Feasibility Study of the Vacuum Infusion Process for Polyurethane Glass Fibre Laminates

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Challenge the future

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Feasibility Study of the Vacuum Infusion Process for Polyurethane Glass Fibre Laminates

Master of Science Thesis

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Abstract

The aim of this thesis is to investigate the feasibility of manufacturing polyurethane glass fibre laminates with vacuum infusion. Thermoset resins classically used for infusion have been polyesters, vinyl esters and epoxies due to their long pot life. Polyurethanes have faster reaction kinetics and therefore the cycle time could be reduced. Additionally, it is expected that polyurethanes bring improved fatigue and impact resistance properties which would enable a re-design of the structures, reducing its thickness. Therefore, the manufacturing costs of large structures, such as wind turbine blades, could be reduced.

Infusion experiments are carried out to research the most appropriate infusion lay-up and procedure for a thermoset polyurethane vacuum infusion process in a lab scale environment. Additionally, polyurethane clear castings are produced to research the behaviour of the neat resin. The quality of these laminates and clear castings are proven by non-destructive inspection and mechanical testing. It is desired by the industry that the polyurethane laminates and clear castings have at least similar mechanical properties as epoxy laminates and clear castings. Therefore, the polyurethane test results are compared with data of epoxy glass fibre laminates and clear castings which are produced simultaneously.

Polyurethane laminates are produced with an experimental formulation which is not fulfilling the main requirements regarding a viscosity lower than 350 cP for the first 45 minutes and a glass transition temperature of 70°C after 6 hours of cure at 70°C. Laminates are produced without visually apparent defects. Ultrasonic inspection shows defects which are confirmed with optical microscopy. These defects are voids and an irregular rough surface at the vacuum bag side of the laminates.

Laminates 25 and 26 are both polyurethane biaxial glass fibre laminates. Laminate 1E is the epoxy reference produced from the same glass fibre. Specimens of these laminates are tested in shear to obtain information about the fibre-matrix behaviour under in-plane shear forces. Specimens of laminates 25, 26 and 1E have a shear modulus of respectively 7556 MPa, 7594 MPa and 7810 MPa. Two polyurethane unidirectional glass fibre laminates (number 29 and 32) and one epoxy unidirectional glass fibre laminate (number 2E) are tested in tensile direction perpendicular to the fibres to obtain knowledge about the fibre-matrix behaviour. Additionally, these laminates are tested in interlaminar shear and give a measure of the strength of the bond between fibres and matrix. Laminates 29, 32 and 2E have a tensile strength of respectively 77 MPa, 79 MPa and 79 MPa, a tensile strain of respectively 0.94 %, 0.90 % and 2.22 % and a tensile modulus of 9706 MPa, 11521 MPa and 11481 MPa. The interlaminar shear modulus of laminates 29, 32 and 2E is respectively 52 MPa, 51 MPa and 47 MPa. Thus it can be concluded that polyurethane biaxial glass fibre laminates have a comparable quality as well as the polyurethane unidirectional glass fibre laminates. There is an indication of higher fibre-matrix bonding of the glass fibres to the epoxy.

Polyurethane and epoxy neat resins specimens are tested in tensile a tensile test and a flexural test for specification of matrix data. The tensile properties of the polyurethane, epoxy and reference epoxy have a tensile strength of respectively 52 MPa, 67 MPa and 67 MPa, a tensile strain to failure of 9.7 % and 11.6 % and 4 % and a tensile modulus of respectively 1002 MPa, 1212 MPa and 3000 MPa. The mechanical properties obtained from the flexural test are for the polyurethane, epoxy and reference respectively 104 MPa, 119MPa and 105 MPa, the flexural strain at flexural strength are respectively 5.4% and 6.3% (no reference available) and the flexural modulus are respectively 2877 MPa, 3124

MPa and 3000 MPa. The polyurethane neat resin specimens show tougher behaviour compared with epoxy neat resin specimens.

Currently the vacuum infusion process with polyurethanes is not feasible for the production of real applications. Although the mechanical properties of polyurethanes are promising, further research is needed to obtain a production method to produce laminates with a consistent quality.

Table of Contents

List of Figures

List of Tables

Preface and Acknowledgments

This report presents the master thesis work, which is part of the Master of Science program Aerospace Materials and Structures within the profile Design and Production of Composite Materials of the faculty of Aerospace Engineering at Delft University of Technology in the Netherlands. The research has been carried out between September 2013 and September 2014 at Dow Europe GmbH in Freienbach, Switzerland.

The report describes the research on the feasibility of the vacuum infusion process for polyurethane-glass fibre structures.

I would like to thank the following staff members: Dr. ir. Otto Bergsma for establishing contact with Dow Europe GmbH, for supervising, critiquing the process and guiding me through the whole process of the master thesis, and Dr. Irene Fernandez Villegas for supervising, supporting and critiquing during the last months of my thesis.

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J. M. Brander

"Never be limited by other people's limited imaginations. If you adopt their attitudes, then the possibility won't exist because you'll have already shut it out.... You can hear other people's wisdom, but you've got to re-evaluate the world for yourself."

-Mae Jemison

Chapter 1

Introduction

1.1 Background

The production of large composites structures is made today almost exclusively by the vacuum infusion technique. Vacuum infusion is a low cost process, particularly suitable for low volume production of large parts [2]. The completion of the impregnation of the reinforcement before resin gelation remains a major challenge [3]. In order to ensure a good impregnation of the reinforcement lay-up, it is required to keep a low viscosity of the reaction mix for an extended amount of time [4]. Thermoset resins classically used for infusion have been polyesters, vinyl esters and epoxies due to their long pot lifes [4, 5].

The industry is continuously looking for ways of reducing manufacturing cost. Specifically the wind power industry needs to keep reducing the wind blade manufacturing cost to be competitive with traditional ways of generating energy [6]. To bring wind power cost/kWh to grid parity versus fossil fuel power, the wind industry needs to develop longer and lighter blades, while simultaneously reducing the total blade costs by more than 10 %. This dual goal is best achieved by radically reducing the bill of materials and drastically increasing the productivity of the blade manufacturing process. Therefore, there is a need in the wind market of a new chemistry, which combines the demand for low cost materials, having very low viscosity, short de-mould times and a low exothermal peak. Thermoset polyurethane is considered as a new chemistry being able to fulfil the previous mentioned requirements.

It is hypothesized that the use of polyurethane in the vacuum infusion process for wind turbine blade production could enable a reduction of approximately 7% blade cost [7]. Additionally, it is expected that polyurethanes bring better fatigue and impact resistance properties versus current resins which would enable a re-design of the structures, reducing its thickness. This would result in a reduction of materials and process times and therefore in an entire reduction of costs.

1.2 Problem Definition

Dow made a proof of concept project to research polyurethanes for the vacuum infusion process. Part of the proof of concept is the development of a polyurethane formulation suitable for the vacuum infusion technique. Additionally, before polyurethane infusion can be applied to real applications such as wind turbine blades, it needs to be tested in a lab scale set-up.

Polyurethanes form a family of polymers which are different from other plastics from the point of view that an urethane monomer does not exist [8]. The polymers are in general created during the manufacture of a particular object [8]. Polyurethanes are produced by the polyaddition reaction between alcohols with two or more reactive hydroxyl (-OH) groups per molecule (diols, triols, polyols) and isocyanates that have more than one reactive isocyanate group (-NCO) per molecule (diisocyanates, polyisocyanates) [8-10].

Commercial polyurethanes have a faster polymerisation compared with the conventional used resins and therefore the pot life for infusing is much shorter. The challenge for Dow is to develop a polyurethane formulation which has a low enough initial viscosity and low reactivity profile to permit the infusion of large structures before reaching a maximum level of viscosity.

Polyurethane systems with low viscosities and long gel times have been developed [5]. The viscosity versus the reaction time for this (soy) polyurethane system is compared with epoxy and vinyl ester and is given in Figure 1-1. It shows that the polyurethane system has a lower viscosity for the first 30 minutes of infusion, with a marked drop in viscosity following mixing due to reduction in H-bonding, which explains the faster infusion rates through glass and carbon fibres [5].

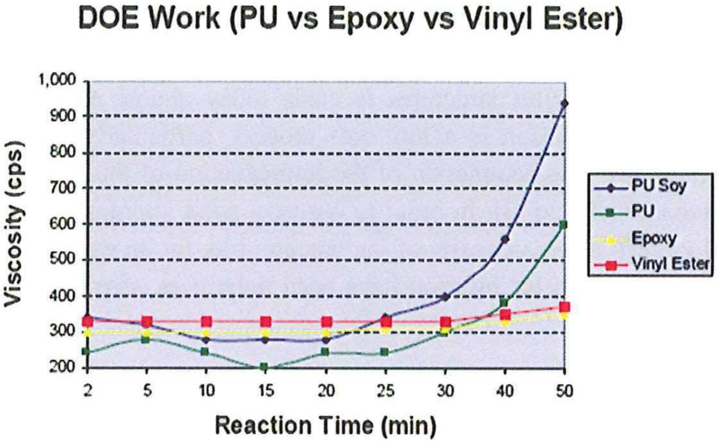


Figure 1-1: Viscosity rise over time of (soy) polyurethane compared with epoxy and vinyl ester currently used in wind turbine blade manufacturing [5].

Another requirement of the polyurethane formulations is that it should build enough cross-linking to be able to achieve the minimum required glass transition temperature.

A recurrent issue on the infusion of polyurethanes is the formation of bubbles that generates voids in the composite structures after gelling. Bubbles could, for example, be formed from entrapped air in the polyurethane and from CO_2 formation due to moisture presence in the reinforcement lay-up of mould substrate. The main causes for void formation are generally due to variations in permeability on a filament and filament bundle scale, outgassing of dissolved gas in the resin, evaporation of volatile components in the resin, shrinkage of the resin and leakage in connections and mould [11]. The void content can be decreased by properly degassing the resin. This leads to a reduced risk of outgassing the resin and an increased capability of dissolving bubbles in the resin that are formed during resin flow [12]. The reinforcement and other materials used in the configuration can be pre-dried before using it in the vacuum infusion process. This removes the moisture from the materials and therefore preventing foaming.

In the industry, soy-based polyurethane and polyurethane with a linear shrinkage of 1.3% are available [5]. Whereas epoxy and vinyl ester have a linear shrinkage of respectively 3.0% and 3.4% [5]. Thus polyurethane shows low linear shrinkage compared to epoxy and vinyl ester. This allows the polyurethane to undergo fast temperature cure without the danger of cracking. Therefore the shrinkage of the resin is unlikely in causing voids in the laminates.

For the polyurethane formulation under development by Dow, there is also a need to conduct a thorough infusion processing performance evaluation and a complete as possible mechanical characterization. The polyurethane formulation described before adheres well to the glass fibres, resulting in an excellent fibre-matrix bond [13]. This contributes to the outstanding mechanical and dynamic properties of the polyurethane composite [13]. The polyurethane formulation exhibits outstanding tensile, compressive and flexural strength, as well as excellent compressive modulus of elasticity and tensile modulus of elasticity in the direction of and perpendicular to the fibre compared with epoxy and vinyl ester resins [5, 13]. This gives an indication that the polyurethane under development by Dow has the ability to have similar mechanical properties as epoxy.

It is hypothesized that void free polyurethane glass fibres can be produced using the vacuum infusion technique. The definition of void free in this specific case is that no voids are visible by visual inspection. Void free laminates are important for the wind turbine blades because voids have an undesirable effect on mechanical-, dielectric properties and surface finish [12]. In order to prove the hypothesis, a research question is defined in section 1.3.

1.3 Research Question, Main Objectives and Sub-goals

The research question is defined as follows:

- What is the most appropriate infusion lay-up and procedure for a thermoset polyurethane vacuum infusion process in a lab scale environment in order to produce defect free laminates with comparable mechanical properties as an epoxy laminate?

The main objectives of the master thesis are:

- The development of a lab scale vacuum infusion process method to discriminate the performance of the different resins;
- The production of defect free laminates;
- The proposition and conduction of a mechanical characterization using an epoxy laminate as control;

In order to solve the research question and to reach the main objectives, the following sub-questions are defined:

- What is the suitable degassing procedure for the components?
- What is the appropriate pre-treatment of the lay-up and substrates?
- What are the key mechanical tests to be performed?
- What is the mechanical response of polyurethane and epoxy based laminates and clear castings?

1.4 Structure of the Report

Chapter 2 describes the requirements of the polyurethane formulation. The methodology is given in Chapter 3. Chapter 4 presents the results and discussion of the neat resin and the vacuum infusions. Chapter 5 describes the test results and discussion of the mechanical testing. Chapter 6 gives the conclusions and recommendations. The report ends with the reference list in Chapter 7 followed by the appendices.

Chapter 2

Polyurethane Requirements

The polyurethane should fulfil the requirements that are defined by Dow in agreement with the wind industry. These requirements are based on the current situation used for producing wind turbine blades with the vacuum infusion technique. The requirements are specified as follows:

Waarom

1. The initial polyurethane viscosity is lower than 150 cP at 25° C.
2. The pot life is defined as the time for the polyurethane to reach 350 cP at 25°C. The desired pot life is at least 45 minutes.
3. The polyurethane has a glass transition temperature higher than 70°C after 6 hours of curing at 70°C.

Additional requirements regarding the polyurethane formulation are:

- No foaming
- Easy to degas
- A water pick-up similar to epoxy
- Low exothermal reaction < 120°C
- Mechanical properties are comparable to epoxy

To discover a polyurethane formulation that fulfils the requirements, different polyurethane formulations are screened for the glass transition temperature versus pot life balance. This is shown with dots in Figure 2-1. Next to every dot the glass transition temperature is written. Currently, all the polyurethane formulations are not in the desired performance zone (green area in Figure 2-1). The reason for this is that it is difficult to decouple the glass transition temperature and the pot life for polyurethane. Raw materials that have a slow reaction time do not have a high glass transition temperature. Besides that, changing one component has influence on the properties of the other components of the formulation. In the case of epoxy, it is not such a challenge to decouple the glass transition temperature and the pot life. This is illustrated with Epoxy A and B in Figure 2-1.

The initial viscosity, pot life (both measured with a Brookfield viscometer RV-PRO) and glass transition temperature after 6 hours curing at 70°C (measured according test standard DIN 53765) are provided for the polyurethane and the epoxy (Airstone 760E resin and Airstone 766H hardener) in Table 2-1.

Property	Polyurethane (required)	Polyurethane	Epoxy
Initial viscosity [cP]	<150	112.5	250.0
Time for 350 cP [min]	45	35	95
Tg after 6h at 70°C [°C]	70	66.0	78.8

Table 2-1: Viscosity, pot life and glass transition temperature (Tg) for the polyurethane and the epoxy [14].

From Table 2-1, it is clear that the current polyurethane formulation is not fulfilling all the minimum requirements and therefore Dow is working on an improvement of the formulation. The polyurethane is thus not yet optimal for the vacuum infusion experiments and mechanical testing. The epoxy is not fulfilling the requirement regarding initial viscosity (Table 2-1). However, this is not problematic, because the viscosity stays below 350 cP for 95 min. Therefore the viscosity is low enough to perform vacuum infusions.

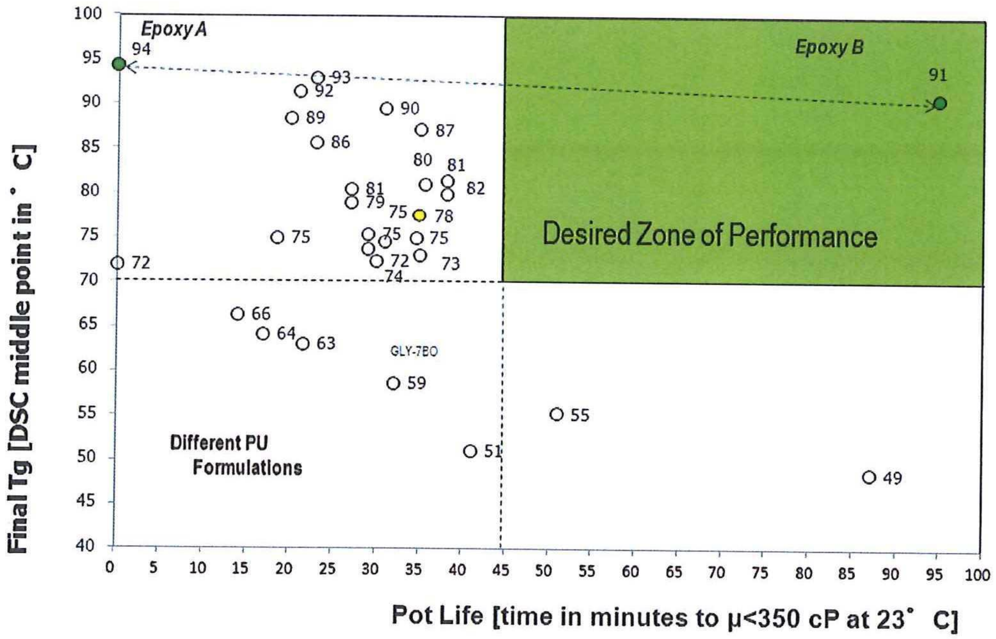


Figure 2-1: Dots show different polyurethane formulations that are screened for glass transition temperature versus pot life. The glass transition temperature is written next to every dot. It is challenging to develop a polyurethane formulation that fits in the desired zone of performance (green area) because it is difficult to decouple the glass transition temperature from the pot life. This is not the case with epoxy as illustrated with epoxy A and B.

Chapter 3

Methodology

3.1 Analysis of the Neat Resin

3.1.1 Water Content Measurement Polyol

The water content of polyol is measured to know if the zeolite (water scavenger) is active. Zeolite is added to the polyol to adsorb residual water. Water could react with the isocyanate part of the polyurethane formulation and finally form carbon dioxide which would lead to bubbles in the system [15, 16]. This is an undesired effect in the vacuum infusion process since it is desired to have full density polyurethane.

The water content of three samples of polyol is measured according to the Karl Fischer Titration method. One sample is taken from a formerly opened jerry can. Two samples are taken from an unopened jerry can. This is done to discriminate the differences between opened and unopened jerry cans.

3.1.2 Increase of Glass Transition Temperature

The increase of the glass transition temperature is measured by differential scanning calorimetry (DSC) according to test standard DIN 53765. This glass transition temperature measurement is used to determine the post curing procedure for the composite laminates and the clear castings The increase of the glass transition temperature of the epoxy is taken from the Airstone 760E:766H technical data sheet [1].

3.1.3 Clear Castings

Clear castings are produced to research the possibility to make clear castings from the polyurethane without bubbles and voids and to investigate the properties of the polyurethane and the epoxy through mechanical testing.

Two methods are used to produce the clear castings. Method 1 is used in Switzerland and can only be used to research if it is feasible to produce clear castings without bubbles and voids and not for mechanical testing. The reason for this is that it is not possible to make clear castings with a constant thickness within tolerances of ± 0.2 mm with the available moulds in Switzerland. In Italy it is possible to make clear castings with these thickness tolerances and therefore Method 2 is developed. This method is used to research the possibility to make clear castings from the polyurethane without bubbles and voids and to investigate the properties of the polyurethane and the epoxy through mechanical testing.

Besides the thickness differences, the influence between open moulds (Method 1) and the closed mould (Method 2) is investigated. It might be that the polyurethane reacts with more moisture in an open mould compared with a closed mould. This could result in different amounts of bubbles and voids in the clear castings.

Method 1

Clear castings are made in metal moulds and in moulds made from Teflon foil and tacky tape to research the influence of the mould material on the polyurethane formulation. It is possible that one of the two moulds has a higher moisture content which could result in foaming. Besides that, the influence of the index (105, 102.5 and 100) on the bubble formation is investigated. The index is the ratio between the amount of polyol and isocyanate, e.g. 110:105. The initial index of 105 is recommended by the industry. It is common to use an excess amount of isocyanate to guarantee that all the polyol reacts with the isocyanate. This results in the highest mechanical properties and glass transition temperature. However, the excess of isocyanate reacts with the environment, e.g. moisture. This can result in foaming of the polyurethane which is an undesired effect.

The required amount of polyol and isocyanate are degassed separately in the resin trap for a minimum of 20 minutes. After that the polyol and isocyanate are added together and manually mixed for 5 minutes. The polyurethane is then degassed in the resin trap for 15 minutes and poured into the moulds (Figure 3-1). The polyurethane is cured overnight at room temperature.

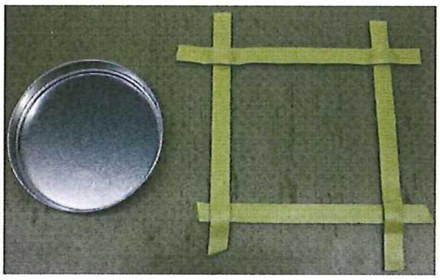


Figure 3-1: The metal mould (left) and Tacky tape/Teflon foil mould (right) used for the clear castings produced with method 1.

Method 2

The clear casting mould is cleaned followed by the application of a Pura external release agent.

Procedure for Polyurethane Clear Castings

The polyol and isocyanate are degassed separately by a high shear mixer (DISPERMAT VE 25) at 1000 rpm for 30 minutes under vacuum (50 mbar). The isocyanate and polyol are poured in a plastic container and mixed under vacuum for 10 minutes at 1000 rpm. The polyurethane is poured in an aluminium mould of 200x200 mm as shown in Figure 3-2. The polyurethane is cured under a vacuum of 50 mbar at ambient temperature for 24 hours. After that, the mould is put into an oven at 70°C and cured for 12 hours.

Procedure for Epoxy Clear Castings

Epoxy resin and hardener are added together and mixed at 500 rpm for 2 minutes. After that, the resin is placed under 50 mbar of vacuum for 10 minutes without agitation. The epoxy is poured in an aluminium mould of 200x200 mm. The epoxy is cured and post cured with the same method as described for polyurethane to keep a similar production process.

Figure 3-2: The mould used for the clear castings produced with method 2.

3.1.4 Infusion Race

The infusion race between polyurethane and epoxy is performed to investigate the flow speed of both formulations. The hypothesis is that the polyurethane infuses faster in the beginning of the infusion compared with the epoxy due to lower viscosity. With increasing time, the epoxy infuses faster compared with polyurethane due to its viscosity that stays low for a longer time.

The configuration used for the infusion race is as follows (tooling side – vacuum bag side):

- 4 layers non-woven biaxial glass fibre, 800g/m^2 (unknown supplier), approximately 40 x 130 cm
- 1 layer of peel ply
- 1 layer of flow mesh

The infusion pressure is 150 mbar and the infusion race is performed at room temperature. Every minute a line is drawn on both laminates so that the infusion distance versus infusion time could be measured after infusion.

A viscosity measurement is carried out with a Brookfield viscometer RV-PRO to proof that the reaction kinetics of the polyurethane is faster compared with the epoxy.

3.2 Vacuum Infusion

The vacuum infusion process is investigated with the polyurethane formulation. Table 3-1 shows the properties that can be changed and that might have an influence on the vacuum infusion process.

Parameters	Influences
Raw Materials	
Polyol and Isocyanate type	The type of polyol and isocyanate can influence reaction kinetics of the formulation and therefore it influences the pot life, glass transition temperature and mechanical properties. Dow works on the improvement of the chemical formulation of the polyol. A standard type of isocyanate is used.
Water scavenger (zeolite) on polyol	The water scavenger takes up the moisture in the polyol. Moisture can cause foaming of the formulation, resulting in laminates with voids.
Index	The ratio between the amount of polyol and isocyanate, e.g. 100:105. The initial recommended index was 105. It is common to use an excess amount of isocyanate to guarantee that all the polyol reacts with the isocyanate. This results in the highest mechanical properties and glass transition temperature. However, the excess of isocyanate reacts with the environment, e.g. moisture. This can result in foaming of the polyurethane which is an undesired effect.
Glass fibre type	The fibre type and direction influences the density and mechanical properties. The type of sizing used on the glass fibres can also affect the mechanical properties. The sizing should be compatible with the polyurethane in order to

	improve the fibre-matrix interfacial strength through physical and chemical bond [17]. Currently there is no sizing available specially made for polyurethane.
Number of glass fibre layers	Influences the infusion time.
Perforated release film	Perforated release film is used in the configuration so that the flow mesh can be easily removed from the laminate after cure. Release film can block the escape of bubbles from the laminate resulting in voids at the pinholes from the release film.
Peel ply	Peel ply leaves a rough surface on the laminate which is favourable for bonding.
Air release agent	Air release agent changes the surface tension and slightly modifies the viscosity of the polyurethane in order to improve degassing.
Processing	
Bubble nucleation material or sparging method used during degassing	By bringing the resin into contact with a bubble nucleation material at reduced pressure prior to the actual infusion leads to a much more effective degassing procedure and a better laminate quality during and after infusion [12]. By sparging, a container is filled with resin. The pressure in this container is reduced to a pressure below the infusion pressure to be used during the vacuum infusion process. At the bottom, air is fed into this container. The air is forced through a very fine filter, thus creating many small bubbles. These bubbles rise through the resin. At reduced pressure, the resin will be over-saturated with (components of) air. The difference in gas concentration between the air bubble and the dissolved gas causes gas molecules to diffuse from the resin into the bubble. This process continues until a new equilibrium situation is reached, e.g. the resin is saturated (but no longer over-saturated) with air [12].
Degassing polyol and isocyanate time (min)	There is a certain time needed to degas the polyol and the isocyanate separately. If this time is too short, the polyol and the isocyanate are not completely degassed. In this case air can be released during vacuum infusion.
Degassing polyol and isocyanate vacuum (mbar)	More air is released from the polyol and isocyanate during degassing if the pressure is closer to 0 mbar. The pressure should be lower during degassing compared with the pressure during infusion in order to prevent outgassing of air during the infusion.
Agitation (rpm)	With agitation the air bubbles in the formulation are destroyed. Therefore the degassing procedure goes faster.
Degassing mixture time (min)	There is a certain time needed to degas the mixture. If this time is too short, the mixture is not completely degassed. In this case air can be released during vacuum infusion.
Degassing polyurethane vacuum (mbar)	More air is released from the polyurethane during degassing if the pressure is closer to 0 mbar. The pressure should be lower during degassing compared with the pressure during infusion in order to prevent outgassing of air during the infusion.
Infusion pattern and direction	The infusion pattern and the infusion direction have an influence on the resin flow and infusion time. The infusion direction of the fibres is visualised in Figure 3-3
Vacuum bag (mbar)	One or two vacuum bags can be used. A second bag can be used for compaction and it can be used as backup in case of leakage of the first bag. The pressure should be close to complete vacuum before the infusion starts. If the pressure is 0 mbar, than there is no leakage in the set-up. In general there is always a small leakage and therefore it is more likely that the pressure will be around 5-15 mbar. Prior to the infusion the pressure is set to the desired infusion pressure. The lower this value, the faster the infusion goes. It is important that the infusion pressure is lower than the pressure used during degassing of the formulation this is in order to prevent outgassing of the resin during the infusion.

Temperature infusion	Room or elevated temperature. Elevated temperature reduces the viscosity of the resin which leads to a decrease of the infusion time, but on the other hand the reaction rate is increased. Therefore the pot life decreases.
Finishing the vacuum infusion process	There are four possibilities to finish the vacuum infusion process: (1) close inlet, (2) close outlet, (3) close both inlet and outlet, (4) increase pressure at outlet (e.g. from 100 to 400 mbar) and subsequently close inlet. Option (4) is most favourable, because this results in an equilibrium pressure at 400 mbar (if the pressure at the outlet is at 400 mbar) [18]. In the case of a leak, a small pressure gradient will remain [18].
Temperature cure and cure time	The temperature of the cure and time of the cure are influencing the degree of polymerization, which influences the glass transition temperature and mechanical properties.
Pre-treatment of glass fibres	The amount of moisture on the glass fibre can be reduced by pre-drying the glass fibres. It is desired that the amount of moisture is as low as possible, since polyurethane always reacts with moisture. This leads to foaming, which is an undesirable effect.

Table 3-1: Process parameters of the vacuum infusion process.

The infusion parameters in Table 3-1 that changed during the infusion experiments are provided in Table 3-2

Infusion parameters	
Index	Number of layers of glass fibres
Air release agent	Glass fibre pre-dried
Infusion direction	Peel ply between glass fibres and flow mesh
Glass fibre type and supplier	Release film between peel ply and flow mesh

Table 3-2: Parameters that changed during the vacuum infusion experiments.

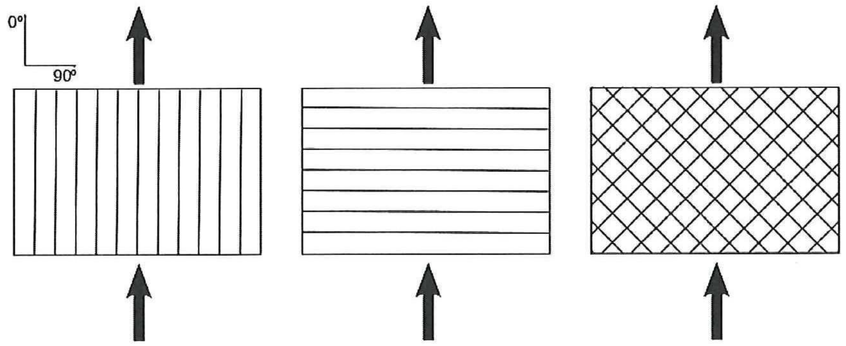


Figure 3-3: The infusion direction of the fibres.

The infusion process starts out by cleaning the infusion table with acetone. After that, a layer of Teflon foil is applied to the table. This layer can be re-used multiple times and guarantees that the laminate can be easily removed from the table after curing. The dimensions of the mould are defined by applying tacky tape to the table. The reinforcement is cut to the dimensions of 60 x 40 cm and placed into the mould in one of the directions as given in Figure 3-3. The materials needed for the configuration are cut to the correct dimensions and placed in the mould. The mould is closed with a vacuum bag. A schematic of the configuration is given in Figure 3-4.

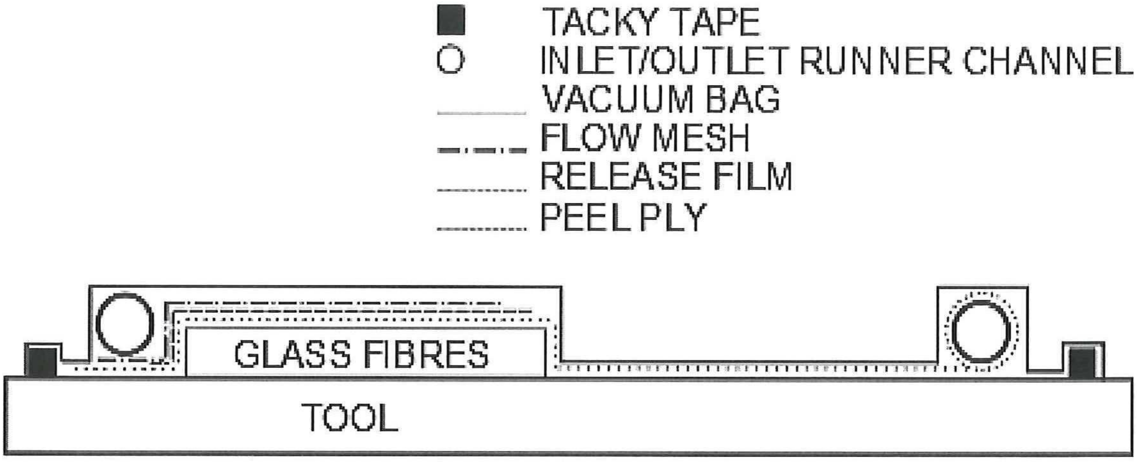


Figure 3-4: General configuration used during the vacuum infusion process.

After closing the mould with a vacuum bag, the inlet tube is closed and the vacuum is applied. The vacuum bag is checked for leakage. Any existing leakages are closed. When a vacuum pressure under 15 mbar is reached and it stays there for at least 20 minutes, a sufficient vacuum level is achieved. The vacuum pressure is then increased to the infusion pressure of 150 mbar. This is done to prevent that the compaction pressure reduces the space between the fibres such that there is less space for the resin to flow around the fibres. This would reduce the ability of wetting the fibres.

The formulation is degassed to prevent the formation of voids. The polyol and isocyanate are separately degassed for at least 20 minutes. This is done to remove as much air as possible before adding the components together. Once the components are added together the viscosity increases rapidly (Figure 1-1) and therefore the time for performing the infusion decreases. The isocyanate is added to the polyol and is then mixed at 390 rpm under a pressure of 30 mbar with a Pendraulik Laboratory Dissolver (sketch Figure 3-5). Mixing with more than 390 rpm is not possible, because the polyurethane splashes out of the open bucket (Figure 3-5). The minimum pressure the Pendraulik Laboratory Dissolver can reach is 30 mbar. After mixing and degassing for 8 minutes, the polyurethane can be used for the infusion.

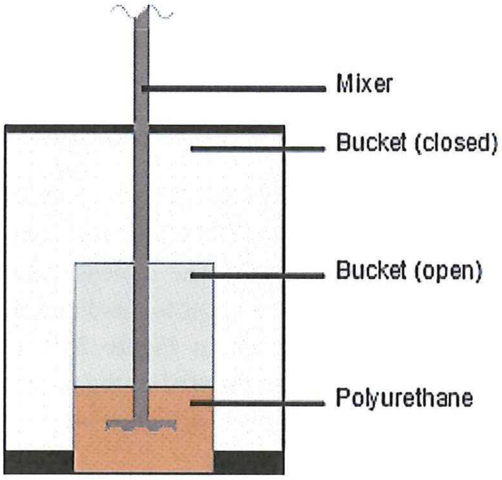


Figure 3-5: Sketch of the Pendraulik Laboratory Dissolver.

The bucket with the polyurethane is placed next to the infusion table and the inlet tube is placed into the bucket. The inlet tube is opened and the infusion has started. The inlet tube is closed after the infusion due to curing of the left over polyurethane in the bucket. If this process happens before the lay-up is completely filled, then it is not possible to produce good quality laminates. Vacuum is removed after gelation of the polyurethane in the reinforcement. The laminate is removed from the vacuum bag the day after the infusion.

3.3 Quality Control

3.3.1 Visual Inspection Laminates

The laminates are removed from the vacuum bag after curing and a visual inspection is performed to investigate the quality of the laminate. The laminate is inspected for bubbles, voids and fibre impregnation. To classify the laminate quality, a scale was developed:

- A *poor quality laminate* is a laminate which has one or more of the following deficiencies spread throughout the laminate: not fully impregnated glass fibres, visible voids and bubbles, rough surface other than that caused by the peel ply.
- An *acceptable quality laminate* is a laminate which has one or more deficiencies at the sides of the laminate. Deficiencies are not fully impregnated fibres, visible voids and bubbles, and a rough surface other than that caused by the peel ply.
- A *good quality laminate* is a laminate without any deficiencies: fully impregnated glass fibres, no visible voids and bubbles, rough surface only caused by the peel ply.

Laminates should have at least an acceptable quality for further testing.

3.3.2 Ultrasonic Inspection

The ultrasonic inspection is performed at the Institut für Werkstofftechnik und Kunstoffverarbeitung which is part of the Hochschule für Technik Rapperswil in Switzerland. The acceptable quality laminates according to visual inspection are then sent for an ultrasonic inspection.

An ultrasonic flaw detector (Olympus Omniscan MX2) is connected with a numerical controlled cutting table as shown in Figure 3-6. In this figure, a water bath is put on the cutting table and the laminate is put inside the water bath. Figure 3-7 gives a detailed photo of the water bath and the phased array probe. The ultrasonic flaw detector can only defects of more than 2 mm in diameter and the type of defects remains unidentified.

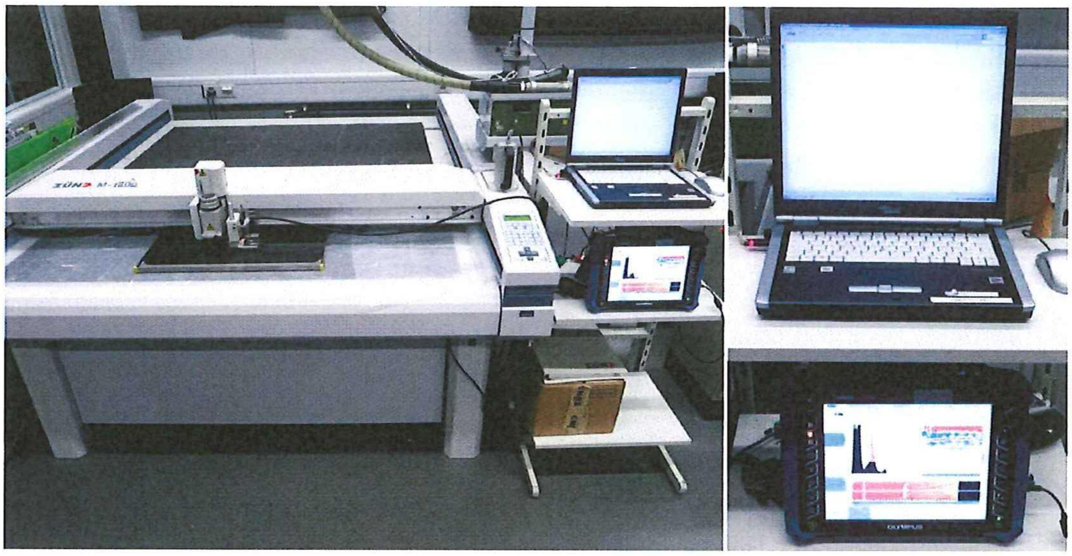


Figure 3-6: The test set-up used during ultrasonic inspection. The ultrasonic flaw detector is connected with a numerical controlled cutting table [19].

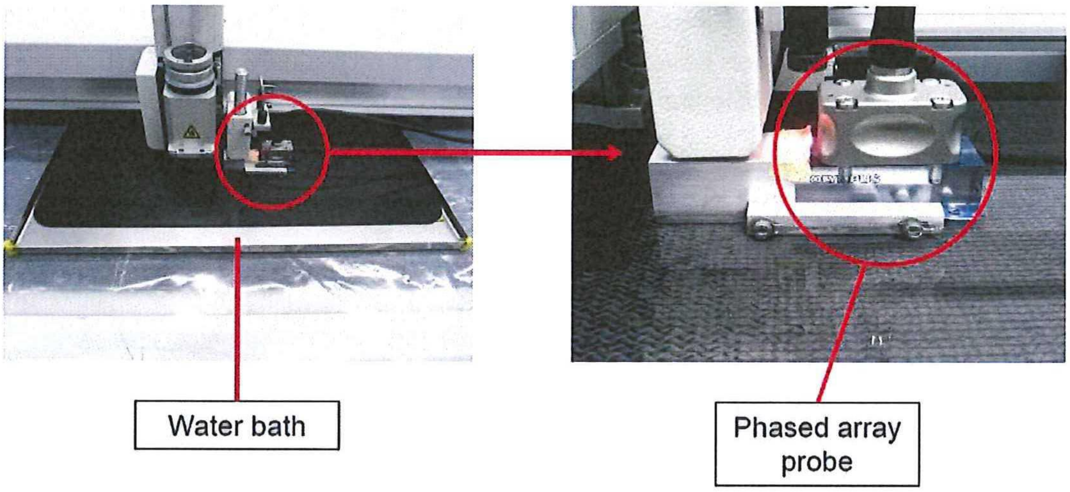


Figure 3-7: Details of the water bath and the phased array probe[19].

3.4 Mechanical Testing

Test specimens from the laminates are water cut at Qualicut AG, CNC-Wasserstrahlschneiden in Uster, Switzerland. The clear casting specimens are cut with a saw at Dow in Correggio, Italy.

Mechanical testing is performed at TEC Eurolab Srl in Campogalliano, Italy, on biaxial and unidirectional composite specimens because it was not possible to perform these tests within Dow. There was an opportunity to test the clear castings specimens within Dow and therefore these are tested at Dow in Correggio, Italy.

The goal is to obtain knowledge about the mechanical properties of polyurethane and the combination of polyurethane and glass fibres. Therefore tests are selected which determine fibre-matrix interface properties, matrix (dominated) properties. Besides that, relatively simple and easy tests are chosen to perform. An overview of the mechanical tests is given in Table 3-3.

The fibre-matrix interface plays an important role in the mechanical behaviour of fibre reinforced composites. To effectively utilize strong fibres as reinforcements of matrix materials, there must be strong adhesion between fibre and matrix to enable introduction of load into the fibres by shear stresses at the fibre-matrix interface [20]. An impregnated fibre bundle is substantially stronger than the dry counterpart; the matrix acts to almost double strength of a fibre bundle, and it also significantly reduces the dispersion of strength in case of tensile loading [20]. The fibre-matrix interface is a critical link in the transfer of load in other loading situations, such as transverse tension, in-plane shear and interlaminar shear [17, 20].

During an in-plane shear test, a highs stress concentration develops at the fibre-matrix interface. The high shear stress at the interface can cause shear failure in the matrix and/or fibre-matrix de-bonding [21]. The shear stress-strain response of fibre reinforced composite materials in non-linear.

For unidirectional specimens in which the fibres are 90° to the tensile loading direction, tensile rupture of the matrix or the fibre-matrix causes the ultimate failure. Matrix craze marks parallel to the fibre direction may appear throughout the gauge length at low loads [17].

Interlaminar shear failure is recognized as one of the critical failure modes in fibre-reinforced composite laminates and depends primarily on the matrix properties and fibre-matrix interfacial shear strengths rather than the fibre properties. The interlaminar shear strength is improved by increasing the matrix tensile strength as well as the matrix volume fraction. The interlaminar shear strength decreases, often linearly, with increasing void content [17]. Fabrication defects, such as internal micro cracks and dry strands, reducing the interlaminar shear strength as well [17].

For tensile testing of composite specimens, a thickness of 2 mm is recommended by the test standard. However, the laminates have a thickness of 3 mm and therefore this thickness is used. The tensile and flexural test for clear casted specimens used rectangular specimens with a thickness of 3 mm instead of 4 mm due to the production method.

Test	Test Standard	Dimensions [mm]	Minimum # of Specimens	Lay-up	Properties Determined
Biaxial Laminates					
Tensile 0°	ISO 527-4	250x25x3	5	Symmetric and balanced	In-plane shear modulus
Unidirectional Laminates					
Tensile 90°	ISO 527-4	250x25x3	5	Symmetric and balanced	Tensile strength/strain to failure/modulus
ILSS	ISO 14130	30x15x3	5	Symmetric and balanced	Interlaminar shear strength
Clear Castings					
Tensile	ISO 527-4	150x10x3	5	-	Tensile strength/strain to failure/modulus
Flexural	ISO 178	80x10x3	5	-	Flexural strength/strain to failure/modulus

Table 3-3: Overview of the performed mechanical tests.

Chapter 4

Results and Discussion

4.1 Analysis of the Neat Resin

4.1.1 Water Content Measurement Polyol

The water content in the polyol is measured to know if the zeolite (water scavenger) is active. The water level of the polyol is provided in Table 4-1. Sample 1 is taken from a formerly opened jerry can (opened 2 days before the measurement). Sample 2 and 3 are taken from a not previously opened jerry can. This is done to discriminate the differences between opened and unopened jerry cans.

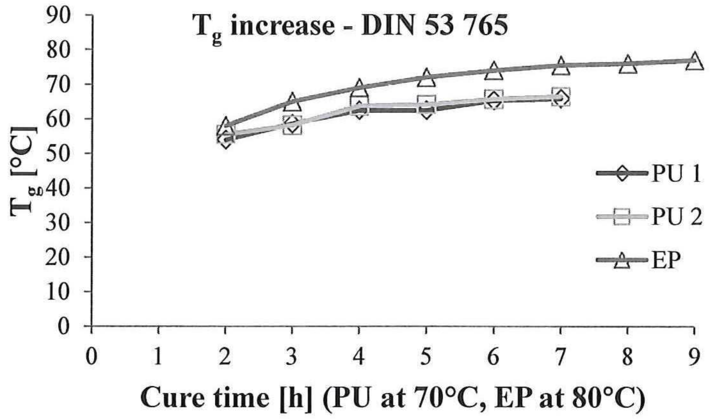
The water content of the polyol is for all samples lower than the low water level threshold (Table 4-1) obtained from test standard D4672-2012 [22]. This means that the zeolite is active. Therefore it is unlikely that water in the polyol reacts with isocyanate. Thus the formation of carbon dioxide is improbable. The water content from a formerly opened jerry can (sample 1) is higher than the water content from the unopened jerry cans. Therefore it might be that the water content is increasing after opening a jerry can and therefore over time the water content could reach the high water reference level. This could eventually results in the formation of carbon dioxide. Therefore it is recommended not to use polyol from a jerry can which is already opened for several weeks. However, performing a weekly water content measurement of the polyol could give more insights about how much water the polyol is absorbing.

Sample	1	2	3	Low water level*	Medium water level*	High water level*
Mass Fraction [%]	0.01042	0.00682	0.00139	0.0281	0.4257	1.6451

Table 4-1: Water content in the polyol. * Reference data is obtained from test standard D4672-2012 [22].

4.1.2 Increase of the Glass Transition Temperature

The increase of the glass transition temperature is measured to determine the post-cure time and temperature of the clear castings and the laminates. Two polyurethane drops (PU 1 and PU 2) are measured at every time interval. These drops are made at the same time from the same cup with polyurethane. The glass transition temperatures of the epoxy are taken from the technical data sheet of this epoxy [1]. The results are given in Figure 4-1. It was expected to observe an increase of the glass transition temperature with every measurement due to an increase of the curing time.



Cure Time [h]	PU 1 [70°C]	PU 2 [70°C]	EP [80°C]
2	53.9	55.5	58.0
3	58.6	58.2	65.0
4	62.5	63.8	69.0
5	62.5	64.2	72.0
6	65.3	65.7	74.0
7	65.9	66.5	75.5
8	-	-	76.0
9	-	-	77.0
24	68.7	68.,3	-

Figure 4-1: Increase of glass transition temperature of polyurethane and epoxy. The polyurethane is cured at 70°C and the epoxy is cured at 80°C. Data of epoxy is obtained from [1].

From Figure 4-1 it can be seen that the glass transition temperature of the epoxy is increased to a higher glass transition temperature compared with the one for the polyurethane. The polyurethane reaches its final glass transition temperature in fewer hours compared with the one for the epoxy since the initial glass transition temperature (polyurethane 53.9°C and 55.5°C versus epoxy of 58.0°C) is closer to the glass transition temperature after 7 hours (polyurethane 65.9°C and 66.5°C versus epoxy of 75.5°C). Therefore it has been decided that the post-curing conditions for the polyurethane laminates is 70°C overnight (16h). The same post-curing conditions are chosen for the epoxy laminates to keep the processing properties as similar as possible.

4.1.3 Clear Castings

Two different methods are used to produce clear castings due to different production facilities in Switzerland (method 1) and Italy (method 2). Clear castings are made with different isocyanate indices. The index is the ratio between the amount of polyol and isocyanate, e.g. 100:105 [23]. In practise, with an index of 100, the polyol cannot always react completely due to the molecular structure of the polyol and isocyanate. Since polyol consist of long molecular chains and isocyanate consist of short molecular chains, the movement of the isocyanate can be blocked by the polyol preventing the reaction between the two. To prevent blocking, an index of more than 100 is recommended. In this case, however, the excess amount of isocyanate can react with the environment, e.g. moisture. This can result in foaming polyurethane, which is undesired for vacuum infusion.

A *good quality* clear casting is one without any visible voids/bubbles. All other castings are considered of *poor quality*.

Method 1

The clear castings are made at Dow Europe GmbH in Freienbach, Switzerland, according to the parameters mentioned in Table 4-2. These clear castings are not post-cured because the castings are not used for further testing.

Parameter	Time [min]
Degassing polyol	>20
Degassing isocyanate	>20
Mixing polyol and isocyanate by hand	5
Degassing polyurethane in resin trap	15
Cure at room temperature	Overnight (16h)

Table 4-2: Processing parameters of the clear castings.

The polyurethane clear castings are made with an index of respectively 105, 102.5 and 100. The polyurethane was poured directly from a cup into Teflon/tacky tape and metals moulds. This resulted in clear castings with bubbles formed by moisture from the air (Figure 4-2). The amount of bubbles decreases for lower index values since less isocyanate could react with moisture (Figure 4-2).

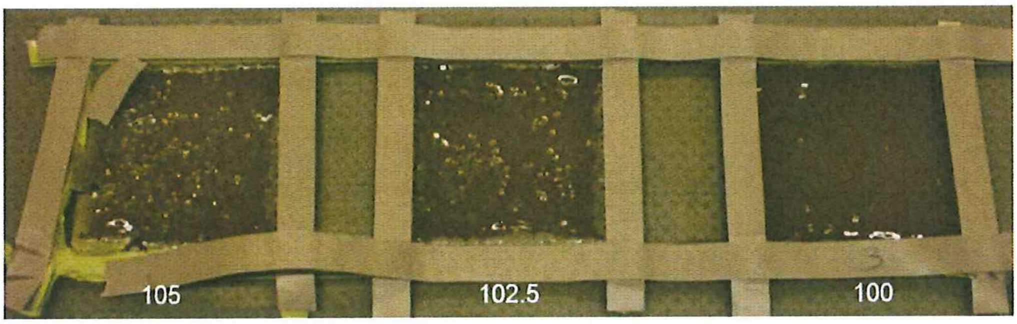


Figure 4-2: Polyurethane clear castings (with different indices) made by pouring the formulation directly from the cup into the mould.

To solve the problem of air entrapment, the polyurethane clear castings were made by pouring the formulation very carefully, using a metal spatula, into the mould. With this method, the polyurethane flows smoothly into the mould without air entrapment resulting in good quality clear castings as illustrated in Figure 4-3. There is no visible quality difference between the clear castings with different indices or the type of mould.

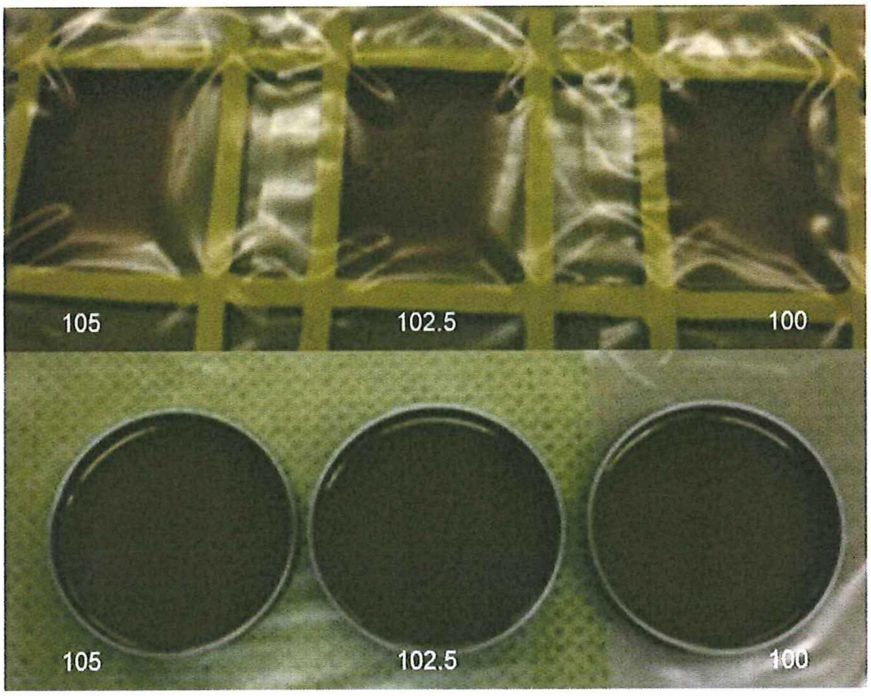


Figure 4-3: Polyurethane clear castings (with different indices) made by pouring in the formulation from the cup very carefully via a metal spatula into the mould.

Method 2

The clear castings are made with an index of 105 according to the parameters mentioned in Table 4-3 at Dow in Correggio, Italy.

Parameter	Time
Degassing polyol (50 mbar) under agitation (1000 rpm) [min]	30
Degassing isocyanate (50 mbar) under agitation (1000 rpm) [min]	30
Mechanically mixing (1000 rpm) polyol and isocyanate under vacuum (50 mbar) [min]	10
Cure at room temperature under vacuum (50 mbar) [h]	24
Post cure at 70°C [h]	12

Table 4-3: Processing parameters of the clear castings.

Good quality polyurethane (Figure 4-4) and epoxy clear castings are produced. The dimensions of the plaques are 200x200x3 mm.

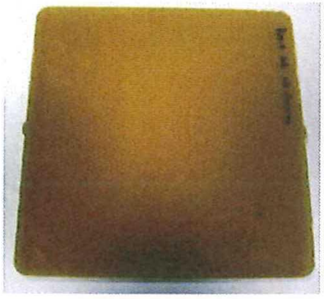


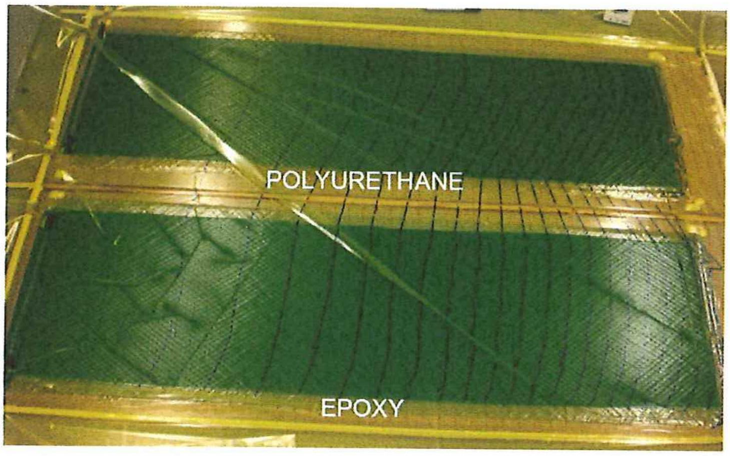
Figure 4-4: Good quality clear casted polyurethane plaque.

From method 1 and 2, it is concluded that it is possible to produce clear castings with a good quality in Teflon/tacky tape and in metal moulds. Care should be taken when pouring the formulation into the mould, because air entrapment needs to be avoided to prevent the formation of bubbles. In case of air entrapment, a lower index results in less bubble formation compared with a higher index due to the lower amount of isocyanate available to react with the water.

The dimensions of the clear castings produced with method 2 are large enough to cut specimens from making them suitable for mechanical testing.

4.1.4 Infusion Race

Figure 4-5 shows the configuration of the infusion race after completion of the race. Lines were drawn on the vacuum bag after every minute of infusion to measure the flow distance versus time. The flow front was not straight as can be seen from the lines in Figure 4-5. This is due to a non-uniform opening of the distribution tubes at the outlet and/or inlet. Therefore the average distance of the left and right side is taken as the infusion distance.



lines?

Figure 4-5: Configuration after the infusion race. Lines were drawn on the vacuum bag after every minute of infusion to be able to calculate the flow distance versus time.

The result of the infusion race between polyurethane and an epoxy based glass fibre laminate is given in Figure 4-6. This figure provides the resin flow distance and the viscosity versus the infusion time.

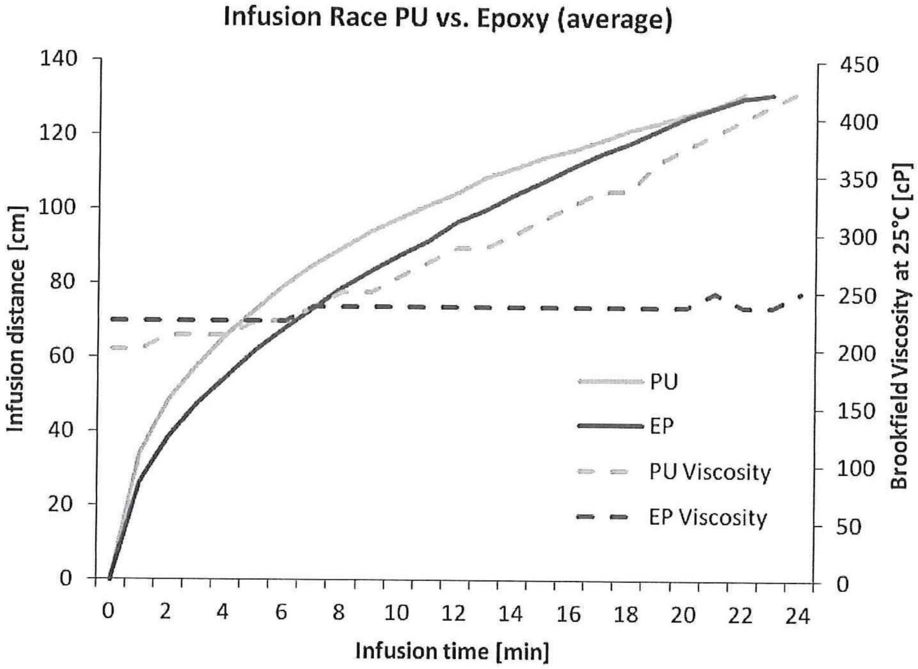


Figure 4-6: Infusion distance and viscosity versus infusion time for polyurethane and epoxy based glass fibre laminates.

The infusion distance versus infusion time of the polyurethane and the epoxy are comparable to each other. The viscosity of the polyurethane increases faster compared with the epoxy. This results in a decrease of the polyurethane infusion speed as given in Figure 4-7. Figure 4-7 gives the difference between the infusion distance of the polyurethane and the epoxy. The maximum difference in distance is at the time where the viscosity of the polyurethane and the epoxy are the same. The difference in infusion distance is decreasing with increasing polyurethane viscosity.

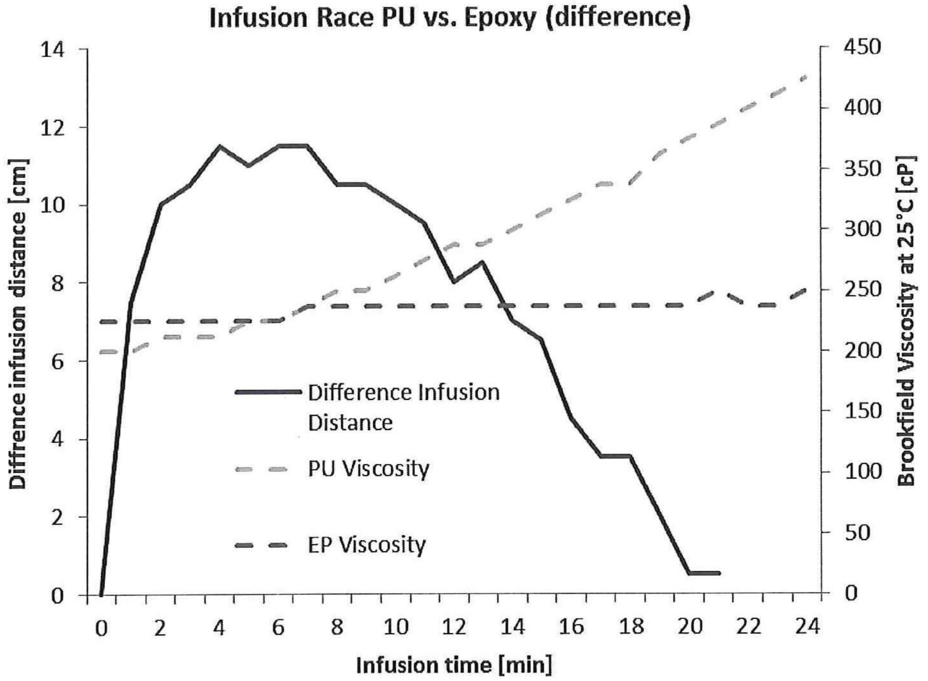


Figure 4-7: Difference infusion distance and viscosity versus infusion time for polyurethane and epoxy based glass fibre laminates.

The relation between the one-dimensional flow of a Newtonian liquid through a porous medium is described with Darcy's law (Equation 4-1) [12].

$$fill\ time = \frac{\varphi \eta l^2}{2\kappa \Delta P} \tag{4-1}$$

Where:

	Fill time [s]
φ	Porosity of the reinforcement [-]
η	Viscosity of the resin [Pa s]
l	Flow distance (length of the rectangle) [m]
κ	Permeability of the reinforcement [m^2]
ΔP	Applied pressure difference [Pa]

By looking at Darcy's law, and assuming that the polyurethane and the epoxy had the same parameters for everything except viscosity, the one with the higher viscosity would have a longer infusion time. This is exactly what happened during the infusion race (Figure 4-6 and Figure 4-7). The first 6 minutes the viscosity of the polyurethane is lower and therefore the infusion distance is higher than the infusion distance of the epoxy. After 6 minutes of infusion the viscosity of the polyurethane is higher than the epoxy and therefore the infusion of the epoxy is faster.

4.2 Vacuum Infusion

Table 4-4 gives the glass fibre lay-up of every laminate. The biaxial laminates produced during infusion 19 to 24 do have a balanced non-symmetrical lay-up. The reason is that less glass fibres are wasted compared with a symmetrical lay-up due to the dimensions of the glass fibre roll. Infusion 25 and 26 are performed to produce laminates with a balanced and symmetrical lay-up.

Lay-up	Laminate
[0/0/0/0]	10, 11, 14, 15, 16, 17, 18, 29, 30, 31, 32
[+45,-45/+45,-45/+45,-45/+45,-45]	19, 20, 21, 22, 23, 24
[+45,-45/+45,-45/-45,+45/-45,+45]	25, 26, 34

Table 4-4: Glass fibre lay-up of every laminate.

The vacuum infusion experiments are performed according to the infusion parameters in Table 4-5. These parameters are changed according to visual inspection during the infusion and gelation of the laminates and after removal of the laminate from the vacuum bag. The potential influencing parameters regarding the vacuum infusion process for the polyurethane glass fibre based laminates are:

- Pre-drying of the glass fibres
- Air release agent
- Peel ply
- Release film
- Indices
- Number of layers of glass fibre
- Types of glass fibres
- Infusion directions

The parameter changes are described below. The configuration of every infusion, the vacuum bag and the tooling side of the laminate are given in a photo overview provided in Appendix A.

There might be moisture in the glass fibres which can lead to the formation of foam in the polyurethane resin which is an undesired effect. Therefore the glass fibres are pre-dried in an oven prior to the infusion for a minimum of 2 hours. Drying temperatures ranging from 80°C to 100°C are used. The glass fibres are sealed 2 to 3 hours before the start of the infusion. The drying time and drying temperature does not seem to influence the quality of the laminates within the above mentioned range.

Air release agents change the surface tension and slightly affect the viscosity of the polyurethane formulation therefore improving degassing [24]. Additionally, an air release agent is used to prevent foam and bubbles formation from moisture during the manufacturing and application. Therefore, the air release agent, BYK-A 535, is added to the polyol before degassing. BYK-A 535 is a silicone-free solution of foam-destroying polymers. The advantage of a silicone free air release agent is that painting or bonding of the part is still possible. This can be difficult or impossible when using an air release agent containing silicone due to the formation of an oily layer at the surface of the laminate [25]. It is recommended to use 0.1-1% additive based upon total formulation [24]. 0.34% and 1% of additive are used to discriminate between a low amount and the maximum recommended amount. Adding 1% of air release agent improves the quality of laminate when compared with 0.34%.

There was foaming of the polyurethane in the peel ply during gelation. Therefore the peel ply was taken out of the configuration to investigate if this would have any effect on the laminate. The lack of peel ply caused the flow mesh to stick to the surface of the laminate. This caused the removal of the flow mesh to be difficult and imprinted the flow mesh into the laminate surface. The amount of bubbles, voids and the fibre impregnation are not improved by not using peel ply in the configuration.

The initial recommendation was to use an index of 105 to have a slight excess of isocyanate. The reasoning behind this and the possible consequences are explained in 4.1.3. The bubble formation in clear castings is lower with a reduced index. Therefore the index used during the vacuum infusions is reduced from 105 to 101. There is no evidence that a lower index results in improved quality of the biaxial laminates. It is possible that the quality of the unidirectional laminates is improved by a reduced index. However, it can also be due to the change from UD 1000 g/m^2 Seartex to UD 950 g/m^2 is responsible for the improvement of the unidirectional laminates.

To research if the polyurethane flows better through a thicker lay-up compared with a thinner lay-up the influence of numbers of glass fibres is tested. Lay-ups of 2 and 4 layers of glass fibres are made to determine the influence of thickness on the polyurethane's flow. It is observed that thicker laminates have a darker colour compared with thinner laminates indicating an increased polyurethane content. However, it can also be that the laminates look darker due to an increased thickness. A fibre volume measurement could give a decisive answer if the fibre volume is decreased, equal or increased and with that the polyurethane content is increased, equal or decreased.

Besides an influence of reinforcement thickness, it is possible that the resin flows easier through unidirectional glass fibres compared with biaxial glass fibres or vice versa. Both unidirectional glass fibres and biaxial glass fibres were tested using infusion to determine the influence of the fibre texture on the resin flow. The infusion directions for different fibres are given in Figure 4-8. The glass fibres have both a sizing suitable for epoxy.

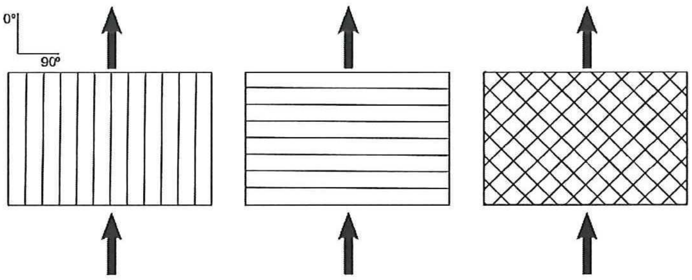


Figure 4-8: The infusion direction for different fibres.

Infusing unidirectional glass fibres following the fibre orientation (left picture in Figure 4-8) enhances the impregnation compared to infusing at an angle of 90° from the fibre orientation (middle picture in Figure 4-8). The reasons for this is that the resin flow at a 90° angle is slowed by the resistance created by the fibres [26]. There is no difference observed in infusing unidirectional glass fibres in 0° compared with infusion biaxial glass fibres in 0°.

Perforated release film was used because it allows a controlled amount of resin to be squeezed of the composite, reducing the resin content and so improving the strength-to-weight ratio of the finished part. Additionally it is used to for easier removal of the flow mesh after curing of the part. The use of perforated release film in the configuration resulted in voids in the laminates at the pinholes of the release film. Therefore the release film was taken out of the configuration to allow easier escape of bubbles from the laminates. This resulted in laminates with little to no voids.

Parameter										
Infusion # is laminate #	10	11	14	15	16	17	18	19	20	21
Index	105	105	105	105	105	105	105	105	105	105
Air release agent	BYK-A 535 0.34%	BYK-A 535 0.34%	BYK-A 535 1%	BYK-A-535 1%	BYK-A-535 1%	BYK-A-535 1%	BYK-A 525 1%	BYK-A-535 1%	BYK-A-535 1%	BYK-A-535 1%
Infusion direction	0°fibres	0°fibres	0°fibres	0°fibres	0°fibres	0°fibres	0°fibres	0° fibres	0°fibres	0° fibres
Glass fibre type and supplier	UD, 1000g/m^2, SEARTEX	UD, 1000g/m^2, SEARTEX	UD, 1000g/m^2, SEARTEX	UD, 1000g/m^2, SEARTEX	UD, 1000g/m^2, SEARTEX	UD, 1000g/m^2, SEARTEX	UD, 1000g/m^2, SEARTEX	BIAX, 800g/m^2, Unknown	BIAX, 800g/m^2, Unknown	BIAX, 800g/m^2, Unknown
# of layers of glass fibres	2	2	2	2	4	2	4	4	4	4
Glass fibre pre-dried	80°C	No	90°C	No	95°C	95°C	80°C	100°C	100°C	100°C
Peel ply between glass fibres and flow mesh	Yes	Yes	Yes	Yes	Yes	Yes	Yes	Yes	No	Yes
Release film between peel ply and flow mesh	Yes	Yes	No	No	No	No	No	No	No	No
Quality	P	P	P	P	P	P	P	G	P	G

Parameter										
Infusion # is laminate #	22	23	24	25	26	29	30	31	32	34
Index	105	102.15	102.15	101	101	101	101	101	101	101
Air release agent	BYK-A-535 1%	BYK-A 535 1%	BYK-A 535 1%	BYK-A 535 1%	BYK-A 535 1%	BYK-A-535 1%	BYK-A 535 1%	BYK-A 535 1%	BYK-A 535 1%	BYK-A 535 1%
Infusion direction	0° fibres	0° fibres	0° fibres	0° fibres	0° fibres	0° fibres	90° fibres	0° fibres	0° fibres	0° fibres
Glass fibre type and supplier	BIAX, 800g/m^2, Unknown	BIAX, 800g/m^2, Unknown	BIAX, 800g/m^2, Unknown	BIAX, 800g/m^2, Unknown	BIAX, 800g/m^2, Unknown	UD, 950g/m^2, SEARTEX	UD, 950g/m^2, SEARTEX	UD, 950g/m^2, SEARTEX	UD, 950g/m^2, SEARTEX	BIAX, 800g/m^2, Unknown
# of layers of glass fibre	4	4	4	4	4	4	4	4	4	4
Glass fibre pre-dried	100°C	100°C	100°C	100°C	100°C	100°C	100°C	100°C	100°C	No
Peel ply between glass fibres and flow mesh	No	Yes	No	Yes	Yes	Yes	Yes	Yes	Yes	Yes
Release film between peel ply and flow mesh	No	No	No	No	No	No	No	No	No	Yes
Quality	G	G	A	A	A	A	P	A	A	G/P

Table 4-5: Infusion parameters.P = poor quality (voids, bubbles, dry spots), A = acceptable quality (voids, bubbles, dry spots only on left, outlet and right side), G = good quality. The first half of laminate 34 has a good quality (inlet to middle of the laminate) and the second half has a poor quality (middle to outlet of the laminate).

4.3 Quality Control

4.3.1 Visual Inspection Laminates

The infusion parameters are provided in Table 4-5. The infusion numbers are directly related to the laminate numbers (e.g. infusion 10 results in laminate 10, infusion 21 results in laminate 21). A sketch of the distribution inlet and outlet tube and the reinforcement is given in Figure 4-9.

Figure 4-11 covers the photos of the laminates. Some glass fibre producers add coloured yarns to the glass fibres to indicate the warp direction of the fibres. These are the vertical lines in laminates 10 to 18 in Figure 4-11.

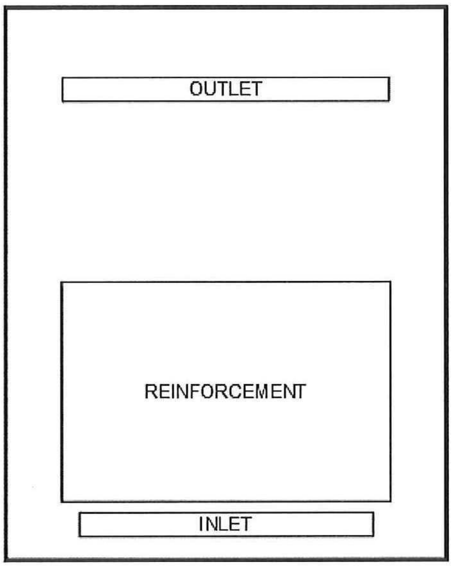


Figure 4-9: Sketch of the distribution inlet and outlet tube and the reinforcement.

Laminates 10 (glass fibres pre-dried) and 11 have voids visible at the location of the pinholes of the perforated release film. During gelation there was foaming of the polyurethane in the peel ply. Laminate 10 has a darker colour compared with laminate 11. This is an indication that pre-drying the glass fibres increases impregnation with polyurethane.

Laminate 14 (glass fibres pre-dried) has a reduced void content compared with laminate 10. Laminate 15 has a reduced void content compared with laminate 10. This is due to the removal of perforated release film in the configuration. In this case the bubbles escape easier from the laminate. It can be that the increase of air release agent (0.34% to 1%) contributes also to the void reduction due to a higher air release during degassing and an improved air release during manufacturing. Laminate 14 is darker than laminate 15. This indicates a higher level of impregnation of the glass fibres with polyurethane by pre-drying of the glass fibres.

Laminates 16 and 18 (both 4 layers of glass fibre) are slightly darker compared with laminate 17 indicating a higher polyurethane content. Although it is also possible that laminates 16 and 18 look darker due to the increased thickness. A fibre volume content measurement could give a decisive answer on this. Laminate 16 and 18 have a reduced amount of voids and dry spots compared to the laminates produced before.

Laminates 19, 21 and 23 are produced with the same method and do not have any defects. Laminate 22 is produced with the same technique as these laminates, but without the use of peel ply in the configuration, this laminate is also without any defects.

Laminates 20 and 24 (both without peel ply in the configuration) have some voids and dry spots on the left and right side of the laminate as well as on the outlet. This is also the case for laminates 25 and 26 (both balanced and symmetrical lay-up, biaxial glass fibres) and laminates 29, 31 and 32 (all three unidirectional glass fibres). The places with defects are the places without flow mesh during the infusion. The inlet side and the middle of the laminate do not have any defects. The initial polyurethane viscosity is lower at the inlet side of the laminate (the initial viscosity is 200 cP and the final viscosity is 300 cP during infusion). This allows the resin to impregnate the fibres easier. Since the resin flows first through the flow mesh, there is more time for the resin to impregnate the fibres under the flow mesh compared with the fibres outside the flow mesh. There is no evidence that peel ply has influence on the quality of the laminate.

Laminate 30 is infused at an angle of 90° from the fibre orientation (Figure 4-8, middle picture). This resulted in a laminate with voids, bubbles and dry spots. It is more difficult for the polyurethane to flow in this direction, due to resistance created by the fibres [26]. The resin flows over and under the fibres and meets resin after the flow around the fibres. Thus there are a lot of opportunities to get empty spots.

Laminate 34 (no pre-drying of the glass fibres because the size is too big for the oven, release film used in the configuration) has no defects from the inlet side to midway the laminate (first part). This is remarkable since, as seen before, pre-drying of the glass fibres was before a key factor in producing (almost) defect free laminates. From midway till the outlet side of the laminate (second part), there are voids at the pinholes of the perforated release film. This is magnified in Figure 4-10. The first part could be without defects due to a lower viscosity (200-225 cP versus 225-425 cP) of the polyurethane (Figure 4-6). The lower viscosity gives more time for the polyurethane to impregnate the fibres completely.

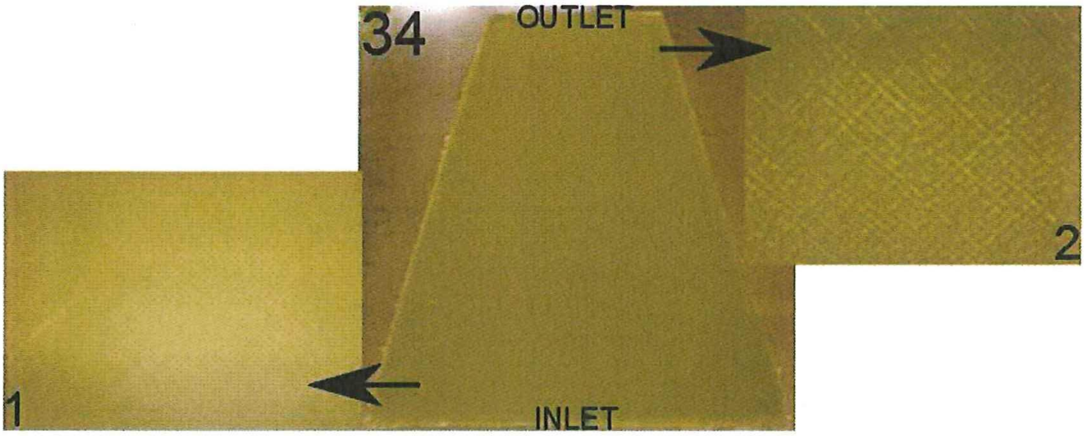


Figure 4-10: Laminate 34. The first part of the laminate, seen from the inlet side, has a good quality. The second part from midway till the outlet side of the laminate is full of voids.

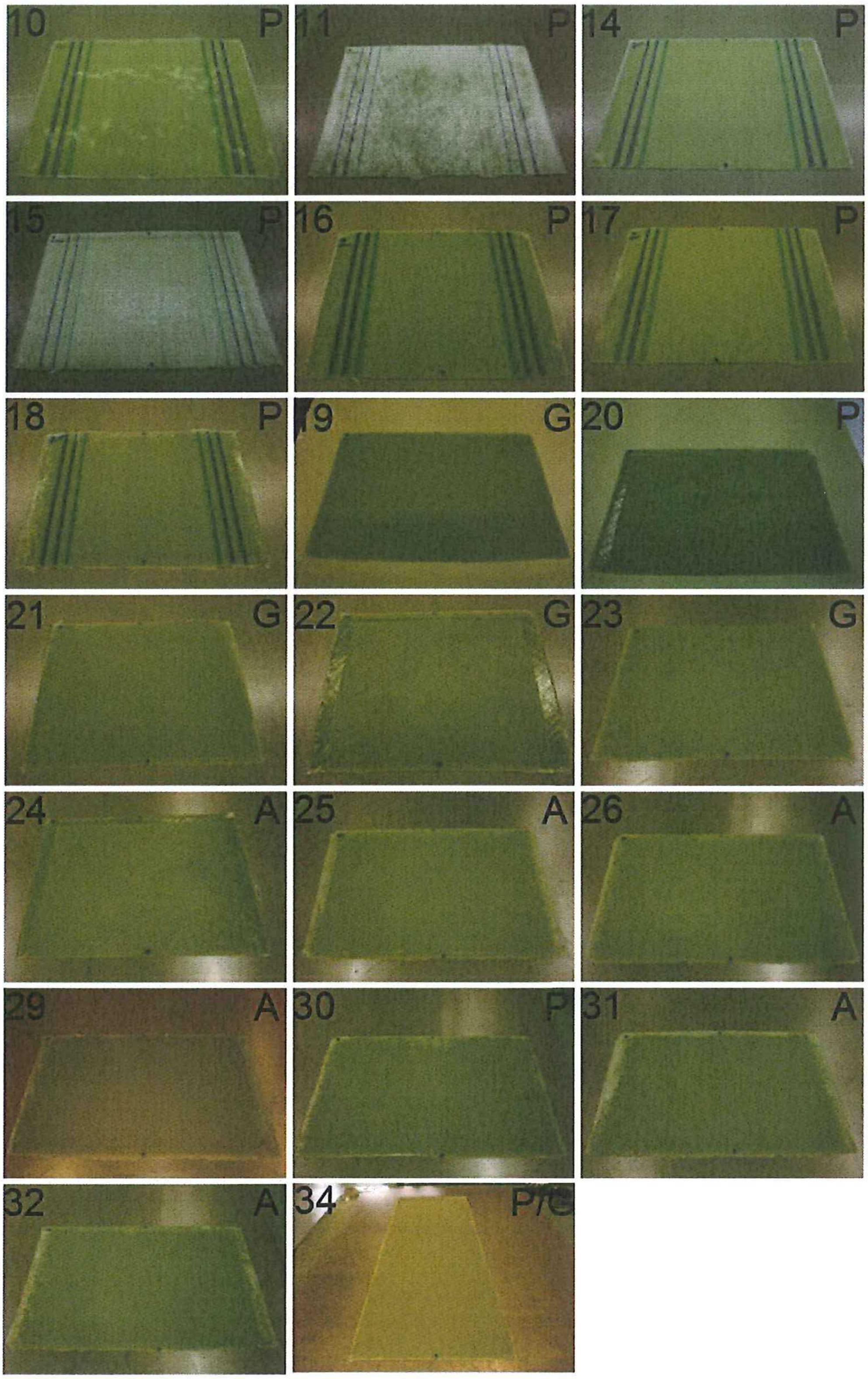


Figure 4-11: Overview of the tooling side of the laminates. P = Poor quality laminates. The fibres are not completely impregnated, dry spots and bubbles are visible. A = Acceptable quality laminates. These laminates have dry areas and voids at the left, top (outlet) and right side of the laminate. The middle and bottom (inlet) parts of these laminates have a good quality. G = Good quality laminates. The fibres are fully impregnated and there are no bubbles and voids visible.

It is possible to produce good quality biaxial glass fibre polyurethane laminates with the vacuum infusion technique as proven by laminates 19, 21, 22 and 23. However, it is not straightforward to reproduce laminates with a constant good quality even if the same method is used. This can be seen by comparing good quality laminates 19, 21 and 23 with acceptable quality laminates 25 and 26 and by comparing good quality laminate 20 with poor quality laminate 22 and acceptable quality laminate 24.

For mechanical testing, it is desired to use laminates with a balanced and symmetrical lay-up. In such a laminate, the normal stress-shear strain coupling for the laminate is zero (balanced) and there is no extension-bending coupling (symmetric). The good quality laminates have a balanced non-symmetrical lay-up, because less glass fibre was wasted in this way due to the dimensions of the glass fibre roll. Therefore, these laminates are not used for mechanical testing. Laminates with a balanced symmetrical lay-up are chosen for further testing from the 'acceptable quality' group. These are biaxial laminate 25, 26 and unidirectional laminate 29 and 32. The implications of this decision are that there is no normal shear-stress strain coupling and there is no extension-bending coupling in these laminates. However, it is expected that the existences of voids in the laminates results in decreased mechanical properties.

4.3.2 Ultrasonic Inspection

Ultrasonic inspection is performed on the epoxy laminates 1E and 2E, and on the polyurethane laminates 25, 26, 29 and 32.

The laminates are ultrasonic inspected by measuring the intensity of the ultrasonic signal through the laminates. A diagram shows the intensity of the ultrasonic signal on the right side of every C-scan (magnified in Figure 4-12). Red means 100% signal strength and white means 0% signal strength. If there is no damage in the laminate, the sound wave travels without any problems through the material (red areas). If there are some delaminations, dry spots or other defects, the sound wave will lose some of its intensity during the travelling and the signal changes its colour. Everything less than 50% of signal strength is defined as defect by the industry and therefore everything green, blue and white are areas with defects.

The inspection results are provided in Figure 4-13 to Figure 4-18. The red rectangular marked areas are the inspected areas of the laminates. The black circles on the pictures of the laminates mark where the laminate number is written on the laminate. The areas in which defects occurs frequently are marked with blue circles. However, if defects occur frequently through the whole laminate it is impossible to mark them separately.

Figure 4-12: Diagram showing the intensity of the ultrasonic signal. Areas with 100% signal strength are red and areas with 0% signal strength are white. Everything less than 50% of signal strength is defined as defect and therefore everything green, blue and white are areas with defects.

Horizontaal vs Verticaal in presentatie

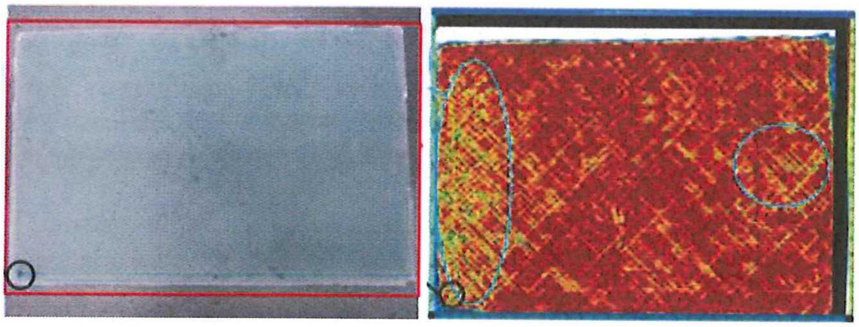


Figure 4-13: Biaxial epoxy laminate 1E is inspected at the tooling side.Blue circles mark the areas with defects.

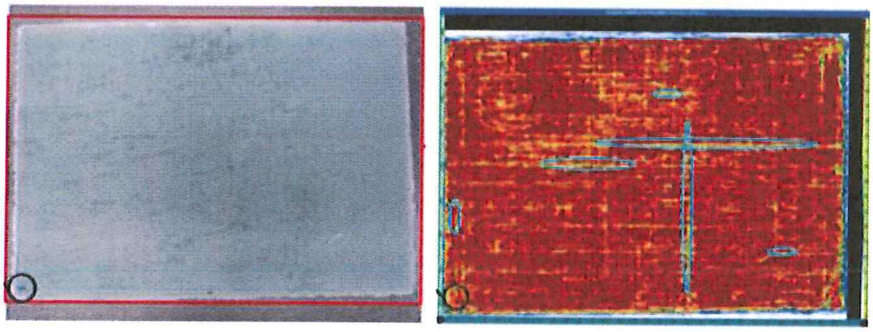


Figure 4-14: Unidirectional epoxy laminate 2E is inspected on the tooling side.Blue circles mark the areas with defects.

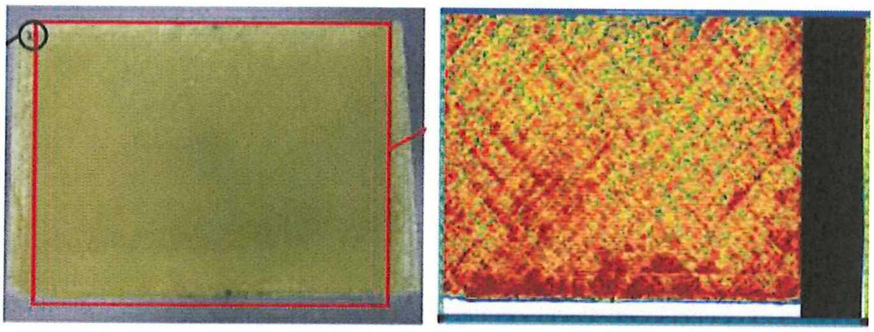


Figure 4-15: Biaxial polyurethane laminate 25 is inspected on the vacuum bag side. The defects are distributed through the laminate. The upper part shows more defects than the bottom part.

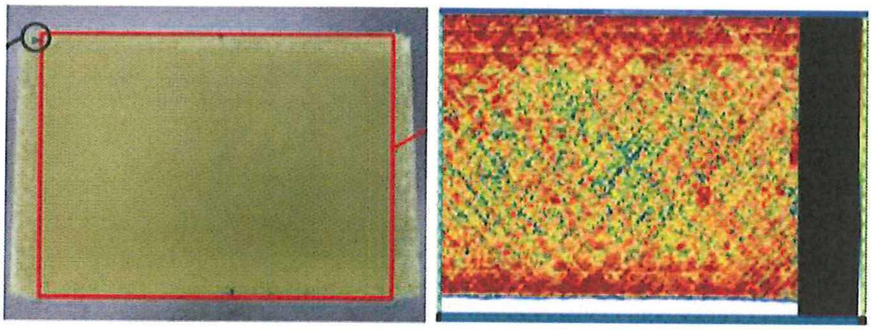


Figure 4-16: Biaxial polyurethane laminate 26 is inspected on the vacuum bag side. The defects are mainly in the central part of the laminate.

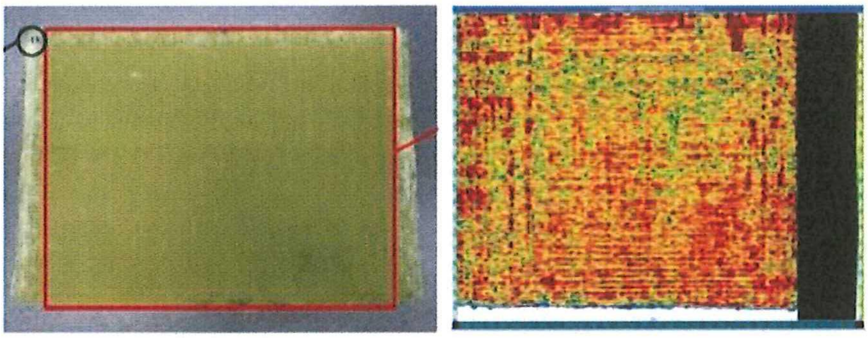


Figure 4-17: Unidirectional polyurethane laminate 32 is inspected on the vacuum bag side. The defects are distributed through the laminate. The upper part shows more defects than the bottom part.

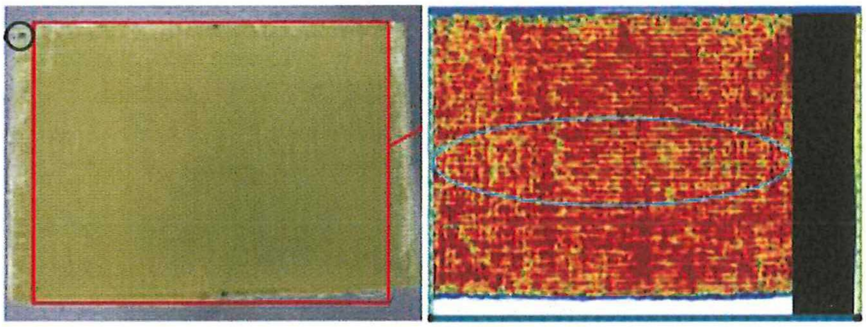


Figure 4-18: Unidirectional polyurethane laminate 29 is inspected on the vacuum bag side. Blue circle marks the area with defects.

The ultrasonic inspection was complicated to perform due to the warping of the laminates. Therefore the flatness of the infusion table was investigated by putting a beam on the table. It was clear that the surface of the table is curved and the distance from the bottom of the beam to the surface of the table was measured. There was about 2 cm in difference and therefore the warping is caused by a non-flat surface of the infusion table. This effect can be increased due to the lay-up of the fibres and shrinkage of the resin. Shrinkage of the resin is not likely due to the low shrinkage of polyurethane [5]. The warping of the laminates leads to difficulties with a proper ultrasonic signal and therefore the test set-up was adapted. It is easier to get a good signal while testing from the vacuum bag side because the smooth tooling side delivers a better echo of the ultrasonic signal. With a rough surface, a higher intensity of signal is needed and this results in loss of accuracy. It can be concluded that the epoxy laminates show some defects, but further testing is possible because specimens can be cut from areas without defects. Polyurethane based laminates 25, 26 and 32 are delivering a strong noise signal. Laminates 25, 26 and 32 are not recommended for further testing. Laminate 29 shows some defects, but further testing seems to be possible since specimens can be cut outside of the area with frequently occurring damage (area outside the blue circle) [19].

Despite the fact that it was not recommended for some of the laminates to do further testing according to the ultrasonic inspection results, these laminates were tested out of interest to gain knowledge about mechanical properties within the proof of concept phase (time restricted phase within Dow). The specimens for mechanical testing are where possible cut from parts of the laminate without blue circles. The use of specimens which have defects has influence on the mechanical test results. It is expected that the mechanical test results have lower values than it would be the case with specimens without defects detected by ultrasonic inspection.

Conclusions

The reduction of the formation of bubbles is achieved by the implementation of different strategies which need to be applied at the same time. Pre-drying glass fibres results in little to no foaming during the infusion and during gelation. The air release agent (BYK-A 535) changes the surface tension and slightly changes the viscosity of the polyurethane and therefore, it improves the air release during degassing. This resulted in improved laminate quality. An index of 101 improves the visible quality of the laminate compared with an index of 105. There are (almost) no bubbles visible in these laminates.

The desired infusion direction is following the fibres in 0° direction. With this method the polyurethane experiences the least resistance by the fibres. The possibility of void formation is therefore the lowest.

Performing infusions without release film and peel ply in the configuration results in laminates with an irregular rough surface. The flow mesh is attached to the laminate after curing and can only be removed by force. This is not desired since it can impact the laminate properties. The perforated release film usually results in voids at the pinholes of the release film since the bubbles are trying to escape at the pinholes of the release film. These bubbles cannot escape before gelation of the polyurethane resulting in voids.

Biaxial glass fibre polyurethane laminates without any defects are produced with the vacuum infusion technique as proven by laminates 19, 21, 22 and 23. However, it is not straightforward to reproduce laminates with a constant good quality even if the same method is used. The laminates produced without any defects are made according to the parameters provided in Table 4-6.

Parameter	
Index	101
Air release agent	BYK-A 535 1%
Infusion direction	0° fibres
Glass fibre type and supplier	BIAX 800 g/m^2 unknown
# of layers of glass fibres	4
Pre-drying glass fibre for minimal 2 hours	100°C
Peel ply between glass fibres and flow mesh	Yes
Perforated release film between peel ply and flow mesh	No
Degassing time polyol and isocyanate	>20 [min]
Degassing and mixing (390 rpm) time polyol and isocyanate	8 [min]
Infusion pressure	150 [mbar]

Table 4-6: Recommended infusion parameters.

Additional Recommendations

Additional recommendations to produce consistent laminates without defects with the vacuum infusion technique could be:

- The vacuum infusion process can be finished with a different method. The used method was keeping the pressure at 150 mbar and closing the inlet naturally by cured left over polyurethane in the bucket. Another method is to increase the pressure from 150 mbar to 400 mbar after infusion and subsequently close the inlet tube [4]. This method results in an equilibrium pressure at 400 mbar. In the case of a leak, a small pressure gradient remains [4].
- Use of bubble nucleation material (e.g. Scotch Brite) or sparging method during degassing of the polyol and isocyanate [12, 27]. By sparging a container is filled with resin. The pressure in this container is reduced to a pressure below the infusion pressure to be used during the vacuum infusion process. At the bottom, air is fed into this container. The air is forced through

a very fine filter, thus creating many small bubbles. These bubbles rise through the resin. At the reduced pressure, the resin will be over-saturated with (components of) air. The difference in gas concentration between the air bubble and the dissolved gas causes gas molecules to diffuse from the resin into the bubble. This process continues until a new equilibrium situation is reached, e.g. the resin is saturated (but no longer over-saturated) with air [12, 27]. Degassing of the resin by exposing the resin to partial vacuum results in a 'de-bubbled' resin and not in outgassing [12]. This is something that cannot be seen during degassing. By adding nucleation material to the resin or by using the sparging method, the resin is more effectively degassed resulting in a better quality laminate during and after infusion [12]. If the infusion pressure is higher than the degassing pressure, there is no risk of outgassing of the resin. There will even be the possibility of dissolving some bubbles, which have been formed during the flow of resin, entrapping air in fibre bundles.

Chapter 5

Results and Discussion

5.1 Mechanical Testing

Laminates produced with the vacuum infusion technique and clear casted specimens are mechanically tested out of interest and to gain knowledge about fibre-matrix and matrix properties within the proof of concept phase (time restricted phase within Dow). The fibre-matrix and matrix behaviour is from particular interest because a possible application of the polyurethane in the industry. A comparison is made between epoxy and polyurethane clear castings and glass fibre laminates.

The most important material characteristic related to the mechanical properties of a polymer is the degree of polymerization and this is indirectly measured by the glass transition temperature [28]. For most polymers at temperatures below the glass transition temperature, the stress-strain relationship becomes linear-elastic, and brittle behaviour is common [28]. This is in general the case for epoxies [29]. Polyurethanes are generally tougher and therefore it is more common to deform elastically at first and then yields into a region of plastic deformation [29]. Suppression of glass transition temperature helps to produce tougher polymers [28]. From the glass transition temperature measurement it is known that the glass transition temperature is lower for the polyurethane compared with the epoxy (Table 2-1 and Figure 4-1). Therefore and from the tougher nature of polyurethane, it is expected that the polyurethane based clear castings and laminates show tougher behaviour.

Voids have a different influence on different mechanical properties. Due to the void content in laminates, a significant reduction was observed for the transverse modulus and the shear modulus of AS44/3502 graphite/epoxy prepregs [30, 31]. However, practically there was no effect of void content observed for the longitudinal modulus [30, 31]. It is expected that the transverse modulus and the shear modulus are influenced by the void content. In unidirectional laminate 32 more defects are detected compared with unidirectional laminate 29, therefore it is expected that the transverse modulus and shear modulus of laminate 32 are reduced compared with laminate 29. The specimens of laminate 25 and 26 are both cut from areas with voids and therefore it is expected that the shear modulus is reduced for both laminates compared with a laminate without voids.

The presence of voids in neat resin specimens will cause problems with premature failures of the test specimen [20].

The dimensions, mechanical properties and strength versus strain to failure graphs of every specimen are provided in Appendix B.

5.1.1 Mechanical Tests for Composite Laminates

Laminates within the 'acceptable quality' group of the visual inspection are selected for mechanical testing. These are biaxial laminates 25 and 26 and unidirectional laminates 29 and 32. Additionally, epoxy laminates 1E (biaxial) and 2E (unidirectional) are mechanically tested and used as comparison material.

The mechanical properties of a composite laminate depend primarily on the strength and modulus of the fibres, the strength and the chemical stability of the matrix and the effectiveness of the bonding between matrix and fibres in transferring stress across the interface [16].

Biaxial Laminates – Tensile Test 0°

A tensile test in 0° direction of the fibres is performed on biaxial (±45°) laminates to determine the in-plane shear response as visualized in Figure 5-1. Test standard ISO 527-4/2/2 is used. This test standard is recommended for the determination of the tensile properties of isotropic and orthotropic fibre-reinforced plastic composites and not for the determination of the shear modulus. The reason that this test standard is used is due to a communication mistake between Dow and Eurolab. The displacement is during testing only measured in longitudinal direction instead of in longitudinal and transverse direction. Therefore equation 5-1, which is used in test standard ISO14129 for the determination of the shear modulus, cannot be used. Therefore the assumption is made that the strain in transverse direction is small and that this strain is similar for both the polyurethane and the epoxy glass fibre specimens. Therefore the shear modulus cannot be compared with data from other sources. The shear modulus is calculated according equation 5-2.

$$G = \frac{\tau'' - \tau'}{\gamma'' - \gamma'}$$

5-1

G Shear Modulus [MPa]
τ' Shear stress at longitudinal shear strain $\gamma' = \varepsilon_{Longitudinal} - \varepsilon_{Transverse} = 0.001$
τ'' Shear stress at longitudinal shear strain $\gamma'' = \varepsilon_{Longitudinal} - \varepsilon_{Transverse} = 0.005$

$$G = \frac{\tau'' - \tau'}{\varepsilon'' - \varepsilon'}$$

5-2

Where:

G Shear Modulus [MPa]
τ' Shear stress at longitudinal shear strain $\varepsilon' = 0.001$
τ'' Shear stress at longitudinal shear strain $\varepsilon'' = 0.005$

The shear modulus and standard deviation are given in Table 5-1 and visualized in Figure 5-2. A typical shear strength versus shear strain to failure graph is given for every laminate in Figure 5-3.

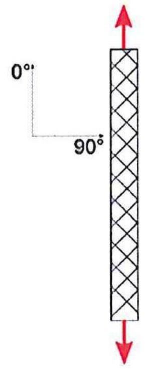


Figure 5-1: Biaxial (±45°) specimen loaded in tensile in 0° direction.

Tensile 0°	Shear Modulus [MPa]		
Laminate	1E	25	26
Number of Specimens	10	10	10
Mean	7810	7556	7594
Standard Deviation	580	553	733

Table 5-1: The shear modulus is obtained from a 0° tensile test on biaxial laminates according to ISO 527-4/2/2.

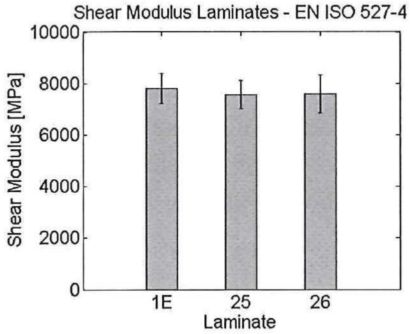


Figure 5-2: The shear modulus is obtained from a 0° tensile teston biaxial laminates according to ISO 527-4/2/2.

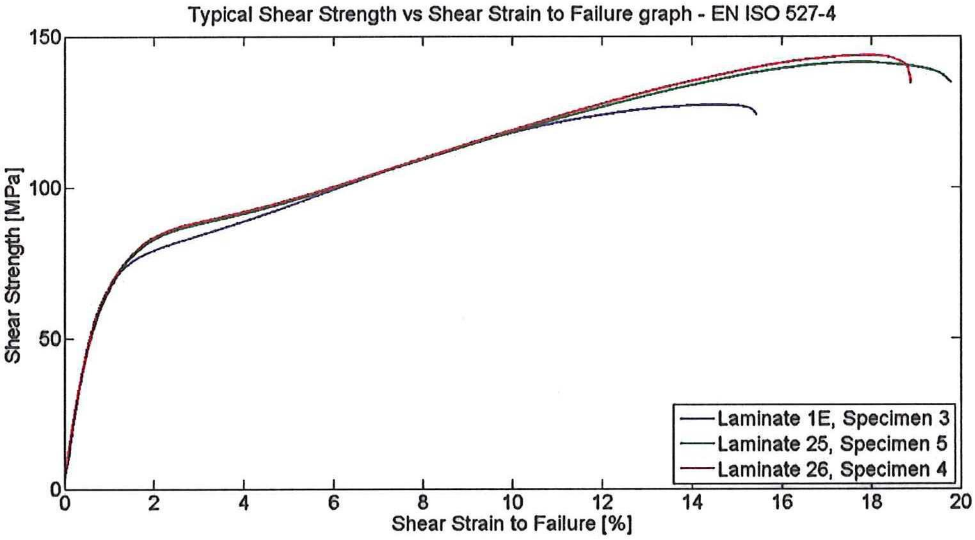


Figure 5-3: Typical shear strength versus shear strain to failure behaviour for epoxy (laminate 1E) and polyurethane (laminates 25 and 26).

Comparison of the polyurethane (laminate 25 and 26) and epoxy (laminate 1E) composite laminates studied here shows that the shear modulus is similar for both types of laminates. Thus the stiffness in the linear-elastic region is comparable. The standard deviation is moderate for the shear modulus of all three laminates. First damage occurs at a shear strain failure of 2% for both types of specimens. This is the point where the curves bend in Figure 5-3.

These results are interpreted differently if another test standard would have been used; this is discussed below. It would have been more suitable to take a test standard into account designed for determination of the in-plane shear response by the ± 45° tension test method, e.g. ISO 14129 or ASTM D3518. These standards state that the test should be terminated at 5% strain if failure of the test specimens has not occurred. The reasoning behind is that with large deformations, extreme fibre scissoring can occur in the specimen for the cases of ductile matrices, weak fibre/matrix interfaces, thick specimens with a large number of repeated plies, or a combination of the above [32, 33]. Such fibre scissoring, if left unbounded, would lead to an unacceptable violation of the assumption in the test method of a nominal ±45° laminate [32, 33].

Taking the above mentioned into account and looking again to Figure 5-3 it can be seen that both materials show large strains and thus large deformations. Since the strains of the polyurethanes are higher compared with epoxy, the polyurethane has a weaker fibre-matrix interface compared with epoxy.

Terminating the test at 5% strain results in the shear strength versus shear strain up to 5% graph provided in Figure 5-4. Figure 5-4 is in principle the same graph as Figure 5-3, except the difference in final strain. This results in similar shear modulus and shear strength for the epoxy and polyurethane glass fibre specimens. There is no indication of tougher behaviour or higher fibre-matrix bonding of one of the two types of materials.

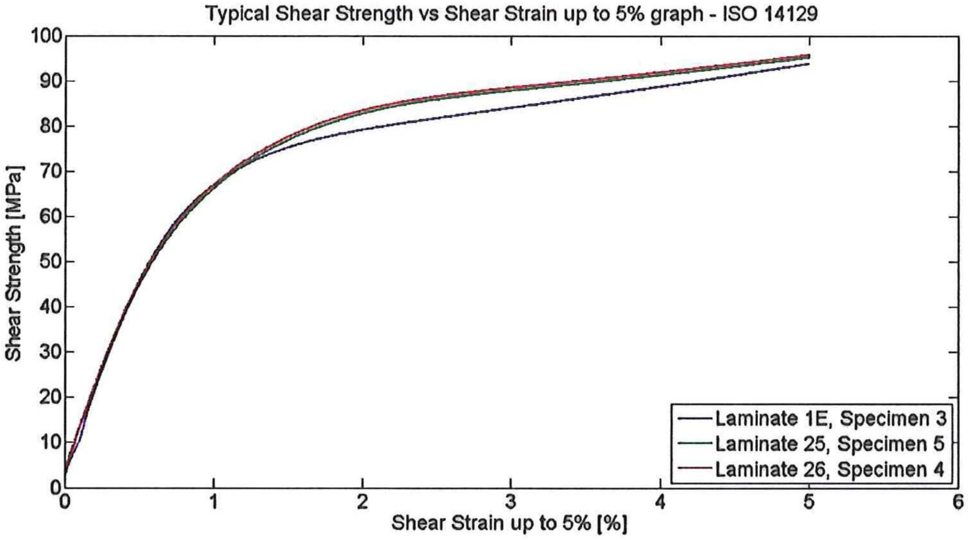


Figure 5-4: Typical shear strength versus shear strain up to 5% behaviour for epoxy (laminate 1E) and polyurethane (laminates 25 and 26).

The specimens would have looked differently after testing if the test was terminated at 5% strain. However, how the specimens would have looked is not possible to examine anymore. Therefore the specimens are discussed after failure. The specimens failed in the gauge section as shown in Figure 5-5. Failure of the specimens occurred first at the vacuum bag side of the laminates. This side of the specimens is more elongated compared with the tooling side of the specimens. Therefore the failed specimens are curved. Optical microscopy on the longitudinal direction of the specimens shows an irregular surface with voids on the vacuum bag side of the specimens and a smooth surface on the tooling side of the specimens as can be seen from Figure 5-6. Before testing there was already a difference in surface smoothness due to the use of peel ply on only one side (vacuum bag side). This, in combination with an increase of void content on the vacuum bag side of the specimens due to outgassing of bubbles resulted in a non-uniform failure of the specimens. Therefore it is expected that the mechanical properties are lower than with uniform failure. Due to premature failure of a part of the specimen, the load needs to be carried by a smaller part of the specimen. The use of peel ply on both sides of the reinforcement during vacuum infusion would have given the laminates a similar surface smoothness on both sides and it might be that fracture behaviour on both sides would have occurred more similar.

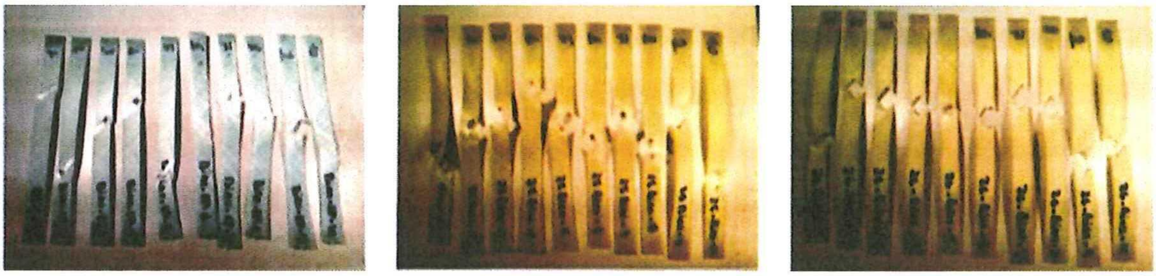


Figure 5-5: The tested specimens after failure are taken from laminates 1E (left), 25 (middle) and 26 (right).

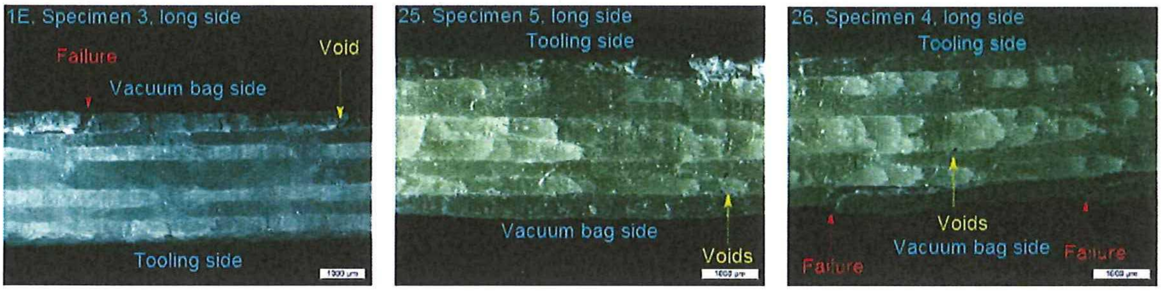


Figure 5-6: Optical microscopy of the longitudinal side of biaxial glass fibre epoxy (left) and polyurethane (middle and right) specimens.

Unidirectional Laminates – Tensile Test 90°

A tensile test in 90° direction was performed on unidirectional polyurethane and epoxy glass fibre specimens according to test standard ISO 527-4/2/2. This test is used as a measure of the matrix and matrix-fibre interface performance. The tensile strength, strain to failure, modulus and standard deviation is provided in Table 5-2 and visualized in Figure 5-7. A typical tensile strength versus tensile strain to failure graph for every laminate is given in Figure 5-8. The stiffness drop of the epoxy in Figure 5-8 is most likely due to the change in strain measurement. The first part is probably measured with an extensometer and the second part is measured by the displacement of the test machine.

Tensile 90°	Tensile Strength [MPa]			Tensile Strain to Failure [%]			Tensile Modulus [MPa]		
Laminate	2E	29	32	2E	29	32	2E	29	32
Number of Specimens	6	5	5	6	5	5	6	5	5
Mean	79	77	79	2.22	0.940	0.900	11481	9706	11521
Standard Deviation	4.64	4.04	3.49	0.147	0.0894	0.0707	516	1214	711

Table 5-2: The mechanical properties are obtained from a 90° tensile test on unidirectional laminates according to ISO 527-4/2/2. Specimens that failed in the grip section are not taken into account.

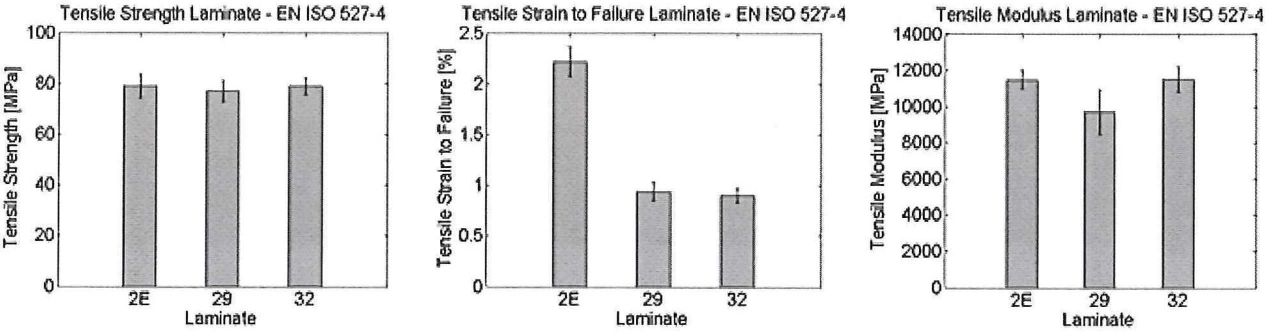


Figure 5-7: The mechanical properties are obtained from a 90° tensile test on unidirectional laminates according to ISO 527-4/2/2. Specimens that failed in the grip section are not taken into account.

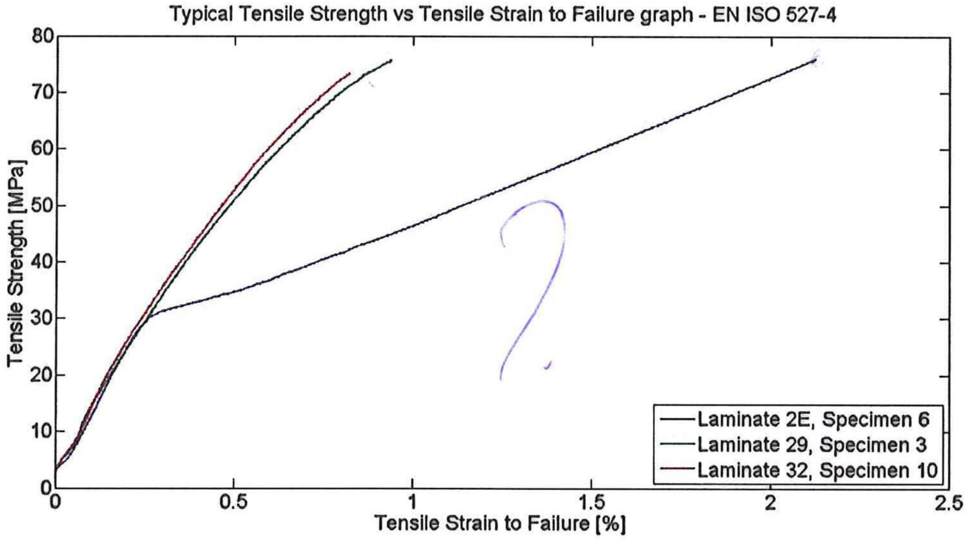


Figure 5-8: Typical tensile strength versus tensile strain to failure behaviour for epoxy (laminate 2E) and polyurethane (laminates 29 and 32).

Comparison of the polyurethane (laminates 29 and 32) with epoxy (laminate 2E) based laminates shows that both types of laminates have similar tensile strength and tensile modulus properties. Polyurethane specimens show a lower tensile strain to failure behaviour compared with epoxy based specimens. Thus the epoxy based specimens elongate more upon failure indicating a stronger bond between epoxy and glass fibres. The tensile modulus has a higher standard deviation for the polyurethane laminates compared with the epoxy laminates.

Figure 5-9 shows the specimens after failure and Table 5-3 provides the failure location of every specimen. Most specimens did not break completely, i.e. the two halves of the specimens are still attached to each other. Not all the specimens failed in the desired gauge section of the laminate. The specimens that failed in the grip section are not taken into account for the calculation of the tensile properties. Failure in the gripping area could have been prevented by using specimens with end tabs [34]. There are no defects observed in the glass fibre epoxy specimens by microscopy (Figure 5-10). The glass fibre polyurethane specimens show several voids spread through the thickness of the specimens by optical microscopy pictures (Figure 5-10). Voids are resulting in reduced tensile modulus [30, 31]. Thus if the void content in the specimens could be reduced, the modulus could be increased.

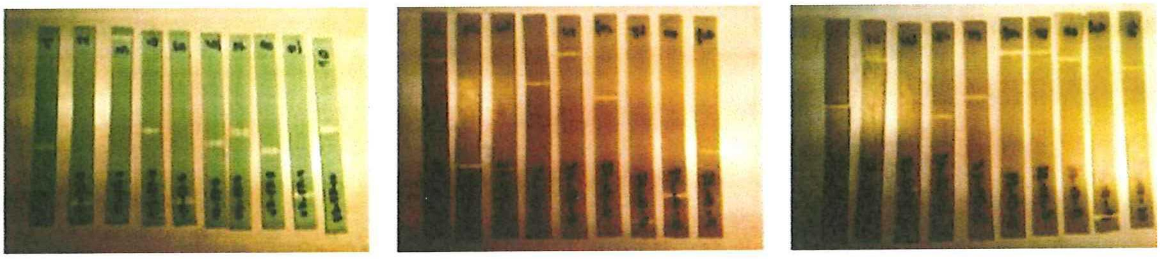


Figure 5-9: The tested specimens after failure taken from laminates 2E, 29 and 32.

Failure	2E	29	32
Gauge Section	1, 4, 6, 7, 8, 10	2, 3, 4, 6, 9	1, 3, 4, 5, 10
Grip Section	2, 3, 5, 9	1, 5, 7, 8	2, 6, 7, 8, 9

Table 5-3: Failure location.

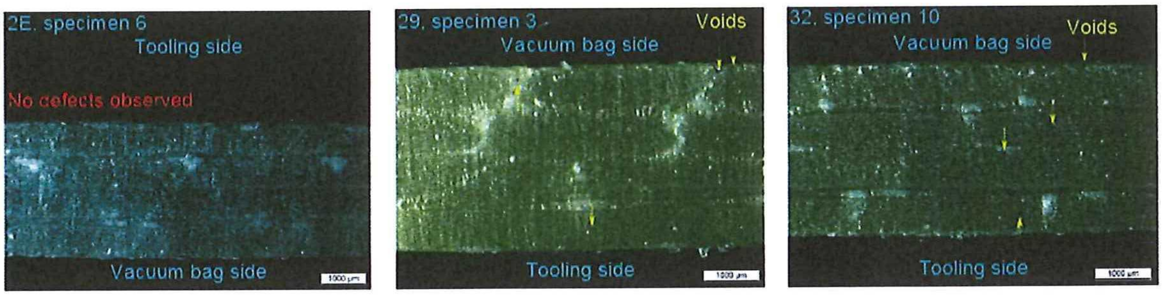


Figure 5-10: Optical microscopy of unidirectional glass fibre epoxy (left) and polyurethane (middle and right) specimens.

Unidirectional Laminates – ILSS

An interlaminar shear strength test is performed on unidirectional laminates according to test standard ISO 14130. The interlaminar shear strength and standard deviation of the data are given in Table 5-4 and visually in Figure 5-11.

ILSS	Interlaminar Shear Strength [MPa]		
Laminate	2E	29	32
Number of Specimens	10	10	10
Mean	47	52	51
Standard Deviation	1.57	1.49	1.49

Table 5-4: The mechanical properties are obtained from an ILSS test on unidirectional laminates according to ISO 14130.

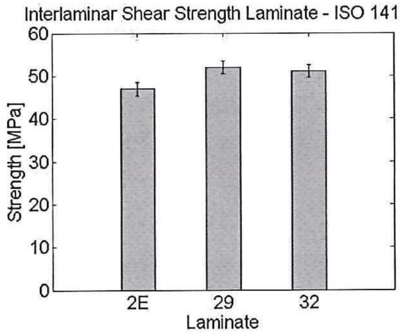


Figure 5-11: The interlaminar shear strength is obtained from an ILSS test on unidirectional laminates according to ISO 14130.

The specimens failed in a combination of shear and compression as shown in Figure 5-12 and Figure 5-13. This is a mixed mode of failure and unacceptable interlaminar shear failure. Therefore the calculated value of interlaminar shear strength is not an interlaminar shear strength value and may therefore only be used to compare test specimens taken from the same material according to test standard ISO 14130. The polyurethane specimens (laminate 29 and 32) have a similar value for the interlaminar shear strength with low standard deviation. This indicates that both polyurethane laminates have an equivalent quality.

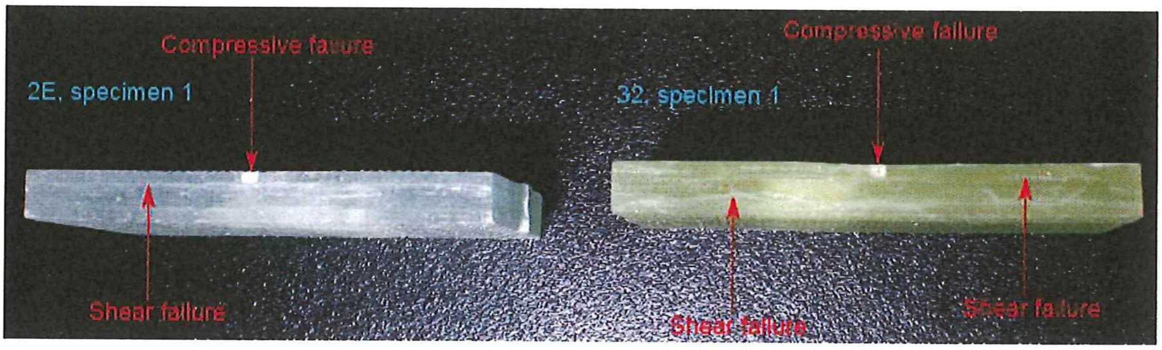


Figure 5-12: Two representative specimens showing a combination of compressive and shear failure.

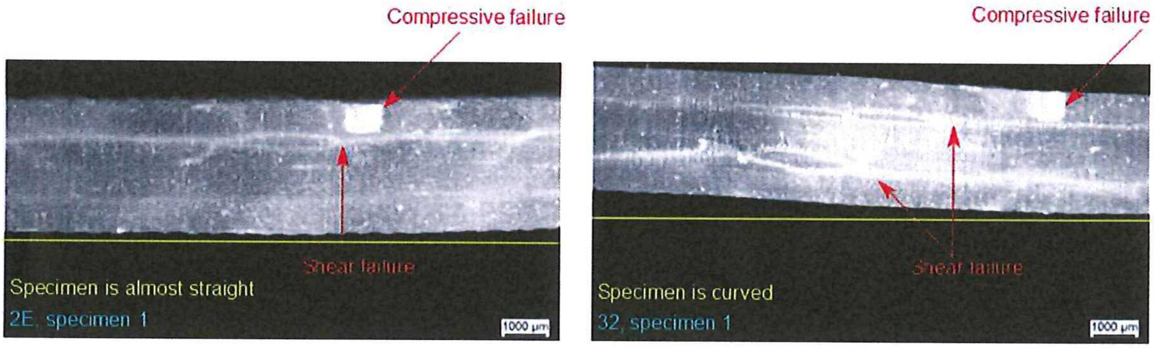


Figure 5-13: Optical microscopy of unidirectional glass fibre epoxy (left) and polyurethane (right) specimens.

5.1.2 Mechanical Tests for Neat Resin

The neat resin properties are not necessarily equivalent to those that will be achieved by the resin in a composite. The fibre-matrix interface region in particular may differ from the bulk matrix [20].

The epoxy and hardener used to manufacture the clear casting specimens are respectively Airstone 760E and 766H. Epoxy reference data is used to compare the mechanical test results of the epoxy and polyurethane specimens. This reference data is taken from the technical data sheet of the Airstone 760E:766H formulation. This reference epoxy is cured for 10 hours at 80°C while the clear castings produced during the thesis are cured for 12 hours at 70°C. Therefore there can be a difference in degree of curing and therefore mechanical properties can be slightly different.

Clear Castings – Tensile Test

A tensile test is performed on clear casted specimens according to test standard ISO 527-4/2/2. Test specimens of 80x10x3 mm are used. The tensile strength, strain (to failure for epoxy specimens), modulus and standard deviation are given in Table 5-5. The polyurethane results are based on four specimens instead of the recommended minimum of five specimens. One polyurethane specimen failed prematurely due to voids. A typical tensile strain versus tensile strain to failure is given in Figure 5-15 for epoxy and for polyurethane neat resin specimens.

Tensile Test	Tensile Strength [MPa]			Tensile Strain to Failure [%]			Tensile Modulus [MPa]		
Specimen	EP	PU	Ref	EP	PU	Ref	EP	PU	Ref
Number of Specimens	5	4	-	5	4	-	5	4	-
Mean	67	52	67	11.6	9.7	4.0	1212	1002	3000
Standard Deviation	0.508	1.45	-	0.807	1.77	-	80.9	81.3	-

Table 5-5: The mechanical properties are obtained from a tensile test on clear castings according to ISO 527-4/2/2. Reference data is taken from the Airstone 760E:766H technical data sheet [1].

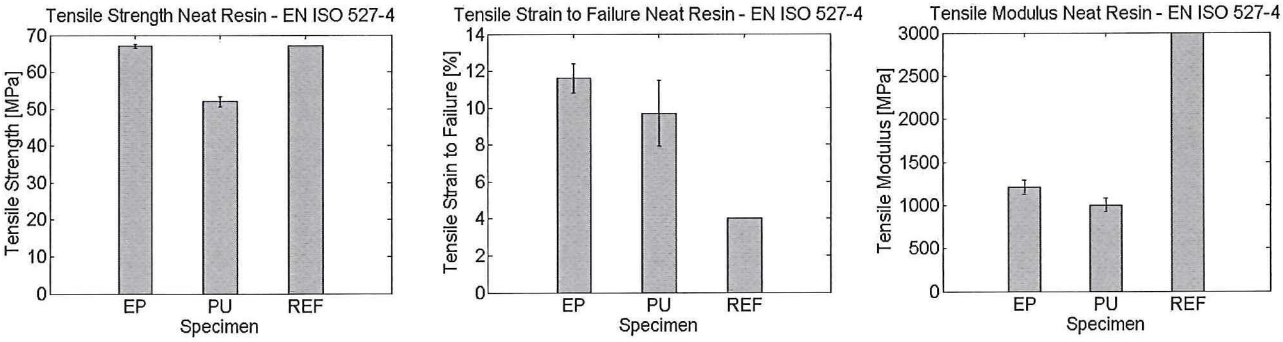


Figure 5-14: The mechanical properties are obtained from a tensile test on clear castings according to EN ISO 527-4/2/2. Reference data is taken from the Airstone 760E:766H technical data sheet [1].

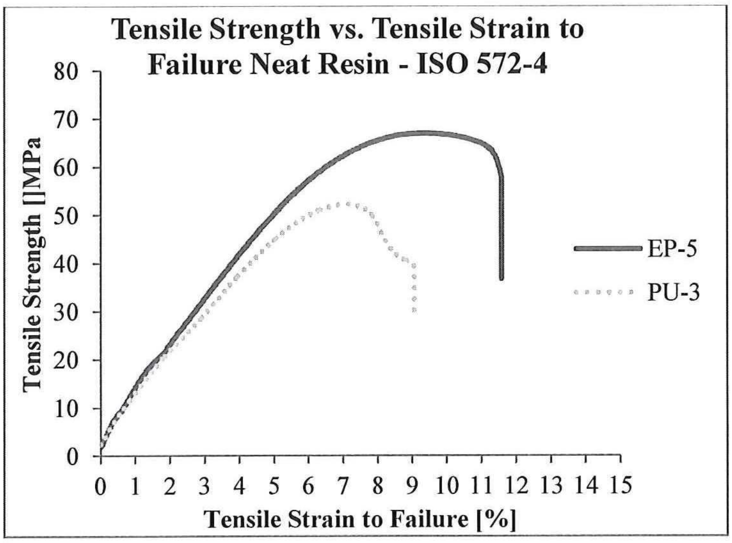


Figure 5-15: Typical tensile strength versus tensile strain to failure behaviour neat resin specimens tested according to ISO 527-4.

Polyurethane specimens have a lower tensile strength and tensile strain to failure compared with epoxy specimens. Thus the polyurethane specimens do not show tougher behaviour which would be expected by the tougher nature of polyurethane. The standard deviation for the tensile strain of the polyurethane specimens is high. The tensile modulus is similar for both materials, about one third of the reference data. A reason for this can be due to the difference in curing cycle. The reference data could have a higher degree of curing due to a curing cycle of 10 hours at 80°C compared with a curing cycle of 12 hours at 70°C. The degree of curing can be measured by performing a DSC measurement on samples with both cure cycles. If the glass transition temperature of both materials is the same, then the degree of curing is also the same. If the glass transition temperature is higher for one of the two currying cycles, then this material has a higher degree of curing. The reference material can be more brittle due to the difference in curing cycle and therefore the tensile strain to failure is lower and the tensile modulus is higher. However, one would expect that the curing cycle would also influence the tensile strength and in this case the epoxy tensile strength is similar to the reference data. Therefore there might be another reason for the low tensile modulus. It could be that something undesired happened during testing. However, this could not be traced. The polyurethane and epoxy specimens show a tensile strain to failure 2 to 3 times as high as the reference data. Thus the ratio of 3 is observed for both the tensile strain to failure and the tensile modulus. This is in agreement with Hooke's law. Hooke's law describes that stress is proportional to load and strain is proportional to deformation (Equation 5-3).

$$E = \frac{\sigma}{\varepsilon} \tag{5-3}$$

Where:

E E-Modulus [Pa]
σ Stress [Pa]
ε Strain [-]

The tensile properties measured in the tensile test perpendicular to the fibres on composite specimens are matrix dominated. Therefore these test results can be compared with the tensile test results on neat resin specimens. The tensile strength of the epoxy composite specimens is 1.2 times higher, the tensile strain to failure is about 5 times lower and the tensile modulus is about 10 times higher than the epoxy neat resin specimens. The tensile strength of the polyurethane composite specimens is 1.5 times higher, the tensile strain to failure is about 10 times lower and the tensile modulus is about 21 times higher than the polyurethane neat resin specimens. The same type of behaviour of tensile properties of composite specimens versus neat resin specimens is observed for epoxy and polyurethane specimens. There is a factor of 2 between the strain to failure and the modulus properties.

Failure occurred for most specimens in the gripping area at locations of voids. Unfortunately it is not possible to trace which specimens failed in the gripping area and therefore all the tested specimens are taken into account with the calculations of the tensile properties. Since it is desired to have specimen failure at the gauge section, it is useful to repeat this tensile test with dog bone shaped specimens and a strain measurement with the use of an extensometer in accordance with EN ISO 527-2. It is expected that the use of dog bone specimen results in failure at the gauge section of the specimens. An extensometer could be used to measure the elongation of the test specimen more accurately than the displacement of the test machine. The elongation is used to characterize the strain.

Clear Castings – Flexural Test

A flexural test is performed on clear casted polyurethane and epoxy specimens according to ISO 178. The flexural strength, flexural strain at flexural strength, flexural modulus and the standard deviation are given in Table 5-6 and visualized in Figure 5-16. The test on polyurethane specimens was terminated at the strength of 42 MPa. This was before failure of the polyurethane specimens occurred and therefore strain value at the flexural strength is provided instead of the flexural strain to failure value. A typical flexural strength versus flexural strain graph is given in Figure 5-17 for epoxy and polyurethane neat resin specimens.

Flexural Test	Flexural Strength [MPa]			Flexural Strain at Flexural Strength [%]			Flexural Modulus [MPa]		
Specimen	EP	PU	Ref	EP	PU	Ref	EP	PU	Ref
Number of Specimens	5	5	-	5	5	-	5	5	-
Mean	119	104	105	6.3	5.4	-	3124	2877	3000
Standard Deviation	1.13	1.35	-	0.5	0.2	-	40.3	52.2	-

Table 5-6: The mechanical properties are obtained from a flexural test on clear castings according to ISO 178. *There was no failure of polyurethane specimens. The reference data is taken from the Airstone 760E:766H technical data sheet [1].

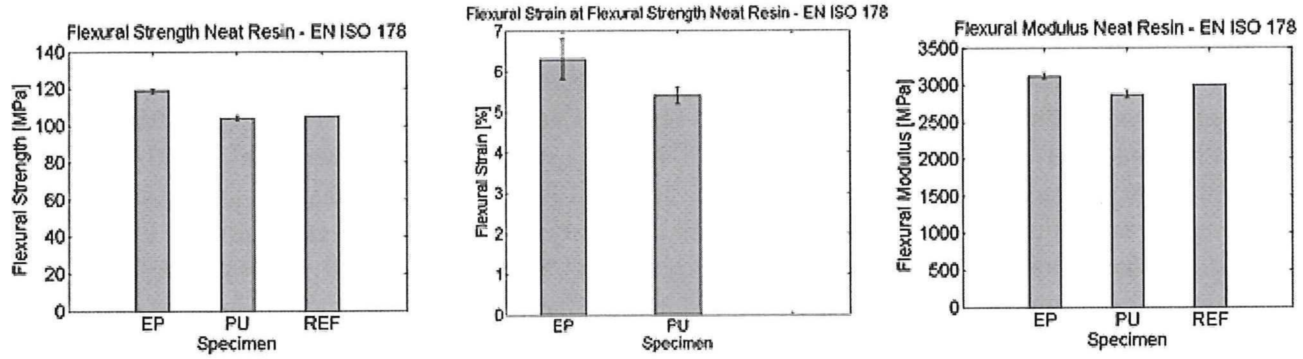


Figure 5-16: The mechanical properties are obtained from a flexural test on clear castings according to ISO 178. There was no failure of polyurethane specimens. The reference data is taken from the Airstone 760E:766H technical data sheet [1].

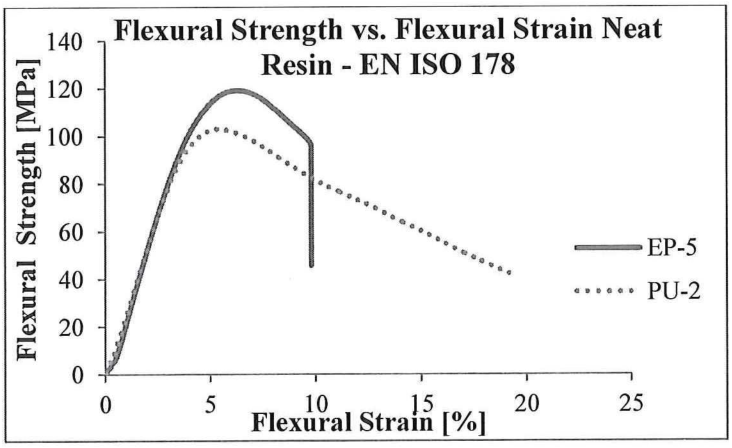


Figure 5-17: Typical flexural strength versus flexural strain behaviour of neat resin specimens tested according to ISO 178.

Polyurethane specimens show a lower flexural strength, a lower flexural strain to at flexural strength and similar flexural modulus properties compared with epoxy specimens. The standard deviation of the flexural strain is low for polyurethane specimens. Comparing the tensile strain (Table 5-5 and Figure 5-15) and flexural strain with each other shows that epoxy specimens show higher tensile strain and higher flexural strain at flexural strength compared with polyurethane specimens. The flexural strength of the epoxy is higher than the reference data. This could be due to the different curing cycles. The flexural modulus properties are comparable for all the materials. Thus the elastic deformation of the materials is similar to each other. The polyurethane specimens show tougher behaviour, while the epoxy specimens behave more brittle.

Conclusions and Recommendations

It is observed that the two polyurethane based laminates show comparable shear, tensile and interlaminar shear properties. Thus it is possible to manufacture polyurethane laminates with reproducible quality regarding mechanical properties. However, this does not mean that the manufacturing process is optimum since there are defects detected by visual and ultrasonic inspection (Chapter 4). The in plane shear strength, the tensile strength perpendicular to the fibres and the interlaminar shear strength of the polyurethane laminates are similar or even higher compared to the epoxy laminates. The shear strain to failure behaviour of polyurethane based laminates is higher and the tensile strain to failure perpendicular to the fibres is lower compared with epoxy based laminates. The shear modulus and tensile modulus are comparable for both types of laminates. The in-plane shear properties and tensile properties perpendicular to the fibres are indicating that there is higher fibre-matrix bonding of the glass fibres to the epoxy. The polyurethane shows tougher behaviour in the flexural test on neat resin specimens. However, this tougher behaviour of polyurethane is not shown in the tensile test on neat resin specimens. In general it can be concluded that polyurethane glass fibre laminates have similar mechanical properties as epoxy glass fibre laminates. This means that one of the additional requirements mentioned in chapter 2, polyurethane based laminates should have similar properties as epoxy based laminates, is fulfilled.

It is questionable if the data obtained from the tensile test on clear castings is correct due to failure in the gripping area and the high difference in tensile strain to failure and tensile modulus properties compared with the reference data obtained from the Airstone 760E:766H data sheet [1]. Therefore it is recommended to redo this test with dog bone specimens according to ISO 527-2.

The fibre volume content influences the mechanical properties of a laminate. Therefore it would be recommended to know if the fibre volumes of the epoxy and polyurethane glass fibre laminates are similar.

Chapter 6

Conclusions and Recommendations

The goal of this thesis was to develop the most appropriate infusion lay-up and procedure for the thermoset polyurethane vacuum infusion process in a lab scale environment. The most appropriate infusion lay-up and procedure should result in visual defect free glass fibre laminates with comparable mechanical properties as an epoxy glass fibre laminate.

- Visual inspections suggest that (partly) defect free polyurethane glass fibre laminates are produced. However, these observations are not confirmed with ultrasonic inspection neither with optical microscopy. Voids and an irregular surface on the vacuum bag side of the polyurethane laminates are detected with optical microscopy.
- From mechanical testing it is observed that the two polyurethane based laminates show comparable mechanical properties and therefore indicating an equivalent quality. The in-plane shear modulus, transverse tensile strength and transverse tensile modulus of the polyurethane and epoxy glass fibre specimens are comparable. The transverse tensile strain to failure of epoxy glass fibre specimens is more than twice the value of the polyurethane glass fibre specimens. The specimens tested in interlaminar shear failed in a combination of shear and compression. This is a mixed mode of failure and unacceptable interlaminar shear failure. Therefore the calculated value of interlaminar shear strength is not an interlaminar shear strength value and may therefore only be used to compare test specimens taken from the same material according to test standard ISO 14130. The in-plane shear properties and tensile properties perpendicular to the fibres are indicating that there is higher fibre-matrix bonding of the glass fibres to the epoxy.
- The polyurethane shows tougher behaviour in the flexural test on neat resin specimens. However, this tougher behaviour of polyurethane is not shown in the tensile test on neat resin specimens. In general it can be concluded that polyurethane glass fibre laminates have similar mechanical properties as epoxy glass fibre laminates. This means that one of the additional requirements mentioned in chapter 2, polyurethane based laminates should have similar properties as epoxy based laminates, is fulfilled.

The best practice to manufacture polyurethane glass fibre laminates with the vacuum infusion technique is defined. The configuration from tooling side to vacuum bag is as follows:

- 4 layers biaxial (±45°) glass fibres, 800g/m^2, unknown supplier
- Peel ply
- Flow mesh

The preparation and infusion parameters are given in Table 6-1.

Parameter	
Index	101
Air release agent	BYK-A 535 1%
Infusion direction	0° fibres
Pre-drying glass fibre for minimal 2 hours	100°C
Peel ply between glass fibres and flow mesh	Yes
Perforated release film between peel ply and flow mesh	No
Degassing time polyol and isocyanate	>20 [min]
Degassing and mixing (390 rpm) time polyol and isocyanate	8 [min]
Infusion pressure	150 [mbar]

Table 6-1: Best practice parameters.

Several recommendations are suggested for further work:

- To perform a fibre volume content measurement on the existing composite laminates. The fibre volume content influences mechanical properties. If the fibre volume content is known, a weighted comparison can be made between the mechanical properties.

- To determine the void content on the existing composite laminates according to test standard ASTM D2734. Information on void content is useful, because high void content can significantly reduce the strength of the composites. Additionally, it can be used as a measure of the consistency of the vacuum infusion process.

- The defects measured with ultrasonic inspection could be investigated with microscopy. Optical microscopy could be used for cross-sectional inspection to detect voids. Stereo microscopy and SEM are more suitable for the inspection of fracture surfaces, to detect voids, delaminations, de-bonding and the type of fracture.

- To redo the tensile test on neat resin specimens according to ISO 527-2 since it is questionable if the data obtained from the tensile test on clear castings is correct due to failure in the gripping area and the high difference in tensile strain to failure and tensile modulus properties compared with the reference data obtained from the Airstone 760E:766H data sheet [1].

- To improve the laminate quality and with that the mechanical properties of the laminates by changing the degassing procedure and the method of finishing the vacuum infusion process.

    - Use of bubble nucleation material (e.g. Scotch Brite) or sparging method during degassing of the polyol and isocyanate [12, 27]. By sparging a container is filled with resin. The pressure in this container is reduced to a pressure below the infusion pressure to be used during the vacuum infusion process. At the bottom, air is fed into this container. The air is forced through a very fine filter, thus creating many small bubbles. These bubbles rise through the resin. At the reduced pressure, the resin will be over-saturated with (components of) air. The difference in gas concentration between the air bubble and the dissolved gas causes gas molecules to diffuse from the resin into the bubble. This process continues until a new equilibrium situation is reached, e.g. the resin is saturated (but no longer over-saturated) with air [12, 27]. Degassing of the resin by exposing the resin to partial vacuum results in a 'de-bubbled' resin and not in outgassing [12]. This is something that cannot be seen during degassing. By adding nucleation material to the resin or by using the sparging method, the resin is more effectively degassed resulting in a better quality laminate during and after infusion [12]. If the infusion pressure is higher than the degassing pressure, there is no risk of outgassing of the resin. There will even be the possibility of dissolving some bubbles, which have been formed during the flow of resin, entrapping air in fibre bundles.

  - The vacuum infusion process can be finished with a different method. The used method was keeping the pressure at 150 mbar and closing the inlet naturally by cured left over polyurethane in the bucket. Another method is to increase the pressure from 150 mbar to 400 mbar after infusion and subsequently close the inlet tube [4]. This method results in an equilibrium pressure at 400 mbar. In the case of a leak, a small pressure gradient remains [4].

- Performing vacuum infusions with a polyurethane formulation that fulfils the initial requirements regarding glass transition temperature (T_g > 70°C after 6h cure at 70°C) and pot life (>45 min before the viscosity is 350 cP at 25°C). There is more time to impregnate the fibres and the bubbles have more time to escape if the pot life is increased. This should result in a decreased void content. Additionally, bigger and or thicker laminates can be infused.

- To infuse lay-ups with more than 4 layers of glass fibres of 800-1000 g/m^2 and or are bigger than 40 x 120 cm. The thickness of the lay-up influences the exothermal reaction of the polyurethane. It could be that thicker and bigger laminates cannot be infused because the pot life is too short. If this is the case, polyurethane is not applicable for large structures.

Chapter 7

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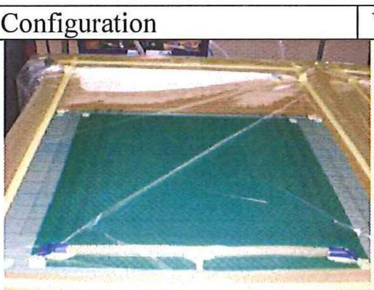
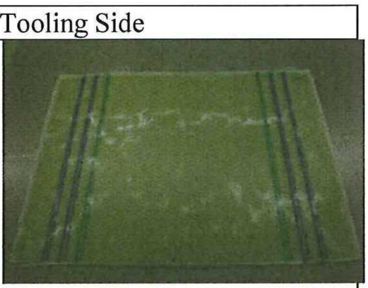
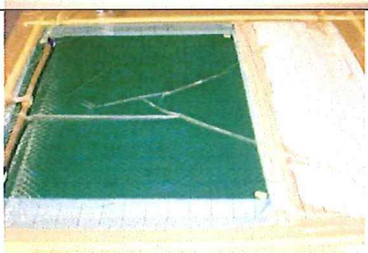
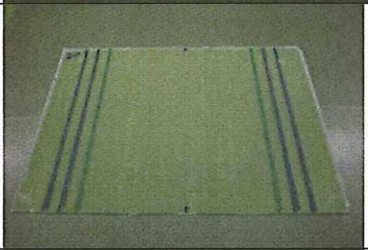
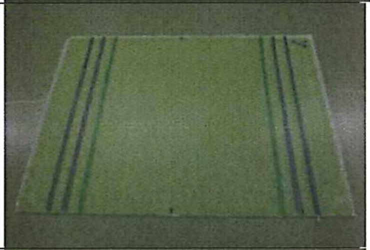
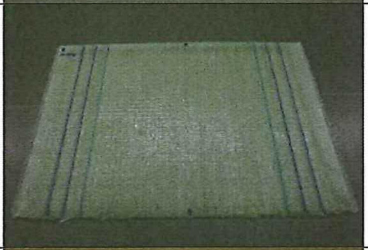
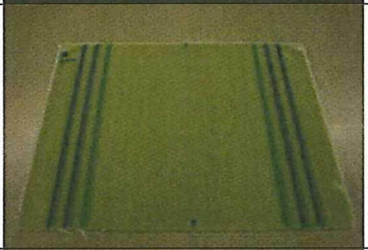
Appendix A.

Photo Overview Vacuum Infusions

#	Configuration	Vacuum Bag Side	Tooling Side
10			
11			
14			
15			
16			
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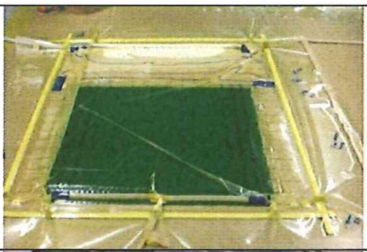
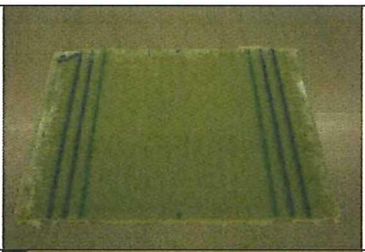
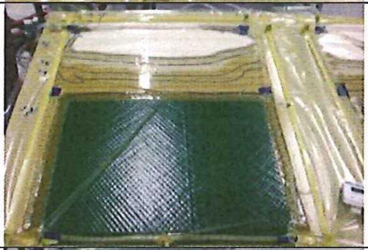
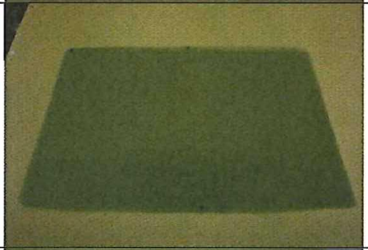
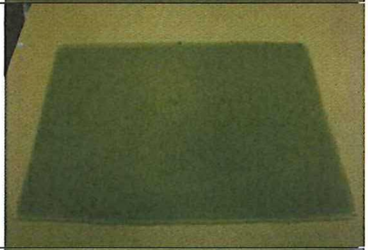
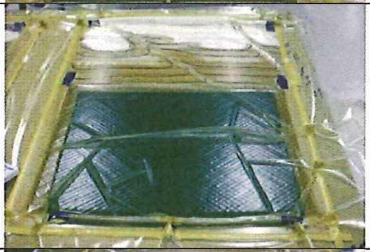
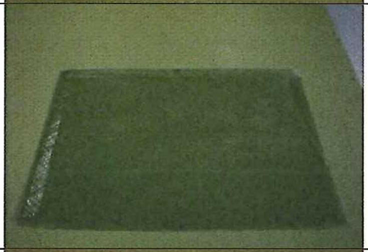
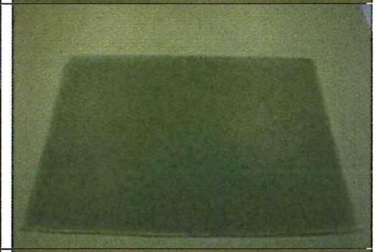
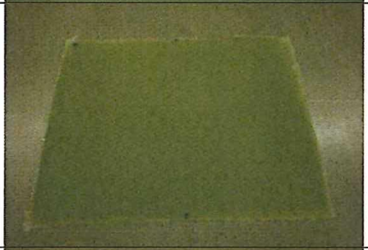
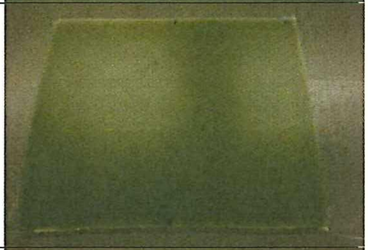
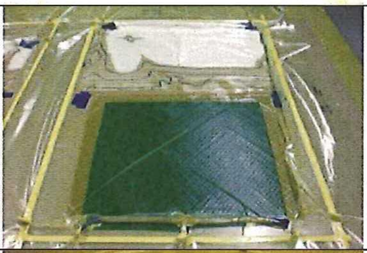
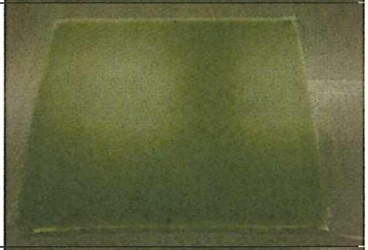
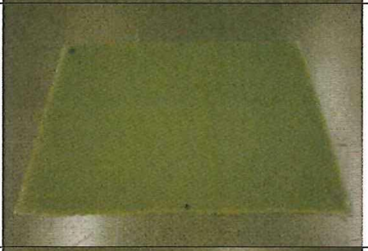
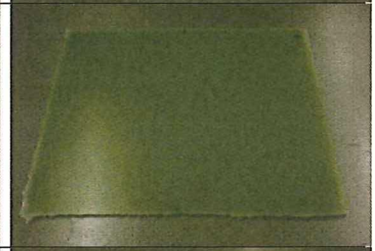
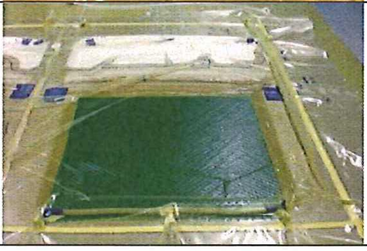
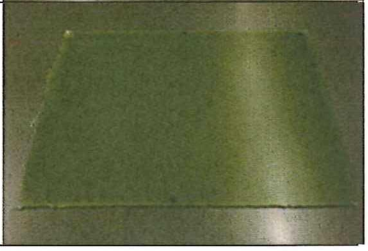
18			
19			
20			
21			
22			
23			
24			

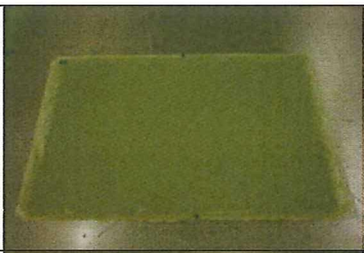
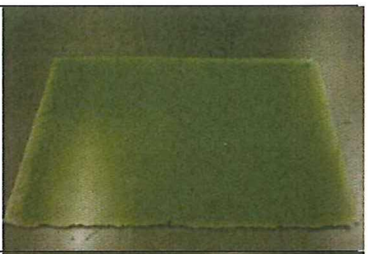
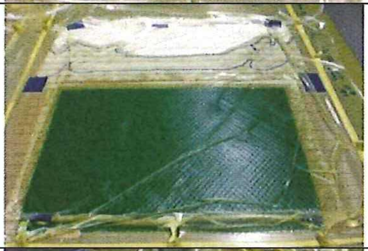
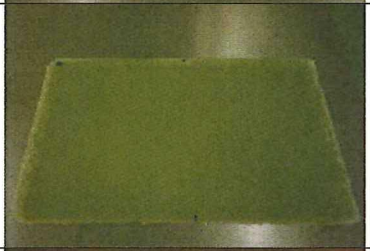
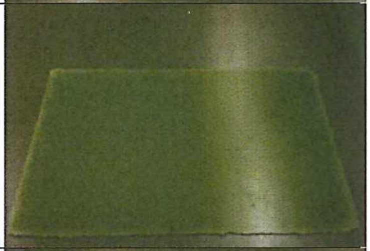
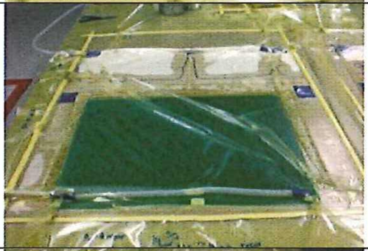
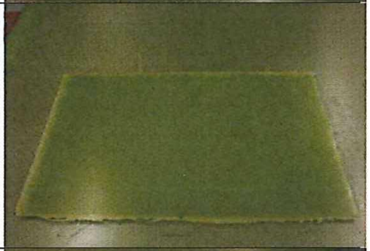
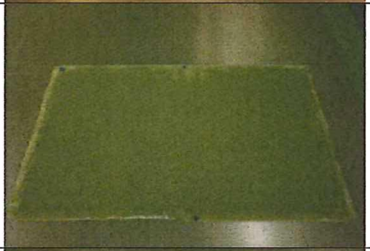
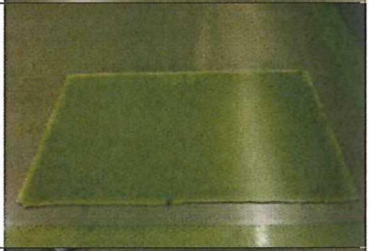
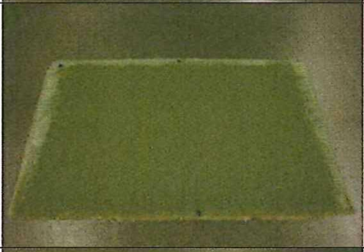
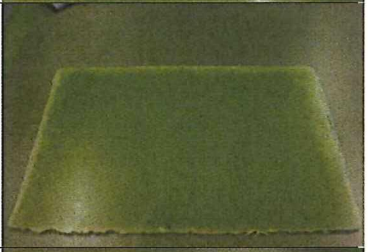
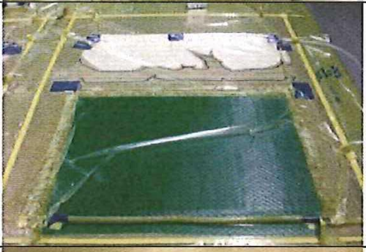
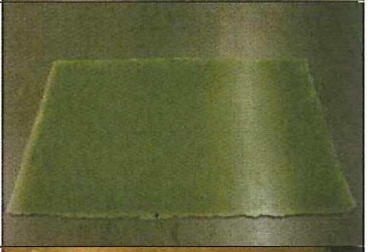
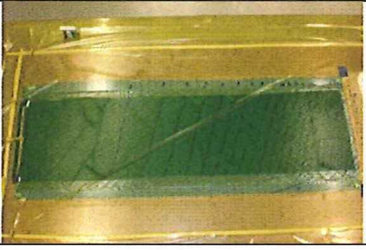
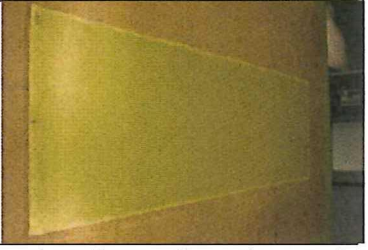
25			
26			
29			
30			
31			
32			
34			

Table 7-1: Photo overview vacuum infusions. From left to right: the infusion number, the configuration used during the infusions, vacuum bag side laminate, tooling side laminate.

Appendix B.

Mechanical Test Results

Composite Laminates - Biaxial Laminates Tensile Test 0° - ISO 527-4

Tensile 0° - 1E	Thickness [mm]	Width [mm]	Cross section [mm^2]	Shear Modulus [MPa]
1	2.96	24.87	73.61	7058
2	2.88	24.88	71.74	7124
3	2.89	24.82	71.72	8825
4	2.88	24.83	71.59	7238
5	2.93	24.89	72.93	8153
6	2.93	24.88	72.82	7280
7	2.90	24.92	72.26	7720
8	2.87	24.86	71.43	8090
9	2.89	24.82	71.64	8297
10	2.89	24.89	71.85	8314

Table 7-2: Dimensions and shear modulus of laminate 1E - 0° tensile test according to ISO 527-4.

Tensile 0° - 25	Thickness [mm]	Width [mm]	Cross section [mm^2]	Shear Modulus [MPa]
1	2.90	24.89	72.18	7437
2	2.83	24.91	70.59	6051
3	2.84	24.82	70.56	7677
4	2.87	24.90	71.54	7638
5	2.88	24.91	71.81	7904
6	2.88	24.88	71.56	7492
7	2.89	24.86	71.84	8156
8	2.89	24.91	71.98	8078
9	2.91	24.88	72.40	7527
10	2.89	24.85	71.83	7595

Table 7-3: Dimensions and shear modulus of laminate 25 - 0° tensile test according to ISO 527-4.

Tensile 0° - 26	Thickness [mm]	Width [mm]	Cross section [mm^2]	Shear Modulus [MPa]
1	2.82	24.89	70.26	7695
2	2.84	24.91	70.82	7585
3	2.81	24.92	70.10	8077
4	2.81	24.89	70.01	8059
5	2.80	24.86	69.62	8042
6	2.80	24.90	69.80	7778
7	2.85	24.91	71.00	7909
8	2.84	24.88	70.73	5460
9	2.84	24.87	70.64	7767
10	2.80	24.90	69.72	7563

Table 7-4: Dimensions and shear modulus of laminate 26 - 0° tensile test according to ISO 527-4.

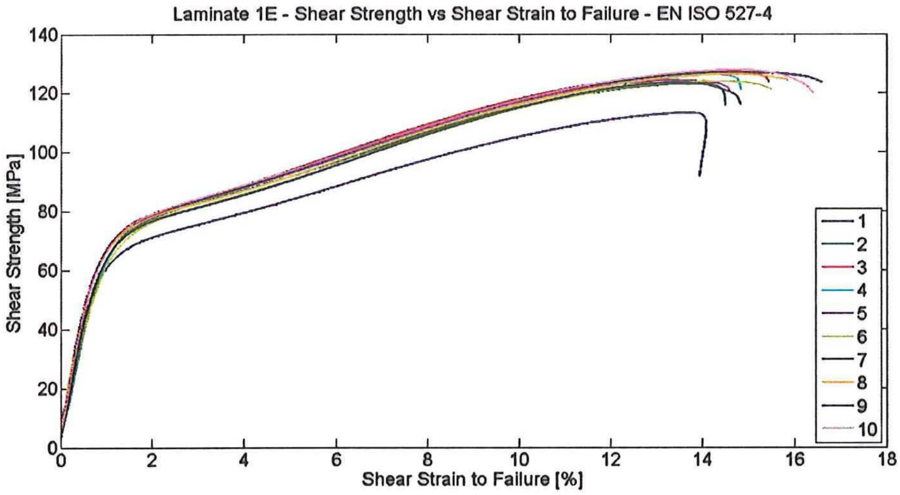


Figure 7-1: Shear strength vs. shear strain to failure for specimens from laminate 1E tested according to ISO 527-4.

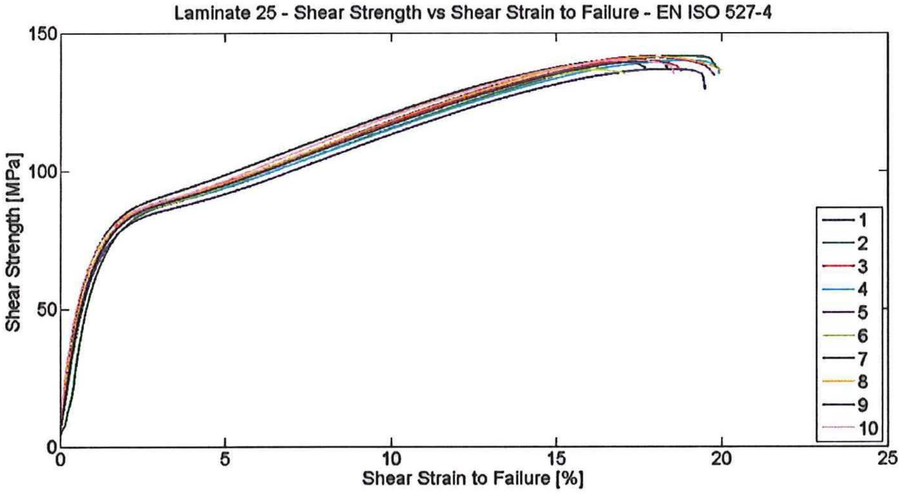


Figure 7-2: Shear strength vs. shear strain to failure for specimens from laminate 25 tested according to ISO 527-4.

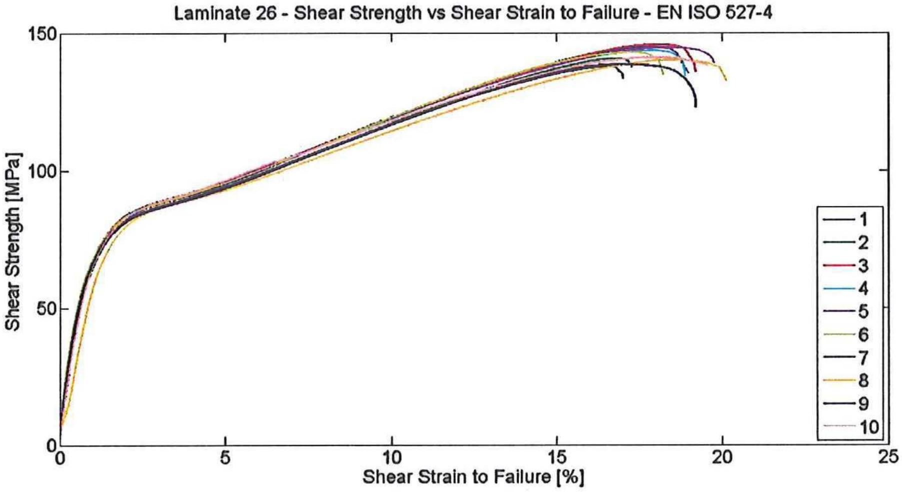


Figure 7-3: Shear strength vs. shear strain to failure for specimens from laminate 26 tested according to ISO 527-4.

Composite Laminates – Unidirectional Laminates Tensile Test 90° - ISO 527-4

Tensile 90° 2E	Thickness [mm]	Width [mm]	Cross section [mm^2]	Tensile Strength [MPa]	Tensile Strain to Failure [%]	Tensile Modulus [MPa]
1	3.12	24.96	77.88	82	2.4	10941
2	3.19	24.89	79.48	78	2.3	10599
3	3.14	24.91	78.12	78	2.0	10649
4	3.14	24.91	78.12	71	2.2	10796
5	3.17	24.3	79.03	71	1.8	12018
6	3.13	24.91	77.87	76	2.1	11916
7	3.13	24.94	7807	84	2.4	11958
8	3.23	24.91	80.38	80	2.1	11866
9	3.11	24.95	77.60	79	2.1	11328
10	3.21	24.89	79.98	78	2.1	11407

Table 7-5: Dimensions and mechanical properties of laminate 2E - 90° tensile test according to ISO 527-4.

Tensile 90° 29	Thickness [mm]	Width [mm]	Cross section [mm^2]	Tensile Strength [MPa]	Tensile Strain to Failure [%]	Tensile Modulus [MPa]
1	3.23	24.95	80.67	75	0.9	8403
2	3.24	24.92	80.75	80	1.0	10433
3	3.24	24.91	80.80	76	0.9	10887
4	3.23	24.93	80.62	71	0.8	9876
5	3.27	24.90	81.43	87	1.2	10850
6	3.22	24.90	80.25	81	1.0	9614
7	3.30	24.87	81.98	54	0.6	9843
8	3.30	24.85	82.10	71	0.8	11614
9	3.33	24.93	83.03	75	1.0	7722

Table 7-6: Dimensions and mechanical properties of laminate 29 - 90° tensile test according to ISO 527-4.

Tensile 90° 32	Thickness [mm]	Width [mm]	Cross section [mm^2]	Tensile Strength [MPa]	Tensile Strain to Failure [%]	Tensile Modulus [MPa]
1	3.06	24.88	79.04	82	0.9	12046
2	3.10	24.88	77.20	61	0.6	11276
3	3.05	24.92	76.08	79	0.9	10591
4	3.13	24.90	77.84	79	0.9	12080
5	3.09	24.95	77.18	81	1.0	10917
6	3.06	24.93	76.20	80	1.0	10412
7	3.05	24.88	75.81	85	1.0	12315
8	3.09	24.92	77.00	73	0.8	9354
9	3.08	24.88	76.64	76	0.9	12231
10	3.08	24.84	76.60	73	0.8	11972

Table 7-7: Dimensions and mechanical properties of laminate 32 - 90° tensile test according to ISO 527-4.

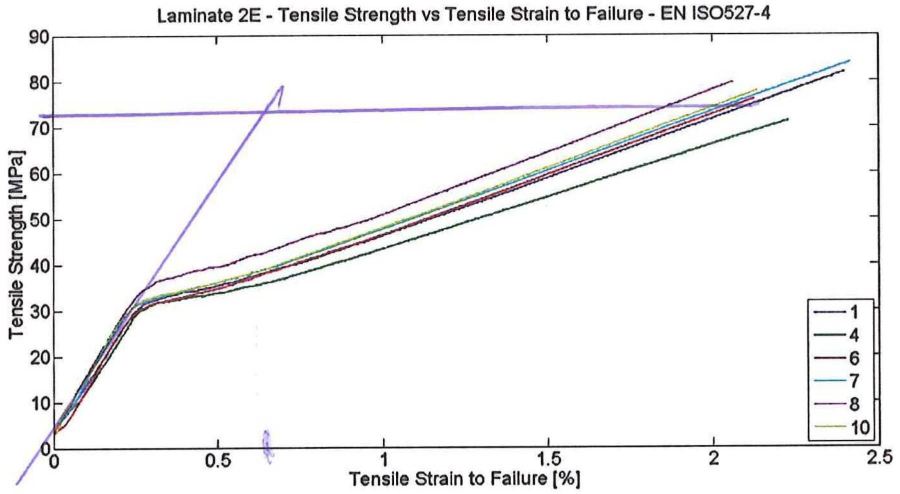


Figure 7-4: Tensile strength vs. tensile strain to failure for specimens from laminate 2E tested according to ISO 527-4.

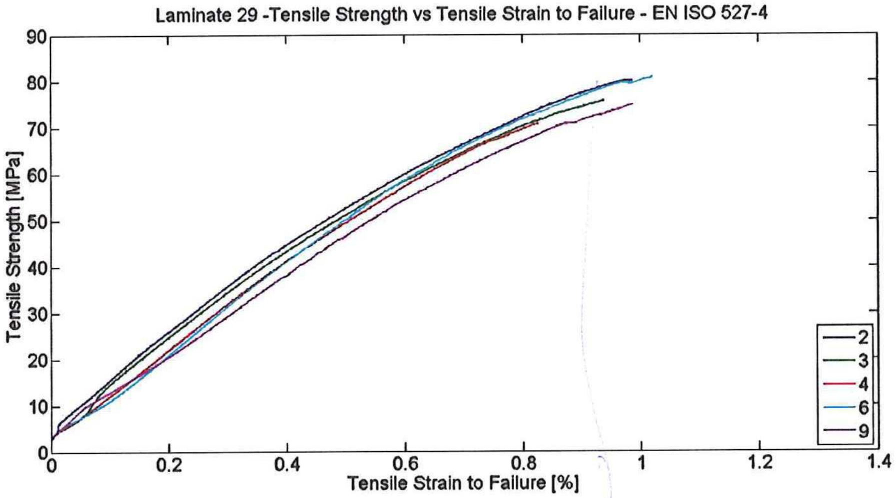


Figure 7-5: Tensile strength vs. tensile strain to failure for specimens from laminate 29 tested according to ISO 527-4.

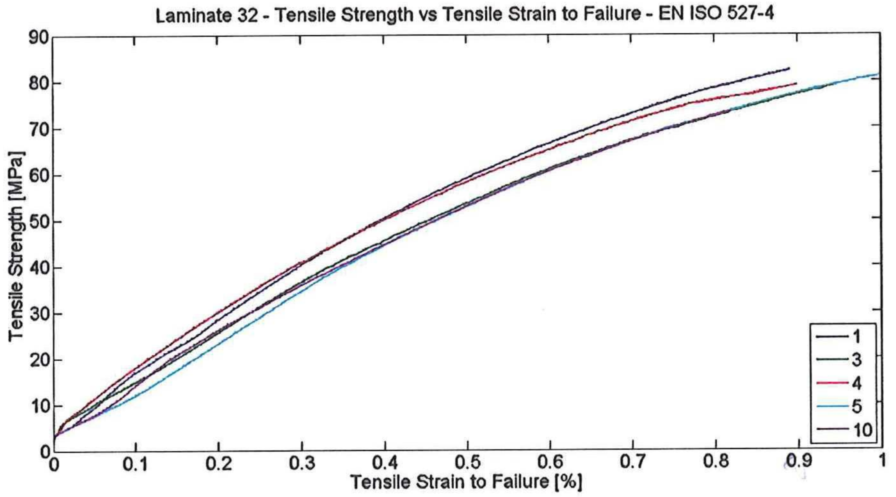


Figure 7-6: Tensile strength vs. tensile strain to failure for specimens from laminate 32 tested according to ISO 527-4.

Composite Laminate – Unidirectional Laminates ILSS

ILSS 2E	Thickness [mm]	Width [mm]	Cross section [mm^2]	Interlaminar Shear Strength [MPa]
1	3.14	14.89	46.75	48
2	3.18	14.89	47.35	47
3	3.18	14.90	47.38	44
4	3.23	14.90	48.13	47
5	3.22	14.93	48.07	49
6	3.19	14.87	47.44	47
7	3.19	14.89	47.50	49
8	3.16	14.91	47.12	46
9	3.11	14.90	46.34	46
10	3.21	14.86	47.70	46

Table 7-8: Dimensions and mechanical properties of laminate 2E - ILSS test according to ISO 14130.

ILSS 29	Thickness [mm]	Width [mm]	Cross section [mm^2]	Interlaminar Shear Strength [MPa]
1	3.22	14.87	47.88	54
2	3.20	14.86	47.55	53
3	3.33	14.88	49.55	52
4	3.23	14.87	48.03	53
5	3.32	14.88	49.40	53
6	3.21	14.86	47.70	50
7	3.20	14.87	47.58	51
8	3.25	14.87	48.33	50
9	3.25	14.87	48.33	51
10	3.30	14.87	49.07	54

Table 7-9: Dimensions and mechanical properties of laminate 29 - ILSS test according to ISO 14130.

ILSS 32	Thickness [mm]	Width [mm]	Cross section [mm^2]	Interlaminar Shear Strength [MPa]
1	3.04	14.87	45.20	50
2	3.05	14.91	45.48	52
3	3.03	14.88	45.09	52
4	3.03	14.87	45.06	51
5	3.04	14.88	45.24	50
6	3.08	14.87	45.80	47
7	3.11	14.88	46.28	50
8	3.05	14.87	45.35	53
9	2.99	14.89	44.52	51
10	3.06	14.87	45.50	50

Table 7-10: Dimensions and mechanical properties of laminate 32 - ILSS test according to ISO 14130.

Neat Resin – Tensile Test

Tensile Test EP	Thickness [mm]	Width [mm]	Cross section [mm^2]	Tensile Strength [MPa]	Tensile Strain to Failure [%]	Tensile Modulus [MPa]
1	3.10	10.65	33.02	67	10.8	1163
2	3.10	10.71	33.20	68	12.1	1137
3	3.10	10.59	32.83	68	12.7	1304
4	3.10	10.48	32.49	68	11.0	1296
5	3.13	10.55	33.02	67	11.4	1160

Table 7-11: Mechanical properties of clear casted epoxy specimens – tensile test according to ISO 527-4.

Tensile Test PU	Thickness [mm]	Width [mm]	Cross section [mm^2]	Tensile Strength [MPa]	Tensile Strain to Failure[%]	Tensile Modulus [MPa]
1	2.92	10.65	31.10	51	9.4	917
2	2.88	10.11	29.12	52	8.1	1038
3	2.84	10.4	29.54	52	9.2	956
4	2.79	9.89	27.59	54	12.3	1098

Table 7-12: Mechanical properties of clear casted polyurethane specimens – tensile test according to ISO 527-4.

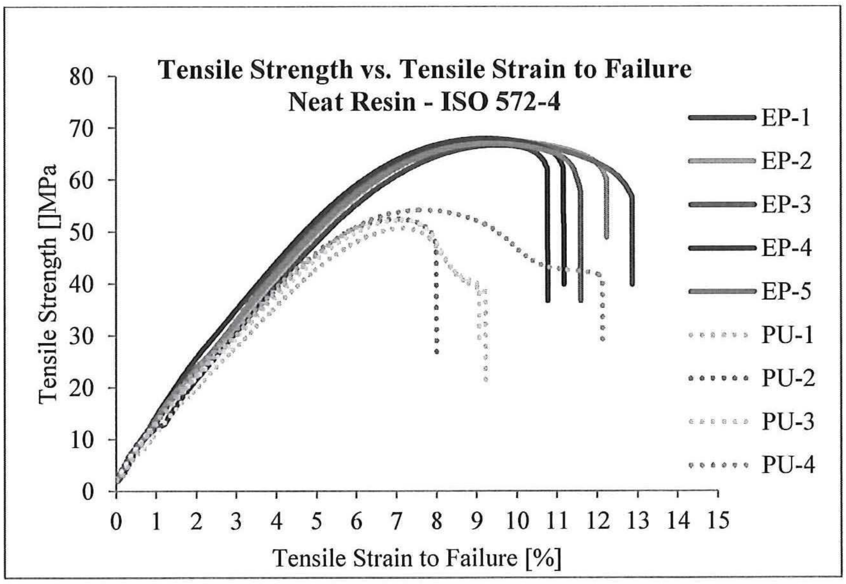


Figure 7-7: Tensile strength versus tensile strain to failure for neat resin specimens tested according to ISO 527-4.

Neat Resin - Flexural Test

Flexural Test EP	Thickness [mm]	Width [mm]	Cross section [mm^2]	Flexural Strength [MPa]	Flexural Strain at Flexural Strength [%]	Flexural Modulus [MPa]
1	3.19	10.62	33.88	119.	6.1	3100
2	3.14	10.4	32.66	117	5.8	3131
3	3.04	10.54	32.04	120	6.3	3190
4	3.17	10.56	33.48	119	6.2	3088
5	3.13	10.4	32.55	119	7.3	3111

Table 7-13: Mechanical properties of clear casted epoxy specimens – flexural test according to ISO-178.

Flexural Test PU	Thickness [mm]	Width [mm]	Cross section [mm^2]	Flexural Strength [MPa]	Flexural Strain at Flexural Strength [%]	Flexural Modulus [MPa]
1	2.95	10.64	31.39	106	5.2	2956
2	2.78	10.85	30.16	103	5.8	2859
3	2.96	10.64	31.49	104	5.5	2813
4	2.89	10.67	30.84	104	5.5	2868
5	2.83	10.75	30.42	103	5.0	2888

Table 7-14: Mechanical properties of clear casted polyurethane specimens – flexural test according to ISO-178.

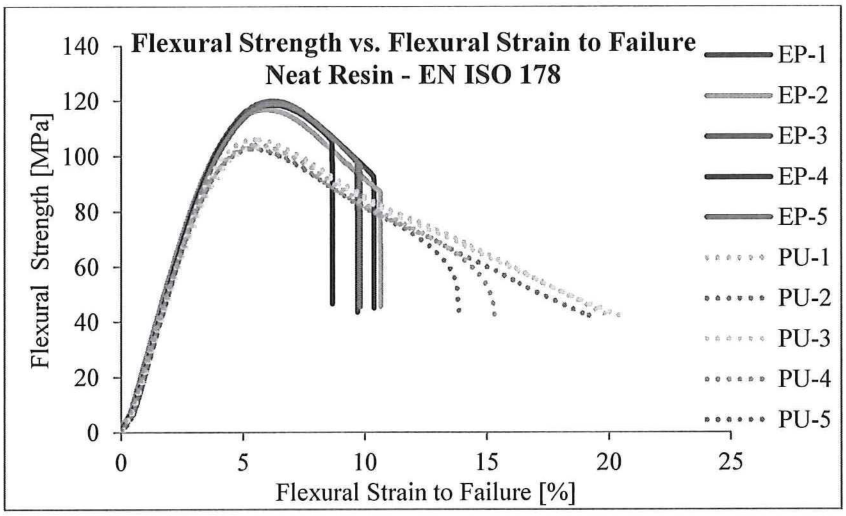


Figure 7-8: Flexural strength versus flexural strain to failure for neat resin specimenstested according EN ISO 178. Failure of polyurethane specimens did not occur.