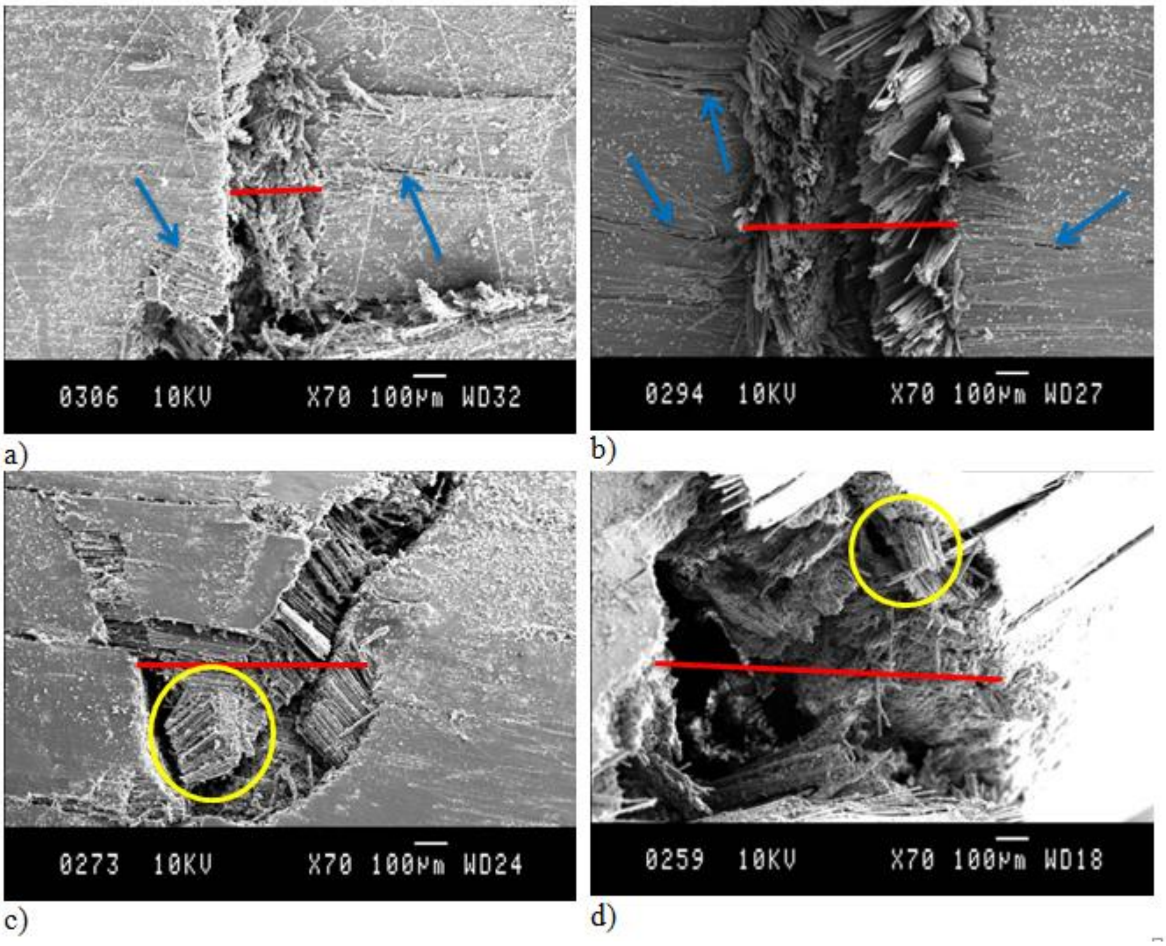


Figure 4.2.2-1 SEM picture of the crack opening a) Pultruded at ambient temperature
b) Pultruded at 60% of Tg
c) Roll-wrapped at ambient temperature
d) Roll-wrapped at 60% of Tg

Table 4.2.2- 1 Crack opening at the fracture (distance of the red line)

	Opening in µm
a)	312
b)	630
c)	750
d)	1062

Temperature effects

Red lines represent the crack opening size. These lengths are measured and listed in table 4.2.2-1 where it is visible that for both processes the crack opening is larger at higher temperature.

Manufacturing process effects

For the pultruded samples a) and b) in figure 4.2.2-1 fibres are clearly visible at the surface and there is no change over from the intact to the damaged area as the separation of single fibres starts even far away from the main crack. This fibre separation near the crack edge is pointed out with blue arrows. The cracks contain many disordered fibres which are separated from each other and are broken into different lengths.

For the roll-wrapped samples c) and d) fibres in the outer layer do not separate as much as for the pultruded samples. The area of fracture is clearly distinguishable from the intact area. The fracture itself presents damaged fibres which are nevertheless aligned in one direction shown in c). Overall the material breaks more into discrete pieces as is highlighted in the yellow circles.

2. SEM Examination: Initial cracks apart the main crack

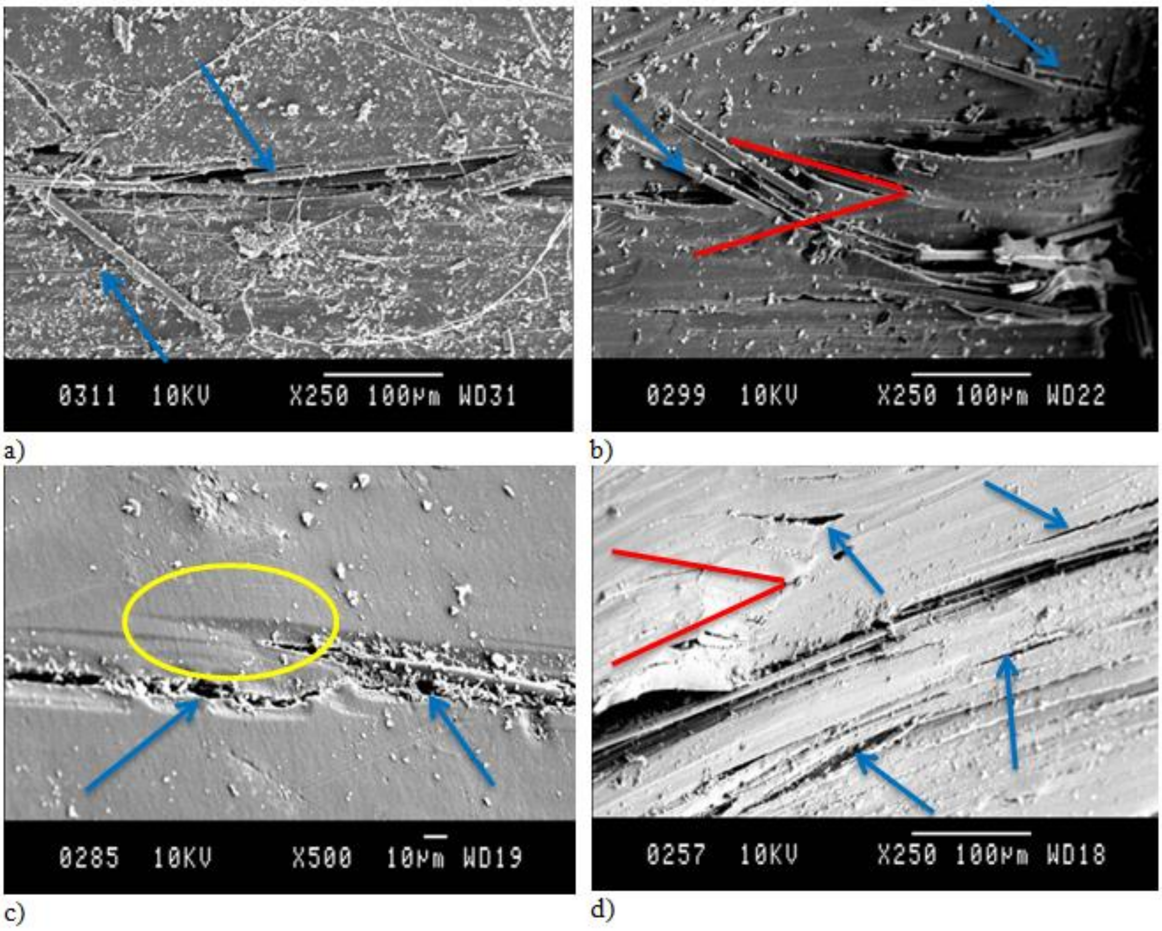


Figure 4.2.2-2 SEM pictures of the initial cracks
a) Pultruded at ambient temperature
b) Pultruded at 60% of Tg
c) Roll wrapped at ambient temperature
d) Roll wrapped at 60% of Tg

A region where fracturing starts is presented in figure 4.2.2-2. Blue arrows point at the initial separation of fibre and matrix called delamination. When comparing the initial cracks note that figure c) is taken at higher magnification and is not exactly comparable with the other three pictures. Even so it provides an insight of the plastic deformation of matrix at the crack tip, pointed out with a yellow circle.

Temperature effects

In samples b) and d), tested at higher temperatures, the fibre direction - as well as the first appearing fracture with fibre separation and delamination - is clearly detectable. Delamination starts with matrix cracking at nearly 45° to the fibre direction as indicated with the red lines.

Manufacturing effects

The pultruded samples a) and b) have more damaged and broken fibres in the regarded area while the fibres of the roll-wrapped samples c) and d) seem to be more intact.

3. SEM Examination: Initial crack opening at the crack tip

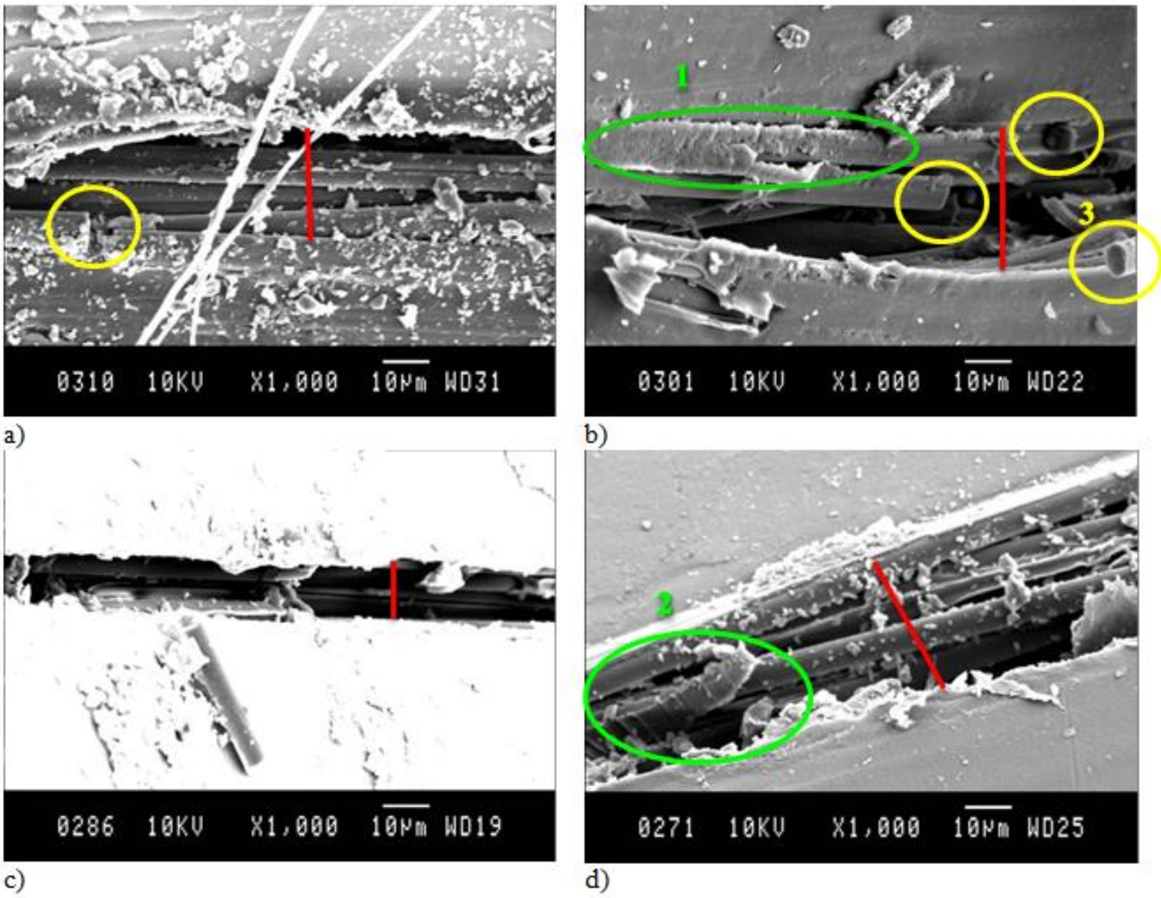


Figure 4.2.2-3 SEM at 1000x magnification of the opening at the crack tip
a) pultruded at ambient temperature
b) pultruded at 60% of Tg
c) roll wrapped at ambient temperature
d) roll wrapped at 60% of Tg

Table 4.2.2- 2 Crack opening at the crack tip (distance of the red line)

	Opening in µm
a)	23
b)	30
c)	13
d)	29

Further Temperature effects

Figure 4.2.2-3 demonstrates that the cracks are orientated along the fibre direction. Comparing the opening distances highlighted with red lines the crack opening is larger for higher temperatures. This confirms observations about the size of the crack opening considered in table 4.2.2-2.

At higher temperatures both samples b) and d) have parts of the matrix with shear lips which indicates plastic deformation near the crack tip. These areas are highlighted with green circles and presented in detail views in figure 4.2.2-4 and 4.2.2-5.

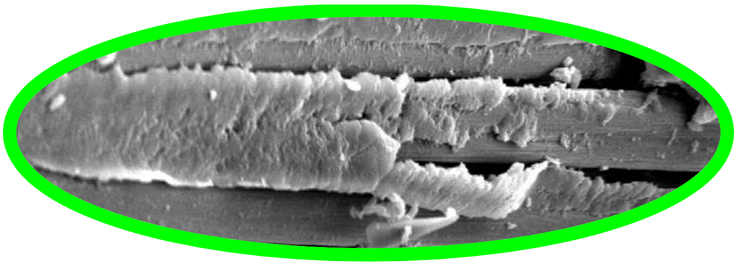


Figure 4.2.2-4 Detail view circle 1

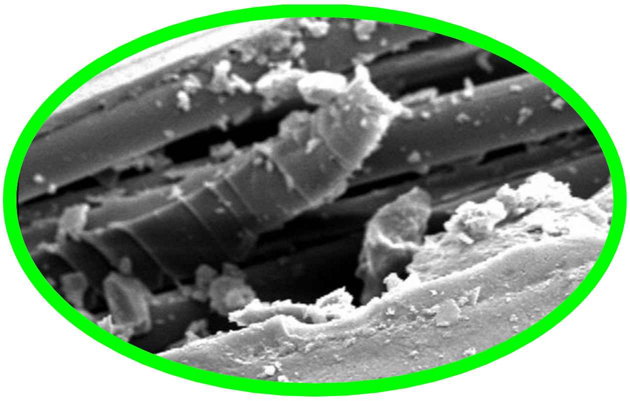


Figure 4.2.2-5 Detail view circle 2

Overall, crack opening at ambient temperature appears along clear fracture surfaces with small brittle matrix pieces around the crack and no visible plastic matrix deformation. The crack opening of the samples at higher temperatures is more surrounded by plastic deformation of nearby matrix.

Manufacturing effects

The roll wrapped samples c) and d) in figure 4.2.2-3 consist of more intact fibres than the pultruded a) and b) where damaged and broken fibres are designated with yellow circles.

One of these fractured fibres is provide in a detail view in figure 4.2.2-6. The fibre breaks normal to its length without necking and the fracture surface itself shows a rough area with deformation.

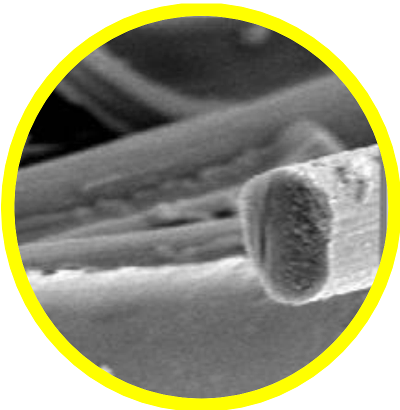


Figure 4.2.2-6 Detail view circle 3

4. SEM Examination: Ribbed area of the roll wrapped samples

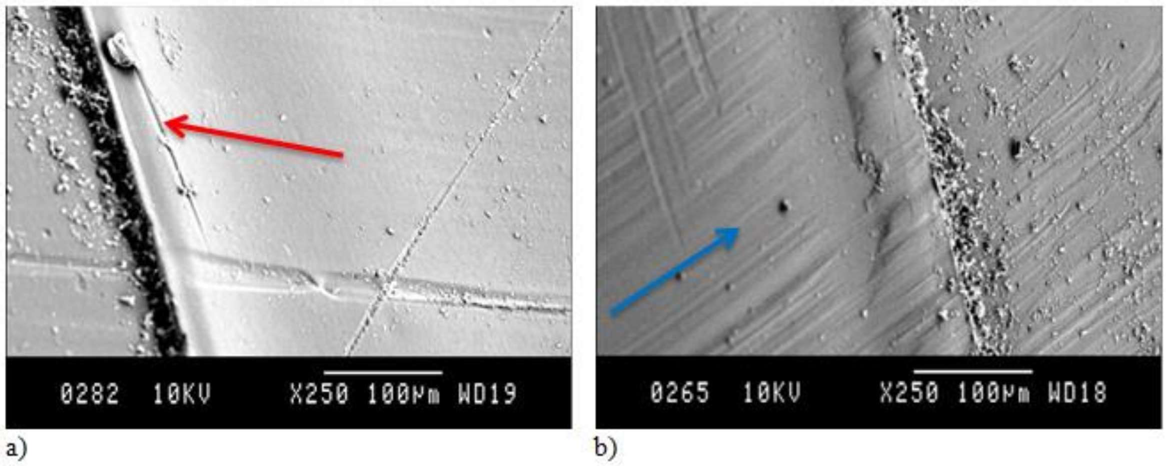


Figure 4.2.2-7 SEM picture of the ribbed area of the roll wrapped-pipe
a) At ambient temperature
b) At 60% of Tg

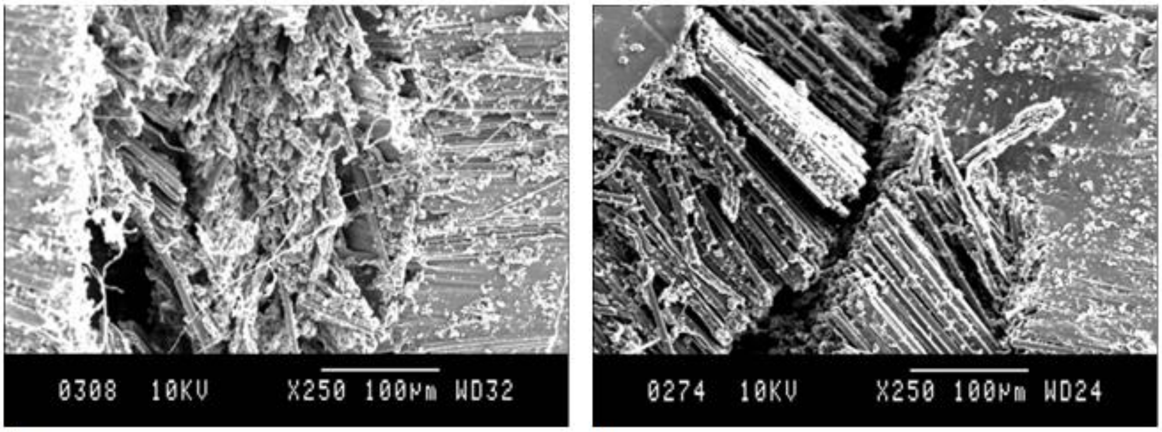
Figure 4.2.2-7 illustrates the surface appearance of the roll wrapped samples where ribs caused by the manufacturing process are visible.

At ambient temperature the surface is flat and plane while the sample at higher temperature demonstrate lines in fibre direction as indicated with a blue arrow.

Even if the examined area of the pipe is apart the fracture a) shows a crack normal to the fibre direction along a rib which is clearly detectable, indicated with a red arrow.

This crack could cause a degradation of mechanical properties even if this crack is not connected to the main fracture.

5. SEM Examination: Fibre-matrix-interface



a) b)

Figure 4.2.2-8 SEM pictures of the fracture area at ambient temperature
a) Pultruded
b) Roll-wrapped

As already mentioned in figure 4.2.2-1 the fracture of the pultruded sample is disordered presented in figure 4.2.2-8 a) and fibres are scattered in all directions. The roll-wrapped sample fibres are still orientated in one direction and it is clearly visible that nearly all fibres were broken along a plane normal to the fibre orientation.

Comparing the appearance of fibre-matrix-interphase, the fibres of a) are covered with remains of matrix material while fibres in picture b) are cleaner with less matrix remains. This indicates a distinctive separation of fibre and matrix at higher temperatures under external loading conditions.

4.3 Hardness-test Rockwell

Table 4.3-1 shows the results of the hardness test HRH with a hard metal ball 1/8" diameter and a preload of 60kN. Each material was tested ten times at ambient temperature.

Table 4.3- 1 Results of the hardness test

Number of test	Pultruded	Roll-wrapped
1	81.3	69.3
2	85.6	67.5
3	87	72.3
4	87	71.6
5	83.7	66.2
6	87.8	67.4
7	84.3	62.1
8	86.6	70.8
9	88.3	73.0
10	84.8	61.8
Average	**85.6**	**66.2**

The average is generated for both materials and the pultruded pipe has higher hardness than the roll-wrapped.

4.4 Tensile test

The tensile test gave no evaluable results because the pipe slipped in the grips. This is the reason why three point bending is used to cause a fracture which can be examined.

4.5 Three point bending test

During testing of both materials at ambient temperature and 60% of Tg the machine records the following data presented in table 4.5-1.

Table 4.5- 1 Test report results three point bending

Sample	Young's modulus [GPa]	Fmax [N]	F at 0.1% plastic deformation [N]	F at 0.2% plastic deformation [N]	Upper yield point [N]	dl at upper yield point [mm]
Pultruded 20°C	19.7	84.0	292	281	344	2.3
Pultruded 60% Tg	13.7	64.7	209	208	212	1.7
Roll wrapped 20°C	39.0	611	481	521	--	--
Roll wrapped 60% Tg	27.3	492	412	453	492	2.5

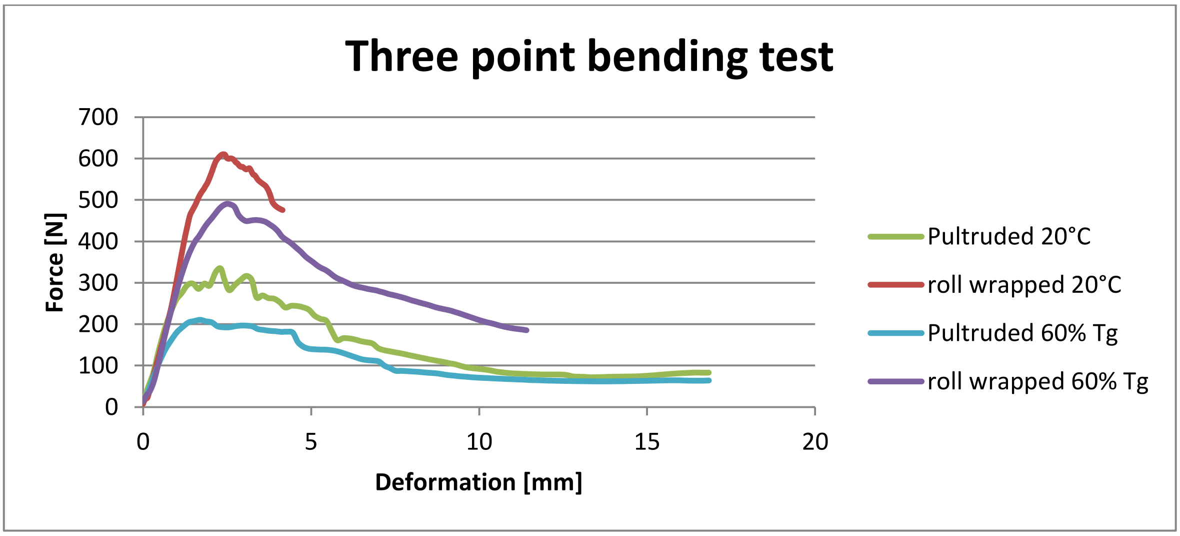


Figure 4.5- 1 Diagram three point bending

The deformation of each sample is presented against the force in figure 4.5-1.

All graphs start with an elastic material behavior where an increase of deformation is linear to the applied force. After linear elastic deformation the pipes break at their upper surface with only small previous plastic deformation.

Comparing the two roll-wrapped samples tested at different temperatures the recorded data listed in appendix 10 represents that the sample at ambient temperature withstands forces up to 610N while the sample at 60% of the glass transition temperature reaches 490N before it breaks. The pultruded sample tested at ambient temperature withstands a maximum force of 320N and the other one tested at higher temperature withstands 210N. These data are not concordant with the test report results summarised in table 4.5-1 and therefore discussed later.

Independent on the temperature, the roll-wrapped samples have higher Young's modulus and can withstand higher forces than the pultruded samples.

Comparing the samples regarded to the testing temperature the plastic deformation starts later at higher temperature which means that more elastic deformation is tolerated before fracture occurs.

The bending test is driven by 2mm/min and stopped after a good visible plastic deformation and fracture at the upper side of the tube occurred. This manual stop explains why the curves end at different times and there are no results provided for the upper yield point of one roll-wrapped sample.

5 Discussion

5.1 Differential scanning calorimetry analysis

For epoxy thermoset resin the literature provide a glass transition temperature range of 75°C-150°C. Hence DSC is done to determine the glass transition temperature of a specific material.

In figure 4.1-1 the range of thermal reaction of the pultruded sample occurs between 136.7°C and 146.2°C. This thermal reaction indicates the range of Tg for a specific material where the mobility of polymer chains changes and energy is needed. To name a single temperature in this range a point in the middle is chosen 138.7°C as Tg.

The range for the roll-wrapped sample shown in figure 4.1-2 is 116.2°C to 135.6°C and Tg is determined at 121.2°C.

A visible earlier decrease of the curve in 4.1-1 around 95°C is not the glass transition temperature. This thermal reaction might be caused by contamination during sample preparation and is not relevant for this examination.

In general, Tg is highly dependent on the degree of cure which means that for a resin cured at ambient temperature the glass transition temperature cannot be as high as for a resin cured at higher temperatures.

Usually materials with a higher Tg have better heat resistance and therefore better mechanical properties at higher temperatures.

Here the pultruded pipe has a higher glass transition temperature and higher hardness which predict good wear resistance.

In this study the strengths and deformation abilities of the roll-wrapped pipes are superior verified by the manufacturers given data and the results of three point bending test which are discussed in chapter 5.4. This disagreement of correlation between Tg and mechanical properties of polymers could be explained in this way, that Tg is a value of the epoxy resin without considering reinforcements and the influence of different manufacturing processes and their parameters. The influence of fibres regarded to their alignment and parameters during the manufacturing process are discussed in the 5.2.

Another reason for the identification of Tg is that the two samples should be tested at a specific temperature at that the material should be able to perform without failure. The influence of extreme temperatures on composite materials is necessary to examine and understand as these materials should be used in offshore applications where insolation let temperatures increase.

It is assumed that the tested temperature range is 70°C to 85°C. For testing, the same temperature influence and achieve information about the material behaviour under similar conditions the same percentage of Tg is chosen.

5.2 Visual investigation

5.2.1 Photographs and optical microscopy

Examinations of as-received pipes present no defects, discontinuities or improper materials. An existence of these defects could create the onset of failure even if the applied load is below the materials maximum strength. With respect to the usage of composites as a replacement of metals the impact of factors that reduce the performance is important to understand and must be known beforehand.

Photographs of pipes deliver an insight of the fibre alignment with respect to the manufacturing process. Different crack behaviours and directions presented in figure 4.2.1-3 occur in consequence of the fibres alignment shown in figure 4.2.1-1 and 4.2.1-2.

Fibres of the pultruded sample are not ideally parallel and the matrix resin is identified as hard and brittle. Lengthways matrix cracks of the pultruded pipe are caused by a low fibre matrix adhesion and a variable distribution of the fibres over the cross-section. This leads to poor load transfer from fibre to fibre and as the load is applied normal to the reinforcing fibres. The weak and brittle matrix has to carry the load and consequently breaks along the fibre direction. Normally load is applied in fibre direction to use the superior strength of fibres which withstands loads better than matrix materials.

Figure 4.2.1-3 a) shows the separation of fibres and many lengthways cracks along the pultruded pipe. The breaking behaviour can be described as brittle because of many cracks next to each other in the same direction and non-existent plastic deformation.

These findings confirm the determination of the pultruded sample as brittle.

The roll-wrapped pipe has equally distributed fibres caused by the prepreg lay-up process shown in figure 4.2.1-2. A crack normal to the fibre direction demonstrates a good fibre-matrix bounding which prevents matrix cracking between fibres. The translaminar cracks form a clear fracture surface without visible separated fibres on this examination level.

Looking at the pipes surface finish the dull looking pultruded sample might experience more stress concentration at the surface because of potential formation of micro-notches due to a rough and uneven surface. These notches promote an onset of cracks and leads to earlier failure.

5.2.2 Scanning electron microscopy

The crack opening mentioned in table 4.2.2-1 and table 4.2.2-2 confirms for both materials that the crack opening is larger at higher temperature.

Temperature has an impact on the mobility of polymer chains and therefore and influence on the brittleness or rather ductility. An increasing ductility of a material caused by temperature provides higher fracture toughness and leads to a higher demand of energy for the formation of a crack opening. Regarded to the crack opening size both materials behave more ductile, but still brittle, at higher temperature through the higher fracture toughness and a small plastic deformation.

Another temperature related appearance is an existence of shear lips shown in the detail view figure 4.2.2-4 and 4.2.2-5. Shear lips indicate plastic deformation and more ductile behaviour as entirely brittle material which has only an elastic deformation followed by fracture. Ductile materials have a yielding range which allows them to deform plastically in order to reduce stress and breaks later after yielding.

Another advice for less brittleness behaviour at higher temperatures is mentioned in figure 4.2.2-2 b) and d) where a first delamination occurs with a 45° angle to the fibre direction. This fracture behaviour appears under tension and shear loading conditions. Brittle material behaviour is represented in a) and b) at ambient temperature where the cracks are in fibre direction.

This figure gives also information about the stages of appearing damage mechanisms. The initial intralaminar cracks are clearly visible in the matrix. After matrix cracking, fibres separate from the matrix and result in delamination. Delamination entails degradation of mechanical composite properties and with an increasing load the reinforcement cannot withstand anymore and the fibres breaks.

It is well known that the strength of the fibres is higher than the strength of the matrix. As mentioned before the applied load in this project is not in fibre direction and therefore the matrix is the constituent which has to carry most of the load referred to chapter 2.4 where calculations confirm that the composite properties are normal to the fibre directions are only dependent on the matrix properties and its volume content.

In figure 4.2.2-1 and 4.2.2-8 an intact area near the fracture of the roll-wrapped pipe is visible which indicates the existence of a crack stopping mechanism. This stopping mechanism is not detectable for the pultruded pipe where fibres separate from the matrix in the area around the fracture. Such a crack stopping mechanism has a particular importance in application where detecting a small crack early could prevent failure of a component.

This material behaviour is a consequence of the manufacturing process and its parameters. The prepregs for the roll-wrapped pipe are produced with an accurateness of their fibre alignment and an excellent fibre matrix adhesion due to the pressure of the shrinkage tape during the curing process.

Furthermore this good fibre matrix adhesion is represented by pieces of broken fibres in figure 4.2.2-1 c) and d) and highlighted with yellow circle.

Considering figure 4.2.2-7 where an initial crack forms along the ribbed surface of the roll wrapped pipe it is possible that these ribs caused by the manufacturing process induce the onset of failure.

Ribs on the pipe surface change the size of the cross-section. When the applied load is distributed equally, changes in the cross-section generate stress differences and stress concentrations. The influence of these ribs could have a similar influence as notches and therefore create a potential onset of cracks.

Applying load on the roll-wrapped sample normal to the fibre direction the fracture behaviour is characterised by an optimal fibre distribution where all fibres carry the load. The fibre next to the force application point experiences the highest stress and hence this fibre collapse first. Related to a good interface the stress is transferred to the next fibres where the same process appears and result in crack growth normal to the fibre direction illustrated in figure 4.2.1-3 c) and d).

5.3 Hardness test Rockwell

For the hardness test HRH an aluminium pin was placed inside the pipe to avoid buckling of the pipe. Without this pin the testing machine generates no data. As the pin is necessary to generate hardness data of this materials the results of this test cannot be considered as an absolute hardness of the tested composite. It is more a combined hardness of the composite pipe reinforced with an aluminium pin. Due to this the data cannot be transformed into other material properties presented in respect to the strength. But nevertheless the data can be compared in reference to their ratio of hardness.

As shown in table 4.3-1 the average hardness of the pultruded pipe is 85.6 HRH and 66.2 HRH of the roll wrapped. Related to this the hardness of the pultruded pipe is 30% higher than for the roll wrapped. This result is independent on the aluminium pin which is in both test pipes the same.

5.4 Three point bending test

Three point bending test is used to identify strength and deformation-behaviour as well as to generate a fracture for optical examinations.

The maximal stress occurs in the edge layer of the pipe. Shear stresses could be neglected because of the long distance between the supports compared to the sample thickness.

Mechanical properties achieved from the bending test are the force at a constant deformation rate. Especially for testing pipes it is important to mention that the load is first applied to an outer fibre layer of the pipe because the tested material is not a solid.

Results of the three point bending test presented in table 3-1 shows that the Young’s modulus of the pultruded pipe given 10-20 GPa by the manufacturers data is reached during the three point bending test where the sample at ambient temperature reaches 19.7 GPa. At higher temperature the Young’s modulus is lower as it was expected because of a softening effect through temperature.

The Young’s modulus for the roll-wrapped pipe should be 70 GPa stated by the manufacturer. The three point bending tests identify a Young’s modulus of 39 GPa at ambient temperature and 27.3 GPa at 73°C. This confirms an increasing ductility with the temperature.

The fact that the Young’s modulus of the roll-wrapped pipe is so much lower than the theoretical values might be caused by the fibre alignment. The manufacturer declares that the roll wrapped pipes consist of carbon fibres in 0° direction down the length of the pipe and glass fibres in 90° direction normal to the length of the pipe. None of the optical and SEM examinations indicate any glass fibre prepregs laid-up in 90° direction. These glass fibres should improve the resistance against bursting.

Because the examinations approve only carbon fibres this could be a potential explanation for a lower Young’s modulus of the tested pipe as the missing glass fibres cannot reinforce the pipe around the circumference.

Figure 4.5-1 presents the required force to create a deformation of the pipe. The linear increase of the graphs in the beginning indicates elastic material behaviour. This elastic zone is followed by a range with plastic deformation. Especially the pultruded pipes which are identified as brittle material with a high hardness in 5.3 present a fluctuation in the area of the highest force. The fluctuation in the diagram of the pultruded pipes is caused by brittle behaviour of the thermoset matrix and the geometry of the pipe.

When a load was applied to the pipe, the outer fibre and matrix at the surface carried the entire load until they transfer it to nearby fibres. Due to this stress concentration on the upper surface of the pipe it collapse with a crack longitudinal to the length of the pipe. This collapse causes a sudden drop in the load and reduces the force momentarily. After this collapse the testing machine detects no resistance against the applied load. This phenomenon happened a few times until a main crack grows and the testing was stopped.

Comparing two kinds of pipes, the roll-wrapped provided better properties regarded to fracture resistance because the lay-up of prepreg induce internal stresses and load.

Table 3-1 shows the recorded data by the testing machine. There are given the maximum force and the upper yielding point. Usually the upper yielding point and the maximum force are the same value as the upper yielding point characterise the highest force before a material starts to fail after its maximum elastic and plastic deformation.

For the pultruded pipe at ambient temperature the presented maximum force is only 25% of its upper yielding point. This mismatch of data could result from an early breaking of the upper pipe surface which is the same cause of fluctuation of the graph in the diagram.

Testing near Tg shows that the epoxy still behaves brittle but with a little more deformation. As the testing temperature would exceed Tg the material behaves ductile related to the mobility of the polymer chains.

5.5 Discussion summary

The two tested materials behave at ambient and higher temperature the way it is predicted for polymer materials. For pipes with fibres in the direction of the length tensile testing might be an appropriate testing method as the load is applied in fibre direction and the fibres are able to reinforce the material and can demonstrate their superior properties mentioned in chapter 2.4.

Tensile testing reproduces the most likely loading cases for this kind of pipe because it is recommended to stress fibre reinforced materials only with tension in fibre direction. Three point bending, which is used due to the gripping problems of the tensile testing machine, stresses the composite not in the way the reinforcements can support the polymer material. Nevertheless three point bending tests at different temperatures confirm an influence of temperature on composite materials.

To verify the influence of fibres during the bending test it could be worth testing the same materials without any reinforcement under same loading conditions to compare the material behaviour and draw conclusions from these results for the influence of fibres in composite materials where load is applied normal to the fibre direction.

An implementation of the composite laminate theory would be useful for the roll-wrapped pipe. But as the manufacturer gives no details about the number of layers in this composite and the optical microscope delivers no useful picture to identify the layers it is not possible to apply this theory in an appropriate way.

In case the number of layers would be known the laminate theory could be used to calculate a theoretical strength of the pipe and compare it to the real strength gained from mechanical tests. The laminate theory is only suitable for composite laminates where plies are laid up with different directions mentioned in chapter 2.5. When the plies are all in one direction the properties of the whole component is a simple addition of single layers.

After visual examination of the roll wrapped pipe it is detectable that the laid-up process of the prepregs was not done the way the manufacturer specified. None of the pictures show a layer of 90° laid-up glass-fibre prepregs. Therefore all layers of the composite have been carbon-fibre prepregs with a 0° direction.

Glass fibres around the circumference of the pipe could have an influence on flexural strength of the pipe and reinforce it against bursting and early failure. Comparing theoretical and tested Young's modulus a difference of nearly 45% appears, which could be caused by missing reinforcement.

The use of composite materials in offshore industry requires a good understanding of the influencing factors and loading cases that for example a pipeline made of composites have to withstand.

In the deep sea, pipes experience many different and complex pressures and stresses as well as many influencing factors such as temperature, salt, moisture and time.

Three point bending test represents only an exemplary model of a loading case where a force is coming from the side of the pipe. To simulate realistic in- service loading cases of pipelines a lot of other models and analysis are necessary. This single exemplary test does not verify a use of carbon composites in offshore industry but it is an accepted and appropriate approach to simulate one loading case and one influencing factor to achieve an insight of the material behavior.

As mentioned before composite materials behave brittle at low temperatures and provide simultaneously higher strength.

In the deep sea the temperature is only a few degrees above 0°C while the temperatures could be much higher on land. Low temperatures might cause high stiffness and stability for the tested pipes. But with these increasing properties on the one hand there are always properties which degrade at lower temperatures. One decreasing factor is ductility which enable a wider elastically and plastically deformation before a material fails. This material behaviour would be desirable for a pipeline in the deep sea where water flow or other unknown loading cases could stress the material above an application range.

Beside the influence of temperature there are more factors influencing the performance of composite materials in the deep sea such as salt, moisture and light. That can result in corrosion and degradation of the properties.

The existence of moisture in a composite material causes expansion depending on the diffusions ability of the used material. This moisture expansion of the polymer matrix increases internal stresses as fibres are not sensitive to moisture. These internal stresses could be used to induce a higher stiffness of the component and qualify it for higher loads. Environmental factors, such as salt and light, can also have an influence on the composite performance as they promote aging and corrosion and therefore reduce the in-service life of a component.

In addition the existence of defects, discontinuities and misuse in service can result in failure which must be prevented to avoid natural disaster. Therefore it is very important to know all factors which can generate failure and how to improve the material.

For this study the manufacturer does not provide any information about the parameters which were used for each manufacturing process. This information would help to understand the influence of temperature or pressure during a process and how it controls mechanical values during testing. With knowledge of these parameters the process could be optimised to achieve a better fibre matrix interface or induce internal stresses which increase the stiffness.

6 Conclusion and recommendation for further work

The primary aim of this study was to investigate the potential damage mechanisms and parameters that affect the composite structure at different temperature levels under flexural loading conditions. Therefore the as-received and fractured composite pipes were characterised in order to identify the fibre-matrix interface, fibre arrangement and understand the damage mechanisms such as fibre and matrix cracking.

The examination of the pipes was carried out by previous characterisation of the as-received material using hardness test, defining the glass transition temperature with the differential scanning calorimetry and study the composite structure by using optical microscope and scanning electron microscope. After the previous characterisation a fracture was caused by applying three point bending.

The first idea to generate a fracture with the tensile test was replaced with the three point bending test after the samples slipped in the grip and create no usable results.

Although the results of the test indicate a comprehensive relationship between the particular results the number of samples was not high enough to state this as generalised behaviour of pultruded or roll-wrapped pipes.

The findings from this study are that failure analysis can be done using SEM, the manufacturing process as well as the in-service temperature has an impact on the material behaviour and properties of the composite.

Visual investigations done with the optical microscope provide only useful pictures of flat and even surfaces. For the fractured pipes the poor depth of focus of an optical microscope at high magnification is inadequately to achieve an insight of the damage mechanisms. Therefore the scanning electron microscope is used for the examination after fracture. SEM pictures deliver detailed information about the composite constituents as well as the occurring damage mechanisms.

The SEM examination of the roll-wrapped samples gives information about the stages of appearing damage mechanisms. It starts with intralaminar matrix cracks followed by delamination and as the stress increases and the reinforcement cannot withstand it anymore it ends in fibre cracking.

Even though the pultruded pipes have a higher hardness and a higher glass transition temperatures which indicate better wear resistance, he roll-wrapped pipes provide the better mechanical properties the for a use of composite pipes in offshore applications at all tested temperatures because of the manufacturing process.

The roll-wrapped pipe is produced with an accurateness of the fibre alignment and an excellent fibre matrix adhesion due to the pressure at the manufacturing. Therefore it can withstand higher stresses because of the optimal fibre distribution where all fibres carry the load.

In addition a crack stopping mechanism occurs in these materials which could be assumed as there are intact areas of the composite near the fracture. Such a crack stopping mechanism has a particular importance in application where detecting a small crack early could prevent failure of a component.

The in-service or testing temperature has an effect on the composite performance. Testing near Tg shows that the epoxy still behaves brittle but with a little more deformation than at lower temperatures. As the testing temperature exceeds Tg the material behaves ductile related to the mobility of the polymer chains.

Higher fracture toughness and small plastic deformation, which leads to an absorption of mechanical energy, confirm the decreasing brittleness at higher temperatures. Other indicators for more ductile behaviour are the appearance of shear lips and delamination in 45° to the fibre direction at temperatures near the glass transition temperature.

Further work in this area would help to establish the findings and confirm them with more testing results.

Recommendation for further work:

- Details of the resin and the manufacturing parameters used for the manufacturing process are required to affirm the evidence of the occurring material behaviour.
- According to the manufacturer the roll wrapped pipe consist carbon and glass fibre prepreg layers laid-up in 0° and 90° direction. But none of the taken photographs shows any indication of fibres orientated normal to the length. Further studies could be concentrated on checking the existence of any layer in this pipe that is orientated in 90°.
- Data collected from only one sample per temperature makes it difficult to verify the results. Further work should be done with more samples to verify the test results
- The experimental investigation should be extended by using more tubes made from other manufacturing processes or layer orientations.
- Further studies could be done by testing the tubes at more temperatures to determine the flexural behaviour of carbon reinforced epoxy tubes depending on the temperature
- Additional work should be done focusing on the fracture and material behaviour under time dependent loading conditions.
- To back up the test results referring to the properties other testing method such as impact or relaxation tests should be done

7 Project Evaluation

Project presentation and selection

- The information lecture about the project was a presentation of the unit-handbook and very useful to know what is expected
- The selection process of the topics was inadequate as I had no chance to see the potential project title before they were uploaded on moodle.
- The idea of creating a own project was not possible for me as I did not know who lecturer is working in which field of study
- Many projects were taken within the first few hours after they were published

Provided information about the project (lectures, moodle, unit-handbook)

- The information in the lectures about how plan a project are useful for people who have no idea how to do a project
- Information about academic writing and other requirements are a helpful guideline
- Changing the unit handbook in the middle of the year (the word limit, size of poster for example) is not helpful and confuses the students

Project management

- Changing the presentation and hand-in dates (from 25.4. to 26.4. and 3.5. to 2.5.) is confusing and give an impression of bad organisation beforehand
- A bit more structure in the unit beforehand and less inconsistent information could prevent misunderstanding

Supervision and support

- I could ask the unit coordinator or my supervisor all the time whenever I had a question about the project
- My supervisor helped and encouraged me to work hard on my project and write a logical good understanding academic report based on my testing results

Lab facilities

- I used the mechanical testing facilities in Anglesea Building and the SEM in Burnaby Building
- The Labs are on a high standard and it is a pleasure to use such modern facilities

List of achievements

Project and learning achievements

- Understanding how to use different mechanical testing facilities (Universal testing machine, Rockwell hardness testing machine, Optical microscope, SEM microscope)
- Preparing of samples for testing and visual examination
- Amplify the knowledge about composite materials, their use and failure
- Understanding of Failure analysis
- The project confirms my way to plan a project. Using project management tools such as mind map, gantt-chart, timetables and so on to structure the project as best as possible.
- Breaking work down into small packages and to set deadlines for them, to make sure being on-time.
- Summarise the project so far from time to time and clarify the aims and objectives
- By reading many academic books and papers I learned many English vocabularies and how to build formal sentences in academic writing.
- While rewriting the report many times I realised an improvement of the technical understanding as well as the use of the language.

References

1. (2001). *ASM Handbook Vol. 21, Composite*. Materials Park, Ohio: ASM International.

2. Schürmann, H. (2005). *Konstruieren mit Faser-Kunststoff-Verbunden*. [Design with fibre-polymer-composites]. Berlin: Springer Verlag.

3. Campbell, F. C. (2010). *Structural Composite Materials.* Materials Park, Ohio: ASM International.

4. Eyerer, P., Hirth, T., & Elsner, P. (2008). *Polymer Engineering: Technologien und Praxis* [Polymer Engineering: technologies and practice]. Berlin: Springer-Verlag.

5. Jones, R. M. (1999). *Mechanics of composite material*. (2nd ed.). London: Taylor & Francis Ltd..

6. Schwartz, M. M. (1996). *Composite materials.* London: Prentice-Hall Inc..

7. Akovali, G. (2001). *Handbook of Composite Fabrication.* Shrewsbury: Smithers Rapra.

8. Hellerich, W., Harsch, G., & Haenle, S. (1996). *Werkstoff-Führer Kunststoffe* (7th ed.) [Material-guide polymers]. München: Hanser Verlag.

9. Advani, S.G., & Sozer, E.M. (2003). *Process Modeling in Composite Manufacturing*. New York: Marcel Dekker, Inc.

10. *Manufacturing Processes & Tube Fabrication Techniques.* (2012). Retrieved 11 20, 2012, from chemical-supermarket: http://www.chemical-supermarket.com/home.php?cat=101.

11. *easycomposites.* (2012). Retrieved 03 16, 2013, from http://www.easycomposites.co.uk/.

12. Bonnet, M. (2009). *Kunststoffe in der Ingenieuranwendung* [Polymers in engineering]. Wiesbaden: Vieweg und Teubner.

13. Smith, P. T., Ross, C. T. F., & Little, A. P.F. (2012). Formulation of Design Charts for Composite Submarine Pressure Hulls. *Journal of Ship Production and Design, 28(1),* 20-41.

14. Roylance, D. (2000). *Laminate Composite Plates*. Unpublished internal paper, Massachusetts Institute of Technology, Cambridge.

15. Totry, E., Gonzàlez, C., LLorca, J., & Molina-Aldareguia, J. M. (2009). Mechanism of shear deformation in fiber-reinforced polymers: experiments and simulation. *Int J Fract*, *158*, 197-209.

16. Milton, G. W. (2001). *Theory of Composites.* Port Chester, New York: Cambridge University Press.

17. Kanchanomai, C., Rattananon, S. & Soni, M. (2005). Effect of loading rate on fracture behavior and mechanism of thermoset epoxy resin. *Polymer Testing, 24,* 886-892.

18. Sridharan, S. (2008). *Delamination behaviour of composites.* Cambridge: Woodhead Publishing Limited.

19. Armanios, E. A. (6th ed.). (1997). *Composite Materials: Fatigue and Fracture*. West Conshohocken: AMERCIAN SOCIETY FOR TESTING AND MATERIALS.

20. O'Brian, T. K. (3rd ed.). (1991). *Composite Materials: Fatigue and Fracture.* Philadelphia: AMERICAN SOCIETY FOR TESTING AND MATERIALS.

21. Hayes, B. S., & Gammon, L. M. (2010). *Optical Microscopy of Fiber-Reinforced Composites.* Materials Park, Ohio: ASM International.

22. Davis, J. R. (2004). *Tensile Testing* (2nd ed.). Materials Park, Ohio: ASM International.

23. Huang, Z. M. (2003). Ultimate strength of a composite cylinder subjected to three-point bending: correlation of beam theory with experiment. *Composite structures, 63,* 439-445.

24. Dhieb, H., Buijnsters, J. G., Eddoumy, F. & Celis, J.P. (2011). Surface damage of unidirectional carbon fibre reiforced epoxy composites under reciprocating sliding in ambient air. *Composites Science and Technology, 71,* 1769-1776.

25. Brown. R. P. (2002). *Handbook of Polymer Testing: Short-Term Mechanical Tests*. Shrewsbury: Smithers Rapra.

26. Pielichowski, K. & Njuguna, J. (2005). *Thermal Degradation of Polymeric Materials.* Sherwsbury: Smithers Rapa.

*Loose translation of German references done by author

Appendices

Appendix 1 Completed project proposal form

Project Proposal – Individual Project 2012 / 2013

STUDENT NAME Nadine Tscheu **REGISTRATION NUMBER** 672817

COURSE BEng Mechanical Engineering **DATE** 1/10/2012

PROJECT TITLE	Macro- and Micro-study of carbon-composite
BACKGROUND A brief introduction to the topic.	The composite material's behaviour would be closely controlled by for example the matrix morphology, fibre/matrix interface, fibre arrangement (direction or orientation). The understanding of this control can be achieved by performing characterization of time-temperature-pressure effects on matrix morphology, on defects, porosity and fibre arrangement (alignment, orientation, fibre distribution, fibre contact). This understanding could be used further to understand the effects of processing parameters on the fibre/matrix interaction and to optimize the properties or the performance of the polymer. This project proposes to study the macro- and micro-structure of polymer composite (such as PEEK) samples which are subjected to mechanical testing at elevated temperature and loading.
AIM Describe the general aim of your project (e.g. investigate ways of catching mice).	Understand the parameters required for the manufacturing process and for the performance optimisation.
OBJECTIVES Describe how you hope to achieve the aim of the project (e.g. literature review; feasibility study, conduct interviews, write a questionnaire, working prototype, experimental plans etc....)	1. To characterise the as-received composite (by Optical and/or Scanning Electron Microscopy, DSC) in order to identify the fibre/matrix interface, matrix morphology, fibre arrangement, and any discontinuity/defects. 2. To review based on the characterisation study the potential damage mechanisms 3. To perform macro- and micro-structure study of the fractured composite to identify the potential damage mechanisms if not to confirm it. Method used includes optical and scanning electron microscopes, strain imaging camera.
PLAN (provisional) Breakdown of the objectives in a timescale.	1. Literature review of composite 2. Study the macro- and micro- structure of the as-received composite 3. Review of the potential damage mechanisms 4. Study the macro- and micro- structure of fractured composite 5. Identify the potential damage mechanisms 6. Report writing and conclusion of the project
ETHICS/ Health and Safety Are there any ethical issues raised on this project? Have you completed the fast track web review: http://ethicsreview.port.ac.uk	No

PROJECT SUPERVISOR NAME: SS **SIGNATURE** Samoras	**STUDENT NAME:** Nadine Tscheu **SIGNATURE** Nadine Tscheu

Certificate of Fast Track Ethics Review

Project Title:	Macro- and micro-study of carbon-composite
Student Number:	672817
Application Date:	03/10/2012 17:53:51

You must download your referral certificate, print a copy and keep it as a record of this review.

It is your responsibility to follow the University Code of Practice on Ethical Standards and any Department/School or professional guidelines in the conduct of your study including relevant guidelines regarding health and safety of researchers including the following:

- University Policy
- Safety on Geological Fieldwork

It is also your responsibility to follow University guidance on Data Protection Policy:

- General guidance for all data protection issues
- University Data Protection Policy

ProjectTitle:
Macro- and micro-study of carbon-composite

SchoolOrDepartment:
ENG

PrimaryRole:
UndergraduateStudent

SupervisorName:
Sarinova Simandjuntak

HumanParticipants:
No

PhysicalEcologicalDamage:
No

HistoricalOrCulturalDamage:
No

HarmToAnimal:
No

HarmfulToThirdParties:
No

Certificate Code: 9B93-2E3B-A878-D8E3-9CED-8565-31F6-6D07 Page 1 / 2

Appendix 2 Completed Interim report form

Interim Report Form

Please use block letters

NAME OF STUDENT: Nadine Tscheu
Student Number: 672817

NAME OF SUPERVISOR: Dr. Sarinova Simandjuntak

DATE: 09/01/13 **AWARD:** MB

PROJECT TITLE: Macro- and micro-study of carbon-composite

REVIEW OF PROGRESS:

- Research: composite material, PEEK, laminate theory, optical microscope, scanning electron microscope, tensile test
- Literature review: containing the research
- Work plan
- Experiment (yellow tube): tensile test, microscopy the damaged area

ACTIONS TO BE COMPLETED BEFORE END OF PROJECT PERIOD

- Experiment: microscopy the received pipe, after the tensile test, and after the relaxation test, documentation of the results/outcomes
- Report: write the experimental methodology, add the damage mechanism to the literature review, write the discussion, conclusion, and the abstract

TIMESCALE (JANUARY TO APRIL)

- January Experiment: microscopy as received, and after tensile test
- February Experiment: microscopy after relaxation test
- March Report writing: experimental methodology, results, discussion, conclusion, and abstract
- April: refine the report, prepare the poster, print the report

Signature of Supervisor:..

Appendix 3 Completed progress monitoring form

Project Monitoring Form

Student: Nadine Tscheu	Student Number: 672817
Supervisor: DR SARINOVA SIMANDJUNTAK	
Title: Macro- and micro-study of carbon-composite	
Award: MB	Year: 3

Students should consult their supervisors and keep them informed about the progress of their work.

Date	Deliverable	Comments	Supervisors Initials
Mid October	**Project Proposal including Plan**	Nadine was well prepared with Gantt Chart & plan.	[signature] 15/10
End October	Progress Review	looking at basic composite, Laminate theory, testing method	[signature] 29/10
Mid November	Progress Review	preparing report, write up on lit. review look @ laminate theory equations.	[signature] 7/11
End November	Progress Review	look @ damage mech. + preparing for microscopic study	[signature] 26/11
Early December	Progress Review	to prepare work plan and updating report. Preparation on microscopy's done.	[signature] 10/12
Mid January	**Interim/Progress Report**	Work progress is very good	[signature] 9/1-13
End January	Progress Review	Discussion on test → Flexural test	[signature] 8/2/13
Mid February	Progress Review	Performed sample prep. for SEM, finished test	[signature] 15/2-13
End February	Progress Review	Added Tg DSC test. Finished all tasks, to perform SEM	[signature] 28/2-13
Mid March	**Project Monitoring Form**	Finished SEM work, to write report	[signature] 15/3-13
End March	Progress Review	Good Progress	[signature] 27/3-13
Mid April	Progress Review	Draft report + posters are ready.	[signature] 23/4-13
Early May	**Full Report**		

Supervisor Signature

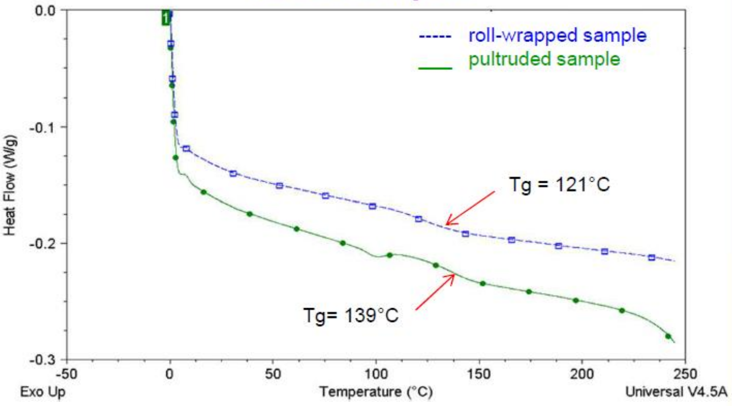
Completed on 15/3/13 [signature]

DR. S. Simandjuntak

This study investigates the potential damage mechanisms and the parameters that affect the composite structure at different temperature levels under flexural loading conditions.

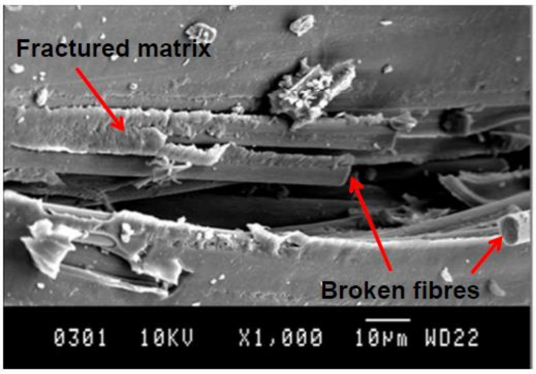
The damage mechanisms are identified by means of macro- and micro-structural studies of carbon reinforced epoxy composites.

DSC-Analysis



DSC analysis of the samples to determine the glass transition temperature (Tg)

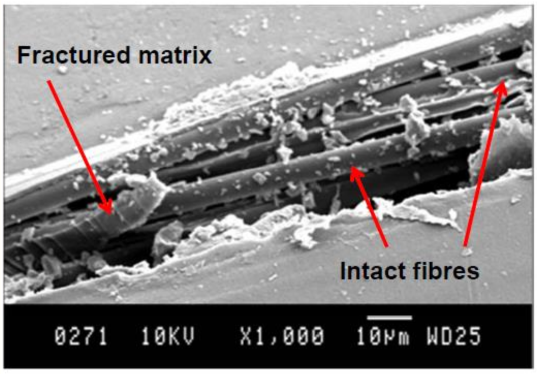
SEM-Analysis



Crack opening of the pultruded pipe at 60% of Tg (1000x magnification)

Roll-wrapped pipe

Pultruded pipe



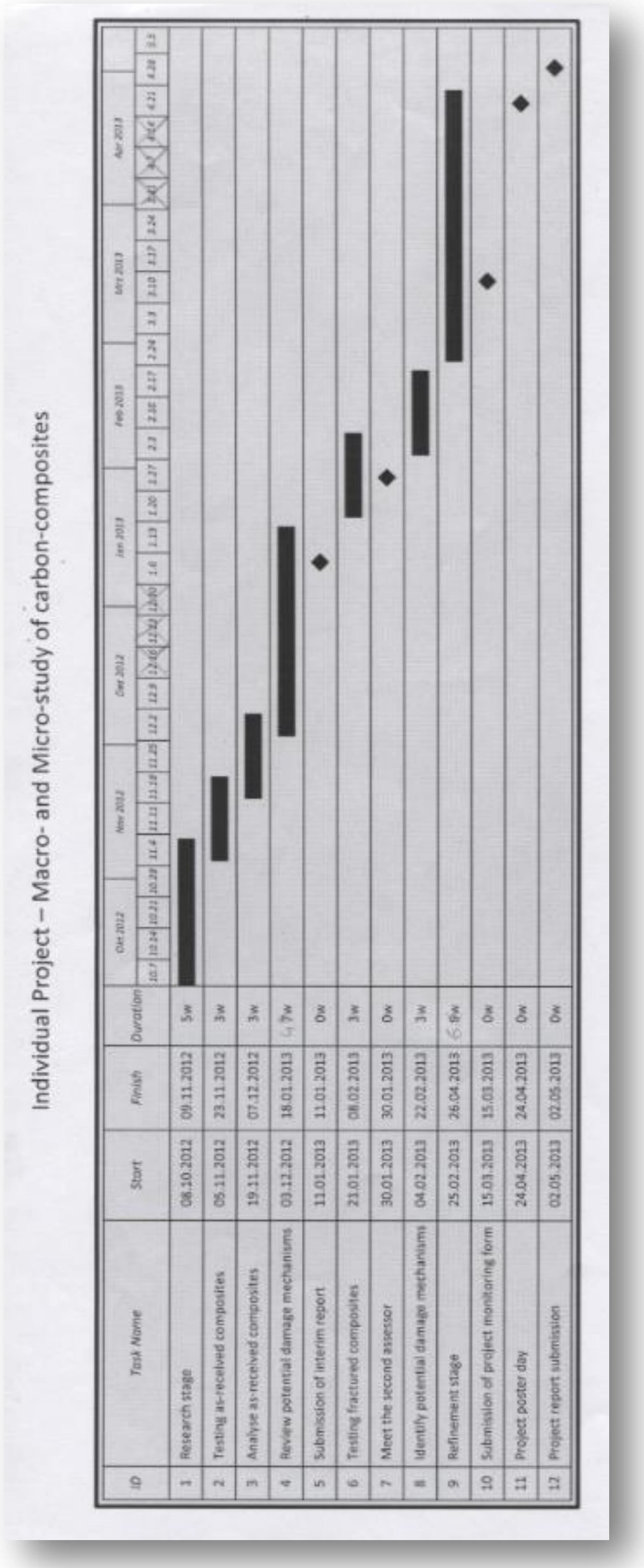
Crack opening of the roll-wrapped pipe at 60% of Tg (1000x magnification)

Conclusion:

- Failure analysis was done by using SEM
- The manufacturing process affects material properties and failure behaviour
- Temperature has an impact on the brittleness
- The pultruded pipe has higher Tg and hardness which leads to better wear resistance
- The roll wrapped pipe provides higher strength and better fracture resistance induced by internal stresses from the lay-up process of the prepregs and a good load distribution from fibre-to-fibre and ply-to-ply

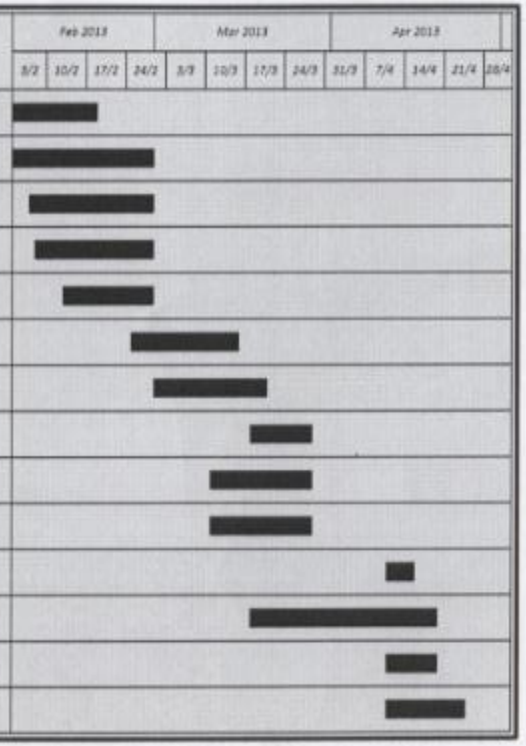
University of Portsmouth	Macro- and Micro-Study of Carbon Composite		School of Engineering
	Name: Nadine Tscheu	Course: BEng Mechanical Engineering	
	Year: 2012/13 Student No.: 672817	Supervisor: Dr Sarinova Simandjuntak	

Appendix 5 Gantt-chart



Individual Project February-April

ID	Task Name	Start	Finish	Duration
1	Improve Literature review	04/02/2013	18/02/2013	2.2w
2	Microscopy of as received pultruded	04/02/2013	28/02/2013	3.8w
3	Tensile Test pultruded	07/02/2013	28/02/2013	3.2w
4	Microscopy of damaged pultruded	08/02/2013	28/02/2013	3w
5	Microscopy of as received roll wrapped	13/02/2013	28/02/2013	2.4w
6	Tensile Test roll wrapped	25/02/2013	15/03/2013	3w
7	Microscopy of damaged roll wrapped	01/03/2013	20/03/2013	2.8w
8	SEM Analysis	18/03/2013	28/03/2013	1.8w
9	Documentation of Tensile Test	11/03/2013	28/03/2013	2.8w
10	Documentation of Microscopy	11/03/2013	28/03/2013	2.8w
11	Improve Methodology	11/04/2013	15/04/2013	.6w
12	Analyse Results	18/03/2013	19/04/2013	5w
13	Conclusion	11/04/2013	19/04/2013	1.4w
14	Improve Report	11/04/2013	24/04/2013	2w



Appendix 6 Pultruded pipe data-sheet

Product Description

Pultruded carbon fibre round tube manufactured from 100% unidirectional carbon fibre.

Dimensions

The tube has an outer diameter (OD) of 10mm and a wall thickness of 1mm leaving an inner diameter (ID) of 8mm.

This tube weighs 42g/m.

Length

Our pulltruded rods are now available in a 1m or 2m length. Please choose the length of rod you require from the drop-down list at the top of the page. 2m lengths are charged at a slight premium (owing to th handling difficulties) so only buy them if you need the extra length.

Please note that due to restrictions imposed by the courier companies it is not possible to ship 2m lengths to some international destinations. If you have a requirement for a length of rod that is over 1m but less than 2m then it might be possible for us to cut the rod to length and therefore ship it to your destination. If this is the situation, please contact us.

Suggested Uses

This 1mm thick medium diameter 10mm tube is ideally suited for use in radio controlled (RC) aircraft, helicopters, U.A.V's and other models but would be equally suited to other precision applications like robotics and automation.

Technical Specification

Property	Direction	Units	Test Method	Value
Tensile Strength	Mpa	Length-ways	ASTM D638/GB1447-83	400-500
	Mpa	Width-ways	ASTM D638/GB1447-83	18-30
Tensile Modulus	Gpa	Length-ways	ASTM D638/GB1447-83	28-40
	Gpa	Width-ways	ASTM D638/GB1447-83	8-12
Flexural Strength	Mpa	Length-ways	ASTM D790/GB1449-83	250-400
	Mpa	Width-ways	ASTM D790/GB1449-83	80-150
Flexural Modulus	Gpa	Length-ways	ASTM D790/GB1449-83	20-30
	Gpa	Width-ways	ASTM D790/GB1449-83	10-15
Compressive Strength	Mpa	Length-ways	ASTM D695/GB1448-83	200-320
	Mpa	Width-ways	ASTM D695/GB1448-83	60-100
Compressive Modulus	Gpa	Length-ways	ASTM D695/GB1448-83	10-20
	Gpa	Width-ways	ASTM D695/GB1448-83	8-20
Short beam shearing strength	Mpa	Length-ways	ASTM D2344	30-40
Barcol Hardness			ASTM D2583	25-45
Relative Density	g/cm3		ASTM D792	1.30-1.50

Table shows typical empiracle values for Easy Composites' carbon fibre pulltruded tubes, strips and box sections.

Pultrusion Explained

Pultruded tubes are the most commonly used type of carbon fibre tube. Pultrusion is a method of manufacturing continuous lengths of fibre reinforced section (in this case tube) by pulling continious strands of carbon fibre through resin and a former before curing the resin all in one process.

Pultruded tubes feature 100% longitudinal fibre orientation which gives them excellent strength along their length but does make them vulnerable to crushing forces. For applications where none-uniform forces are likely to be encountered, including crushing forces, roll wrapped carbon fibre tubes should be considered as an alternative.

The pultrusion process allows incredible tiny diameter tubes to be produced (as small as 2mm OD).

Appendix 7 Roll-wrapped pipe data-sheet

Product Description

Carbon fibre tube can be used to replace or substitute aluminium or steel tubes in a wide range of projects. Their superior mechanical properties mean that tubes of the same weight as an aluminium or steel tube can be much stronger, or that tubes of the same strength can be much lighter.

This tube is manufactured predominantly using high modulus (T700) unidirectional pre-preg carbon fibre oriented to provide maximum strength in the lateral (length-ways) axis but the use of pre-preg reinforcement oriented at 90° also ensures that the tube has good crush-strength; ideal for real-world applications.

The use of unidirectional fibres and maximum strength in the lateral axis does mean that the tube is not strong across its diameter as it is along its length so should be used in such a way as to avoid unnecessary crushing forces across the tube.

Roll Wrapping

The phrase 'roll wrapped' refers to the process used to manufacture these tubes. The pre-preg carbon fibre is first laid up around a mandrel in multiple layers. Once the reinforcement is in place it is spiral wrapped with heat-shrink tape (this is what gives roll-wrapped tubes their characteristic ribbed appearance) before the whole assembly is oven cured.

Roll wrapped carbon fibre tubes offer superior strength in general use to 'pulltruded' tubes (which have all their fibres running in exactly the same direction). This is because forces on a tube are rarely exclusively in straight compression or tension, whereby the unidirectional fibres of a pulltruded tube are not ideal.

Mechanical Properties & Specification

The tube is available the following lengths: 500mm, 1000mm, 1500mm, 2000mm and 2500mm. Please choose the length you require from the drop-down list.

Internal Diameter (ID, mm)	Wall Thickness (mm)	Outer Diameter (OD, mm)	Stiffness KNm2	Weight Kg/m
8.00	1.00	10.0	0.03	0.05

Composition

Our roll wrapped carbon fibre tubes are manufactured from special high-modulus Toray T700 unidirectional pre-preg carbon fibre oriented at 0° (down the length of the tube). Crush/burst strength is provided by unidirectional E-Glass oriented at 90° (around the section of the tube).

Layup schedule

[0, 90, 0, 90, 0]

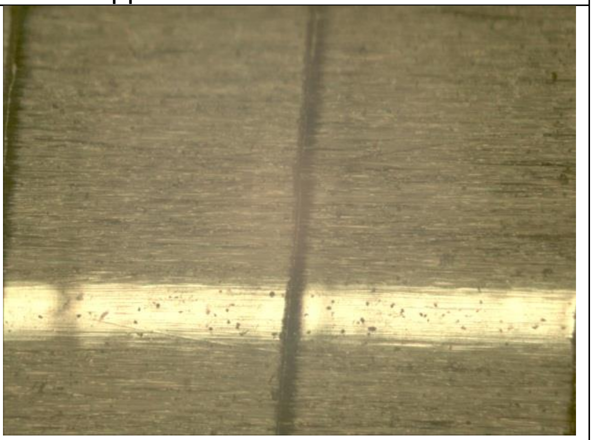
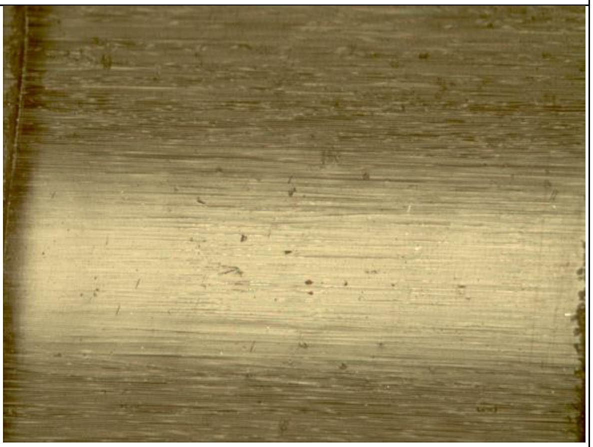
0° layers are 300gsm Toray T700
90° layers are 300gsm E-Glass ~UD (80/20)

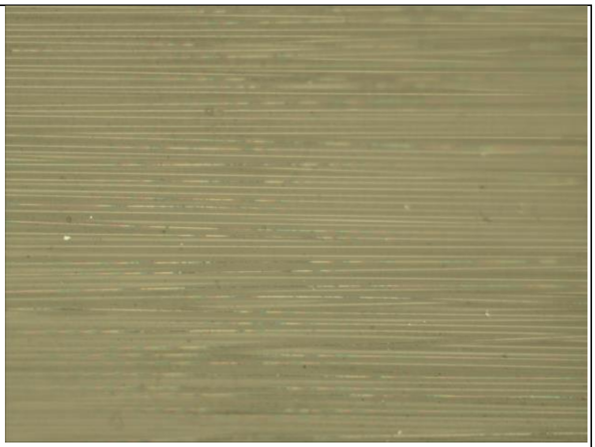
Comparison with Steel and Aluminium Tube

Property	CF Fabric (50% at 0°/50% at 90°)	CF Uni-Directional	Steel	Aluminium
Density (g/cc)	1.60	1.60	8.0	2.7
Youngs Modulus 0°(GPa)	70	135	207	72
Youngs Modulus 90°(GPa)	70	10	207	72
Ult. tensile Strength 0°(MPa)	600	1500	990	460
Ult. comp. Strength 0°(MPa)	570	1200	990	460
Ult. tensile Strength 90°(MPa)	600	50	990	460
Ult. comp. Strength 90°(MPa)	570	250	990	460

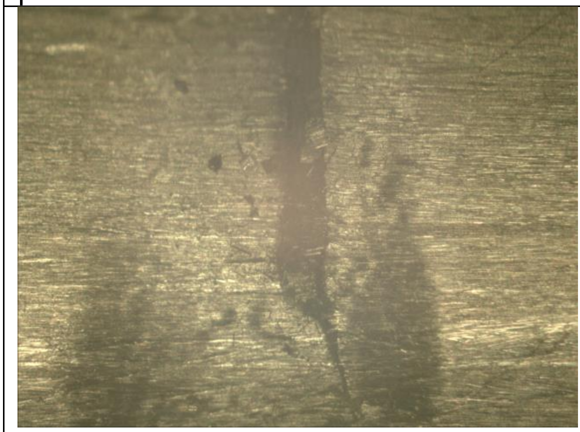
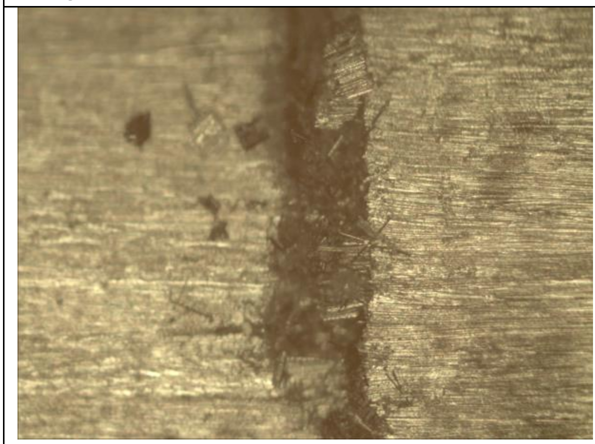
Appendix 8 Optical microscopy pictures

As-received:

Pultruded	Roll-wrapped
	 X2.5
 X5	 X5
 X10	

 X20	 X20

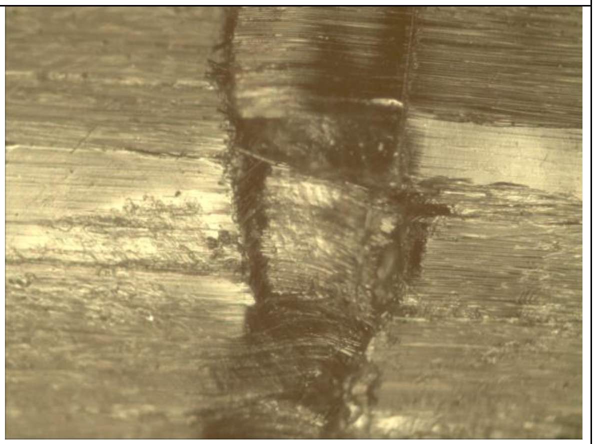
After three point bending (at ambient temperature)

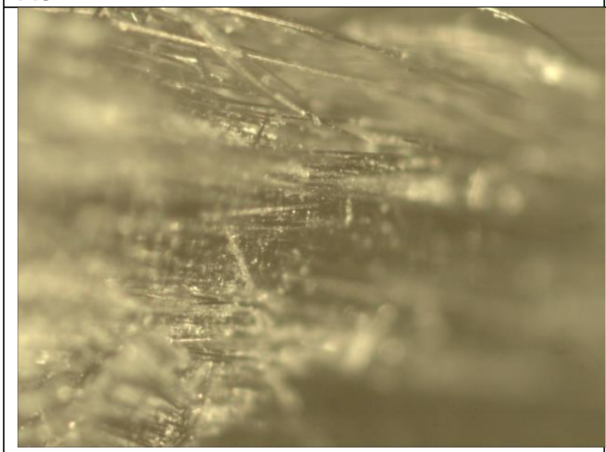
pultruded	Roll wrapped
 X2.5	
 X5	 X5

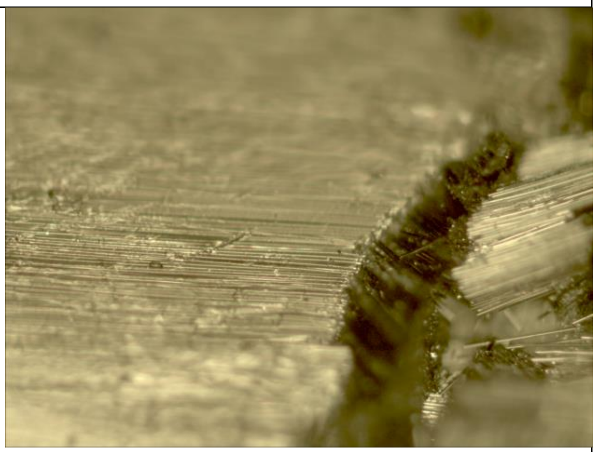

X5

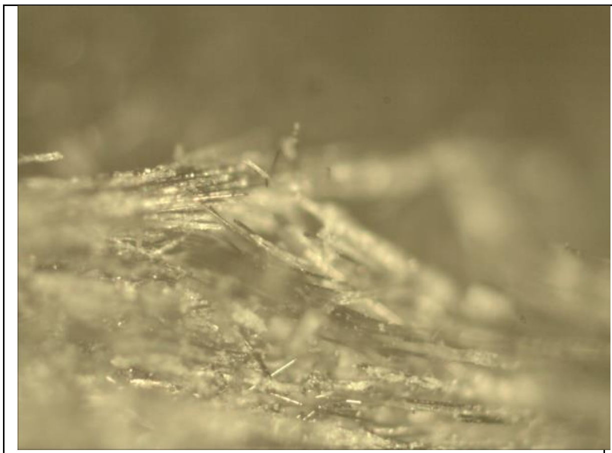
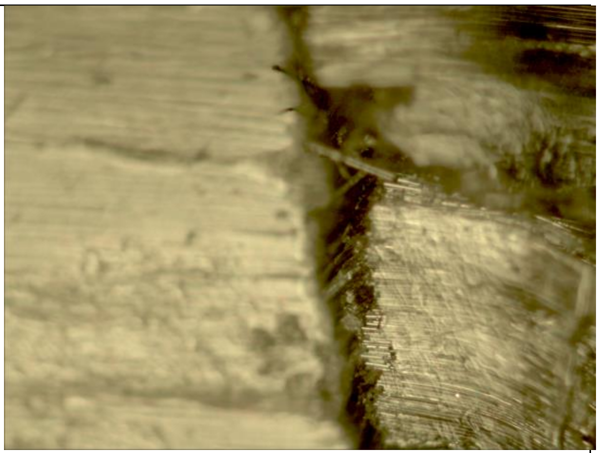
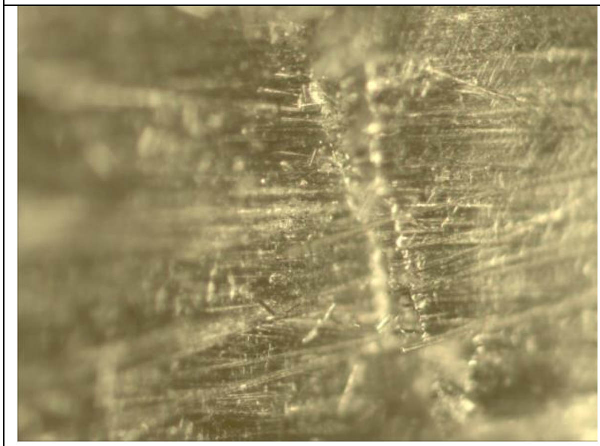
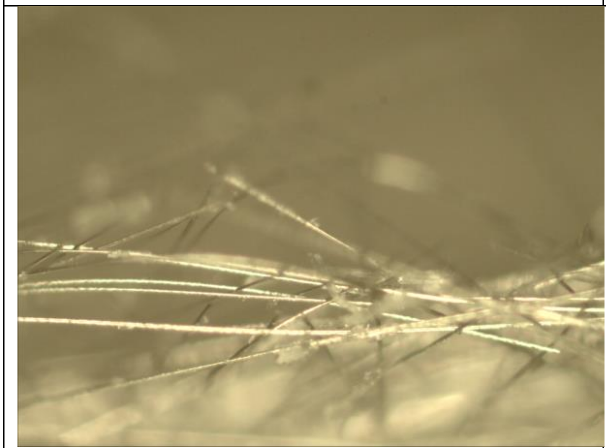

X5

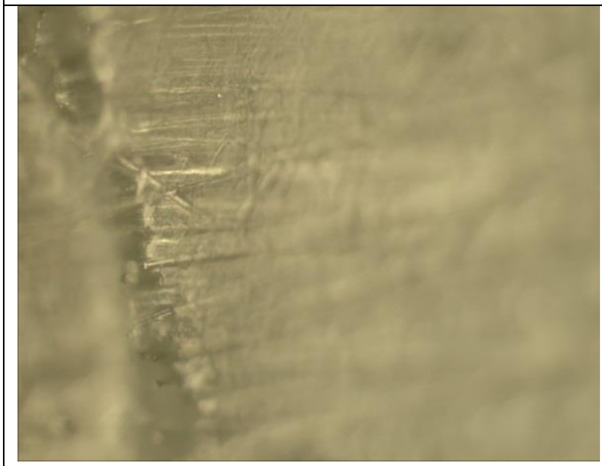
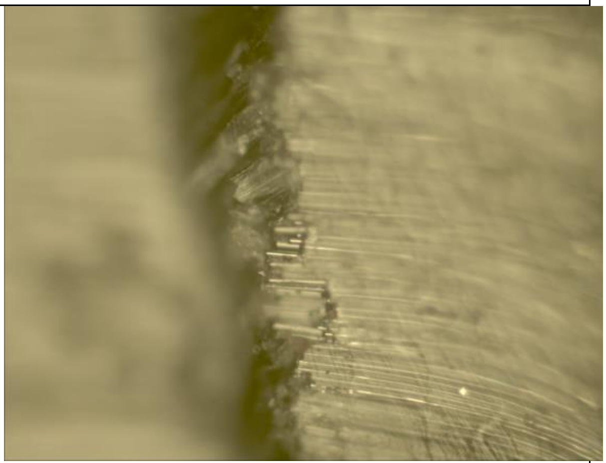

X5


X5


X10


X 10

 X10	 X10
 X10	
 X10	

 X10	
 X20	 X20

Appendix 9 Data of three point bending test

Specimen 1		Specimen 2		Specimen 3		Specimen 4	
Deformation	Standard force	Deformation	Standard force	Deformation	Standard force	Deformation	Standard force
mm	N	mm	N	mm	N	mm	N
0.000799	8.815891	0.00893	13.52072	0.003745	13.72352	0.008685	16.83771
0.047637	16.05302	0.142356	46.13577	0.08749	26.95993	0.14118	36.27114
0.132542	28.58103	0.308265	84.17946	0.20428	38.96448	0.313896	73.54721
0.218571	51.30065	0.464148	136.1119	0.318334	62.26789	0.486568	109.4071
0.305238	77.32004	0.629387	184.6536	0.430643	97.39926	0.659292	138.8307
0.391947	103.4781	0.805021	225.7116	0.533566	132.6833	0.831983	161.0942
0.477739	129.1759	0.980782	258.6423	0.633683	167.9458	1.004669	180.6048
0.560271	154.9246	1.144939	274.9221	0.740749	203.2251	1.177392	193.4432
0.624764	180.7953	1.320002	294.7961	0.852434	237.5652	1.350063	204.2303
0.687532	205.9483	1.483043	297.8668	0.967074	271.1914	1.522763	207.5919
0.751814	228.7544	1.649105	285.2646	1.081473	303.296	1.695224	210.4897
0.81762	238.8402	1.823141	298.1016	1.196706	332.6889	1.867381	206.9636
0.886079	252.479	1.980454	293.4192	1.313776	359.6483	2.040069	204.4953
0.957829	269.9296	2.156268	325.3944	1.431262	381.9877	2.212754	194.9075
1.036509	285.8966	2.308499	334.4491	1.548789	400.1719	2.385489	192.5754
1.121593	297.9362	2.422801	306.5509	1.666333	412.829	2.558136	192.4759
1.199962	298.4357	2.558472	282.1178	1.783874	428.4003	2.730856	194.9461
1.284675	313.0679	2.732522	295.0218	1.901353	442.4082	2.903574	196.6775
1.371369	317.3255	2.900321	306.7887	2.018897	453.6566	3.076224	196.4842
1.458066	317.7207	3.073811	316.5028	2.136434	465.7408	3.248951	194.6135
1.543557	319.9925	3.231189	306.8189	2.253972	477.7715	3.420259	188.09

1.625509	318.1424	3.374222	264.7531	2.371464	486.2038	3.59276	186.0135
1.7122	291.882	3.550035	269.4027	2.488995	490.4928	3.765474	183.977
1.797919	282.2941	3.725836	263.0012	2.60654	488.9194	3.938151	183.073
1.88322	279.6267	3.90162	261.3146	2.723977	482.6728	4.110851	181.42
1.969898	275.7816	4.068819	252.8628	2.833279	464.3959	4.283565	181.899
2.052727	276.5748	4.229834	240.2196	2.950371	453.8923	4.454618	179.5183
2.090982	276.3605	4.404085	244.3686	3.067895	448.9234	4.606356	156.7184
2.168856	269.2922	4.579854	243.8752	3.185405	450.0123	4.773485	145.2767
2.255572	268.4594	4.755628	240.8032	3.302926	451.3531	4.946195	140.5338
2.342287	269.2312	4.931148	235.0139	3.420452	451.1728	5.118857	139.211
2.42891	270.9965	5.103252	220.4775	3.538001	449.5811	5.291592	138.6572
2.515599	274.1754	5.279066	212.6837	3.655513	446.4353	5.46428	138.2851
2.590395	276.1548	5.454881	208.0179	3.77302	440.3014	5.636948	136.7109
2.676317	274.4699	5.615257	182.9111	3.890554	433.8102	5.80967	133.7362
2.763013	273.6907	5.772329	161.4902	4.008091	424.6289	5.98236	129.4589
2.849708	267.8057	5.948155	166.0793	4.125634	412.167	6.155046	124.6273
2.93639	260.5245	6.123968	165.7454	4.243105	404.324	6.327763	119.9618
3.023127	258.8092	6.299722	163.1373	4.360654	398.1379	6.500447	115.4394
3.109756	259.4613	6.475548	158.9812	4.476966	390.2004	6.673146	113.1625
3.196432	262.5513	6.651345	156.4117	4.59334	381.9678	6.845847	112.0246
3.282776	264.9776	6.827136	153.214	4.710836	373.4885	7.01855	109.9057
3.369497	267.2953	6.996652	141.9782	4.828358	363.5001	7.179524	99.863
3.456114	273.479	7.169223	137.2582	4.945904	356.0415	7.344843	94.12289
3.54284	279.4383	7.345006	134.463	5.063416	349.2769	7.512055	87.53217
3.629533	281.7619	7.520827	131.7121	5.180946	341.6151	7.684747	87.37653
3.716225	284.7012	7.696615	129.1776	5.298452	335.7522	7.85742	86.60009
3.80291	291.7542	7.872417	125.9959	5.415987	331.324	8.030153	85.73991
3.889642	294.0337	8.048217	123.0249	5.533517	325.073	8.202829	84.89452

3.975632	292.5368	8.224026	119.9814	5.651044	317.3995	8.375527	83.49466
4.061805	296.2844	8.399812	117.0351	5.768541	311.3247	8.54825	82.36375
4.148473	299.3263	8.575631	114.1859	5.886071	307.0671	8.720916	81.39741
4.235191	304.37	8.751413	111.5031	6.003606	302.7497	8.893635	79.37519
4.321897	310.0208	8.927201	109.0961	6.12115	298.1889	9.066339	77.14335
4.407583	305.659	9.103035	105.9035	6.238623	294.1678	9.239015	75.86601
4.494047	285.4491	9.278786	103.3989	6.35616	291.0908	9.411721	74.26189
4.57629	238.9586	9.454612	99.25762	6.473707	289.0117	9.584429	73.20751
4.65434	227.697	9.630419	95.74889	6.591233	286.9871	9.757116	72.03617
4.738578	228.393	9.806182	93.74642	6.708742	284.7218	9.929803	71.12273
4.819304	230.0098	9.982008	92.21176	6.826254	283.0932	10.10252	70.36797
4.900647	228.7619	10.15781	90.55735	6.943804	281.3175	10.2752	69.81256
4.960552	227.7384	10.33359	88.4496	7.061326	278.6551	10.44789	69.01889
5.029711	227.9935	10.5094	85.56489	7.178846	276.192	10.62061	68.31782
5.116183	228.2068	10.68521	83.64669	7.296371	273.0308	10.79328	67.79836
5.202891	225.0899	10.86099	82.15833	7.413895	270.8538	10.96599	67.22421
5.289589	222.8216	11.03681	81.12101	7.531434	268.6394	11.13871	66.60635
5.3763	223.9332	11.21261	80.35616	7.648968	266.0431	11.31138	66.11107
5.462919	223.4898	11.38838	79.82613	7.766463	263.4644	11.4841	65.32849
5.549655	221.6772	11.56422	79.33595	7.883984	260.451	11.65679	64.85819
5.636354	219.6051	11.73999	78.94992	8.001526	257.2551	11.82949	64.48737
5.723033	214.6872	11.9158	78.61291	8.119063	254.7263	12.0022	63.96454
5.809726	214.3972	12.09161	78.61181	8.236546	251.9158	12.17488	63.63384
5.896453	214.4604	12.26739	78.4756	8.354069	249.4213	12.34759	63.28044
5.983088	214.1068	12.4432	78.47471	8.471625	246.8873	12.52028	63.00927
6.067659	213.357	12.61753	77.08172	8.589131	243.9077	12.69299	62.70662
6.149748	201.1384	12.79046	73.9251	8.706639	240.8823	12.86567	62.47894
6.23643	198.1397	12.96621	73.16692	8.824171	238.4997	13.03838	62.29897

6.323115	195.8987	13.14203	72.78731	8.9417	236.4211	13.21108	62.1417
6.409839	191.2127	13.31783	71.80385	9.059228	234.5158	13.38376	61.92345
6.495981	188.2088	13.49361	72.01207	9.176753	232.0913	13.55647	61.90098
6.564902	183.0398	13.66942	72.38884	9.29427	229.0823	13.72915	61.89695
6.650827	159.7747	13.84522	72.9154	9.411784	226.2302	13.90185	61.90281
6.737561	159.8995	14.02101	73.11928	9.529323	222.97	14.07457	62.13191
6.824245	159.0233	14.19682	73.38911	9.646866	219.6518	14.24724	62.20914
6.910931	158.1314	14.37262	73.60262	9.764356	216.5216	14.41995	62.51273
6.996954	156.8922	14.5484	73.78141	9.881876	213.172	14.59267	62.80112
7.054454	155.5912	14.72422	74.23552	9.999432	210.0304	14.76533	63.01896
7.138075	154.1056	14.90001	74.94823	10.11696	207.1876	14.93806	63.22803
7.224794	152.3258	15.0758	75.93652	10.23445	204.5275	15.11076	63.55482
7.311458	150.6748	15.25163	76.99668	10.35198	202.4566	15.28344	63.88207
7.398147	149.3451	15.4274	78.21382	10.46953	199.9865	15.45615	64.21634
7.48483	148.0377	15.60322	79.41284	10.58704	197.1921	15.62886	64.49077
7.571555	146.8153	15.77902	80.45662	10.70456	194.7538	15.80153	64.54312
7.658254	145.3862	15.95481	81.37645	10.82208	192.638	15.97423	64.39042
7.74491	142.2349	16.13061	82.10632	10.93961	190.8661	16.14694	63.92577
		16.30642	82.78845	11.05714	189.2969	16.31962	63.66222
		16.48219	83.25082	11.17467	188.1116	16.49231	63.55163
		16.658	82.78658	11.29217	186.9017	16.66503	63.65025
		16.83381	83.06599	11.40831	185.3448	16.8377	64.05075
		9.003056	83.62823	6.245793	179.812	8.837634	64.17549

Appendix 10 Three point bending test report

18.02.13

Zwick / Roell

Test report

Customer	:	Specimen type	:
Job no.	:	Pre-treatment	:
Test standard	:	Tester	:
Type and designation of Material	:	Notes...	:
		Machine data	:
Specimen removal	:		

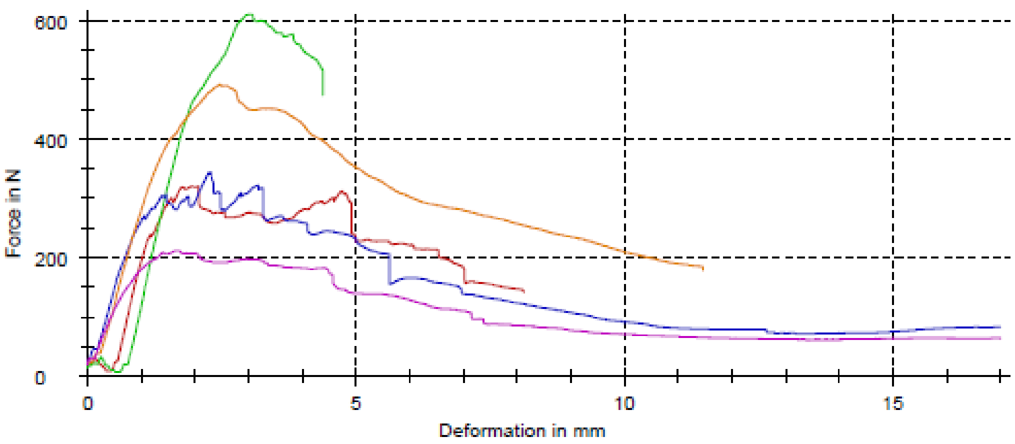
Pre-load : 20 N
Speed, E-Modulus : 2 mm/min
Test speed : 2 mm/min

Test results:

Nr	E_{mod} GPa	F at 0.1% plastic deformation N	F at 0.2% plastic deformation N	Upper yield point N	dl at upper yield point mm
1	26.3	315	320	321	2.0
2	39.0	481	521	-	-
3	19.7	292	281	344	2.3
4	27.3	412	453	492	2.5
5	13.7	209	208	212	1.7

Nr	F_{max} N	dL at F_{max} mm	F_{Break} N	dL at break mm	d_a mm	d_i mm	S_0 mm²
1	321	2.0	-	-	10	8	28.27
2	611	3.0	-	-	10	8	28.27
3	84.0	17.0	-	-	10	8	28.27
4	492	2.5	-	-	10	8	28.27
5	64.7	15.9	-	-	10	8	28.27

Series graph:



18.02.13

Zwick / Roell

Statistics:

Series n = 5	E_{mod} GPa	F at 0.1% plastic deformation N	F at 0.2% plastic deformation N	Upper yield point N	dl at upper yield point mm
$\bar{x}$	25.2	342	356	342	2.1
s	9.45	106	128	115	0.4
ν	37.51	31.09	35.89	33.70	16.82

Series n = 5	F_{max} N	dL at F_{max} mm	F_{Break} N	dL at break mm	d_a mm	d_i mm	S_0 mm²
$\bar{x}$	314	8.1	-	-	10	8	28.27
s	242	7.7	-	-	0.000	0.000	0.00
ν	77.05	95.06	-	-	0.00	0.00	0.00