The effect of X-core foam processing on surface defects in carbon fibre-epoxy sandwich composites

Redefining sandwich manufacturing with X-core

D.O. Berckmoes

In cooperation with Donkervoort Automobielen B.V.

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by

D.O. Berckmoes

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In cooperation with Donkervoort Automobielen B.V.

Student number:	4146565	
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Thesis committee:	Ir. J. Sinke,	Supervisor, TU Delft, Structural Integrity and Composites
	Dr. Ir. O.K. Bergsma	TU Delft, Structural Integrity and Composites
	Dr. J.C. Bijleveld,	TU Delft, Novel Aerospace Materials
	Ing. R. Grooten,	Company supervisor, Donkervoort Automobielen B.V.

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Preface

Before you lies a Master's thesis, the synthesis of what has been six years of studying Aerospace Engineering at the Delft University of Technology. Nine months of research, manual labour and strenuous typing have been dedicated to this very report on the X-core process. X-core is being developed by Donkervoort Automobielen, which is principally the only Dutch sports car manufacturer. My passion for sports cars initially got me to do an internship at Donkervoort, but it was X-core and the potential it offers that convinced me to do a thesis research. It has been challenging at times due to failing equipment and inconsistent test results whilst it always seems like a race against the clock. But I have gained a lot of valuable hands-on experience and it was exciting to explore the boundaries of this process. Being part of the development of X-core has been very rewarding and I look forward to seeing it develop into a commercialized process.

I wish to express my most sincere gratitude to Roel Grooten for always being open to discussion, inspiring me to ever work at the best of my capabilities and, most of all, for entrusting me with this project. A special thanks also goes to my supervisor Jos Sinke for taking his time to help me make the most of my research and providing me with valuable feedback.

Many thanks are also due to Matthijs Eversdijk for all his support, insights and experimental data. It was nice to know that I was not the only one struggling with a thesis, it was enjoyable working together and, frankly, I believe we both pushed each other to a higher level. I am also indebted to Stijn Vial and Quintus Boechem for laying the foundation for my research and for helping me familiarize with X-core in the workshop. Finally I would like to acknowledge the contribution of my colleagues at Donkervoort; thank you for borrowing your tools, your time and for sharing your knowledge to help me solve my problems.

D.O. Berckmoes
Lelystad, June 2017

Abstract

X-core is a thermo-expandable syntactic foam that has been developed by Donkervoort automobielen to enable a novel process for the manufacture of composite sandwich structures. This process is unique because the compaction pressure on the laminate is delivered from inside the component by the expansion of Expancel® DU microspheres in the foam. This allows for the fabrication of seamless components with mould-defined features all-around in a single-step operation. However, the quality of the cured products is not yet comparable to that of the vacuum bag cured components at Donkervoort. The main problem is the visible presence of surface defects in X-core components. The objective of this thesis is to develop processing guidelines that help to reduce the surface defects in X-core sandwich structures.

In an exploratory study sandwich samples with carbon fibre prepreg facings and a 270 g/L X-core foam are subjected to a variety of processing conditions. A new prepreg system containing c-m-p CP0093 resin and a two-ply laminate with a 245 g/m^2 3k fabric and 420 g/m^2 12k fabric was proposed for a first process window study. Eighteen samples are manufactured in a designed experiment to determine the effect of cure temperature, heat-up rate, core thickness and Expancel® DU content in X-core on the surface porosity content. The results indicate that no samples could be manufactured with a surface porosity lower than 0.68% within the defined range of the process variables. It had however been proven that a porosity content of 0.025% is achievable within this range of conditions when a two-ply laminate is used solely composed of 245 g/m^2 3k fabric. A porosity content 0.68% is easily detectable with the naked eye and impairs the surface finish, whereas 0.025% is nearly invisible.

The difference is attributed to the out-gassing of interstitial/entrapped air in X-core into the laminate. When a vacuum film is used between the laminate and the core (an impermeable barrier) the surface porosity can be mitigated almost entirely. The 3k ply has a smaller out-of-plane permeability than the 12k ply, so it is expected to act as a more effective barrier, which helps to reduce surface porosity. A second process window study is performed with a vacuum film barrier layer between the core and laminate, solely focussing on the effect of DU volume content. The results show that a viable process window can be identified when an effective barrier layer is used.

Based on these results processing guidelines are formulated. The parameters that mainly affect the surface finish are DU content, the weave of the fibres in the different plies of the laminate and the extent of mould filling with X-core. The DU contents should be as high as possible (increased pressure), but care should be taken that the tooling can cope with the pressure. Tightly woven fabrics are more effective to prevent penetration of X-core through the weave and to reduce surface porosity. Finally, it is important to fill the mould 100% with X-core, there should always be a tendency to reduce air entrapment in the core or between the core and facings.

Floor panels for the Donkervoort D8 GTO RS are manufactured following these new guidelines and a new two-step cure cycle. There is still a large surface porosity content (2.5-4.5%) but other surface defects like X-core penetrating through the weave, waviness of the surface ply and large air inclusions are no longer present. Because the floor panel is a flat and horizontal surface the porosity was easily mitigated by applying one layer of transparent filler. For complex geometry components this will not be achieved as easily and more time is required for rework. Further research is therefore recommended in addressing the main source of porosity in X-core structures: entrapped air inside the core and between the core and the laminate.

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Introduction

Donkervoort automobielen is a Dutch car manufacturer that was founded in 1978 by Joop Donkervoort. The company specializes in exclusive high-end sports cars that have found their origin in Colin Chapman's famous Lotus Seven. At Donkervoort a "*No Compromise*" philosophy is pursued; anything that may impair the on-track performance of the car is simply stripped out or reduced to a minimum. Their latest model, the D8 GTO RS, reflects all of that: A lightweight hybrid steel space frame stiffened by carbon fibre/epoxy body panels, a powerful turbocharged 2.5 L Audi five-cylinder engine and practically no electronic assists that interfere with the driving experience.

In the sports- and race car industry the use of carbon fibre reinforced plastics (CFRP) has become prevalent in load-bearing components. CFRPs offer superior strength- and stiffness-to-weight ratios over steel and aluminium alloys, this allows sports car manufacturers to reduce the mass of the chassis to the advancement of the car's performance. At Donkervoort the use of carbon fibre in structural components took off with the launch of the D8 in 2007. It was also the company's first production car that was fitted with a door that is made using carbon fibre. The door is a sandwich beam where the core is completely enclosed by the laminate.

It is specifically this structure that has lead to the development of X-core: Donkervoort wanted to have a process that could make sandwich structures with mould defined features all-around in a one-shot process. The idea was to make use of an expanding core material that can compact the laminate from the inside out in an enclosed mould.

X-core is a unique thermoset syntactic foam containing Expancel® thermo-expandable microspheres which allows it to expand at elevated temperatures. In its current form it is a 300 kg/m^3 foam that is proven to be capable of providing an expansion pressure of anywhere up to 16 bar. It is typically co-cured to carbon fibre/epoxy prepreg laminates at a 100°C temperature, the products do not need to be vacuum bagged or cured in an autoclave.

X-core has proven that it can achieve its intended goal, but the process is not flawless. A large problem that is faced in the manufacture of X-core structures is the visible presence of surface defects, which especially impairs the aesthetics of 'bare carbon' products. These defects include surface porosity, waviness or distortions in the surface ply and penetration of X-core through the weave. Preventing these flaws helps to prevent rework and to save costs, but that requires a more thorough understanding of the process and the effect of processing conditions on the surface quality of these structures. Therefore the objective of this research is to:

Formulate processing guidelines for repeatable one-shot production of X-core sandwich structures with co-cured fibre reinforced polymer facings by increasing the understanding of the relationship between process conditions and surface defects in these structures.

This report presents the details of a research that is performed in a time-frame of nine months. The steps that are taken to achieve this goal are summarized in a flow chart in Figure 1.1. The research starts by exploring a large variety of process variables. Then, with each step the study progresses the contribution of different variables is assessed and the research is narrowed down by identifying the most influencing parameters.

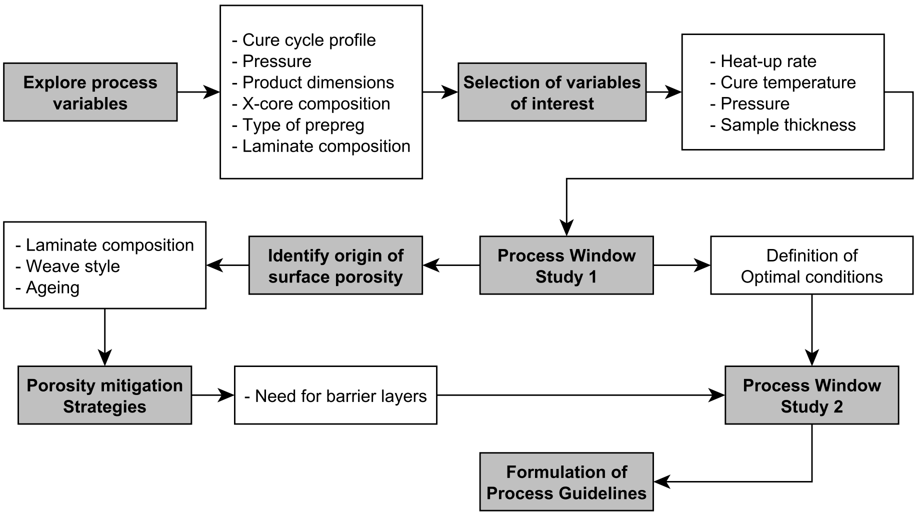


Figure 1.1: *Flowchart of the steps that are taken to achieve the research objective.*

The report start out with a brief literature review on composite sandwich structures and porosity in fibre reinforced polymer laminates in Chapter 2. Chapter 3 covers the concept of X-core and how it has developed over the years. This is followed by a description of the process itself in Chapter 4, where the constituents of X-core and the integrally heated tooling technology that is used for making these products are detailed. Up to this point in the report the background has been covered, the description of the research itself starts in Chapter 5 where the methodology and experimental set-up is presented. The remainder of the report follows the flowchart that is presented in Figure 1.1. It starts in Chapter 6 where the relevant process variables are identified and selected for subsequent research. The first process window study is covered in Chapter 7, it focusses on the effect of the cure cycle, pressure and core thickness on the surface porosity. The data from this study suggested that surface porosity could not be prevented under the conditions of the experiment. This problem initiated a study into the origin of the surface porosity in the X-core process, it is the topic of Chapter 8. Subsequently, in Chapter 9 a new study is performed to explore whether a process window could be defined at all within the bounds of the defined process variables. All of the results are then combined to formulate processing guidelines, which are presented in Chapter 10. Based on these guidelines a set of floor panels is manufactured in order to assess their effectiveness, the result of this procedure is presented in Chapter 11. Finally the report is concluded in Chapter 12, followed by recommendations for further research in Chapter 13.

2

Background on sandwich structures in composites

Sandwich structures are used in many weight critical applications like aircraft, spacecraft and sports equipment. The main design philosophy of sandwich constructions is increasing the flexural stiffness and strength of structural panels by adding a relatively small amount of weight. This 'weight addition' is the core material, it is used to separate stiff and strong face sheets that carry most of the applied load. This principle is explained in more detail in Section 2.1. X-core is a core material, more specifically it may be categorized in the group of syntactic foams, these foam materials are covered in Section 2.2.

The research presented in this report directs the attention towards the face sheets of the sandwich, which is a carbon fibre/epoxy composite in this case. In Section 2.3 the constituents of this composite and the curing process using pre-impregnated fibres (prepreg) is described. Section 2.4 addresses voids and porosity in fibre composites. Their origin, their effect on the properties of the composite and possible mitigation strategies are discussed. Finally, the chapter is concluded in Section 2.5, where the background on sandwich structures is coupled to the research presented in the remainder of this thesis.

2.1. The principles of sandwich construction

Thin monolithic plates offer good in-plane properties, but the out-of-plane properties are comparatively poor. Sandwich structures offer the solution to this problem whilst introducing a minimal weight penalty. The concept of its working principle, the influence of adding a core material on the flexural properties and the failure modes of sandwich panels are covered in this section.

2.1.1. The sandwich concept

A sandwich panel consists of two strong and stiff face sheets that are separated by a relatively lightweight and thick core material that is bonded to the facings with an adhesive. These elements of the sandwich structure are shown in Figure 2.1 for a panel with a honeycomb core.

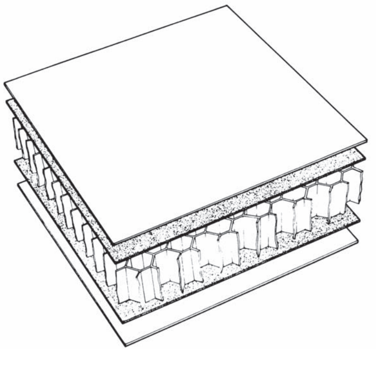
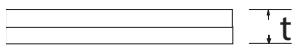
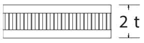
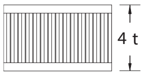


Figure 2.1: *A sandwich structure with a honeycomb core, including two facings and two layers of adhesive [1].*

Separating the face sheets with the core material is an effective measure to increase the area moment of inertia of the panel. The contribution of the facings to the inertia increases quadratically when the distance between the midplane of the panel and the facing is increased (Steiner's Theorem). The flexural stiffness of a panel is proportional to the inertia and the direct stresses in the facings due to bending is inversely proportional to the flexural stiffness. This means that increasing the core thickness yields disproportionally large increases in the flexural properties of the panel. Especially when a lightweight core material is used the mass penalty can be small and the gain in structural efficiency is considerable, see Table 2.1 for an example. Commonly used core materials are polymer foams, balsa wood and aluminium or Nomex (aramid) honeycombs.

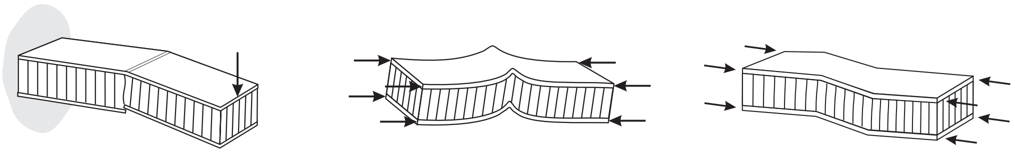
Table 2.1: *An example comparing structural efficiency of sandwich panels and a monolithic plate [1].*

			
Relative Bending Stiffness	1	7.0	37
Relative Bending Strength	1	3.5	9.2
Relative Weight	1	1.03	1.06

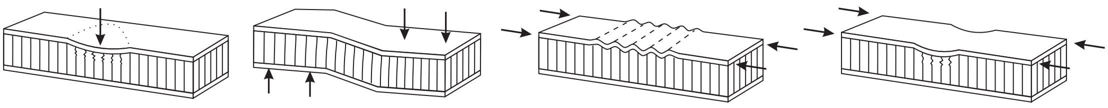
The facings are generally the stiffest and strongest element of the structure, all in-plane loads (shear, tension, compression) are predominantly carried by the facings. For out-of-plane loads the facings carry the direct stresses, the load is transferred between the facings by shear through the adhesive and the core. The core also supports most of the out-of-plane compression loads. The core material can be used to serve several other functions as well, for instance acoustic damping, thermal insulation and vibration damping. Integration of functions can yield additional weight savings in structures, which, combined with the improved flexural properties, has rendered sandwich panels a popular choice for many applications.

2.1.2. Failure modes of sandwich panels

Predicting the in-plane and out-of-plane stiffness properties of a sandwich panel is relatively straightforward and may be done through classical laminated plate theory. However, the prediction of the strength is complicated due to the composite nature of the sandwich structure. There are more failure modes than for monolithic laminates: besides the known failure modes of the facings there are also new modes related to core failure, adhesive failure and local instabilities. Some of the most common failure modes are depicted in Figure 2.2



(a) Skin compression failure (b) Panel buckling (c) Shear crimping



(d) Local impression (e) Core shear failure (f) Intra cell buckling (g) Skin wrinkling

Figure 2.2: *Common failure modes of sandwich panels [2]. Modes (b), (c), (f) and (g) are buckling related failure modes (instability), whereas (a), (d) and (e) relate to failure of the constituent material.*

Under tensile or compressive loadings, which may also be introduced by bending moments, there may be cohesive failure of the facings. The failure load can be predicted, but its value may vary significantly dependent on the chosen failure criterion, e.g. Tsai-Hill, Tsai-Wu, maximum stress, maximum strain and Puck [3]. Under in-plane compression loading there may also be instability related failure modes that can precede compression failure of the facings, see Figures 2.2b, 2.2c, 2.2f and 2.2g. Panel buckling is principally the same as column buckling, it is not different for a sandwich panel than for a monolithic plate. Intra cell buckling only occurs in cellular core materials like honeycomb, in intra cell buckling the core and the adhesive bond remain intact but the facings become unstable and the load carrying capability of the structure is impaired. In skin wrinkling the core is either crushed by the face sheet that kinks inwards or the skin kinks outwards and there is failure of the adhesive bond or cohesive failure in the core.

The local impression (local crushing) and core shear failure modes can exist under out-of-plane loading (Figures 2.2d and 2.2e). The failure loads can be approximated based on the shear/compressive strength of the core and the size of the area on which the load is exerted.

A major point of concern in sandwich composites is barely visible impact damage (BVID), which may for instance be caused by hail storms or dropping tools during fabrication or maintenance. It is not easily detected with the naked eye but it can significantly impair the structural integrity of the sandwich structure [4]. A study by P.M. Schubel et al. [5] revealed that delamination damage may reduce the compressive strength to less than half the original undamaged strength in carbon fibre/PVC foam sandwich panels. These delaminations are not easily detected through visual inspection, an ultrasonic C-scan was used to determine the presence of delaminations and the extent of the damage.

2.2. Syntactic foams

X-core is a syntactic foam, which is in itself a composite material. By definition a syntactic foam is a material containing hollow fillers in a matrix. Polymer matrices and in particular thermoset polymer matrices have been most widely accepted in syntactic foams. They are manufactured by mixing hollow fillers into the polymer, after which the mixture can be cast into a mould where it solidifies. Similar to ordinary polymer foams a blowing agent can be added to the mixture as well, which allows the foam to expand into a mould cavity. This is principally what is done in X-core, it is explained in more detail in Chapter 3.

Syntactic foams can be easily distinguished from ordinary polymer foams; the microballoons that are embedded in the matrix are so small that the material appears like a homogeneous solid, in ordinary polymer foams the pores that are caused by the blowing agent are easily seen with the naked eye [6]. The properties of syntactic foams can be tailored by changing the fillers and their properties; for instance the volume fraction of fillers, the size of the fillers, the microballoon material or the wall thickness of the microballoon. Pinisetty et al. has gathered compression strength data of epoxy/hollow glass microsphere syntactic foams in literature and summarized them in the graph in Figure 2.3. It shows that the relation between foam density and compressive strength is by approximation linear.

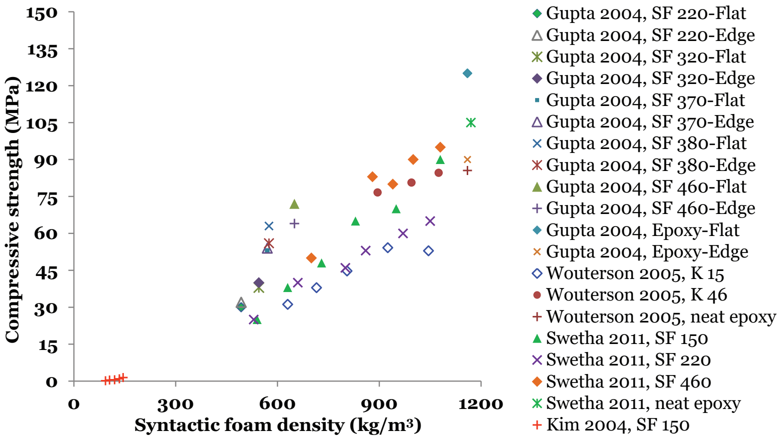


Figure 2.3: *Compressive strength versus density for a variety of epoxy/hollow glass microsphere syntactic foams found in literature, summarized by Pinisetty et al. [7].*

In general syntactic foams are characterized by an excellent hydrostatic strength, good impact properties and damage tolerance compared to other closed-cell foams. They are predominantly used in the off-shore industry in buoys, small boats, submarines and flotation in offshore drilling. [6].

2.3. Manufacturing of composite laminates

The sandwich structure in this research consists of X-core foam and a carbon fibre reinforced epoxy laminate for the facings. This section focusses on the facings, X-core is covered in more detail in Chapters 3 and 4. The description of the manufacturing process of the facings starts in this section with the constituents materials: epoxy and carbon fibre. The fibres can be purchased in a pre-impregnated format where the epoxy is already incorporated, which is also known as a prepreg. This format is often used in processes like autoclave curing and Vacuum Bag Only (VBO) curing, and it is also used in the X-core process. This section covers prepregs and the process by which they are cured.

2.3.1. Constituents of the laminate

In Fibre Reinforced Plastics (FRP) a polymer matrix is used to support and embed fibres that are much stronger and stiffer. It is by definition a composite material; there is a distinct interface between the two materials. The fibres are the main load-carrying element, the polymer matrix keeps them in the desired orientation, transfers the loads between the fibres (especially in-plane and inter-laminar shear loads) and protects them from the environment. The discussion is confined to epoxy matrices and carbon fibres because of their relevance for this research.

Epoxy

A common starting material for epoxies is diglycidyl ether of bisphenol A (DGEBA). At each end of this molecule there is an epoxide group, which is a ring containing two carbon atoms and one oxygen atom. An amine-based hardener is used to start the cross-linking reaction, the amine groups (NH_2) react with the epoxide groups. These hardeners contain at least three functional amine groups, allowing them to form the junctions in a network structure of interconnected DGEBA polymer chains. There is no by-product in this reaction, there is only the cross-linked polymer [8].

Epoxies are either supplied as a pre-mixed system with a limited shelf-life or as a two-component system of resin and hardener that need to be mixed in the right proportions. The epoxy in X-core is a DGEBA based epoxy that is supplied as a two component system. It is for these systems important that the user strictly adheres to the required weight or volume ratio for mixing the components. The mixing ratio affects curing time and cross-linking density, not complying to this ratio leads to a deviation of the mechanical properties from the values that are indicated on the technical data sheet for the cured polymer.

Pre-mixed epoxies are found in prepregs, which are discussed in Section 2.3.2. In this case the components have been mixed by the supplier, guaranteeing the right mixture ratio. This does however initiate the polymerization reaction, so the reaction needs to be slowed down to extend the shelf-life. For prepregs this is achieved by keeping them in cold-storage, at about -23°C.

There is a large variety of base materials, curing agents and additives for epoxies, which renders it a versatile matrix material. In general, their principal advantages and disadvantages compared to other thermoset matrices (e.g. polyester, vinyl ester, bismaleimides) are:

- (+) High strength
- (+) No emission of volatiles
- (+) Low shrinkage during cure
- (+) Excellent chemical- and solvent resistance
- (+) Excellent adhesion to a wide variety of materials and tendency to wet surfaces easily

- (-) High cost
- (-) Long time to cure

Carbon fibre

The widespread use of carbon fibre in all commercial applications has been limited so far, mainly due to their high cost. They are predominantly used in the aerospace industry and in sporting goods because they offer a high specific strength and an exceptionally high specific stiffness, which is desirable for weight-critical applications. In addition they boast a very low coefficient of thermal expansion and a high fatigue strength compared to metal alloys. They do however suffer from a low failure strain and low impact resistance compared to metals [8]. These fibres are also known for their electrical conductivity, which poses problems like galvanic corrosion when combined with metals or shorting of electrical circuits.

Different types of carbon fibres are commercially available in a wide variety of mechanical properties, they are often characterized by the magnitude of the tensile modulus. Generally, low modulus fibres have a higher strength, lower cost and lower density. Typical properties of high strength (HS), Intermediate modulus (IM) and High Modulus (HM) PAN-based carbon fibres are provided in Table 2.2. Properties of commonly used glass fibres and aramind fibres (kevlar) are also provided in the table as a reference.

Table 2.2: *Properties of common glass fibres, PAN-based carbon fibres and kevlar fibres [9]*

Fibre material	Tensile modulus [GPa]	Tensile strength [MPa]	Density [kg/m^3]	Failure strain [%]	Filament diameter [μm]
E-Glass	76-78	3100-3800	2550	4.5-4.9	10
S-Glass	88-91	4380-4590	2490	5.4-5.8	10
HS Carbon	220-241	3450-4830	1800	1.5-2.2	6-8
IM Carbon	290-297	3450-6200	1800	1.3-2.0	5-6
HM Carbon	345-448	3450-5520	1900	0.7-1.0	5-8
Kevlar 49	131	3600-4100	1440	2.8	12
Kevlar 149	179	3400	1470	2.0	12

2.3.2. Pre-impregnated fibres (prepregs)

Fibres that have already been impregnated with catalysed resin are commercially available, they are referred to as pre-impregnated or 'prepreg' systems. Prepregs are made with both woven fabrics and unidirectional tapes and are most commonly impregnated with epoxies. The main advantage that they offer is consistency in the manufacturing process: There is always the same resin content and the same resin-to-hardener ratio.

After impregnation the degree of cure of the epoxy is advanced to the 'B-stage'. This is the moment in the polymerization reaction where the cross-linking has already started, but the density of the cross-links is still very low. The cross-link density directly influences the viscosity of the polymer, at the B-stage the epoxy is a tacky resin that strongly adheres to the fibres and it is viscous enough to prevent flow at room temperature. Cold storage is necessary to slow down the curing reaction, this helps to extend the shelf life to 6-12 months where it would only be a matter of days or weeks at room temperature [8]. When the prepreg is taken out of cold-storage it needs to be conditioned at room temperature first. This is done whilst the prepreg remains in a sealed plastic bag to prevent condensation of water onto its surface. The amine-based hardeners are hydrophilic and attract moisture, which causes void formation when curing at elevated temperatures. The curing process is covered in Section 2.3.3, the theory of void formation is discussed in Section 2.4.1.

2.3.3. The curing process for prepregs

Curing prepregs requires pressure, temperature and time. Temperature and time define the 'cure cycle', the way in which the pressure is applied and the magnitude of the pressure is characteristic of the process. Two widely used processes in the composites industry for curing epoxy based prepregs are autoclave curing and Vacuum Bag Only (VBO) curing. In both processes the prepreg is cut to the correct size and collated onto the product mould. The uncured laminate is then covered with release film, bleeder, breather and a vacuum bag, these are referred to as the consumables, see Figure 2.4a.

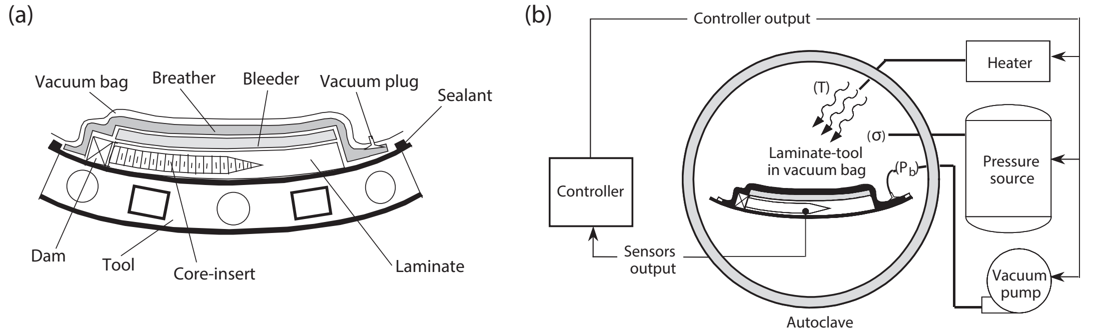


Figure 2.4: *(a) Cross-sectional view of a product on a mould including consumables, ready for autoclave processing. (b) Schematic representation of typical autoclave processing system [10].*

The vacuum bag is evacuated and the part is placed in an oven (VBO) or an autoclave. An autoclave is a pressurized oven, in composite production it is typically pressurized to 7 bar [11], see Figure 2.4b. The additional pressure helps to suppress void formation and growth in the laminate. Autoclave curing offers low void content products and a high degree of repeatability, it is a well-understood process that benefits from a lot of gained experience through widespread industrial use, it remains to be the benchmark for composite production [11].

In VBO curing the laminate is solely compacted by the vacuum pressure, which eliminates the need for an autoclave. Autoclaves require a massive capital investment and consume a large amount of energy compared to an industrial oven. However, without the external pressure much of the driving force behind void growth suppression is removed. In order to make low void content laminates the use of specialized VBO prepregs is necessary [12].

Figure 2.5 illustrates typical autoclave/VBO cure cycles and the corresponding viscosity profiles of the epoxy. A typical autoclave/oven cure cycle is subdivided into four stages (Figure 2.5a):

1. Initial ramp-up
2. Intermediate dwell
3. Ramp-up to cure temperature
4. Cure

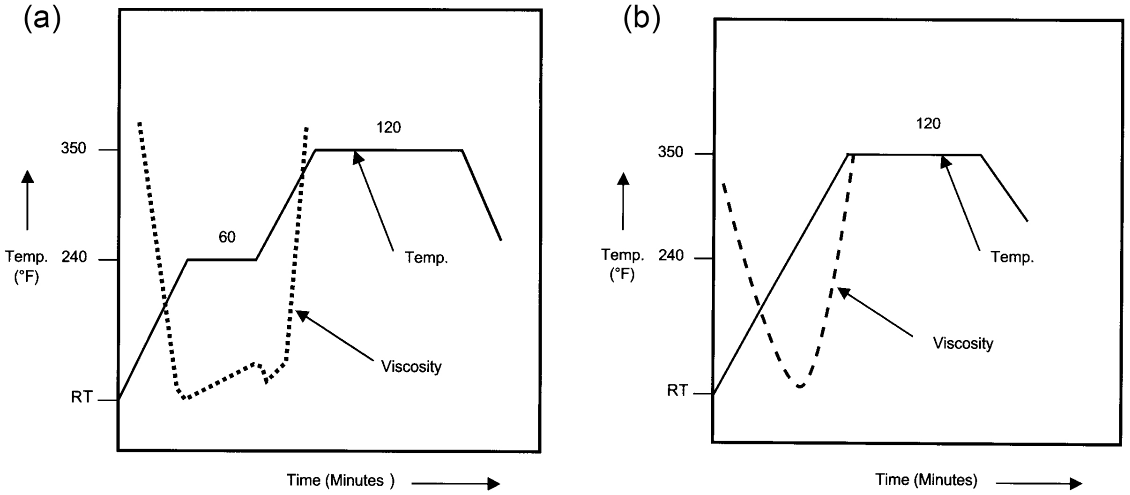


Figure 2.5: *Typical cure cycles and corresponding viscosity profiles for a cycle with (a) an intermediate dwell and (b) straight ramp-up [11].*

In the first stage the temperature is increased and the viscosity of the prepreg drops dramatically, this is referred to as softening of the matrix. The second stage is an isothermal dwell which extends the gel-time to allow more time for resin flow and entrapped air and volatiles to escape the resin whilst it still has a low viscosity. Additionally, the isothermal dwell can also be an effective means to homogenize the temperature of the product at some stage in the cure cycle. During the isothermal dwell the viscosity slowly increases as the polymerization reaction progresses. This dwell is still commonly applied in VBO processing to allow more time for volatile extraction. In autoclaving the dwell was used for bleeding-out excess resin, but the industry has moved to net resin content prepregs which has eliminated its use and the cycle in Figure 2.5b is used [11].

After the dwell there is another ramp-up to bring the prepreg to the cure-temperature where the cross-linking reaction is completed. At the start of the ramp-up the viscosity will initially drop as the temperature increases, but it will quickly rise dramatically as the cross-linking reaction accelerates. The average molecular weight of the resin goes to infinity, which is referred to as gelation; the resin has transferred from a liquid state to a solid state [13]. The gelation may take place in the third or the fourth stage of the cure cycle, the fourth stage is the final isothermal dwell where the cross-linking reaction is completed and the resin solidifies.

2.4. Void formation and mitigation in fibre composites

In the manufacture of composite laminates there is a desire to minimize the voids and porosity in the laminate. 'Voids' usually refer to large pores whereas the word 'porosity' is used for small pores. This section covers the theory of void formation, its impact on the properties of the composite and strategies to mitigate them.

2.4.1. Theory of void formation

In literature three main causes are identified as possible contributors to porosity: entrapped air, dissolved moisture and volatiles. There is however no consensus regarding the relative impact of each factor [14]. The uncured epoxy in the prepreg has a natural tendency to absorb moisture and air is easily entrapped during ply collation. The amount of air that is entrapped depends on many variables: the prepreg tack, the room temperature viscosity of the resin, the degree of impregnation of the prepreg, surface smoothness of the prepreg, whether the plies have been debulked and the number of debulk cycles, ply drop-offs, etc. [11]. Volatiles can originate from solvents that are used in prepreg production, but nowadays a hot-melt process is often used to impregnate the fibres and small concentrations of solvents are no longer present.

Moisture is typically the most predominant volatile in hot-melt prepregs. The amount of moisture that is absorbed by the prepreg is dependent on the ambient relative humidity and the rate of absorption is dependent on the ambient temperature [11]. The moisture vaporizes due to the combination of a vacuum pressure (lower boiling point of water) and the elevated temperature of the cure cycle. Void growth will potentially occur when the vapour pressure exceeds the hydrostatic resin pressure. So, the higher the hydrostatic resin pressure the more void growth and nucleation is suppressed.

It is however important to realize that the applied pressure in the process is not necessarily the same as the hydrostatic resin pressure. The resin pressure may be lower or, in the worst case, there can be practically no pressure at all. The pressure can drop as a result of resin bleed-out. Resin can bleed out into a bleeder fabric, through parting lines in a mould or into the core material as the viscosity drops during the cure cycle. As it bleeds out an increased amount of the applied pressure is carried by the fibre bed and the hydrostatic resin pressure drops.

In summary, a high resin pressure helps to keep volatiles dissolved and helps to prevent void growth before the resin gels, over-bleeding of resin should be prevented to maintain sufficient hydrostatic resin pressure.

2.4.2. The impact of voids

The presence of voids in the laminate adversely affects the mechanical properties and the surface finish of the composite. Experimental data suggests that voids in the composite laminate mainly impact the matrix-dominated mechanical properties [15]. The effect of the void volume content on the transverse tensile strength and InterLaminar Shear Strength (ILSS) is shown in Figure 2.6.

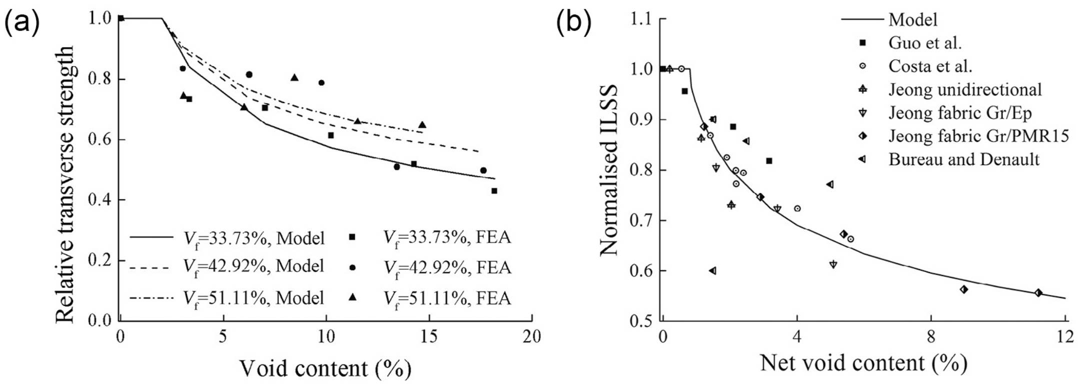


Figure 2.6: *The effect of void content on (a) transverse strength and (b) ILSS. Experimental data (and regression models) are compared to RVE FEA results [15].*

The results in both Figures 2.6a 2.6b indicate that there is a critical level of void content before the strength of the laminate is affected, it is around 2 vol% for transverse tensile strength and 1 vol% for ILSS. The matrix dominated transverse and in-plane shear modulus are similarly adversely affected by increasing void contents. Additionally, the fibre volume fraction plays an important role; high fibre content laminates are less susceptible to the effect of transverse strength (matrix dominated) reduction due to increased void contents [15].

Surface porosity refers to voids that appear at the surface. It is believed that the main source of surface porosity is air entrapment between the prepreg and the tooling surface. It is mostly found in underlap regions of the woven fibres [14]. The results in Figure 2.6 show that a 1-2% void volume content has a negligible effect on the mechanical properties, but a similar void content at the surface significantly impacts the aesthetic quality of the laminate. Surface porosity is responsible for a considerable amount of rework in the industry, mostly because it causes pinholes as the lacquer is drawn into the pores and it may even cause blistering of the coating when the product is baked in an oven [16].

2.4.3. Void mitigation strategies

A number of strategies are proposed in literature to reduce the void content in prepregs that are cured in a variety of processes. This section provides a brief overview of the most important findings of these studies.

Increase the processing pressure

In Section 2.4.1 the theory of void formation was discussed, it was stressed that void formation and growth are strongly related to the hydrostatic resin pressure. Generally, a higher processing pressure is more likely to result in a higher hydrostatic resin pressure. So, a higher processing pressure helps to achieve more suppression of void formation and growth. F.Y.C. Boey and S.W. Lye [17] investigated the effect of increasing processing pressure on the void content in glass fibre/epoxy prepregs cured without application of a vacuum. They found that significant void reduction only occurred above an applied pressure of 4 bar. At pressures beyond ~10 bar the porosity content decreases asymptotically. The void content was eventually reduced to 3% at a pressure of 70 bar.

However, processing at such pressures requires very strong and expensive tooling and equipment. The addition of a vacuum helps to reduce the void content at low pressures as well. In autoclave processing with vacuum the trend still exists that increasing pressures leads to lower void contents, so higher pressures are still preferred [18, 19]. L. Hamill et al. [14] points out that especially surface porosity in VBO processing is reduced when the room temperature vacuum time is increased. For thick laminates it is also beneficial to do intermediate de-bulk cycles during ply collation; every few plies the product is vacuum bagged for a few hours to remove air that is entrapped between the plies.

Choose the right prepreg format

In VBO processing only the vacuum pressure is acting on the laminate and it is still possible to manufacture components with void volume contents of 3% or less. This does however require a different prepreg format, often referred to as VBO prepreg, semi-preg or Out-of-Autoclave (OoA) prepreg. These prepregs are partially impregnated, which leaves 'Engineered Vacuum Channels' or EVaCs that allow for easier air and volatile extraction [12, 20]. These prepregs have a higher in-plane permeability than standard fully-impregnated autoclave prepregs. The EVaCs are automatically impregnated when the viscosity of the resin drops during processing. For VBO processing this new format directly helps to consistently achieve high quality products, comparable to autoclaving. However, when key processing parameters such as vacuum pressure, temperature and the arrangement of the consumables deviate from their allowable range the void content can quickly become unacceptable [12].

Reduce moisture absorption

A primary cause of void formation in prepregs is dissolved moisture. Especially in low pressure processes the relative humidity of the ambient atmosphere can have a significant effect on the void content. In low pressure processes the water vapour pressure is more likely to exceed the resin pressure. A study by L.K. Grunenfelder and S.R. Nutt [21] showed that for VBO processing the relative humidity should be kept below 45% to keep the vapour pressure below the vacuum pressure. In the same process the void volume fraction was less than

0.1% when the relative humidity is below 45%, whereas it was 2.6% at 90% relative humidity. In autoclave processing the pressure is high enough to suppress void formation due to dissolved moisture, no significant difference is observed at higher relative humidities (the void content is always below 0.1%).

Modify the surface ply

The surface ply has a strong influence on the surface porosity of the cured composite. The main source of surface porosity is believed to be air entrapment between the tooling surface and the first ply, so measures to reduce air entrapment help to reduce surface porosity. One measure that has been proven to be effective is making use of UD plies at the surface [22, 23]. These plies are not woven, which eliminates the problem of air entrapment in the underlap regions of the woven fabric. However, UD plies may not give the desirable surface finish for bare carbon products: At Donkervoort 2x2 twill weave fabrics are preferred because they are found to be more visually attractive.

It is also found that an increased out-time of the prepreg that is placed at the surface helps to reduce surface porosity in VBO processing [14]. The increased out time results in a reduced tack and leads to less air entrapment at the part-tool interface. The drawback is however that the reduced tack complicates the lay-up process as it makes it more difficult to keep the plies in the desired position. This also only works when a vacuum is applied, the reduced tack effectively helps to create EVaCs at the part-tool interface. As such the air can be extracted more easily during a room temperature de-bulk cycle.

Cure cycle optimization

The cure cycle influences the curing reaction of the polymer inside the prepreg. An intermediate dwell can be introduced in the cycle to extend processing time when the viscosity of the resin is low (see Figure 2.5). This is done because air and volatiles are extracted more easily when the viscosity is low. Hernández et al. cured a prepreg in a compression moulding process at 2 bar, without applying a vacuum pressure on the laminate. They found that making use of an intermediate dwell in this process drastically reduced void content as opposed to the straight ramp-up cycle suggested by the manufacturer of the prepreg.

After the dwell the temperature is increased, at which stage the viscosity initially drops and the void pressure increases. As a result the void volume content may increase just before the resin gels [19, 24]. This problem can be mitigated by increasing the pressure during the intermediate dwell. Or, the dwell time is varied and the pressure is increased at the end of the dwell. L. Liu et al. [18] found that there is an optimum dwell time in this situation. But this is of course dependent on the chosen resin system in the prepreg. The optimum dwell time is strongly related to the dwell temperature and the gelation time of the resin at that temperature.

2.5. Conclusion

The concept of sandwich constructions and their potential for weight savings was introduced and the failure modes of these structures were examined. These failure modes play an important role when the strength and stiffness of X-core samples is assessed. In the X-core process presented in this report a carbon fibre/epoxy prepreg laminate is co-cured to the core in a one-shot process. There is interaction between core and laminate, but the curing reaction of the polymer itself is still the same. The viscosity evolution of the epoxy in the prepreg is directly related to the cure cycle and affects the void volume content of the laminate. Achieving a low void volume content is a primary concern in composites manufacturing; matrix-dominated mechanical properties such as the transverse tensile strength and ILSS decrease with increasing void contents. In addition, voids that appear at the surface (referred to as surface porosity) impact the aesthetic quality of the laminate, they may cause pinholes in the lacquer or even blistering of the coating.

Voids originate due to dissolved moisture, entrapped air and volatiles. The application of pressure is a necessity during the cure of the composite laminate: The hydrostatic resin pressure needs to be higher than the void pressure or vapour pressure to reduce void growth and void formation. Increasing the processing pressure generally helps to reduce the void content, but care must be taken to prevent over-bleeding of the resin. Other measures to reduce the void content include changing the prepreg format, reducing moisture absorption by the prepreg and optimizing the cure cycle. Surface porosity in particular may be mitigated by making use of non-woven surface plies or increasing the out-time (reduce the tack) of the prepreg.

3

The X-core concept and its development

X-core is a novel type of core material that enables a new and unique process in the manufacture of sandwich structures. Donkervoort began exploring the concept of X-core in 2007, motivated to develop a one-shot technique that could manufacture the door beam of the D8 GT, a sandwich structure with mould-defined features all around.

This chapter sets out to provide an explanation of the working principle of X-core in Section 3.1, followed by a brief summary of previous research into X-core and their findings in Section 3.2. This chapter concludes in Section 3.3 with a review of concurrent research and the state-of-the-art in X-core production, which entails the door window frames and floor panels of the new Donkervoort D8 GTO RS. So far, X-core has demonstrated to be capable of achieving its intended goal and Donkervoort continues to invest in its development with the aim to improve the process and to make it suitable for other industries.

3.1. The basic principle of X-core

X-core is being developed and patented by Donkervoort. It is in essence a thermoset syntactic foam containing a physical blowing agent. In the X-core process the foam can be co-cured with fibre reinforced thermoset polymer facings to form sandwich structures in a one-shot operation. Its defining property is its ability to volumetrically expand at a certain temperature. Restraining this expansion in a closed mould system results in a pressure that is exerted on the surfaces of the mould, which can be harnessed to compact a composite laminate. Exerting the pressure from inside the component enables the manufacture of complex near-net-shape structures with mould-defined features and high quality surfaces all-around.

X-core is capable of expanding through a special type of blowing agent: hydrocarbon gas filled thermoplastic microspheres. At elevated temperatures the thermoplastic shell softens, allowing the microsphere to expand as it is subjected to an increased internal gas pressure at these temperatures. These microballoons are a product of AkzoNobel, they are sold under the tradename Expancel®. The basic principle of Expancel® is illustrated in Figure 3.1.

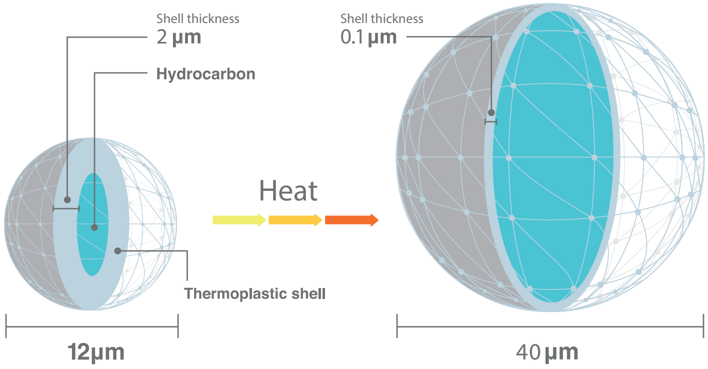


Figure 3.1: *The expansion mechanism of Expansel® microspheres [25].*

Different types of Dry Unexpanded (DU) Expancel® microspheres are readily available, their unexpanded diameters range in sizes from $6\,\mu$m to $40\,\mu$m and the onset of expansion temperature ranges from 80°C to 173°C, depending on the type of DU. The volume of the microsphere keeps increasing beyond the onset of expansion temperature, until the maximum allowable temperature is reached. Up to this point the expansion can be substantial, the expanded microsphere in the illustration in Figure 3.1 takes up almost 40 times as much volume as it initially did, AkzoNobel even reports that microspheres can expand up to 60 times their original volume [25]. There is also a maximum temperature; at some point the walls of the microsphere will become too thin and the microspheres start to deflate and collapse. This temperature is known for each type of DU and is provided in the datasheets.

Besides thermally expandable microspheres X-core also contains lightweight fillers, in fact more than 50% of its volume is generally made up by fillers. These fillers are introduced to reduce the density of the foam. A variety of fillers has been applied in X-core, currently a combination of Hollow Glass Microspheres (HGMs) and pre-expanded Expancel® is used. The pre-expanded microballoons do not expand or shrink in the range of temperatures wherein X-core is processed, they are used as a lightweight filler.

3.2. Previous Research

Research into X-core was initiated by a desire to cut the processing time and costs for the door beam of a Donkervoort D8 GTO. This door beam contains a CNC milled solid core of Divinycell H45 polymer foam ($48\,\text{kg/m}^3$). It has mould defined features all-around and may be required to be a 'full visual' component, meaning that the carbon fibre weave should be visible anywhere in the component. Figure 3.2 shows two views of the door beam CAD model, its position in the car as viewed from the cockpit can be seen in Figure 3.12 on page 20.

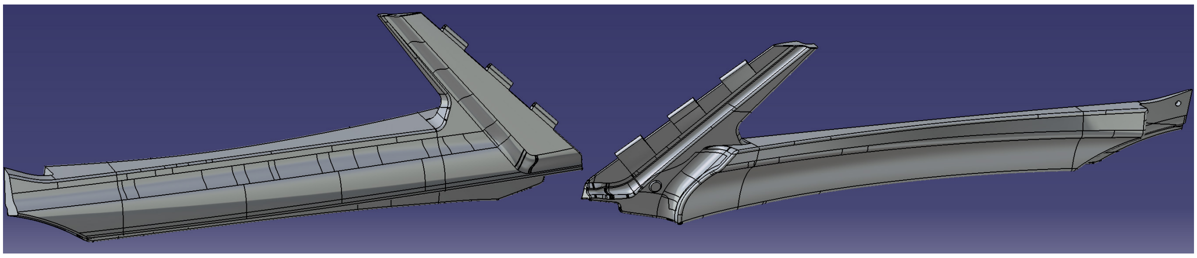


Figure 3.2: *The outside (left) and inside (right) of the passenger side door beam as viewed in CATIA CAD software.*

The door beam is manufactured in two steps. The firs step involves laying-up two thin carbon fibre outer shells that enclose the entire product, they are cured under vacuum in an oven. In a second step the core is wrapped in prepreg and glue film (excess resin) and placed between the cured outer shells. The door beam is then placed in its mould, the mould is vacuum bagged and subsequently the whole product is cured in an oven. It is only in a two-step process that mould defined features and high quality surfaces can be achieved. It is very likely that there may be porous or resin rich areas between the core and facings, mostly because it is difficulty to control the tolerances between the prefab core, facings and inner mould dimensions.

It was then argued by Donkervoort that an expanding core material might provide a means to have a one-shot process. The expansion can be used to compress the skins and the problem with manufacturing tolerances is circumvented; any location where the core and facings do not contact is simply filled up by the expanding core.

3.2.1. J. Houwers - 2013

The first study into expanding cores for sandwich structures was carried out by J. Houwers in 2013 [26]. The proposed solution for cutting processing time and costs for the Donkervoort D8 GTO door beam was to use an expanding core material, which is now known as X-core. The first X-core formula was solely based on gas filled thermoplastic microspheres: Expancel®. Two variants of Expancel® were used: Dry Unexpanded DU 031 40 and Dry Expanded DE. The latter are pre-expanded microspheres, they are generally used as a filler due to their exceptionally low density. More detailed information on these Expancel® products is provided in Section 4.1. At first they were combined in a 7:1 DU/DE weight ratio, which was based on a similar research by L. Vaikhanski and S.R. Nutt [27]. They found that using solely DU to generate a foam yields foams that display large crack formation. the addition of DE helps to increase the degree of mould filling and prevents cracking. The microspheres are in this case welded together as they soften at elevated temperatures, there is no binder material present. The resulting foams are depicted in Figure 3.3.

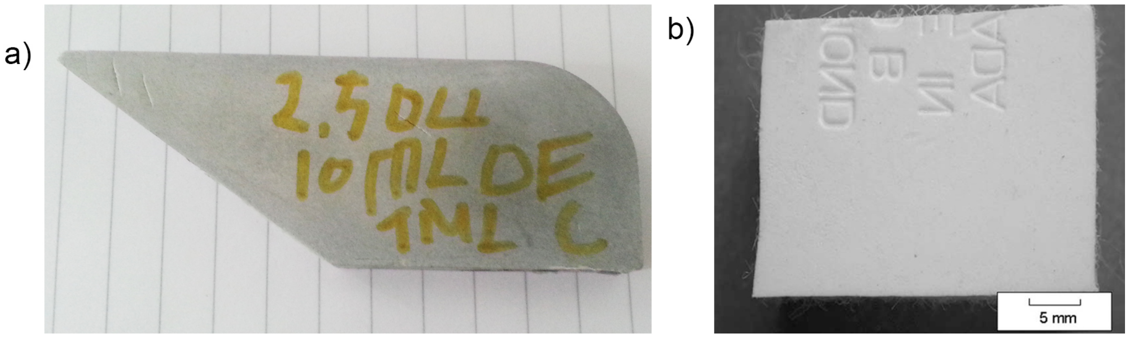


Figure 3.3: *Samples foamed using 7:1 weight ratio of Expancel® DU to DE by a) J. Houwers [26] and b) L. Vaikhanski and S.R. Nutt [27].*

The core material made by J. Houwers (Figure 3.3a) shows strong resemblance to the core made by L. Vaikhanski and S.R. Nutt (Figure 3.3b). It is very lightweight but it is not very strong and stiff; the material is elastic and the stiffness is comparable to a squash ball. An additional problem was faced with this foam once it was reheated; a sandwich sample with carbon fibre epoxy facings was cured in the mould, de-moulded and subsequently reheated to 105°C upon which it failed catastrophically as the core re-expanded, see Figure 3.4.

Figure 3.4: *This sample with Expancel® DU/DE core was post-cure re-heated to 105°C outside of its mould, which initiated re-expansion resulting in catastrophic failure [26].*

Figure 3.5: *Gear shifter knob made in a one shot process using X-core containing epoxy, Expancel® DU and Expancel® DE [26].*

The proposed solution was to add epoxy to the foam. The epoxy serves as a matrix, a binder material that is strong enough to prevent the foam from re-expanding and it provides a large contribution to the mechanical properties of the foam. This helped to solve the problem and it made the foam easier to handle as it is now a paste rather than a powder before it is cured. The consequence was however a drastic increase in density, but it was expected that the addition of epoxy also leads to a substantial increase in mechanical properties.

This X-core formulation showed to be very promising; J. Houwers proved that small products like a gear shifter knob could be manufactured with a relatively good surface quality and good laminate compaction in a one-shot process, see Figure 3.5. There is a small amount of porosity at the surface, but this was easily removed by filling the pores with a transparent epoxy. Scaling up the process to the door beam would however not yield satisfactory results due to the high amount of porosity in the laminate. He recommends that the process needs more fine-tuning and that improvements could be made by choosing a different epoxy resin.

3.2.2. F. Minde - 2014

The feasibility of the X-core process and its application in the manufacture of door beams was further explored by F. Minde in 2014, as part of a Master's Thesis at the Delft University of Technology [28]. He used a different type of epoxy and added lightweight spherical PolyStyrene (PS) foam beads to X-core, which helped to reduce the density and hence the weight penalty of using X-core instead of Divinycell H45. The volume fractions of the constituents where determined through tests such that strength criteria were met at an as low as possible density.

An X-core foam was formulated with a density of 168 kg/m^3 and a shear and compressive strength of 0.64 MPa and 1.27 MPa respectively. At this density the required strength values were met, namely 0.56 MPa and 0.6 MPa for compression and shear respectively. F. Minde managed to manufacture a door beam in a one-shot process using this foam, it reduced the part costs by 65% whilst still satisfying the imposed strength criteria. The part weight would however increase by 23% due to the higher density of X-core (compared to Divinycell H45) and the surface quality was still affected by a large amount of porosity. The lay-up of the door consists of a 245 g/m^2 3K 2x2 twill weave carbon fibre semi-preg for visual applications, backed by a 420 g/m^2 12k 2x2 twill weave carbon fibre prepreg for reinforcement. It is just a two ply laminate and it can be manufactured with a low void content in a vacuum bag only process. In order to improve the surface quality F. Minde investigated four different approaches:

1. **Application of a vacuum:** The X-core product is prepared and moulded as usual, the mould is closed and subsequently vacuum bagged in its entirety. The part is cured in the oven whilst a vacuum is applied. There was no significant decrease of porosity, it was deliberated that out-gassing of the core would only take place at elevated temperatures when the viscosity of the epoxy in the prepreg is too high to allow for evacuation of the air.
2. **Addition of a dry fabric:** In line with the hypothesis that was proposed after the vacuum bag research it was decided to add a layer of dry fabric between the laminate and the mould surface. The reasoning was that this would provide a path for the air to escape the mould, but it lead to a poor surface quality because there was not enough excess resin in the prepreg to wet this dry layer.
3. **Barrier layers:** Placing a barrier layer between the core and facings removes the hypothesized problem of degassing from the core into the prepreg, simply because the flow path is blocked. The use of a 0.023 mm thick PET foil yielded a significant improvement of surface quality, as can be seen in Figure 3.6. However, no peel tests were performed and there was some uncertainty regarding the quality of the core-to-face bond with the addition of a plastic foil.
4. **Different grades of Expancel®:** Different types of unexpanded and expanded Expancel® and their effect on the quality of the foam and the facings was investigated. F. Minde used 031DU, 551DU, 461DU (which are unexpanded), 551DE and 920DE (expanded). The combination of 031DU and 920DE would provide the smoothest surfaces without discolourations. Currently, these two grades of Expancel® are still the main components of X-core; more information is provided in Section 4.1

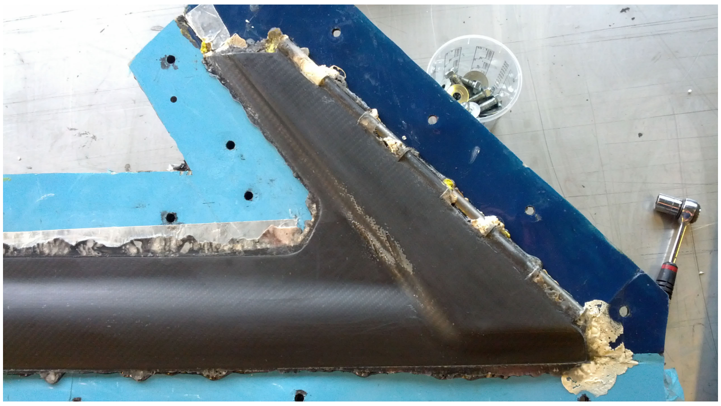


Figure 3.6: *Door beam produced with a 0.023 mm PET foil barrier layer between the core and facings gives a low amount of porosity [28].*

F. Minde also suggested that the surface porosity could be reduced by changing the lay-up of the facings or the epoxy systems of the prepregs. In addition he identifies the PS beads as a weak link and recommends to replace them by another filler in the same size range, mostly because of their poor thermal stability.

3.2.3. S. Vial - 2016

In 2016 a second Master's thesis research was devoted to X-core, when S. Vial investigated the material properties and how they are affected by the constituents [29]. The constituents were altered to improve the processability and mechanical properties of X-core. The epoxy resin was changed to a type with a less severe exothermic reaction and a low room temperature viscosity. Hollow glass microspheres (HGMs) were also introduced to replace the polystyrene beads, which was necessitated by a new strength requirement: The new X-core formulation should have a compressive strength of at least 3.6 MPa. The addition of HGMs lead to a ~50% increase in density of the foam, but their use was justified as the compressive strength of the foam increased by ~150% and they have an excellent thermal stability. Expancel® 031DU and 920DE are the remaining constituents besides the HGMs and epoxy that complement the X-core foam.

S. Vial altered the volume fractions of the constituents in a designed experiment and subsequently related them to the density and mechanical properties. A mathematical model was developed that helps to identify which volume fractions of constituents lead to the highest compressive strengths for a range of densities. The response surface of this mathematical model is shown in Figure 3.7, the black dots represent the points of maximum strength for a range of densities between 400 kg/m^3 (left) to 150 kg/m^3 (right) in increments of 10 kg/m^3. The density is a function of the volume fractions of the constituents.

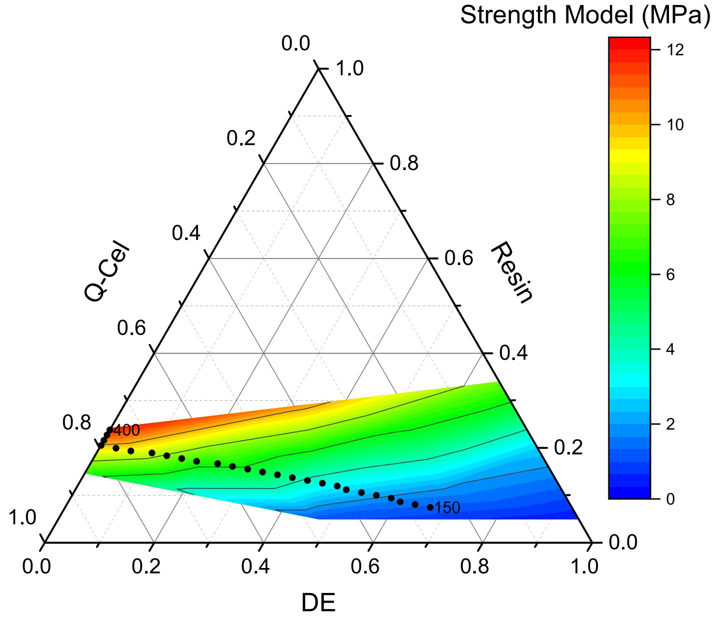


Figure 3.7: *Relation between compressive strength of X-core and the volume fractions of its constituents (with 4 vol% DU). the black dots represent the points of maximum strength for densities ranging from 400 kg/m^3 (left) to 150 kg/m^3 (right) in increments of 10 kg/m^3. [29].*

Theoretically, the lightest possible foam to satisfy the strength criterion (3.6 MPa) would have a density of 239 kg/m^3. It is heavier than the foam developed by F. Minde, but the specific strength has doubled. In addition, with the new epoxy system the X-core can be moulded and cured directly after mixing the constituents, previously the core was made as a prepreg which needed to be aged for 24 hours prior to being processed.

The results also point out that the use of Q-cel in combination with DE can lead to more optimal foams in a range of densities between 150 kg/m^3 and 400 kg/m^3. In fact, foams with a density greater than 370 kg/m^3 would benefit from containing just Q-cel as a filler and no DE at all. Below this density the highest specific compressive strengths are achieved by adding DE, the lower the density the higher the DE volume fraction and the lower the Q-cel volume fraction.

In an additional study S. Vial proved a linear relation between the DU (Dry Unexpanded Expancel®) volume content and the pressure generated at the surface of the mould, indicating that a typical autoclave pressure of 7-8 bar is easily achieved at 10 vol% DU, see Figure 3.8. The discrepancy between the 'low pressure' data and 'high pressure' data points was thought to be a result of mould deformation or collapsing of fillers.

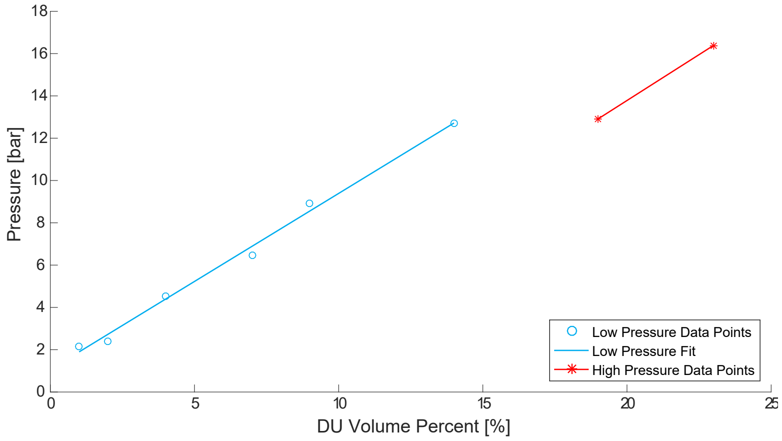


Figure 3.8: *Relation between Expancel® DU volume content and pressure generated by X-core at the mould surface [29].*

An X-core foam with a density of 330 kg/m^3 and a compressive strength of 7.08 MPa was specifically formulated for the one-shot manufacture of floor panels and door window frames of the Donkervoort D8 GTO RS. It is heavier than the ideal mixture and is stronger than required, but it was developed before the model was finished. The surface finish of the components showed clear improvement over earlier attempts at making X-core door beams (J. Houwers and F. Minde).

He demonstrated once again the feasibility of this process, especially for the door window frames that have a complex geometry and mould-defined features all-around, see Figure 3.11 in Section 3.3. Surface porosity is however still an issue: The products are made with a VBO prepreg that has a glass fibre surface ply, the surface porosity is visible as white spots against a dark carbon fibre background. This makes it easily visible in 'bare carbon' components and is thus required to be removed. This can be achieved by sanding away the glass fibre surface ply and applying a transparent filler.

S. Vial concluded that surface porosity requires rework after demoulding, which is costly and partly offsets the important benefit X-core offers by enabling a one-shot process. The use of PET film between the X-core and the laminate would help to remove most of this porosity, but it also clearly led to delaminations and its use could therefore no longer be supported. He recommends further research into the effect of processing conditions on the surface quality of X-core structures and suggests a number of alternative strategies for void reduction:

1. **Thin PET foil:** The DU content was higher in the past, it is suggested that the decrease in pressure yields a reduced amount of mechanical interlocking of the softening PET film and the adherents. A thinner film might offer the solution.
2. **Vacuum:** Similar to F. Minde it is suggested to vacuum bag the entire mould, or otherwise to allow the mould to vent the entrapped air more easily.
3. **Flow medium:** Instead of using dry fibres (as F. Minde did) it is suggested to use a peelply as a flow medium to remove entrapped air between the mould surface and the laminate. A peelply consumes less resin and is removed afterwards, it does however impede the surface quality of the product and only works for relatively flat surfaces.
4. **Different fibres and lay-up:** S. Vial suggests that surface quality can potentially be improved with different prepregs: a different weave, impregnation or resin for instance.

3.3. X-core: state-of-the-art

At the moment of writing X-core has been researched for more than four years, in that time it has grown from concept to product. Its was first intended for the manufacture of door beams, but instead the focus moved to floor panels and door window frames. The former served a demonstration of the concept; floor panels are relatively simple of geometry but could still take advantage of the X-core process. The latter is a true demonstration of the capabilities of X-core; the core is completely enclosed by carbon fibre, there are mould-defined features all-around and this complex geometry component is made in one-shot. This section covers a description of both components and their features, it concludes with a brief review of concurrent research.

3.3.1. Floor Panels

The X-core floor panels are part of the cockpit floor of the Donkervoort D8 GTO RS. There is a driver side and a passenger side panel, they span the distance between the seat and the end of the foot-box. The panels are flat, 8 mm thick and have sharp 90° corners all-around, see Figure 3.9. The floor panels are the first large-scale products that are manufactured in one-shot and installed in a road legal car. It was a demonstrator of the capabilities of X-core: there is sufficient pressure to push the fibres into the sharp corners, see Figure 3.10, the facings are mould-defined on both sides (flat, smooth) and no trimming operations are required after demoulding.

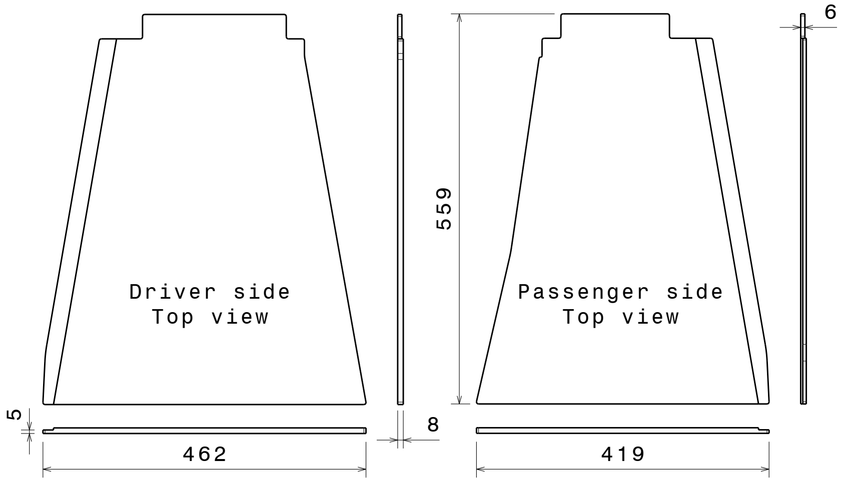


Figure 3.9: *Drawings of the driver side (left) and passenger side (right) floor panels*

Figure 3.10: *Photograph of the top-right corner of the passenger side floor panel.*

Getting the process to work for the floor panels required integrally heated tooling (IHT), the floor panel mould is the first IHT that was made at Donkervoort. Since then more IHTs have been developed for making X-core car components and samples, it plays an important role in its development. IHT and the temperature control system used at Donkervoort is covered in more detail in Section 4.3.

The surface finish of the floor panels does however not meet all requirements, surface porosity is clearly visible in the component and there is waviness of the surface ply. The waviness results in dark spots or dark lines in the product due to the change in reflection of the incident light. In some floor panels there has also been insufficient compaction of the facings and distortions in the weave.

3.3.2. Door window frames

The door window frames of the Donkervoort D8 GTO RS are the first car components that were specifically designed for the X-core process. Their shape is very complex; the mould needed to be divided into eight different pieces to allow the component to be de-moulded. Figure 3.11 shows two views of the door window frame CAD model, its position in the car as viewed from the cockpit is shown in Figure 3.12 (part 3).

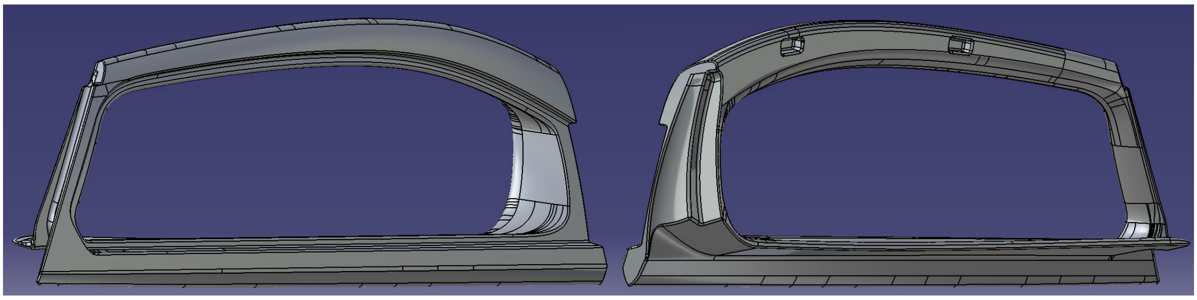


Figure 3.11: *The outside (left) and inside (right) of the driver's side door window frame as viewed in CATIA CAD software.*

The door window frames have a more consistent finish than the floor panels, distortions of the weave and insufficient compaction of the laminate is not observed. It does however suffer from a large amount of surface porosity, which again needs to be removed by sanding and applying a transparent filler. Additionally, in regions where the weave is stretched the X-core may find its way to the surface of the component. This results in white spots (X-core is white), which is also undesirable for a bare carbon finish.

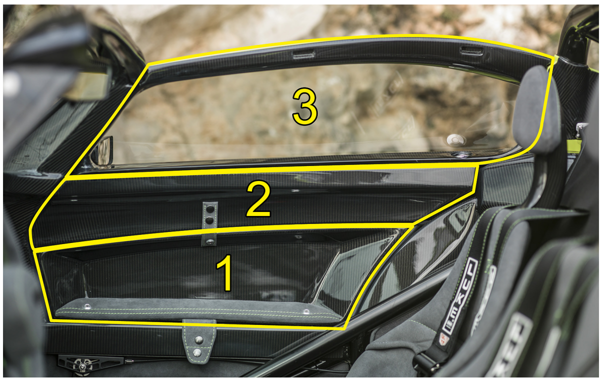


Figure 3.12: *Donkervoort D8 GTO RS passenger door as viewed from the cockpit, definition of the door components: 1) Lower door, 2) Door beam, 3) Upper door or door window frame.*

3.3.3. Concurrent research

This study focusses on the surface porosity content of X-core structures, which has been a recurring problem in previous years and still is problematic in production. But the development of X-core is far from finished, there are more challenges in the process that need further exploration. A concurrent study by M.P.J. Eversdijk [30], who is also graduating at the faculty of aerospace engineering at the Delft university of technology, is devoted to understanding the physical behaviour of X-core during the cure cycle. This study aims at gaining a better understanding of the relation between the cure cycle profile and the physical appearance and mechanical properties of the core. A transient heat transfer model is developed that can help to predict the temperature-time evolution of the foam, which takes into account the exothermic reaction of the epoxy in X-core. These temperature differences in the foam in turn affect the expansion of the core material, which affects the compaction of the laminate in a sandwich and the homogeneity of the density of X-core. M.P.J. Eversdijk has also designed and built a test set-up in an effort to measure the pressure that is delivered by X-core. He postulates that the pressure may not only be dependent on the Expancel® DU content, but also on the relative composition of the other constituents and the temperature.

Additionally, Donkervoort is working on using the X-core process for manufacturing the entire three-part door (Figure 3.12). An integrally heated mould is currently being developed for the door beam. At this point in time the X-core formulation is relatively heavy compared to Divinycell H45 foam (~300 kg/m^3 versus 48 kg/m^3). The specific strength of X-core is higher, but that is not relevant since the Divinycell foam fulfils the strength requirement and is lighter. As a compromise Donkervoort is opting for a hybrid process: a lightweight foam that is smaller than the actual core size is used to lower the part weight, the remaining gap between the core and the facings is filled with X-core. This allows for taking advantage of the X-core process and for the use of a much cheaper solid foam core because the tolerances may be very large.

For the lower door a different approach is taken, for this component a 100 kg/m^3 X-core formulation is developed based on S. Vial's model (Figure 3.7). There is no strength requirement for the foam, the lower door is in its current form a hollow structure. It is manufactured by making two shells (outside and inside door shell) which are bonded together after they have cured. The use of X-core is in this case very similar to bladder inflation moulding, the main difference being that the core remains inside the components and becomes part of the structure. The X-core process is applied to reduce manufacturing costs, but preliminary results have even indicated that a weight reduction can be achievable.

Finally, tests with X-core have been done on the rear chassis of the D8 GTO RS. A new hybrid process that combines vacuum bag processing and X-core is investigated. The X-core is used to fills the gaps between the foam core and the laminate and to provide extra compaction pressure in areas that are hard or impossible to reach with the vacuum bag. It has effectively helped to make the rear chassis in a one shot operation and its feasibility has been proved, but there are still problems with insufficient laminate compaction around the foam core and the study is still ongoing.

4

The X-core production process

This chapter covers both the material and the process that is enabled by X-core. Section 4.1 covers the properties of the constituent materials and the reasoning behind their selection. Then, in Section 4.2 a specific X-core mixture is discussed that is used in the production of X-core components of the D8 GTO RS. But it is not just the formulation of the material itself that has enabled the new process, new manufacturing procedures needed to be introduced at Donkervoort as well. These procedures relate to moulding of the core material and the integrally heated tooling technology. The integrally heated tooling is covered in Section 4.3, the procedure to mix the constituents of X-core and the method that is used to mould it are covered in Appendix A.

4.1. Constituents of X-core

X-core is a lightweight mouldable syntactic foam, there is not one general formulation with fixed properties; the density and mechanical properties can be altered simply by changing the volume fractions of the constituents. It is the constituents themselves that define X-core: Lightweight fillers, epoxy resin and thermally expandable microspheres. The specifics of these constituents are covered in more detail in this section.

4.1.1. Thermo-expandable microballoons

The basic concept of X-core (see Section 3.1) relies on Expancel® thermo-expandable microballoons. It is these microballoons that allow X-core to expand in volume and allow it to exert pressure when it is enclosed in a mould. In X-core the 'Dry Unexpanded' Expancel® 031 DU 40 grade is used, it comes as a powder that is easily mixed into the epoxy, the binder material. The properties as provided by AkzoNobel are presented in Table 4.1.

Table 4.1: *Properties of Expancel® 031 DU 40, obtained from AkzoNobel [31].*

		Expansion Temperature		Expanded	
	Particle Size D(0.5) [μm]	Tstart [°C]	Tmax [°C]	Density [g/L]	Solvent Resistance[1]
031 DU 40	10-16	80-95	120-135	≤ 12	3

[1] On scale from 1-5, 5 being excellent solvent resistance.

The density of unexpanded DU in X-core has been determined at Donkervoort to be 370 g/L. Out of all DU microspheres this variant has the lowest onset of expansion temperature and its expansion range is very compatible with epoxy based prepregs and the capabilities of the ovens in the composites department of Donkervoort. The maximum expansion temperature indicates at which temperature the microballoon starts to deflate as a result of over-expansion. The Expansion curve of 031 DU 40 is shown in Figure 4.1. It shows that the expansion starts very suddenly around 85-90°C and a further increase in temperature results in a non-linear expansion up to about 125°C.

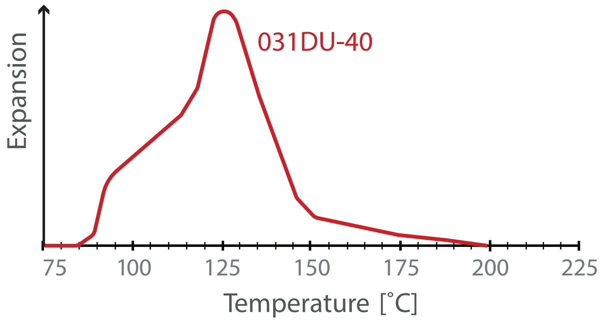


Figure 4.1: *The expansion curve of AkzoNobel Expancel 031 DU 40, the data on the y-axis is not available. [25]*

Relying solely on the expansion of DU to develop a foam is not recommended. A study by L. Vaikhanski and S.R. Nutt [27] revealed that DU based foams results in a high amount of crack formation and a low quality foam. Adding pre-expanded thermo-expandable microspheres gives a higher volume of material prior to foaming and yields high quality foams. The general idea is that most of the volume is pre-expanded, only a small amount of unexpanded material is added to fill-up the entire cavity and provide pressure to weld the microballoons.

AkzoNobel also produces pre-expanded Expancel®, it is referred to as DE (Dry Expanded) and comes as an exceptionally lightweight white powder. There are different grades of DE, they vary in particle size and density. For X-core the 920 DE 80 d30 variant is used, its properties as provided by AkzoNobel are presented in Table 4.2. The density of DE is determined to be approximately 15 g/L.

Table 4.2: *Properties of Expancel® 920 DE 40 d30, obtained from AkzoNobel [32].*

	Particle size D(0.5) [μm]	True Density[1] [g/L]	Solvent Resistance[2]
920 DE 80 d30	55-85	30±3	5

[1] The density of a single Expancel® DE microsphere
[2] On scale from 1-5, 5 being excellent solvent resistance.

For convenience Expancel® 031 DU 40 and Expancel® 920 DE 40 d30 will be referred to as DU and DE for the remainder of this report.

4.1.2. Hollow glass microspheres

Hollow Glass Microspheres (HGMs) are a secondary filler in X-core that is used in combination with Expancel® DE. The HGMs used in X-core are Q-cel® 5028, they are manufactured by Potters Hollow Spheres. In terms of size they are similar to 920 DE 80 d30, but they exhibit a higher density and strength. Their known properties are provided in Table 4.3.

Table 4.3: *Properties of Q-cel® 5028, obtained from Potters [33]*

	Particle Size Mean, Range [μm]	Bulk Density[1] [g/L]	Effective Density[2] [g/L]	Max. Working Pressure [MPa]
Q-cel 5028	65, 5-115	160	280	5.17

[1] The density as measured by weighing a container with a certain volume of Q-cel, includes interstitial air.
[2] The particle density as measured by liquid displacement; the density of a single microsphere.

For the determination of the weight of Q-cel that needs to be added to X-core, based on the required volume fraction, it should be assumed to have a density of 160 g/L (the bulk density).

4.1.3. The binder material

The Expancel® and Q-cel® microspheres typically make up 70-80% or more of the volume of an X-core foam. There is some degree of efficient packing due to the size variation of the spheres, but interstitial voids are inevitable. A resin, or binder, is added to fill up these interstitial voids and form a network structure that connects the microspheres. The binder material in X-core is a DGEBA (DiGlycidyl Ether of Bisphenol A) based epoxy. Its addition results in a drastic increase in strength, stiffness and thermal stability. However, the density of the epoxy is 1150 g/L, which is much higher than any of the other constituents (Q-cel is 160 g/L and DE is 15 g/L). So, its addition also leads to a significant density increase of the foam, in the order of 200 g/L^3.

A number of criteria were considered when selecting the epoxy:

1. The chemical reaction should not be too exothermic to prevent thermal degradation of the thermoplastic microspheres.
2. The pot life should allow for more than enough time to mix and mould the X-core.
3. The resin system should be processable in the expansion range of Expancel® DU, which is between 80 and 120°C.

A number of resin systems were proposed by Air Products and Chemicals, temperature logs of reactions carried out at 60°C and viscosity curves are shown in Figures 4.2a and 4.2b for a number of hardeners that were added to an Ancarez® 4010 epoxy base resin.

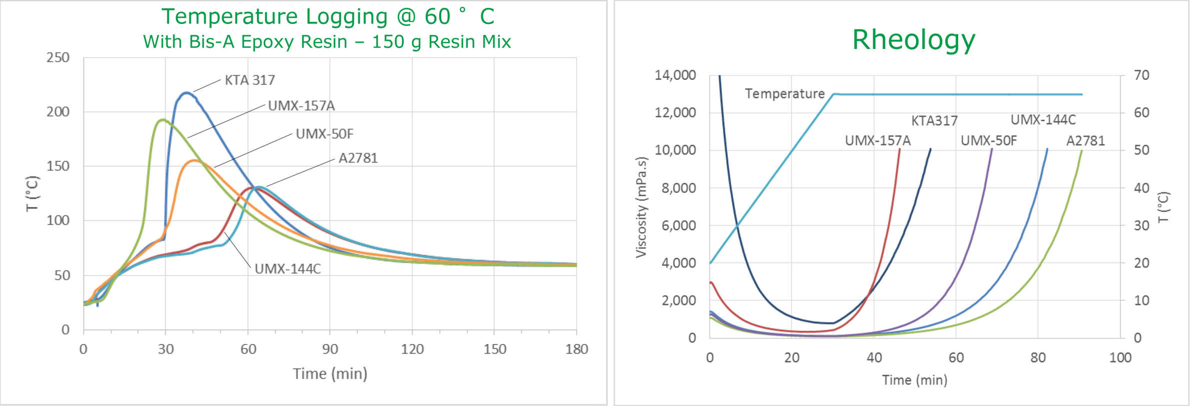


Figure 4.2: *a) Temperature logging of 150 grams of DGEBA based epoxy with different hardeners at 60°C. b) Viscosity evolution of DGEBA based epoxy with different hardeners (1.5°C/min, 65°C). Courtesy of Air Products and Chemicals Inc.*

The epoxy resin system in X-core is Ancarez® 4010/UMX-144C, it was chosen because it has a low reactivity, a workable potlife and a low room temperature viscosity. The latter is beneficial for mixing the constituents at room temperature. Practise has taught that using this resin system guarantees that X-core is still workable within 3 hours after mixing.

Before 2015 the KTA 317 hardener was used in combination with Sicomin SR 8500, which is in fact an epoxy systems for 'in house' prepregging. It has a very high reactivity combined with a strong exothermic reaction, as is seen in Figure 4.2a. In addition, this prepregging resin is required to be aged for 24 hours prior to curing it and has almost no flow after that time (high viscosity). With the new 4010/144C system this ageing process is not required, so X-core can be mixed and directly processed. As a final note: 144C is actually the laboratory code of the resin, officially it is the Ancamide™ 3399 by Air Products. So, the epoxy resin in X-core is Ancarez® 4010 / Ancamide™ 3399.

4.2. The X-core 330 mix

The X-core mix that has been used for the manufacture of floor panels and door window frames is a 306 g/L formulation. In the remainder of this report it will be referred to as the 330 mix, the 'pseudo' composition of this mix without DU is given in Table 4.4.

Table 4.4: *Volume percentages of the constituents of the 330 mix.*

	Resin	Q-cel	DE
330 mix	18.73%	49.55%	31.71%

The final composition depends on the desired volume content of DU, all the other components will then change according to Equation 4.1.

$$v[\%] = \frac{(100 - v_{DU}[\%])}{100} \cdot v_{pseudo}[\%] \tag{4.1}$$

It is called the 330 mix because for a long time its density was believed to be approximately 330 g/L. During his research S. Vial changed the estimated density of Q-cel in the X-core mix from 250 g/L to 160 g/L. This was done because the calculated density based on the volume fractions of the components in X-core was always higher than the measured densities of the samples, this change helped to reduce the difference. In fact, the 330 mix used to have an estimated density of 380 g/L, this changed to 345 g/L by changing the Q-cel density. He then initiated a new stage in his research where an empirical model was developed based on measured densities of 12 newly formulated samples. He found that, according to this model the density of the 330 mix is 334 g/L (when it contains 4 vol% DU). But this number is actually wrong because the volume fractions are no longer correct, this may be explained as follows:

the quantity of Q-cel, resin and DU that needs to be mixed in the X-core is measured based on weight, DE is based on volume (see Appendix A for more information on mixing). For the 330 mix a certain quantity of each of the components is required, dependent on the volume of X-core. For example, the door window frame requires 491 g resin, 14.2 g DU, 181 g Q-cel and 724 mL DE. These quantities are not allowed to change relative to each other because that would define a different X-core foam. Q-cel is measured based on weight, so the mass cannot change. However, when the density is redefined the estimated volume of this component does change, and so does the volume of X-core. The expected density of Q-cel in X-core was lowered from 250 g/L to 160 g/L in order to have a better prediction of the density of X-core. This means that the expected volume of Q-cel increases, which affects the volume fraction of all the constituents in X-core. This problem is worked out in Table 4.5 for the door mix, keeping in mind that the measured quantities cannot change:

Table 4.5: *The effect of Q-cel density on the volume fractions of the constituents in X-core*

		Before Q-cel density changed			**After Q-cel density changed**		
	Door mix composition	Density [g/L]	Estimated Volume [mL]	Volume fractions [%]	Density [g/L]	Estimated Volume [mL]	Volume fraction [%]
Resin	491.23 g	1150	427.16	22.33	1150	427.16	18.41
DU	14.16 g	370	38.26	2	370	38.26	1.65
Q-cel	180.91 g	250	723.66	37.83	160	1130.72	48.74
DE	723.66 mL	15	723.66	37.83	15	723.66	31.20

Plugging in the new volume fractions in the validated density model of S. Vial then yields a predicted density of 306 g/L. This discrepancy was not identified at the time, it is rectified here because it affects the predicted values for density and strength of the mixture.

Three different mixes are defined for production at Donkervoort, they are all based on this 330 mix, but vary in DU contents, see Table 4.6. The original DU percentages were actually 1%, 2% and 3.5% for the floor panel, low pressure door mix and high pressure door mix respectively. The numbers are affected by the changed estimated density of Q-cel.

Table 4.6: *DU volume contents of the 330-mix variations used for floor panel and door window frame production.*

	Floor Panel mix	Low pressure door mix	High pressure door mix
DU content	0.82%	1.65%	2.89%

4.3. Integrally Heated Tooling (IHT)

At Donkervoort almost all carbon fibre components are made through Vacuum Bag Only (VBO) processing and the components are cured in an industrial oven. The cure cycle of these VBO components is not compatible with the X-core process; there is a room temperature dwell and a 65°C dwell, which will cause X-core to cure before it could even expand. So, these products cannot be combined in the same oven cycle. In addition, temperature control of the mould is difficult to achieve because it is indirectly heated by convection of the hot air in the oven and running an entire oven for just one component is a waste of energy.

An alternative to oven heating was especially required when it came to the manufacture of the floor panels. The internal pressure delivered by X-core on the flat facings results in a large force on the mould, as such the mould is made from a stiff aluminium and steel construction that weighs 60 kg. This is a lot of thermal mass which responds slowly to a change in temperature, resulting in a heat-up rate that deviates significantly from the ramp-up rate of the surrounding air in the oven.

The solution Donkervoort came up with is integrally heated tooling (IHT). The set-up for IHT at Donkervoort is illustrated in Figure 4.3. The mould contains a resistance heater, current is send through it and regulated by a PI (Proportional-Integral) temperature controller that is connected to a Windows computer. The whole system connects to a standard 230 V power outlet, the controller is provided by Twenco CME B.V., and is still under development. The IHT and development of tooling for Donkervoort was part of an internship performed by the author, it is not subjected to confidentiality [34].

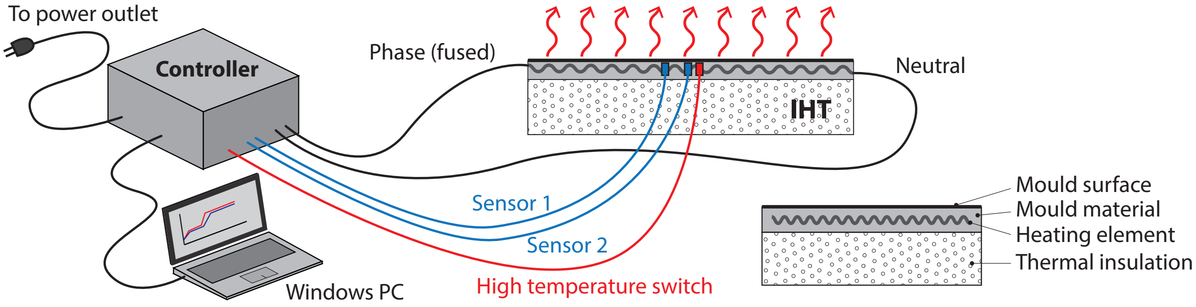


Figure 4.3: *A graphic representation of the set-up for the integrally heated tooling at Donkervoort*

Originally the temperature controller was developed for heating wind turbine blade moulds with nichrome resistance wires. At Donkervoort the nichrome is replaced by carbon fibre braids because the electrical resistivity of carbon is higher. This allows for the use wider 'wires' for the same resistance per metre. This is beneficial because it introduces the heat over a larger area and allows for placing the braid closer to the mould surface. In addition the carbon fibre braid is easily integrated in fibre composite moulds and can be processed by wet lay-up, the process by which all composite tooling at Donkervoort is manufactured [34].

Having the heater close to the mould surface was especially beneficial for the floor panel mould. The heating element is integrated in a flat laminate that is backed with thermal insulation. As such the heavy steel and aluminium construction is thermally insulated from the heat and their thermal mass nog longer plays a large role in the ramp-up of the cure cycle. The photograph in Figure 4.4 shows a decomposition of one part of the floor panel IHT.

Figure 4.4: *The floor panel IHT during production. From left to right: the original metallic mould lid, the thermal insulation, the laminate containing the carbon fibre braid heating element*

This IHT is however not flawless, measurements have indicated that a 30°C temperature difference exists in the mould when it is dwelling at 110°C, which is the cure temperature. This may however be reduced significantly through proper design of the heating element, which was at the time outside the scope of the study.

The standard cure cycle that is use for both the manufacture of door window frames and floor panels is depicted in Figure 4.5. The cure time for floor panels is 4 hours, for door window frames it is 8 hours. Some parts of the door window frames were not fully cured after 4 hours, this is mainly because a large temperature difference exists in this mould as well. To be safe, the cure time is prolonged to 8 hours. The double ramp-up was introduced to limit the temperature overshoot in the transition from the ramp-up to the dwell, based on measurements in the floor panel mould [34].

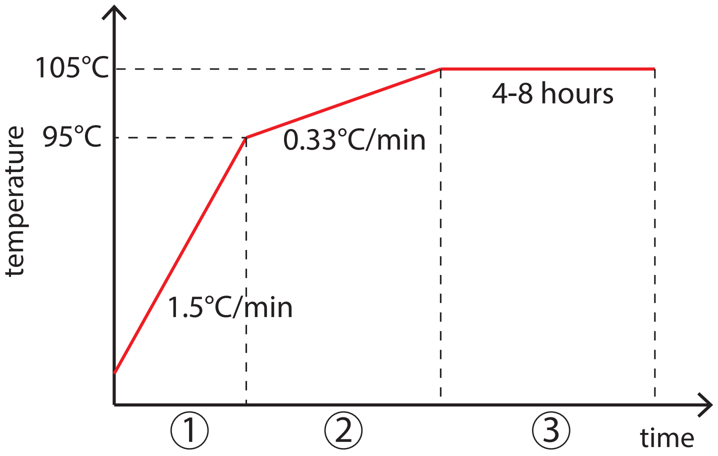


Figure 4.5: *The three-stage cure cycle that is used in the production of X-core structures*

Typically there are three sensors in the mould: two temperature sensors and one safety switch. Two sensors are used as a safety measure, in case one might fail or give false readings. The moulds at Donkervoort make use of type J thermocouples, but the system is compatible with other sensors as well. The safety switch is a normally closed bimetallic switch that will open at a pre-defined limit temperature and stops the temperature controller from providing current. The cure cycle is controlled from the computer, the computer can also be used to record and store temperatures, PI tuning variables and controller outputs.

This controller in combination with a dedicated IHT is extensively used to manufacture samples for this study. The design and capabilities of this IHT are covered in detail in Section 5.4. The constituent of the X-core formulation that are covered in this chapter are still used, but a new formulation is suggested for this study. The reasoning behind this changed formulation and the constituents of this foam are provided in Section 6.2.5.

5

Methodology and experimental set-up

This chapter covers the methodology of this study and the experimental set-ups that are used to manufacture samples and to determine their properties. The proposed methodology in Section 5.1 is a general outline wherein the effort of relating process variables to the quality of the X-core samples is described. This includes a description of the experimental set-ups, treated in Section 5.2 and 5.3. The design and manufacture of tooling that was required to carry out this research is presented in Section 5.4, Section 5.5 covers the procedure of producing samples with this tooling.

5.1. Research methodology

The goal of this study is to develop processing guidelines for the manufacture of X-core sandwich structures. These guidelines should provide information on which processing conditions are preferable for one shot production yielding high quality components: Components with a low surface porosity and good mechanical properties. It is to be established through experiments which surface porosity content is acceptable, it may be impossible to completely eliminate porosity at the surface. In terms of mechanical properties it is desirable that the sandwich structure fails through a cohesive failure mode; the adhesive bond between core and skin should not be the weakest link.

The proposed method is to manufacture representative samples, subject them to a variety of processing conditions and subsequently relate these variables to the surface porosity content and the mechanical strength or failure mode of the specimen. How the adhesive bond strength is assessed and how the surface porosity is measured is treated in Sections 5.2 and 5.3 respectively. X-core/carbon fibre sandwich samples that are to be subjected to these tests need to be manufactured. The dimensioning of the samples is dependent on the testing methods and practical considerations such as material usage per experiment. How the moulds for sample production are designed and manufactured is covered in Section 5.4.

In the next step in this study it is identified which variables may influence the surface porosity and it is decided which are to be researched. To this end small scale tests are performed to rule out the influence of some variables or to underline the importance of others. At this stage, the scope of this study is also reduced to certain combinations and variations of process variables; introducing more parameters requires additional tests and this number increases when variables influence each other. However, the number of tests can be limited through a designed experiment. In a designed experiment a special reduced set of experiments that contains the most information is used to approximate reality through a mathematical model. This strategy is employed to increase the number of process variations that can be researched.

The variations in process parameters are then coupled to the surface porosity content by manufacturing a pre-determined number of representative samples. This process contains an iteration loop and an additional study into porosity mitigation strategies. Some special processing conditions also require an evaluation of the adhesive bond strength, for instance when a barrier layer is placed between the core and facings. Based on the outcome of these tests a number of processing guidelines is formulated. With those guidelines in mind a synthesis of the study follows by manufacturing the floor panels of the Donkervoort D8 GTO RS.

5.2. Mechanical tests

The aim of the mechanical tests is to determine the failure mode and compare the strength of specimens that are manufactured in different processing conditions. It is preferable to test the strength of the sandwich structure through a standardized method, to that end a three-point flexure test in accordance with the ASTM test standard C393 was opted for. According to paragraph 4.2 in this test method the only acceptable failure modes are core shear and failure of the bond between the core and facings [35]. The specimens should be dimensioned according to the standard to ensure these failure modes. Specimens failing in core shear or adhesive failure are regarded to be successfully manufactured specimens.

5.2.1. Test specimen dimensioning

The specimen size depends on the strength and thickness of the facings. The thicker and stronger the facings the larger the support span length in the three point flexure test should be, irrelevant of core thickness, see Equation 5.1 [35].

$$S \leq \frac{2k\sigma t}{F_s} \tag{5.1}$$

Where S is the support span length, σ the expected ultimate strength of the facing, t the facing thickness, F_s the estimated core shear strength and k is a strength factor, typically set to $k = 0.75$ to ensure core failure. The facings are made of carbon fibre prepreg fabrics, a High Strength (HS) carbon fibre/epoxy fabric typically has a tension/compression allowable of 900 MPa along one of the fibre directions [36, Appendix A]. A knock-down factor of 0.8 is applied to account for scatter in material properties (B-basis value), yielding 720 MPa. Additionally a possibility of a 5° fibre misalignment during production is taken into account, further reducing the strength to 530 MPa using the Tsai-Hill failure criterion for first ply failure. These values are conservative, but the odds of encountering facing failure is actually reduced by underestimating the strength.

The shear strength F_s must be estimated, there is only compressive strength data available on the current X-core formulation. This estimate is based on the ratio of shear strength to compressive strength of other polymer foams. To that end the data of five different foams made from different polymers and made by different suppliers is used as a reference, see Table 5.1. The highest density foams of the particular series are presented, keeping in mind as a reference that the 330 mix is 306 g/L.

Table 5.1: *Properties of high density commercially available polymer foams [37–41]*

Name	Polymer	Density [g/L]	Compressive strength F_c [MPa]	Shear strength F_s [MPa]	F_s/F_c
Rohacell 200WF	PMI	200	9	5	0.56
Gurit PVC250	PVC	250	6.88	4.37	0.64
Divinycell H250	PVC	250	6.2	4.5	0.73
Corecell M200	SAN	200	4.4	2.95	0.67
Gurit G-PET200	PET	200	3.56	1.81	0.51

Based on the data in Table 5.1 the shear strength of X-core is estimated to be 67% (two-thirds) of the compressive strength. It is thereby assumed that the material is homogeneous and that the shear strength of the entire core can be represented by a single value. The non-homogeneous nature of X-core has been identified during research at Donkervoort, but its effect on the properties of the core is outside the scope of the current research. An estimate of the compressive strength of a certain X-core foam can be obtained from empirical models developed by S. Vial [29]. It is unlikely that the shear strength of the foam is higher than the compressive strength and the amount of conservatism built into the strength estimation should help to prevent facing failure in the test, even if the shear strength turns out to be higher than expected (provided that the facings have properly cured and are free of damage).

Making the shear strength a function of compressive strength F_c and filling in $k = 0.75$, Equation 5.1 can be rewritten as follows:

$$S \leq \frac{2.25 \cdot \sigma t}{F_c} \tag{5.2}$$

A new tooling system is also required for the manufacture of these samples, the dimensions of this mould are therefore dependent on the required span length for the three-point flexure test. In order to start designing this tool the desired span length needs to be determined. The process of laminate and X-core composition selection for the flexure specimens had not been finished at the time the tooling was designed. Instead it is designed based on a typical laminate that Donkervoort uses in most components and the X-core formulation used in production of floor panels and door window frames. This means a lay-up of 245 g/m^2 3K carbon fibre, backed by a 420 g/m^2 12K carbon fibre fabric, both plies are a 2x2 twill weave. A delaminated X-core floor panel sample (release film between core and facing) with this lay-up is measured to have a thickness of 0.75 mm. The 330 X-core mix in this panel has an estimated compressive strength of 7.08 MPa. The estimated strength of the facings is 530 MPa (Section 5.2), this is true when the fibres are aligned along the length of the bending specimen. Combining this yields:

$$S \leq \frac{2.25 \cdot 530 \cdot 10^6 \cdot 0.75 \cdot 10^{-3}}{7.08 \cdot 10^6} \longrightarrow S \leq 126.3\,\text{mm}$$

It was decided to round it off to 125 mm. According to the ASTM C393 test standard for three point bending the specimen length should be at least 50 mm more than the span length [35], yielding a specimen length of 175 mm. It was later found that the determination of the compressive strength is erroneous, this is related to the Q-cel density problem that was addressed in Section 4.2. When the volume fractions of the components are adjusted the empirical strength model of S. Vial [29] predicts a strength of 6.25 MPa. The moulds had already been manufactured when this was realized, but it is not necessarily problematic. An increase in maximum allowable span length changes nothing because it may be smaller, a decrease can easily be accommodated by reducing the span length, which does not affect the specimen length. So, this calculation serves as an indication for the size of the specimens and it is not necessarily constricting the choice of laminate or X-core formulation. Making the samples 175x175 mm allows for extracting multiple flexure specimens and introduces a number of symmetry planes that can be exploited. The design of the mould for manufacturing these samples is based on these dimensions, the design is covered in detail in Section 5.4.

Finally, the ASTM C393 standard describes that the width of the specimen should be at least two times the thickness. So, when five flexure specimens are to be extracted from a single plate the maximum allowable thickness is about 15 mm (depending on the width of the saw-cut).

5.2.2. Testing apparatus and set-up

For the three-point flexure tests the 10 kN Zwick Roell test apparatus in the Delft Aerospace Structures and Materials Laboratory (DASML) of the TU Delft is used. The ASTM standard loading fixture with rotating support bars is not available at the lab, instead a fixture is used with 10 mm diameter cylindrical steel bars. The problem with this set-up is that the small diameter of the cylinder results in a high local compression pressure which may cause local indentation of the foam core. In order to prevent this a 3 mm thick and 25 mm wide steel pressure pad is inserted between the steel cylinder and the sample, see Figures 5.1 and 5.2.

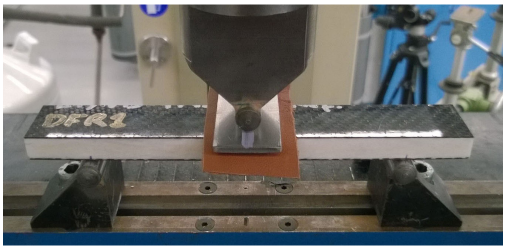


Figure 5.1: *Photograph of the fixture, sample and pressure pad.*

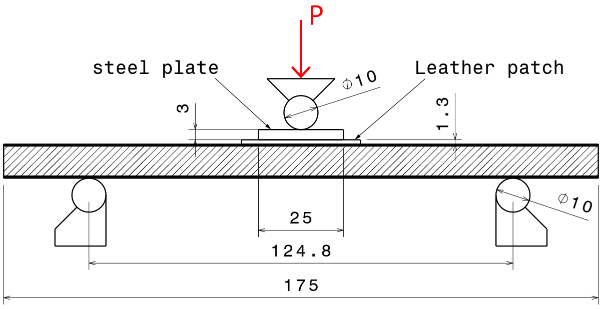


Figure 5.2: *Dimensions of the different components of the thee-point flexure set-up.*

The set-up in Figure 5.2 shows the span length that is measured prior to the test, this value is to be used for stress calculations. The steel pressure pad was chosen to be 25 mm wide because that is the same size as the loading bar in the ASTM standard fixture. A 1.3 mm thick and 35 mm wide piece of leather is placed under the steel plate to prevent it from damaging the facings. The supports are not adapted, the sample rests directly on the steel cylinders. Tests showed that no indentation of the core/facings is observed at the supports.

The pre-load is set to 10 N, this ensures that the pressure pad and leather patch are compacted. 10 N is less than 1% of the failure load of initial tests, the effect of this pre-load on the deflection of the sample is considered to be negligible. The testing speed is set to 1 mm/min, for the tests that are performed in this research this typically yields a testing time of ~2.5 minutes. The test stops as soon as the measured force drops to a value that is lower than 80% of the highest recorded value. All samples that are tested in this report failed in core shear or at the core-to-facing bond under these test conditions, which is required as per the test standard.

The samples are cut from larger panels using an automated cutting machine with a 2 mm wide diamond coated circular saw. At every pass of the blade 100 μm of material is removed, coolant is used to limit heating of the material. After cutting the samples are air-dried for at least two hours at room temperature. The width and length of each sample is measured and averaged over three locations. The thickness is measured at five locations, evenly spaced over the length of the sample. With these dimensions and the load-displacement data of the Zwick testing apparatus the core shear ultimate stress F_s^{ult} is determined using the following equation:

$$F_s^{ult} = \frac{P_{max}}{(d+c)\cdot b} \tag{5.3}$$

Where P_{max} is the highest load that is recorded, d is the sample thickness, c is the core thickness and b is the sample width. The core thickness is equal to the sample thickness minus the combined thickness of the face sheets ($c = d - 2t$). Because the facings are co-cured it is difficult to accurately measure their thickness, in this case the test standard allows for the definition of a nominal thickness value for the laminate.

5.3. Visual inspection

Donkervoort has mainly expressed a desire to achieve a class-A finish in their composite products. The fewer flaws the components have right after demoulding the less work is required to achieve high quality finished products. Part of this problem is that a large portion of the carbon fibre bodywork is only coated in a transparent lacquer, thus exposing the carbon fibre weave. Ideally, this means that waviness of the weave or other distortions are not allowed and that the surface of the product needs to be resin rich and free of surface porosity. In production of floor panels and door window frames a significant amount of small pinholes and surface porosity is easily visible with the naked eye.

The surface porosity content is expected to vary for different processing conditions. In the current research it is important to develop a standardized test method to quantify the defect content of the surface such that the effect of changes in processing conditions can be accurately and repeatedly correlated to the surface quality of different samples.

In order to analyse the X-core composite sandwich samples it is necessary to have a fixture or jig that allows for making multiple photographs of each sample whilst ensuring the same lighting conditions, such that repeatability can be guaranteed. The design and use of this jig is covered in Section 5.3.1. Section 5.3.2 covers the procedure of digitally processing these photos to assess the surface porosity content.

5.3.1. Alignment jig for photographing samples

Most surface defects such as porosity, dry spots, wrinkles and resin-rich areas are visually detectable. This means that a digital camera and some digital post-processing software can be used to determine the amount, size and distribution of visible flaws. Illumination is critical for visual inspection, most importantly the angle of the incident light and its intensity. It is important that the light is correctly oriented to allow for the most optimal conditions for the detection of flaws [42]. The goal is to achieve the highest possible contrast between flaws and the surrounding pristine surface. This lowers the detection threshold for surface flaws in the samples. The principle of defect detection using an optical camera is demonstrated in Figure 5.3.

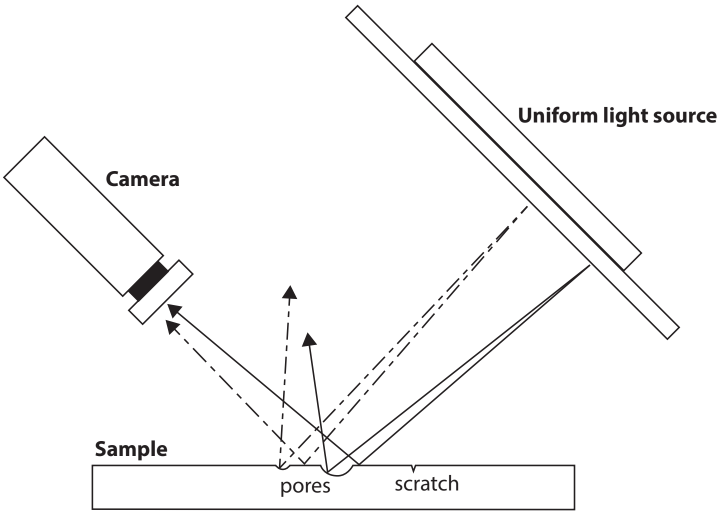


Figure 5.3: *Schematic of the physical principle of light scattering that provides the contrast between pores and the pristine surface.*

By orienting the camera and light source at some angle with respect to the sample it is possible to detect flaws that affect the smoothness of the surface. A small hole (pore) or scratch will scatter some of the incident light which means that some of the light is not captured by the camera and the picture will locally show a darker spot. It is important that the light source is constant in order to repeatedly make pictures with the same brightness. When the brightness varies the intensity threshold value for pore detection moves for each image, so determining the porosity content would require to redetermine these thresholds for each photograph. The brightness of the photograph is also dependent on the shutter speed and ISO sensitivity setting of the camera, so these setting should be the same for every photograph as well.

For photographing the 8.7 megapixel camera in a Nokia Lumia 925 is used, this particular phone is used because it is directly at the author's disposal. It showed to be more than capable of making sharp images in the conditions required for these measurements. In addition it allows for manually setting the shutter speed and sensitivity. It was found that a good contrast between pores and pristine surface was achieved with a 1/100 second shutter speed and an ISO sensitivity of 200. For the light source a 400W 60x60 cm LED panel is used, such a large size is needed to achieve uniform lighting across the sample.

The camera, LED panel and sample need to have a certain relative position, which is not known a priori. That is why a jig was designed that allows the operator to change a variety of angles and dimensions and subsequently fix everything in place once a desirable contrast between pores and pristine surface is found. A render of this set-up is shown in Figure 5.4, a drawing in Figure 5.5 shows the final orientation of the set-up wherein all photos for this study are taken.

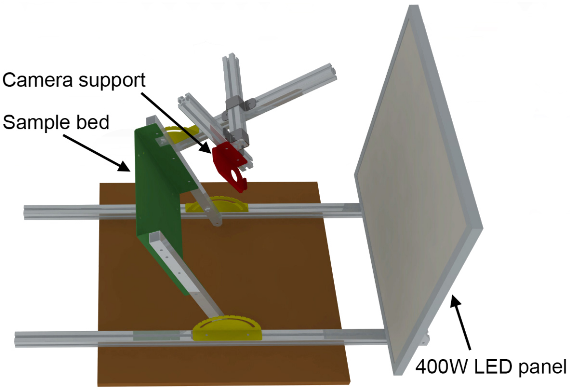


Figure 5.4: *A render of the CAD model of the optical inspection set-up.*

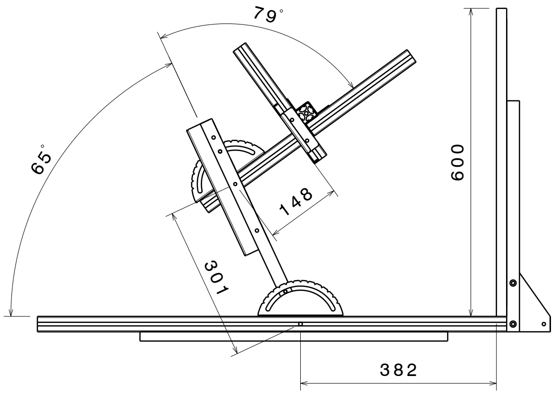


Figure 5.5: *The configuration of the optical inspection set-up in this drawing corresponds to the actual dimensions (in millimetres) and angles used during measurements.*

5.3.2. Digital image processing

The goal of processing the photographs is to identify which parts of the image represent a void. The goal of the photographing jig was to enable the user to find a certain relative orientation of the light source, sample and camera that yields the highest contrast between pores and the pristine surface of the laminate. This is important when it comes to digitally processing the samples, because the software can be used to make a distinction between the two based on brightness. Principally, the digital image processor works as follows:

1. **Import the raw image:** it is preferable to import a high quality photo with an as high as possible contrast between the brightness of the flaws (pores) and the surrounding pristine surface.
2. **Crop the image:** if needed, leave out segments of the image that should not be part of the analysis.
3. **Convert to HSV:** convert the image from the standard colour model, often RGB (Red, Green, Blue), to HSV (Hue, Saturation, Value), also known as HSB (Hue, Saturation, Brightness). The HSV colour model is a cylindrical representation of the cartesian RGB colour model, it may be seen as a coordinate reference system transformation; the colours of the image remain the same but the value of the 'coordinates' and the reference system have changed.
4. **Apply a brightness filter:** The 'hue' and 'saturation' colour spaces do not need to be filtered, only the bandwidth of the 'value' or 'brightness' colour space needs to be narrowed. The process of finding the cut-off values of the brightness-filter is done manually, but once they are set they do not require to be changed for each new photo. The filter is however sensitive to changes in the photograph; changes in shutter speed, camera sensitivity, distance to the sample and relative position of the camera and light source all affect the brightness and contrast in the photo. This stresses the importance of having a fixture to position the sample and the camera, it eliminates the tedious process of changing the filter for every new photo.

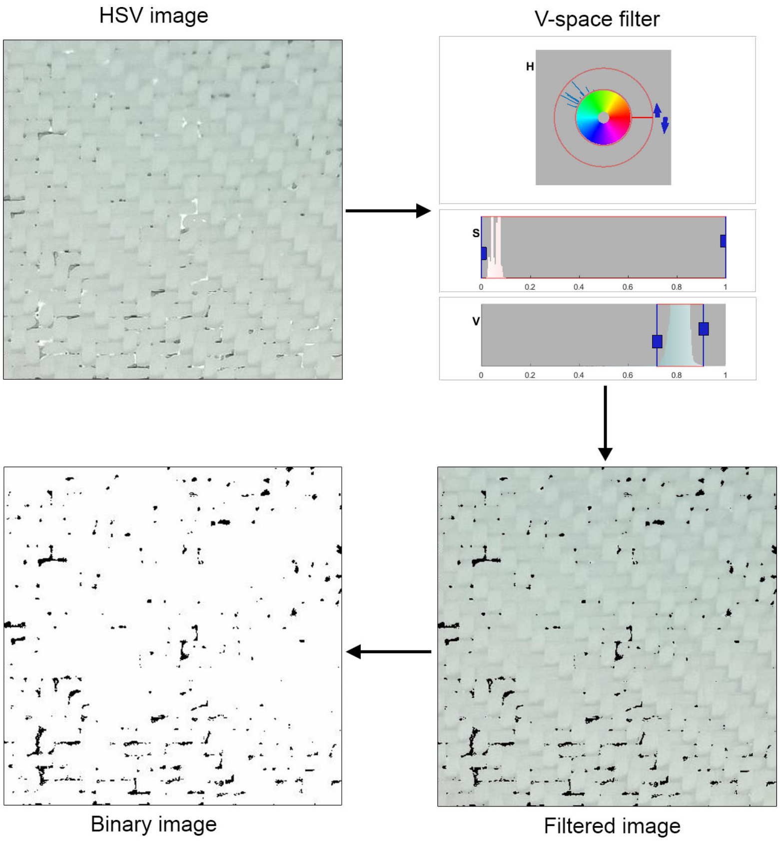


Figure 5.6: *The digital image processing procedure*

The cut-off values in the 'value' colour space are changed such that all pores are filtered out without affecting the pristine surface, see Figure 5.6. Coincidentally it turned out that subsurface pores covered by a thin layer of transparent resin are also easily detectable: under certain conditions they show up as white spots in the photograph that are brighter than the surrounding surface.

5. **Convert to binary**: All pixels that are outside the bounds of the filter will be represented as black (zero) pixels, the remaining pixels will be white (one), see Figure 5.6. The binary image is represented by a two-dimensional matrix filled with ones and zeros. If the original image was 400x400 pixels, then the binary 'image' is a 400x400 matrix.
6. **Determine surface porosity:** The surface porosity content can be determined by counting the number of zero entries (black pixels) in the matrix and divide that by the total amount of entries in the matrix.

Generally, if the value colour space spans from 0 to 1, the lower and upper bound of the filter were positioned at 0.73 and 0.89 respectively for the measurements in this study. The original photos are 1920x1080 pixels, they were cropped to a 400x400 pixel area, which represents an area of approximately 40x40 mm. Photographing a larger area was found to be difficult, the camera is at an angle with respect to the surface of the sample which complicates focussing to form a sharp image over the entire area. In this case one pixel is about 0.1x0.1 mm, so smaller pores may not be detected. But after comparison of the binary images and original images there was no necessity to increase the resolution of the photos; all visible pores seem to be detectable.

5.4. Tooling for sample production

In this section the mould that is specifically designed for this research is described. This mould is referred to as the 'tile mould', it is used to make samples for optical inspection and for performing 3-point flexure tests. The samples are square, thus taking advantage of symmetry. If the sample and the process (e.g. temperature, pressure) is symmetric than it is expected that the result behaves in a symmetric manner as well. Asymmetry in the result might indicate the influence of parameters that were not accounted for at first. It is also designed to accommodate a carbon fibre heating element and integrated type J thermocouples such that it can be heated using the PI temperature controller. In this set-up it can be programmed to any desired heating rate and temperature up to 3°C/min and 120°C. Higher heat-up rates should be possible but were never tested, it only takes about 40% power to achieve 3°C/min between 110 and 120°C.

The mould allows for manufacturing different sample thicknesses. The proposed design has two stiff lids that contain the heating elements and makes use of stackable walls that are sandwiched between these lids. The walls are L-shaped, a set of walls forms a square cavity between the lids. Figure 5.7 shows an exploded view of the tile mould, Figure 5.8 shows a detail drawing of the heating element, mould lid and a wall section.

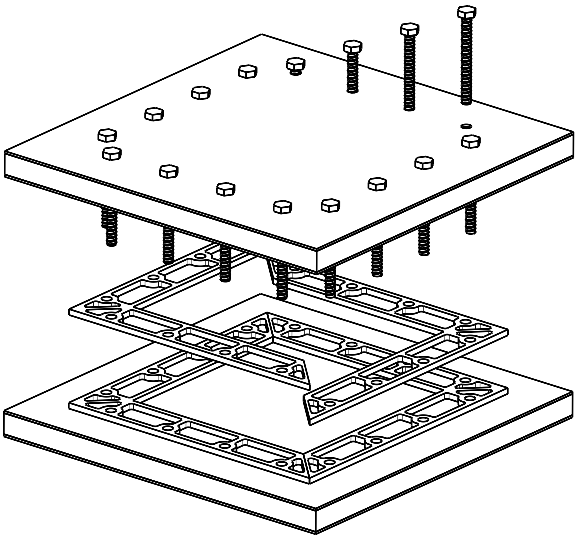


Figure 5.7: *Drawing of exploded view of the tile mould.*

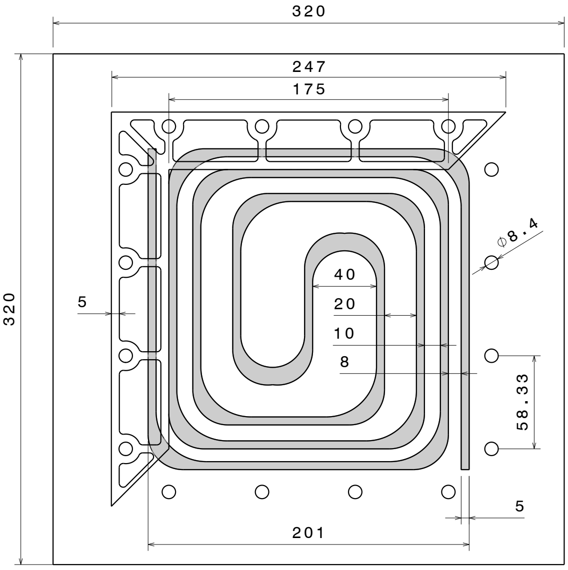


Figure 5.8: *Technical drawing of some of the features of the tile mould. The gray pattern in the figure is the electrically conductive tracing.*

The lids and walls

The lids of the mould are 320x320 mm, this is slightly larger than the outer dimensions of the walls, which provides some space to add thermal insulation. The lid is a stiff sandwich panel consisting of carbon fibre/epoxy, glass fibre/epoxy and an 18 mm thick MDF (Medium Density Fibreboard) core. The carbon is used for reinforcement, the glass fibre is used to electrically insulate the carbon fibre heater from the carbon fibre reinforcement. The total thickness of the laminate is 26 mm and it is estimated to deflect just 0.13 mm at its centre when subjected to a 10 bar pressure on a 175x175 mm surface. The core material is selected based on the required compressive strength to cope with the local pressure exerted by the 16 M8 bolts, see Figure 5.7. At 10 bar the force exerted on one bolt is 1.9 kN, when a stiff 20 mm washer is used the local pressure on the laminate is approximately 7.6 MPa. So, the required compressive strength is 7.6 MPa, which is relatively high for a polymer foam (see Table 5.1 for some typical values). Therefore it was decided to use MDF, it has a compressive strength of 10 MPa and it is cheap compared to polymer foams.

For the design of the walls the following requirements [R] and wishes [W] are formulated, the latter are ranked from most important to least important:

- **[R]** The walls should have sufficient strength to cope with a 10 bar pressure exerted on the surface that has an interface with the sample.
- **[R]** The walls should allow for different sample thicknesses, preferably an as large as possible range
- **[W]** The surface temperature of the sample-wall interface should be as close as possible to the setpoint temperature of the IHT.
- **[W]** The thermal mass should be minimized; the material should have a low specific heat capacity and/or the design needs to be lightweight.
- **[W]** The costs should be minimized

The combination of these factors lead to the aluminium design that is visible in Figures 5.7 and 5.8. Each wall is 5 mm thick, so it allows for stacking up to any desired thickness in increments of 5 mm. They are manufactured by water-jet cutting of a 5 mm sheet. This is a relatively cheap and fast process compared to laminating a composite wall and it ensures that all walls are equally thick. The thermal conductivity of aluminium is also ~50 times higher than a typical glass or carbon epoxy laminate [11]. This helps to transfer the heat from the mould lids to the sides of the specimen. The high conductivity is also a drawback as the walls are also more easily cooled down from the outside, but that is counteracted by cutting large pockets into them. This reduces the in-plane heat flow and reduces the thermal inertia of the walls. In addition, the heat loss is reduced because there is room for placing an effective thermal insulator between the mould lids, surrounding the walls.

The tracing pattern

Uniformly heating a flat plate using a resistance heater does not necessarily lead to a uniform temperature at the surface. This is especially demonstrated in the floor panel mould, where the centre is about 30°C hotter than the outer edges. This is because the heat losses are smaller at the centre than at the sides. Heat moves from a hot to a cold location, the centre of the mould is simply further away from the ambient (cold) atmosphere than the edges. A priori it is not known what is realistically achievable in terms of temperature homogeneity in the mould. In terms of scale a 1°C difference at a 100°C temperature is considered to be excellent whereas 10°C is not acceptable for this study.

As a countermeasure to the heat losses, it is decided to vary the spacing of the carbon fibre braid tracing, see Figure 5.8. This braid has an 8.9 mm diameter and weighs 2.75 g/m at a 45° fibre angle. The diameter and weight decrease when the fibre angle is reduced, which happens when the braid is stretched. The braid is approximately 5 mm wide when stretched. In the stretched state its resistance is measured to be approximately 12 Ω/m. The power of heating is proportional to the resistance (Joule's first law), so varying the spacing between adjacent parts of the tracing is a measure to vary the heat input. The pattern was designed such that the maximum heat input occurs near the outer edges, where the conduction losses are higher and where the thermal inertia of the walls tends to reduce the heat-up rate locally.

In order to leave some margin during manufacturing and to prevent the tracing from shot-circuiting a minimum allowable spacing between the braids of 8 mm is set. The braid can be placed with a relatively high accuracy. This is achieved by printing out a template, spray painting it onto the cured laminate, applying tack-spray and carefully laying down the dry carbon braid on the painted pattern. This is followed by impregnation with epoxy, covering it with a 160/,g/m^2 glass fibre weave and subsequently curing it under a 950 mbar vacuum, the result of which is seen in Figure 5.9. Inspection of the position confirmed that the required pattern matches with a maximum deviation of ~2 mm.

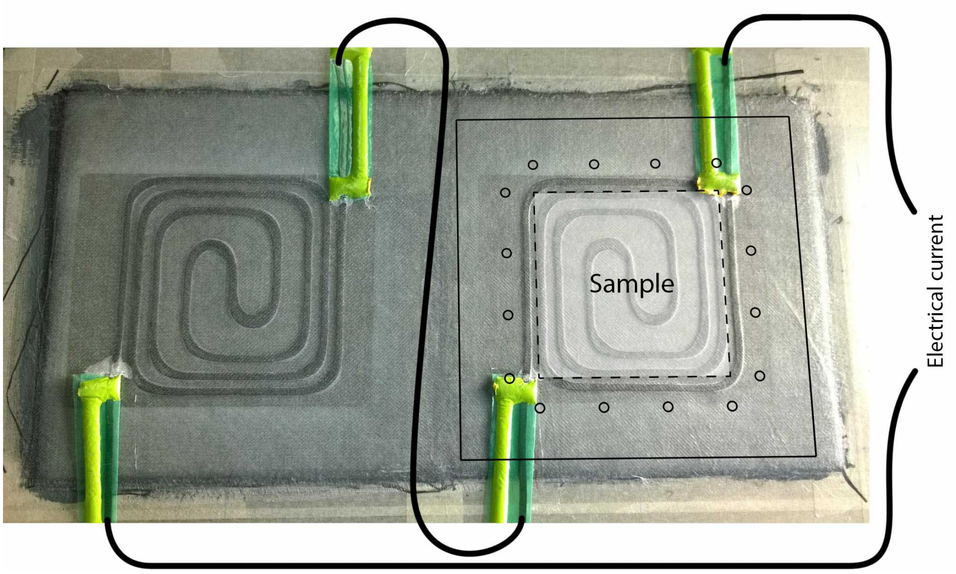


Figure 5.9: *The carbon fibre braid tracing in the tile mould, correctly positioned and cured. The ends of the braid are bagged and sealed to prevent impregnation, this is required for follow-up steps in the process.*

The outer track is actually beyond the dimensions of the specimen, acting as a thermal barrier to minimize conduction losses at the outer perimeter of the specimen. The total length of the tracing is approximately 2.3 metres, which yields about 27.6 Ω of resistance considering a 12 Ω/m resistance of the braid. This totals 55.2 Ω when the moulds lids are connected in series to the temperature controller. Connecting them in series ensures that the same amount of current flows through each mould lid, which means that the same amount of heat should be generated, provided they contain the same length of braid. The resistance is high enough to keep the peak current below 4.25 A, where 7.5 A is the maximum that is allowed by the temperature controller.

Optimization of the tracing pattern for the most homogeneous temperature inside the mould is considered outside the scope of this study. Instead it was argued that there are a number of measures at our disposal that, in combination with the proposed pattern, can help to achieve a more homogeneous temperature in the mould. These measures include:

- Making use of strong thermal conductors between the mould surface and the sample to improve the flow of heat to colder locations, e.g. aluminium alloys and copper have high thermal conductivities.
- Thermally isolate the mould from the surroundings, this allows the mould to heat up entirely and not just the surface, which limits the heat losses.

Tests on the finished mould pointed out that the temperature differences at the mould surface are in the order of 30°C and that the previously suggested measures to improve temperature homogeneity are a necessity. These tests were carrier out by M.P.J. Eversdijk [30] in a parallel study. He used 13 Arduino compatible DS18B20 sensors, placed in a rectangular grid covering the mould surface. Inside the mould was a cured 20 mm thick X-core sample wherein the sensors were embedded. The temperature readings were interpolated, which gives the temperature graphs that are shown in Figure 5.10.

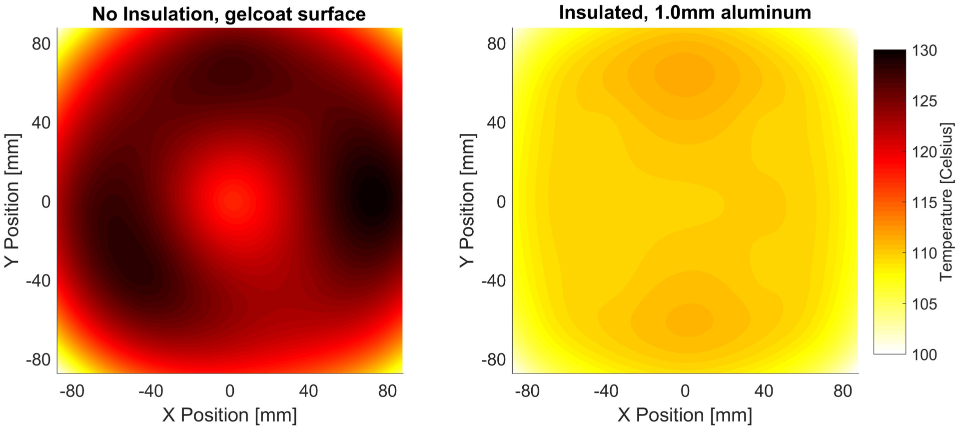


Figure 5.10: *These temperature plots of the tile mould are made by interpolation of temperature data. The graphics represent the data near the end of the ramp-up, where the temperature difference between sensors is maximal.*

The temperature was raised from room temperature to 110°C at a rate of 1.5°C/min, the image represents a point in time near the end of the ramp-up, where the temperature difference between sensors is maximal. The image on the left in Figure 5.10 is the original condition, on the right the mould is thermally insulated in its entirety and a 250x250 mm aluminium sheet with a 1 mm thickness is placed between the sample and the mould surface. The insulation is formed by covering the mould in six layers of 340 g/m^2 dry bleeder (A photograph of the set-up is shown in Figure 5.13). The results indicate a clear improvement of the temperature homogeneity: The maximum temperature difference between sensors in the original situation was 28.5°C, whereas it is just 5.5°C with the addition of insulation and aluminium plates.

It is however important to note that the corners are still about 10°C colder than the centre of the mould near the end of the ramp-up. This difference in temperature is taken into consideration in the analysis of the samples. The photographs are cropped to a small region of the sample, four regions are analysed per facing of the sample, see Figure 5.11. This way the maximum difference during the cure cycle of the analysed regions is just 2.3°C, the data is obtained from the measurement presented in Figure 5.10. In this case the maximum deviation from the cure cycle (110°C cure) is also just 1.4°C at maximum. During the dwell at 110°C the temperature difference in this region drops to below 1°C, which is considered to be acceptable. The pattern of the photographed regions in Figure 5.11 originates from rotating the sample by 90° after each photo is taken, the camera remains in a fixed position.

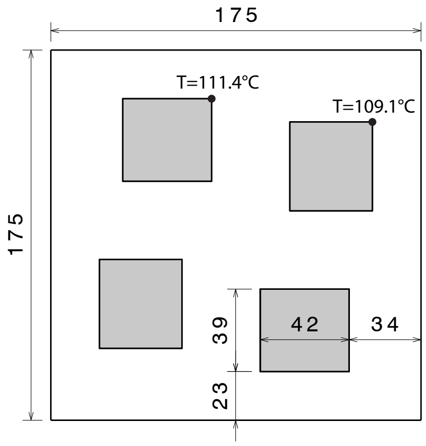


Figure 5.11: *The sizes of the photographed areas of the sample, the highest and lowest temperatures are indicated.*

The added benefit of the use of aluminium plates is that they are easily polished or even replaced when damaged. It is important that the surface finish of the mould is flawless; any scratches or contamination on the mould surface is transferred to the sample. When the sample is photographed these flaws will also create dark lines/dark spots on the image, which will be falsely identified by the digital image processor as porosity. Aluminium is easily polished and can give a desirable mirror-like finish. This was achieved by first sanding the plates using water resistant sanding paper up to grain size 360, followed by a polish with a polishing compound that is generally used for the initial-buff of aluminium alloys (brown compound). This gives a mirror-like finish, but it does leave some scratches. These scratches are however small enough that they are not picked up by the camera in the inspection jig.

5.5. Production of samples in the tile mould

The tile mould is specifically designed and manufactured for this research and it is used in most of the experiments, the general procedure for manufacturing a sample in this mould is outlined in this section. Unless otherwise mentioned this procedure is the standard for every tile mould sample.

The prepregs for all samples that belong to a certain measurement series are cut simultaneously, for the tile mould this requires cutting of 175x175 mm patches. The laminate for each sample is then stored individually in a sealed plastic bag and put in cold storage (-23°C). This is done to limit the effect of ageing between tests, because at most three samples can be manufactured in one day when two moulds are used; the moulds can only be used sequentially because there is one temperature controller. This still saves time because it is no longer required to wait for the mould to cool down and to de-mould the sample before a new one can be fabricated. The bags are removed from the freezer approximately 90 minutes before the test, the prepregs remain in their sealed container as they condition to room temperature to prevent condensation of water onto their surface.

The walls and the polished aluminium tooling plates of the tile mould are coated with three layers of Loctite® 700-NC™ mould release agent. It leaves a high-gloss finish, there is no mould build-up and it has a high temperature resistance [43].

the bottom tooling plate and the walls are placed on one lid of the tile mould and momentarily secured in place with a few bolts. The bottom laminate is placed in the cavity on the aluminium tooling plate and compacted. The mould cavity is subsequently overfilled with X-core, the excess is removed by scraping it off with a spatula. As a result the X-core is now well compacted and the mould cavity is 100% filled, see Figure 5.12. The top laminate is placed over the X-core, the bolts that secured the walls to the bottom lid of the tile mould are removed and the second aluminium tooling plate is placed on top of the sample and walls. The second lid of the tile mould is installed and the entire mould is closed with 16 M8 bolts. The mould is then connected to the temperature controller: the power cables, thermocouple cables and ground are hooked up. An insulation blanket in the form of six layers of 340 g/m^2 bleeder is used to envelop the mould, see Figure 5.13.

The cured samples are de-moulded and labelled. The labelling is done in accordance with a naming convention, it is described in Appendix D.1. The samples are then cleaned with a glass cleaner; it helps to remove grease (fingerprints) and dirt, it leaves no traces on the sample and is not aggressive to epoxy.

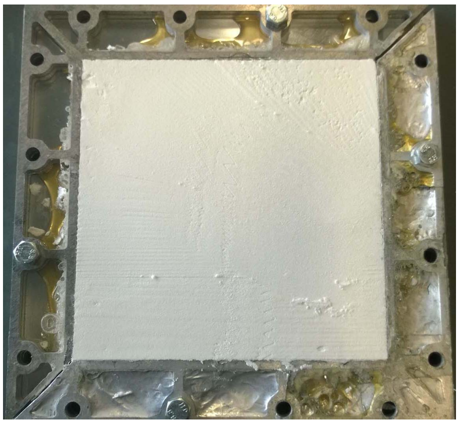


Figure 5.12: *The X-core in the tile mould fills the mould cavity entirely, excess material was scraped off with a spatula.*

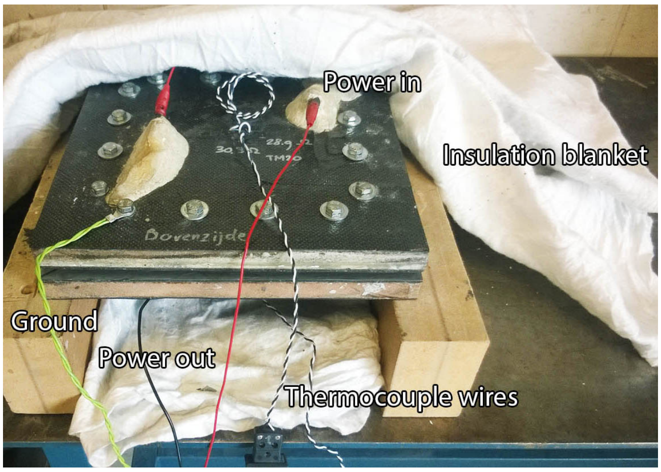


Figure 5.13: *Photograph of the experimental set-up of the tile mould.*

6

Exploration of X-core process variables

This chapter covers the identification of process variables and assessing their relevance for the X-core process. The identification of the relevant process variables is covered in Section 6.1. In addition, the scope of the research is set; it is limited to a subset of all possible combinations of process variations. The goal is to identify which subset of variables and which variations of these variables contain the most information for the current research. This is done based on exploratory tests in which small samples are prepared, the results of these tests and the selection of the process variables that are to be researched is covered in Section 6.2. The chapter concludes in Section 6.3 with a summary of these process variables. The tests that are performed in this study have been labelled according to a naming convention that is presented in Appendix D.1, it explains how samples can be identified by the different elements of their label.

6.1. Identification of variables

The X-core process involves co-curing of an epoxy based syntactic foam with a carbon fibre-epoxy prepreg and the laminate compaction pressure is triggered by the inflation of thermally expandable microballoons in X-core. Generally, curing prepregs is well-understood and widely researched, but the combination with X-core yields some important differences and introduces additional process variables.

Different grades of X-core can be made by altering its composition. The composition affects the density, strength and processability of the core material. Mainly the latter is of importance; lower density mixes generally contain less epoxy and are therefore more dry, which makes mixing (homogeneity of the mix) and application more difficult. In addition, the epoxy that is currently used in X-core, the Ancarez® 4010/Ancamide™ 3399 system, should be compatible with the resin system in the prepreg. The viscosity profile of the prepreg resin provides information about the desirable range of temperatures and ramp-up rates for processing, which should be compatible with the expansion temperature range of Expancel® 031 DU 40 as well.

There is a variety of prepregs for different applications, an important factor by which they can be classified is the degree of impregnation; partial impregnation has proven to be effective in producing VBO composite components with low void contents, whereas autoclave prepregs are generally fully impregnated [12]. Furthermore, different weave styles and areal weights are available for the fibres in the prepreg. At Donkervoort carbon fibre twill (2x2) weaves are generally used, a 245 $\mathrm{g/m^2}$ 3k weave for visual layers and a 420 $\mathrm{g/m^2}$ 12k for reinforcement. In the discussion on void mitigation strategies in Section 2.4.3 it also became apparent that the porosity content in the prepreg can be affected by the out-time (reduced tack) and relative humidity, which are variables that may be more difficult to control in the process. The composites department at Donkervoort does not have a controlled humidity level, so this is very much weather dependent.

Variations of temperature, heat-up rates and dwell times are known to affect the amount of porosity and mechanical properties of the composite laminate. It is however not guaranteed that the cure cycle is the same anywhere in the product; it may be expected that the centre of the core does not heat up at the same rate as the outer edges of the core, which are almost directly in contact with the heated tooling. The microspheres in the X-core contribute to decreasing the thermal conductivity of the material, hence delaying the flow of heat to the centre of the core material. Additionally, the polymerization reaction of epoxy is an exothermic reaction, so heat will be released by the core itself at some stage during the cure cycle. This may lead to a local increase in temperature, especially near thicker locations of the part since more volume of epoxy produces more heat. Principally, thickness variations lead to variations in temperature and hence variations in pressure application; some parts of the foam may expand before others. This could affect the density of the core material and the homogeneity of the pressure that is applied at the tooling surface. The thermal properties of X-core and how it affects the temperature distribution during the cure cycle is covered in a concurrent thesis study that is carried out by M.P.J. Eversdijk [30].

The magnitude of the pressure is very important, pressure is required to compact the laminate, extract or disperse voids and suppress void growth. In the X-core process the pressure originates from expansion of the core material. The expansion itself depends on factors like the composition of X-core, on the geometry of the component (thickness variations) and the extent of filling of the mould cavity. It is known that varying the amount of unexpanded microspheres directly influences the pressure exerted by the core on the facings. Additionally, if the mould cavity is underfilled the X-core needs some of the expansion to fill the entire space before it can apply any pressure on the laminate, so some of the pressure resulting from expansion is lost.

The thickness and stacking sequence of the laminate varies for different components. The fibre orientation can vary for aesthetic reasons (visible fabric, surface ply) or mechanical reasons. The thickness of the laminate or the number of plies can also play an important role. Thick laminates are often intermediately debulked during ply collation to remove as much entrapped air as possible and hence reduce the void content of the cured laminate [11].

The next step involves quantifying the impact of these variables on the void content of co-cured X-core sandwich structures. This is partly achieved through tests on small samples and partly by relating to similar research in literature. In summary, the main parameters that have just been identified to affect production of X-core components are:

- **Type of prepreg**
    - Prepreg out-time and tack
    - Resin system; viscosity profile, gel time, cure temperature/time
    - Type of weave; weave style, areal weight, bundle size
    - prepreg impregnation; semi-preg/full prepreg
- **Cure cycle**
    - Cure cycle profile
    - Dwell/cure times
    - Ramp-up rates
- **Pressure**
    - Extent of mould filling with X-core
    - DU volume content in X-core
- **Laminate**
    - Laminate thickness
    - Stacking sequence; varying fibre orientations, different weaves
- **X-core composition**
    - X-core epoxy content (wet/dry mix)
- **Product geometry**
    - X-core volume, core thickness
    - Thickness variations

6.2. Variable selection for the current research

The main variables that have been summarized in the previous section are a general identification, in this section a more detailed review of each variable is presented. Variables that can be expressed numerically (e.g. ramp-up rate, DU content) are quantified and their range of interest in this study is determined. The determination of numerical values, prepreg systems, X-core composition and laminates is based on tests with representative samples or based on available literature.

6.2.1. Prepreg selection

There is a large variety of prepregs, often tailor made for certain processes; e.g. autoclave, vacuum bag only and hot press-moulding. The resin in the prepreg is selected based on properties such as processing viscosity, cure temperature, gel time, glass transition temperature and shelf-life. Furthermore the type of weave, fibre material, areal weight, thickness, resin content and degree of impregnation can be of importance when selecting the right prepreg for the required application.

Measurements by S. Vial [29] have shown that X-core is capable of applying a 15 bar pressure at the tooling surface in an enclosed mould, which is achieved by adding an increasing amount of Expancel® DU. The pressure generation of X-core is covered in more detail in Section 6.2.3. The ability to achieve such pressures makes this process more comparable to compression moulding or autoclaving rather than Vacuum Bag Only (VBO), which is currently done at Donkervoort for all carbon fibre prepreg car components. This also means that the currently used prepreg systems are tailored for VBO and might not be suitable for X-core processing. The pressure is only generated at temperatures between 80°C and 120°C, above 120°C processing is still possible but the DU microballoons start to deflate. Donkervoort has several types of prepreg in storage, some of which are compatible with autoclave processing at these temperatures.

Most important is the visual layer, the outer layer of a component. This has to be a 245g/m^2 3k carbon fibre 2x2 twill weave in order to have a consistent finish throughout the vehicle. The most commonly used prepreg at Donkervoort for this application is a semi-preg with Cytec VTF261 resin. This prepreg is semi-impregnated to aid in air extraction in VBO processing and contains a thin impregnated glass fibre surface layer which helps to reduce surface porosity. Donkervoort also owns a very similar semi-preg with c-m-p CP012 resin which is actually designed for short cure cycles and autoclave processing. Finally, there is a batch of fully impregnated prepreg with c-m-p CP0093 resin which, according to c-m-p, is suitable for all processes and displays very good surface properties. All of the previous mentioned prepregs have a 245g/m^2 3k carbon fibre 2x2 twill weave, which means they all have 6 bundles per centimetre.

A fourth prepreg that is investigated is a Deltatech 12k carbon fibre prepreg with DT806R resin. The fibre architecture is not correct, but the resin system seemed promising because of its flexibility; according to Deltatech the DT806 resin is a versatile low viscosity epoxy suitable for VBO, autoclave and press moulding. All these prepregs are readily available at Donkervoort and could therefore be tested directly. Properties of all four resin systems in these prepregs are provided in Table 6.1.

Table 6.1: *Properties of prepreg epoxy resin systems used in test SP-FP3 [44, 45].*

Resin	Cure Temperature [°C]	Cure Time [min]	Pressure [bar]	Min. viscosity [1] [mPa·s]	Max. T_g [°C]	Shelf life @21°C
VTF261	65-120	960-60	0.98	NA	100 [3]	3 weeks
CP012 [2]	120-165	30-8	2.0-8.0	1700	190	4 weeks
CP009 [2]	80-170	640-4	0.8-6.0	350	138	2 weeks
DT806R	65-140	960-6	0.9-6.0	900	135	3 weeks

[1] Measured at constant heating rate of 2°C/min
[2] Data obtained from `http://c-m-p-gmbh.de/products/`, accessed on 19-05-2017, and from data sheets (not publicly available).
[3] Maximum use temperature, T_g not available.

Four samples were prepared in the floor panel sample mould (Appendix B.3, Figure B.4), they all contain the same X-core mixture: the low pressure door frame 330 mix, see Tables 4.4 and 4.6 for more information. In brief, it has a density of 306 g/L and contains 1.65 vol% DU. There is only one layer of prepreg on each side of the foam core. The cure cycle starts with a ramp-up of 1.5°C/min up to 95°C, followed by a second ramp-up of 0.33°C/min to 105°C and a 4 hour dwell at 105°C. This is the standard cure cycle for floor panel production, it is depicted in Figure 4.5. The test is designated as test 'SP-FP3', for the naming convention of samples the reader is referred to Appendix D.1. Photographs of the bottom facing of the cured samples are shown in Figure 6.1.

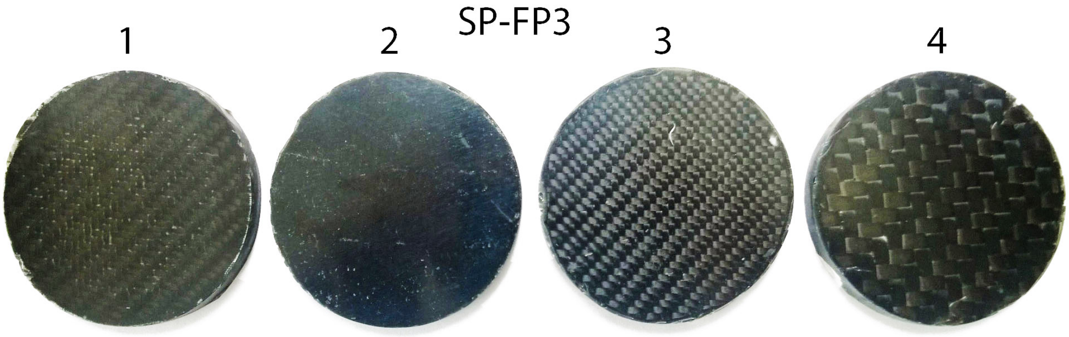


Figure 6.1: *Photographs of the samples of test SP-FP3, from left to right: Cytec VTF261, c-m-p CP012, c-m-p CP0093 and DT806R.*

The CP0093 resin has quite a favourable viscosity profile as the point of minimum viscosity ranges from about 110°C to 120°C with a heat up rate varying between 1-4 °C/min. This is favourable considering that X-core expands from 80°C onwards and can be safely used up to 120°C. So, after the onset of expansion the pressure drastically increases and a few moments later (depending on the heat-up rate) the viscosity of the prepreg is at a minimum, thus promoting resin flow, laminate compaction and void suppression. The adopted cure cycle seems to favour the c-m-p CP0093 resin system, which displayed the least amount of porosity on both facings when compared to the other specimens. There is of course a wide range of prepregs, but the availability of the c-m-p CP0093 prepreg and preliminary test results renders it the favourable candidate for this research.

6.2.2. Cure cycle variations

The design of the cure cycle is largely dependent on the prepreg used in the X-core sandwich composite, all of the samples so far had been made using semi-preg with the Cytec VTF261 resin. The introduction of a new prepreg and process window research provides an opportunity to quantify the effect of the cure cycle design on the quality of X-core products. It is expected that the cure cycle plays an important role in the development of voids in X-core, especially since the expansion of X-core is temperature dependent. A typical four-stage autoclave/oven cure cycle is shown in Figure 6.2. The cycle can be described entirely by the six parameters that are indicated in the figure.

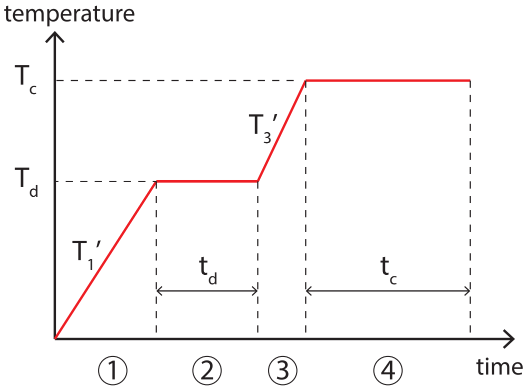


Figure 6.2: *Schematic representation of a typical autoclave cure cycle with an intermediate dwell.*

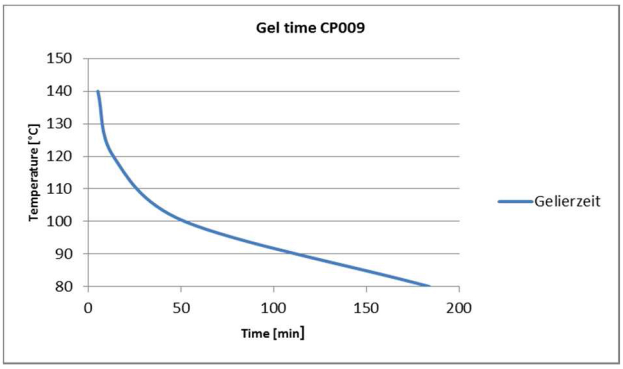


Figure 6.3: *Gel time vs temperature for c-m-p CP009 prepreg resin, courtesy c-m-p gmbh.*

The curing process itself is explained in more detail in Section 2.3.3. The cure temperature and time depends on the resin in the prepreg, but it can also be limited by the tooling or the maximum capacity of heating equipment. Expancel DU 031-40 can only be safely used up to 120°C, beyond that temperature the microballoons start to deflate. The isothermal hold in the fourth stage is required to complete the cross-linking reaction and thus cure the resin. The absolute cure temperature mainly influences cure time; the higher the cure temperature (T_c) the shorter the cure time (t_c) can be. The datasheet of c-m-p CP009 resin system contains information on gel time, cure time and viscosity, see Figures 6.3, 6.4 and 6.5 respectively. It can be read from Figure 6.4 that it takes about 60 minutes to achieve a 98% degree of cure at 120°C and that this time is doubled when curing at ~105°C.

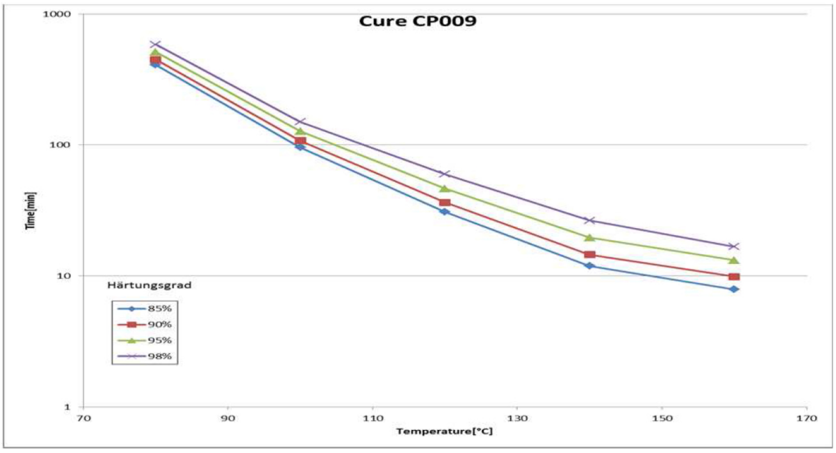


Figure 6.4: *Degree of cure vs temperature for c-m-p CP009 prepreg resin, courtesy c-m-p gmbh.*

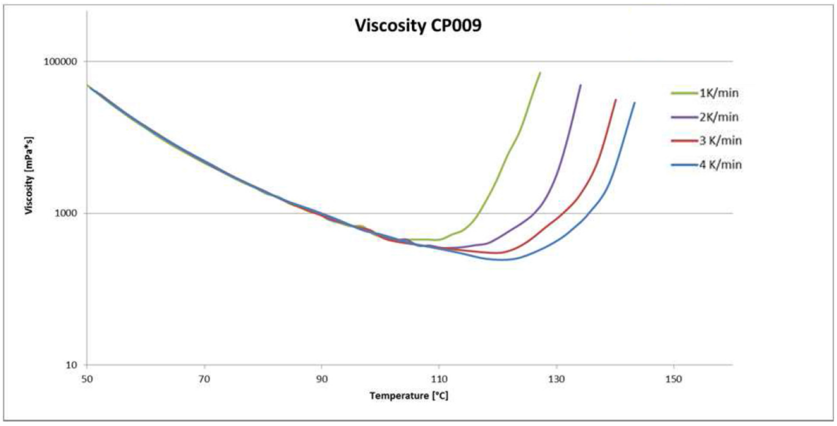


Figure 6.5: *Viscosity vs temperature for different heat-up rates of c-m-p CP009 prepreg resin, courtesy c-m-p gmbh.*

The heat-up rate is limited by the available thermal power of the heating system and it affects the viscosity of the resin in the prepreg. It is desirable to minimize viscosity during the isothermal dwell in stage 2 of the cure cycle because a low viscosity promotes resin flow and volatile removal [11]. It is clear from Figure 6.5 that the viscosity depends both on the heat up rate and the temperature.

The dwell time (t_d) may be varied to allow more time for the resin to flow or entrapped air to escape the product. After some time however the epoxy starts to gel and extending the time of the dwell beyond the gel point does not significantly contribute to resin flow or volatile extraction. Additionally the dwell needs to be carried out at temperatures in excess of 80°C, because pressure from the expanding X-core is required to remove or disperse voids and compact the laminate. In order to have some safety margin it is best to have $T_d \geq 90$°C, the upper limit is the cure temperature (120°C), which effectively skips steps 2 and 3 in the cure cycle of Figure 6.2. At 90°C the gel time is approximately 120 minutes (see Figure 6.3), so $t_2 \leq 120$ minutes.

According to the data sheet of c-m-p CP009 the processing windows is between 80°C and 170°C. Additionally, according to the graph in Figure 6.4, the cure time at the minimum dwell temperature of 90°C is approximately 300 minutes, or 5 hours. Ramping up to 120°C and curing at that temperature saves processing time, but it also introduces extra processing variables. It is therefore suggested, for the purposes of this research, to eliminate the 'cure' portion of the cycle. The dwell of step 2 is just extended until the product has fully cured, which has effectively turned it into a two-step cycle. So, the intermediate dwell still exists, but it is extended and has effectively become the 'cure' portion of the cycle.

As a result the variables dwell time t_d, dwell temperature T_d and ramp-up to cure T_3' have been eliminated. Finally the cure time can be selected based on the cure temperature and the plot in Figure 6.4; the time should be at least greater than what is required to fully cure the prepreg.

This leaves just two process variables concerning the cure cycle, namely T_1' and T_c. There is no viscosity data available on heating rates below 1°C/min and beyond 4°C/min for the CP009 resin (see Figure 6.5). It is however decided to cap the maximum heating rate at 3°C/min. Heat-up rates in excess of 2°C/min had never been tested at Donkervoort with the integrally heated tooling and temperature controller, there are concerns that high heat-up rates may damage the tooling.

The minimum heat-up rate is mostly limited by the viscosity of the resin and the desire to reduce processing time. Clearly, lower rates result in higher viscosities (Figure 6.5), but it is not yet known how high the viscosity can be allowed to be. It is decided to set the minimum heat-up rate equal to 1°C/min, this is because this is the lowest rate for which there is still viscosity data available. Table 6.2 shows the cure variables and their range, the effect of these variables on surface porosity within the indicated range is investigated in this study.

Table 6.2: *Cure cycle variables and their range.*

Variable	min. value	max. value	units
T_1'	1.0	4.0	°C/min
T_c	90	120	°C

6.2.3. Variables that affect pressure

In an autoclave the required pressure can be selected by the operator and can be varied during the cure cycle, but in the X-core process the pressure is applied when the temperature is above the onset of expansion temperature. The magnitude of the expansion also depends on temperature and contrary to gases the expansion of 031 DU 40 microspheres is not linearly related to temperature, see Figure 4.1 in Section 4.1.1. An implication of this expansion curve is that the pressure can vary inside the mould cavity if the temperature is non-homogeneous and if X-core does not behave like a perfect fluid or gas. S. Vial also found the processing pressure to be non-uniform in test samples [29]; pressures varying from 6 bar to 10 bar were measured using four laterally spaced Tekscan™ Flexiforce™ HT201 sensors in a 130x60x30 mm sample. This measurement was done on a 330 mix with 14 vol% DU, the samples were cured in a preheated oven at 100°C.

The effect of non-homogeneous pressure distributions on product quality is considered outside the scope of this research. The distribution of pressure is considered a product of the process that is dependent on several process variables like temperature, DU volume content, the homogeneity of the foam and the extent of mould filling. It is instead proposed to consider the effect of the DU volume content and extent of mould filling as process variables. It is easier to control the microsphere content in the mix or the extent of mould filling than to control the pressure in the sample. It is expected that DU can be dispersed homogeneously in X-core when it is mixed in at the stage where the viscosity is still very low: just after mixing the resin. The problem of temperature variations in the tile mould is addressed at the end of Section 5.4. The temperature is not perfectly homogeneous at the surface of the sample, but once again this is hard to control. Changing the cure temperature and the ramp-up rate will also affect the absolute difference in temperature, so the variation in pressure due to temperature is considered to be a product of the cure cycle.

Degree of mould filling

In order to investigate the effect of the extent of mould filling samples were produced in the X-core sample mould, a description of this mould is provided in Appendix B.1. Four samples (130x60x30 mm) were prepared with facings (130x60 mm) of one layer 245 g/m^2 3K carbon fibre 2x2 twill weave semi-preg with Cytec VTF261 resin. This test was conducted before test SP-FP3 in which the prepreg with c-m-p CP0093 resin was considered to be the favourite candidate for this research. The semi-preg with Cytec VTF261 was chosen because it had been used in all X-core products up to that point. Each sample contains the 330 floor panel mix (0.82 vol% DU). Filling of the mould with X-core is done as follows for each sample:

1. **Underfilled + flaw:** The mould cavity is completely filled with X-core after which a 1 mm thick layer is scraped off. In addition a trench is dug into the X-core, approximately 20 mm wide, 3 mm deep and 90 mm long, running along the length of the sample
2. **Underfilled:** The mould cavity is completely filled with X-core after which a 1 mm thick layer is scraped off, which means it is ~97% filled.
3. **Overfilled:** The mould cavity is completely filled, in addition the material that is removed by digging the channel sample 1 is added on top of sample 3, meaning that it is ~102% filled.

For clarification, the photographs of the samples prior to placing the prepreg facings are shown in Figure 6.6. This test is referred to as test SP-SM1.

Figure 6.6: *Photographs of the amount of X-core in each sample of test SP-SM1*

The samples were cured by placing them in an oven preheated to 100°C for a time period of 2 hours. The samples were subsequently demoulded, cleaned and photographed. Figure 6.7 shows photographs of a part of the top facing of the samples and digitally processed binary images that show the voids/pores as white pixels on a black background.

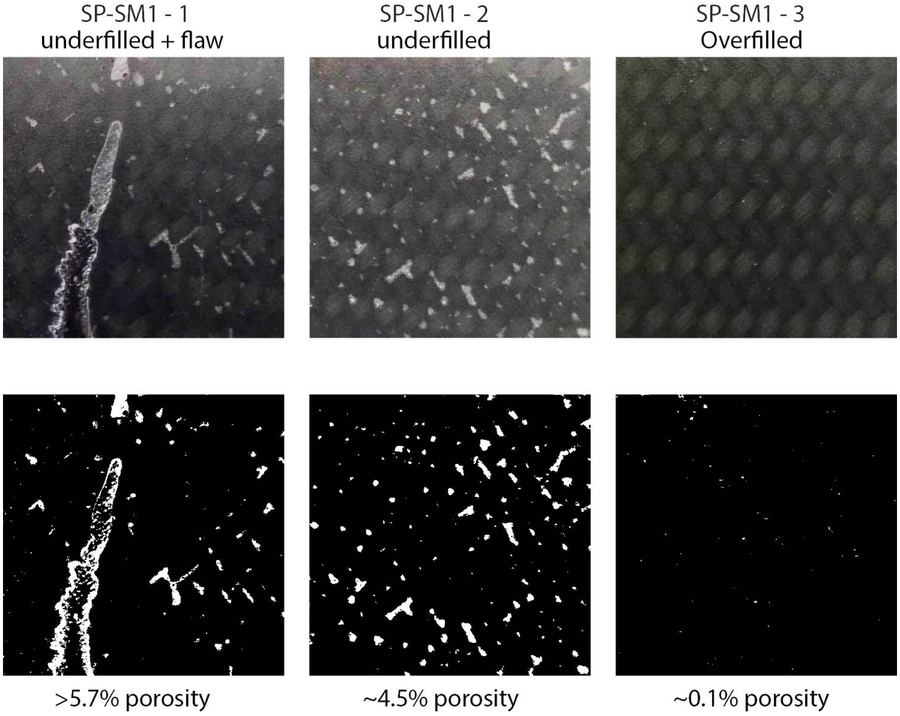


Figure 6.7: *Photographs of part of the top facing of specimens of Test SP-SM1 and digitally processed images of the surface porosity.*

At this stage of the research there was not yet a standardized photography set-up and digital image processor, but the procedure remains the same and it can still be used to get an indication of the surface porosity for a comparative study. The porosity in sample 1 is underestimated since the digital processor cannot distinguish flaws that are much larger compared to the size of the fibre bundles in the weave. But the trend is still clear; overfilling yields high quality surfaces and introducing a large flaw in the component can even lead to insufficient expansion of the core material and thus insufficient compaction of the laminate. It is also troubling that slightly underfilling the mould yields a very poor surface quality in this experiment (sample 2). The results indicate that surface porosity is strongly related to the extent of mould filling, it may be postulated that this is partly due to a different pressure in each of the samples. there can also be other mechanisms at work, but given the large difference in porosity it is clear that mould filling should be standardized.

It is suggested that the easiest way to fill the mould is to fill it 100%, which means it is actually slightly overfilled because it does not take into account the thickness that is taken up by the laminate on one side of the core. This is also currently done for the door window frames, where a dedicated X-core mould is used to ensure that the volume of the core prior to closing the mould corresponds to the volume of the final product (see Appendix A.2 for an explanation of this procedure). Slight overfilling has until now not caused problems with closing the mould, X-core allows for some extra compaction if needed.

DU volume content

S. Vial [29] managed to find a linear relation between the DU content in the X-core mix and the pressure at the facing of a 130x60x30 mm sample inside the X-core sample mould. These samples were cured by placing them in a preheated oven at 100°C. Above the expansion temperature the samples would almost immediately achieve maximum pressure and retain this pressure at the cure temperature. The processing pressures can be approximated beforehand if it is assumed that this relation still holds when using the same mix in a different geometry mould. The relation was formulated as follows:

$$P = 1.0653 + 0.8324 \cdot v_{DU} \tag{6.1}$$

Where v_{DU} is the volume content of Expancel 031 DU 40, expressed as a percentage. This relation holds for DU volume contents up to 14%. The relevant data and the linear fit are presented in Figure 3.8 (Previous research). Two problems are however identified in this relation:

1. The measurements by S. Vial were done by mixing a fixed volume of X-core, aiming at 95% filling of the mould. But, in the current study the aim is to always have a 100% mould filling. Pressure is a product of expansion, if some expansion goes lost to fill the mould cavity it may be expected that Equation 6.1 underestimates the pressure for 100% mould filling.

2. As stated in Section 4.2, S. Vial changed the estimated density of Q-cel in X-core, which changes the volume fractions of all components. This relation was formulated assuming a density of 250 g/L for Q-cel, later on this changed to 160 g/L, which results in a decrease of the DU volume fraction. The quantity remains the same, it just affects the volume fraction and hence the equation.

The change in DU volume content as a result of the difference in the density of Q-cel can be accounted for in Equation 6.1. To that end a linear regression is made relating the 'old' DU content to the 'new' DU content, see Figure 6.8. Equation 6.1 then changes as follows:

$$P = 1.0653 + 0.9934 \cdot v_{DU} \tag{6.2}$$

A test is conducted to quantify the effect of DU volume content on the porosity of the laminate. It is expected that the porosity will drop with increasing pressure, which is the same as increasing DU volume content. Pressure helps to keep volatiles dissolved, suppresses void growth and is for the X-core process the only mechanism to force air out of the laminate. In this test four samples are prepared in the floor panel sample mould. All samples contain the 330 mix with a varying DU content:

1. 330 mix with 2.5 vol% DU
2. 330 mix with 5.0 vol% DU
3. 330 mix with 7.5 vol% DU
4. 330 mix with 10.0 vol% DU

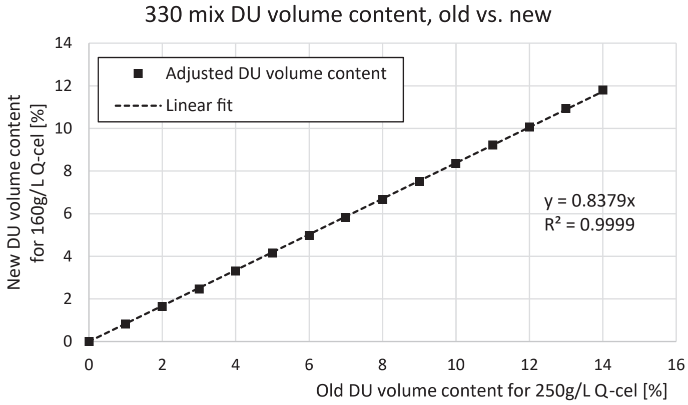


Figure 6.8: *Regression of the DU volume content of the 330 mix, assuming a density of 250 g/L for Q-cel, versus the DU content of the 330 mix, assuming a density of 160 g/L for Q-cel.*

These DU contents should, according to Equation 6.2, correspond to a range of pressures between 3.5 bar and 11 bar. This test is also conducted with the selected prepreg system: the 245 g/m^2 carbon fibre twill weave with c-m-p CP0093 resin. Each sample contains one layer of this prepreg on each facing. The lower bound was set at 2.5%, but it should in fact be realized that as low as 1.65% had already been investigated: Sample SP-FP3-3 in Figure 6.1. The upper limit is set to prevent damage to the tooling, the weakest link in the floor panel mould is the thermal insulation that sits between the heating element and the metallic lids of the mould. It is a slab of Airex C71-75 foam, which has a minimum compressive strength of 1.3 MPa [46] or 13 bar; there is still some margin, but caution is maintained.

The samples are cured in a new 2-step cycle, rather than the 3-step cycle that was previously used for the manufacture of X-core car components (Figure 4.5), this is more coherent with what is proposed in Section 6.2.2. The new cycle has a ramp-up rate of 1.5°C/min, it continues up to 110°C, where it is followed by a 120 minute dwell. It is designated as the 'X-core cycle', it is depicted in Figure 6.9. This cycle is designed with the CP0093 resin in mind. The cure time is shortened to 120 minutes, which should warrant at least a 98% degree of cure at any temperature above 105°C, based on the data in Figure 6.4.

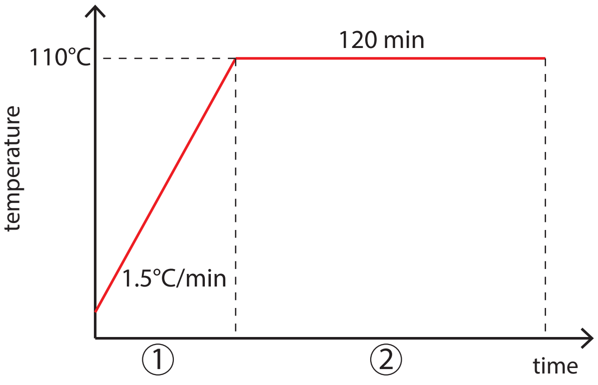


Figure 6.9: *The X-core cycle.*

Photographs of the samples of this DU content test, designated as test SP-FP7, are digitally processed, the binary images and the corresponding porosity content are shown in Figure 6.10.

In terms of statistical significance the data-set is very small, there is only two measurements per sample: the top facing and the bottom facing. Based on the results two conclusions may however be drawn:

1. The porosity in the bottom facing is significantly higher than in the top facing
2. The porosity is generally lower for the high DU content samples, samples 3 and 4.

Striking is the difference in porosity between the top and bottom facing, there is some process related phenomenon at play that is not fully understood. It might be that the process is not symmetric, for instance because the heat-up of the floor panel mould is unbalanced. This is believed because in subsequent tests with the tile mould this consistent behaviour is not reproduced.

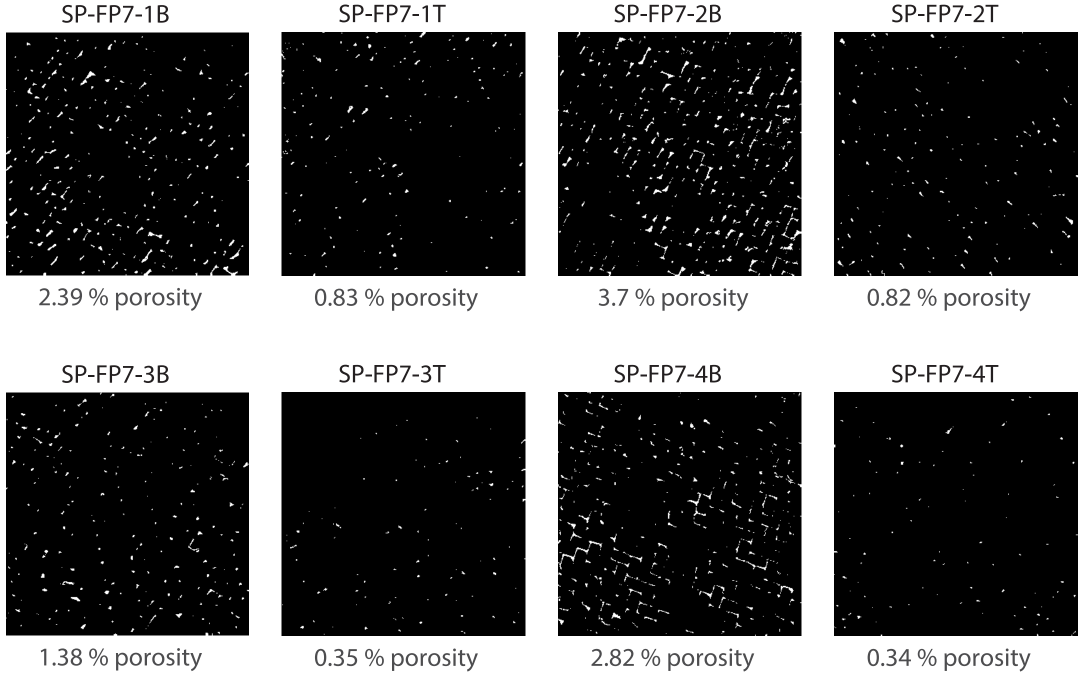


Figure 6.10: *Binary images and corresponding porosity content of DU volume variation test, designated as test SP-FP7. The last two digits in the name above each figure indicate sample number and whether it is a photograph of the top (T) or bottom (B) facing.*

On average, the porosity in samples 1 and 2 is higher than in samples 3 and 4. So, the porosity decreases as the pressure increases, but the trend is far from linear. This can however be supported by a study by F.Y.C. Boey and S.W. Lye. In this study glass fibre/epoxy prepregs are cured under pressure in an autoclave or isostatic press, without the application of vacuum. The results show a plateau where the porosity is high for low pressures, and beyond a certain pressure (~4 bar) there is a significant drop in porosity, see Figure 6.11 [17]. After this drop the porosity decreases asymptotically with increasing pressure.

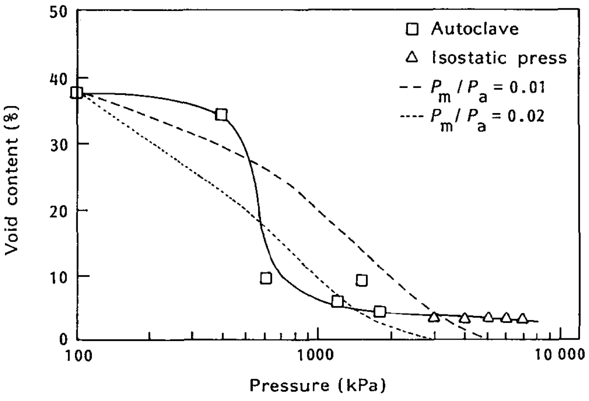


Figure 6.11: *Pressure versus porosity content in glass fibre/epoxy prepregs cured in an autoclave or isostatic press, without the application of a vacuum [17].*

The tile mould, which is to be used to do process research, is designed for a 10 bar pressure. A limit was needed to be defined to design the tooling at Donkervoort, this pressure is higher than typical autoclaving pressures (8 bar [11]) and was therefore expected to be sufficient. The results of test SP-FP7 (Figure 6.10) indicate that low porosity contents are already achievable at 7.5% DU content, increasing to 10% does not necessarily yield significant improvement. According to Equation 6.2 this should correspond to a pressure of 8.5 bar, but it is possibly higher because of the higher degree of mould filling. This is why 7.5 vol% DU is set as the upper limit for process research using the tile mould. The lower limit was set at 1.5%, which is similar to the low pressure door mix (Table 4.6) and corresponds to an estimated pressure of 2.6 bar. Based on these initial experiments lower DU contents were not expected to deliver defect-free surfaces. The limits are summarized in Table 6.3.

Table 6.3: *The range of DU volume content.*

Variable	min. value	max. value	units
v_{DU}	1.5	7.5	vol%

6.2.4. Laminates and stacking sequences

Variations in the laminate are related to the number of plies, weave types, the thickness and stacking sequence. It is common practice at Donkervoort to use a 245 g/m^2 3k 2x2 twill weave carbon fibre prepreg tailored for application as a surface layer in conjunction with a 420 g/m^2 12k 2x2 twill weave carbon fibre for reinforcement. Most components have a lay-up that consist of one 3k surface layer and one or more layers of 12k carbon fabric. The orientation of the 3k layer is solely dependent on aesthetics whereas the orientation of the 12k plies is tailored to the direction and type of loading.

For application at Donkervoort the laminate needs to consist out of at least one layer of 245 g/m^2 3k 2x2 carbon fibre twill weave at the surface. This fabric has 6 bundles per cm, other aerial weights or bundle sizes of the fibres result in an inconsistent visual finish of the component. The reflection of light on the carbon fibre bundles gives it a distinct look: Alternating bright and dark lines that run in parallel over the component. Changing the number of bundles per centimetre changes the width and spacing of these lines and the appearance of different components no longer matches.

The effect of laminate composition and thickness was investigated in test SP-FP8, wherein four samples were manufactured in the floor panel sample mould:

1. 1 Ply 245 g/m^2 (3k) carbon/epoxy prepreg with c-m-p CP0093 resin, X-core 330 mix with 10 vol% DU
2. 2 Plies 245 g/m^2 (3k) carbon/epoxy prepreg with c-m-p CP0093 resin, X-core 330 mix with 10 vol% DU
3. 1 Ply 245 g/m^2 (3k) at the surface plus 1 ply 420 g/m^2 (12k) carbon/epoxy prepreg with c-m-p CP0093 resin, X-core 330 mix with 2.5 vol% DU
4. 1 Ply 245 g/m^2 (3k) at the surface plus 1 ply 420 g/m^2 (12k) carbon/epoxy prepreg with c-m-p CP0093 resin, X-core 330 mix with 10 vol% DU

Sample 3 was added for comparative purposes, to check whether the addition of the 12k ply leads to a change in surface porosity at low pressures as well. The results of this test are shown in Figure 6.12.

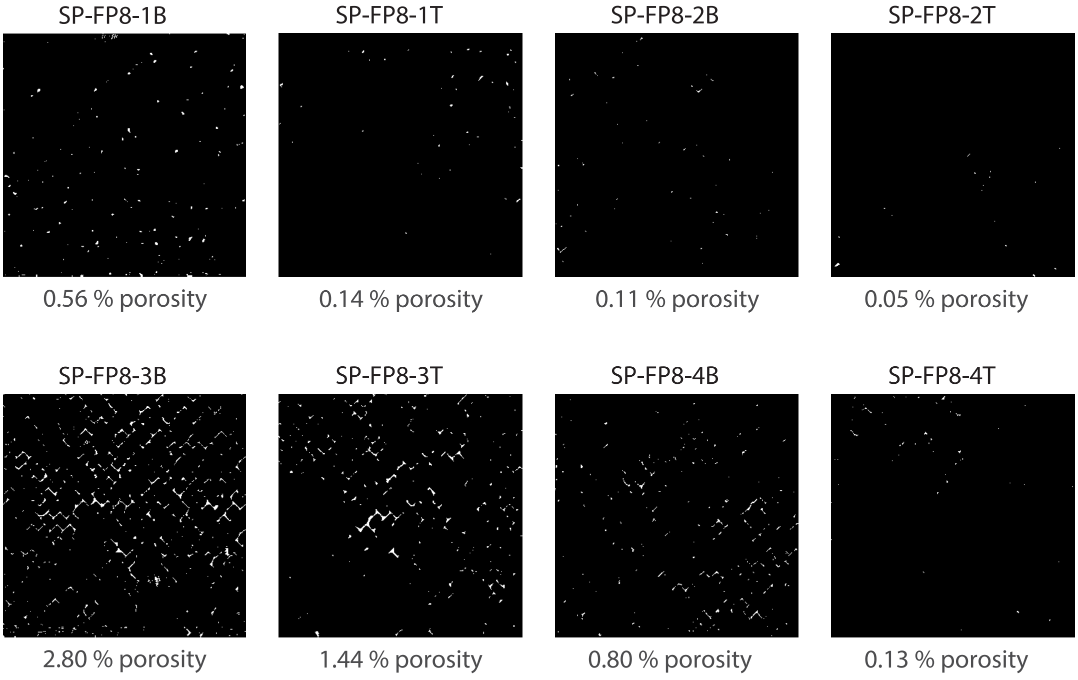


Figure 6.12: *Binary images and corresponding porosity content of laminate variation tests, designated as test SP-FP8. The last two digits in the name above each figure indicate sample number and whether it is a photograph of the top (T) or bottom (B) facing.*

First of all, the addition of one extra layer of 3k yields the best results: This is the first sample that displays a near-perfect surface finish in the X-core process. The 3k/12k laminate performs worse than the 3K or 3K/3K laminate, both for 2.5% DU (compare to sample SP-FP7-1) and for 10% DU (compare to samples SP-FP7-4, SP-FP8-1, SP-FP8-2). Based on the results it was expected at the time that this was due to the fact that the 12k had exceeded the out-time, it had very little tack compared to the 3k (same resin system), but it could not be determined how old the prepreg was. However, the results in Chapter 7 and Chapter 8 actually indicate that the difference in porosity is more likely to be caused by the difference in the weave of the fabric.

The test results also show that it is beneficiary to add an additional ply to the laminate. The extra layer helps to reduce the protrusion of X-core through the weave, which contaminates the surface and gives an aesthetically poor finish. This is especially clear when photographs of samples SP-FP8-1 and SP-FP8-2 are compared, see Figure 6.13.

Figure 6.13: *Comparison of surface finish of samples 1 and 2 in test SP-FP8.*

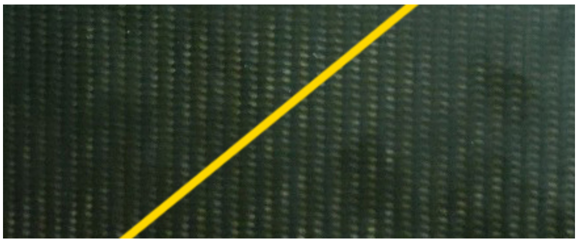


Figure 6.14: *Alternating parallel arrangement of dark lines that are visible at the surface.*

The 3k/3k lay-up gives the most promising results, but there is a preference for investigating the process window with 3k/12k lay-ups. The latter is much cheaper; it requires approximately three plies of 245 g/m^2 3K to achieve an equivalent laminate as a 3k/12k lay-up (735 g/m^2 vs. 665 g/m^2 for the latter). The 12k fabric costs 29 €/m^2 and the 3K fabric is 25.50 €/m^2, so the costs of the laminate per square metre is lower and time is saved during ply collation, which saves labour costs.

Some interesting surface phenomena can potentially result when a 12K fabric is used directly behind the surface ply; in some floor panels there are areas of the surface that show parallel lines rotated 45 degrees with respect to the 3K fabric. These lines are alternating dark and light, but the resin is not pigmented and their width corresponds to the size of the weave pattern of the 12K twill weave, see Figure 6.14.

In conclusion, the 3k/12k lay-up is investigated first because it is economically more attractive. On the other hand, it is also interesting to investigate 3k/3k lay-ups because they display a better surface finish: no waviness is observed and the surface porosity content is lower. These lay-ups result in comparable costs with respect to 3k/12k, but they are thinner and therefore mechanically inferior. Some non-structural applications at Donkervoort may benefit from this lay-up because it is lighter and the decreased surface porosity is expected to enlarge the process window. Table 6.4 provides a description of the 3k/3k and 3k/12k laminates that are used in this study.

Table 6.4: *Definition of laminates*

Laminate name	Fibre material	Type of Resin	Weave style	Fibre aerial weight [g/m^2]
3k/12k	carbon / carbon	c-m-p CP0093	2x2TW / 2x2TW	245 / 420
3k/3k	carbon / carbon	c-m-p CP0093	2x2TW / 2x2TW	245 / 245

Unless otherwise mentioned it should be assumed that the orientation of the plies in the laminate is always (0/90): The fibres are parallel to the edges of the tile mould samples.

6.2.5. X-core composition

Core material optimization is outside the scope of this research, it has been the topic of a previous study at Donkervoort (S. Vial [29], see Section 3.2.3) and its results are used to define the X-core composition for this study. The 330 mix is the benchmark, but there is reason to opt for a different composition. First of all the strength model developed by S. Vial allows for finding the mix with the highest compressive strength for a given density. In addition, Donkervoort benefits from making the X-core process work with a lighter foam, so if there is a lighter formulation that also yields promising results it is the preferred option.

Tests were carried out in a concurrent study by M.P.J. Eversdijk [30] to determine which X-core formulation provides, based on current knowledge, the most attractive set of physical and mechanical properties for application in the manufacturing process at Donkervoort. Six X-core formulations were proposed that range in densities from 230 to 330 g/L in steps of 20 g/L. All formulations have, according to the strength model, the highest specific compressive strength for their respective density. X-core is moulded manually, the comparison between mixtures mainly focussed on the processability of the foam: The selection criteria were based on formability, mouldability and stickiness, or tack of X-core. This is a qualitative study, the samples are directly compared by manual tests and their performance is measured relative to each other.

From the test it was found that the mixture with a density of 270 g/L displayed the best balance of processability and density. The strength model of S. Vial [29] predicts a compressive strength of 4.72 MPa for this mixture with 7.5 vol% DU. Compressive tests performed by M.P.J. Eversdijk confirmed this value within a maximum deviation of 3.97% over four samples. This mix is from now on referred to as the '270 mix', its pseudo composition (composition without DU) is provided in Table 6.5. The true composition, when the DU content is known, can be determined by using Equation 4.1.

Table 6.5: *Volume percentages of the constituents of the 270 mix.*

	Resin	Q-cel	DE
270 mix	14.9%	52.8%	32.3%

Compared to the 330-mix (Table 4.4) the main difference is the lower resin content which is compensated with a slight increase in DE and Q-cel. The lower compressive strength compared to the 330 mix (6.25 MPa) also affects the maximum span length for the samples in the three-point bending tests (Equation 5.2). However, in this case it is assumed that a lower compressive strength yields a lower shear strength, which in turn increases the maximum allowable span length. It is a maximum allowable, so 125 mm is still small enough to ensure core- or interface failure in the three point bending test. There is however no warranty that the 270 mix delivers the same quality of surface finish. Therefore a test is conducted in the tile mould whereby two samples are made. The sample identification label is different from this test onwards, the details are described in Appendix D.1. This test series is designated as XC-TM, more tests follow in the remainder of the research, a description of the first two samples is provided below:

- **XC-TM1:** 10 mm thick sample with 330 mix, 7.5 vol% DU, 3k/3k laminate
- **XC-TM2:** 10 mm thick sample with 270 mix, 7.5 vol% DU, 3k/3k laminate

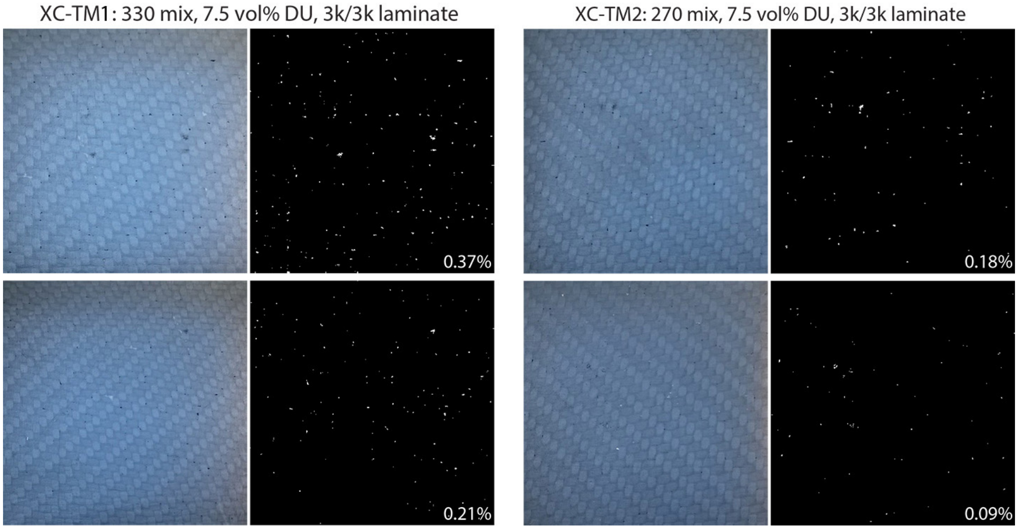


Figure 6.15: *Comparison test between 270 mix and 330 mix, original photographs and binary images including surface porosity percentage are shown.*

Both samples are cured with the X-core cycle: 1.5°C/min to 110°C (see Figure 6.9). The 3k/3k laminate was opted for because at the time there was no 12k prepreg with c-m-p CP0093 in storage at Donkervoort.

Two photographs are taken of the top surface of each sample and are subsequently analysed digitally, the result of which is presented in Figure 6.15. Sample XC-TM1 serves as the benchmark and allows for assessing whether the same results can be obtained when the process is scaled up from a 67 mm diameter by 8 mm thick sample to a 175x175x10 mm sample. The bottom surfaces were not analysed because of a contamination on the mould that would appear as porosity on the photographs. After these tests the aluminium tooling plates of the tile mould were polished, effectively eliminating this problem.

The results indicate that there is no significant difference between the two samples. Sample XC-TM2 actually performs slightly better: a mean porosity of 0.14% versus 0.29% in XC-TM1. But, considering all process variations and that only two photographs are used for the comparison the difference is statistically insignificant. More photographs can be taken in the next tests if the aluminium tooling plates are cleaned and polished and the alignment jig for photographing of the tile mould samples is finished (Section 5.3.1).

6.2.6. The effect of product dimensions

The final parameter of influence that is to be addressed in this research is product dimensions. Problems mainly arise when components are relatively large: it becomes harder to heat the products up homogeneously and the internal pressure generated by X-core results in large forces on the mould construction. X-core is a foam and even in its uncured state its volume is for the most part taken up by hollow gas-filled microballoons. This has a thermally insulating effect, which helps to delay the flow of heat to the core of the material. Conversely, the exothermic heat generated by the reaction of the epoxy in X-core in combination with the thermal insulation may lead to a temperature gradient in the core as well. The thicker the core the more pronounced these effects are expected to be. The thermal behaviour of X-core during the cure cycle is researched by M.P.J. Eversdijk [30].

The test standard for three point bending (ASTM C393 [35]) prescribes that the core thickness has to be at least 10 times the thickness of the facings. So, the minimum sample thickness is, assuming the 3k/12k laminate, $0.75 \cdot 10 + 0.75 \cdot 2 = 9.0$ mm, which is rounded off to 10 mm. The absolute maximum thickness is 40 mm, this is because the test standard dictates that the sample width should be at least twice the thickness. Just two test samples can be extracted from a 175x175x40 mm plate, which is considered the bare minimum in this research. Two samples still offers repetition of results whilst guaranteeing the same processing conditions. More samples is preferable, the determination of confidence bounds in the results is then more accurate and the results are more statistically significant. The range of sample thicknesses h that are to be researched are provided in Table 6.6.

Table 6.6: *The range of sample thicknesses.*

Variable	min. value	max. value	units
h	10.0	40.0	mm

6.3. Summary of variables

In this chapter the process parameters that could influence the surface quality of X-core sandwich structure were identified, after which the choice of variables that are to be researched in this study is explained. One of the parameters that is going to be fixed is the prepreg system: For all samples a carbon fibre prepreg that is impregnated with c-m-p CP0093 resin is going to be used. For the core material the 270 mix is proposed, a new X-core composition that has a lower density than the 330 mix and comparable processing characteristics. It is also decided to always pursue a 100% degree of mould filling with X-core, insufficient filling should especially be prevented. The laminate is restricted to two permutations: the 3k/3k and the 3k/12k lay-up. The 3k/3k lay-up has a lower aerial weight and has shown promising results in terms of low surface porosity contents, the 3k/12k laminate is economically more attractive.

The process variables that are not fixed are the cure cycle, Expancel DU volume content in X-core and the sample thickness. The cure cycle is simplified to a two-step cycle, the only variables are heat-up rate (1-3°C/min) and cure temperature (90-120°C). The cure time is directly taken from the datasheet of the prepreg resin, it is solely dependent on the cure temperature. The DU volume content is varied from 1.5% to 7.5%, this is estimated to correspond to a pressure range of approximately 2.6-8.5 bar. The sample height is varied from 10 mm to 40 mm, this range is related to the requirements for the three-point flexure test in accordance with the ASTM C393 test standard.

7

Process window study 1

In this first process window study the tile sample mould is used to manufacture samples with variations in the cure temperature and heat-up rate of the cure cycle, Expancel® DU volume content and sample thickness. In this study the variations of these four process parameters are related to changes in the surface porosity content of the cured samples. The goal of this process window study is to identify trends between the variables and the outcome and, if possible, to identify whether a process window exists wherein X-core structures can be manufactured with a high quality surface finish.

It is first established what a high quality surface finish is, a reference is required to define where the limit is. The definition of the benchmark surface porosity value is covered in Section 7.1. Subsequently Section 7.2 covers Design of Experiments (DoE) and how it helps to determine which samples should be manufactured to maximize the information that can be obtained from a specified number of test samples. Finally, a description of the experiment, the results and the validation are presented in Sections 7.3, 7.4 and 7.5.

7.1. Benchmark for high surface quality

A ’high surface quality’ is not a clear definition, at this stage it is not measurable; it is unknown what value of surface porosity content may be attributed to it. A realistic impression of what is possible with the X-core process is obtained when examining samples XC-TM1 and XC-TM2, see Figure 6.15 (see Appendix D.1 for an explanation of sample names). It is however necessary to replicate the results in order to get a fair assessment of the porosity content using the optical inspection jig and methodology outlined in Chapter 5. This is necessary because the aluminium tooling plates of the tile mould were not polished and had some contamination, which impairs the porosity measurement.

A new sample is manufactured following the same procedure as for sample XC-TM2, but now using the polished aluminium tooling plates. The sample is labelled XC-TM3, a photograph of its top facing is shown in Figure 6.1, where top is defined as the top facing during the cure of the sample. Upon first inspection it shows to be of high quality and it is aesthetically much more appealing than samples XC-TM1 and XC-TM2.

There are some small pinholes scattered over the surface of the sample, so the surface porosity content is not exactly zero. After digital processing the porosity content of the sample is determined, the values are provided in Table 7.1. This sample is a clear demonstration of the capabilities of the X-core process and sets the benchmark for subsequent research. This value is not considered to be a requirement, it only serves as a visual reference of what is realistically achievable and what is considered a ’high quality’ surface finish at Donkervoort. 0.025% is not a target for for the process window, it is a reference value to which the results of this study can be compared.

Table 7.1: *Average surface porosity and standard deviation of sample XC-TM3*

Sample name	Mean surface porosity [%]	Standard deviation [%]
XC-TM3	0.025	0.020

Figure 7.1: *Photograph of sample XC-TM3, 3K/3K laminate, 7.5 vol% DU.*

7.2. Design of Experiments (DoE)

The expectation is that products can only be successfully manufactured for a certain range of the four process parameters heat-up rate (T_1'), Cure temperature (T_c), sample thickness (h) and DU volume content (v_{DU}). The surface porosity is the response of the experiment, the process parameters are the factors. The goal of the experiment is to relate the factors to the response, this allows for the identification of trends, for example that porosity decreases with increasing DU content.

Initially it is however unknown how this trend behaves, it is not clear whether the relation between DU content and surface porosity is by approximation linear, quadratic or a higher order. Intuitively the strategy is to change one separate factor at a time within the determined range and measure the corresponding responses. But this approach requires a large number of experiments, as is illustrated by the following example:

Consider that a quadratic relation between the factors and the responses is expected. The bare minimum of data points to fit a quadratic relation is three, this is defined as the number of levels. To capture the entire measurement interval these data points would be the maximum, minimum and for instance the mean value of the factors. There are four factors, each with three levels, so the total number of tests to run when changing one factor at a time is $3^4 = 81$. If a higher accuracy is desired the number of data points, or levels, can be increased to four, which results in $4^4 = 256$ tests.

Running this number of tests might be possible, but it is not the most efficient approach. The better option is a designed experiment, in a designed experiment a special reduced set of tests is used to approximate reality through a mathematical model. This is beneficial because reducing the number of tests saves time and costs, especially when the outcome of the experiment is not strongly affected. The mathematical model helps to fill the 'gaps' by interpolating test results. In this model the response y is related to factors x and coefficients β. A linear model with m factors is defined as follows:

$$y_i = \beta_0 + \beta_1 x_1 + \beta_2 x_2 + ... + \beta_p x_m \tag{7.1}$$

or in matrix notation:

$$y = X\beta \tag{7.2}$$

Where $\beta_1, \beta_2, ..., \beta_p$ are the regression coefficients of the model and X is the model matrix. The first step in a designed experiment is to select the model, at this stage the correlation between the factors and responses is to be estimated. There is a trade-off to be made: higher order models can represent the outcome more accurately but also require more tests to accurately fit the response. It should first be considered what the implication is of the order of a polynomial: A first order polynomial, or linear model, can only have maxima at the extremities, a second order (quadratic) model can have a local optimum and subsequently every order higher can capture an additional local optimum. So, the question is whether there is reason to believe whether there can be local optima and how many there could potentially be.

In this case the quadratic model is proposed, the advantage of this model is that it can capture a local optimum and that it still requires a relatively low number of tests. The approach is then to first fit the proposed quadratic model and assess the accuracy of the fit. Additional tests can always be performed to add data points to the test matrix, thus improving the accuracy of the quadratic model or to allow for fitting a higher order model if the data suggests that such a trend exists. On the other hand, the order of the model can always be reduced if the trend shows to be linear. The quadratic model in this research would be as follows:

$$y = \beta_0 + \beta_1 T_1' + \beta_2 T_c + \beta_3 h + \beta_4 v_{DU} + \beta_5 (T_1')^2 + \beta_6 (T_c)^2 + \beta_7 (h)^2 + \beta_8 (v_{DU})^2 \tag{7.3}$$

As mentioned earlier, a quadratic model requires at least 81 tests to cover all permutations of the combinations of test factors. This is where the concept of Design of Experiments (DoE) comes in as an effective tool to reduce the number of test runs. In DoE the most informative combination of factors is chosen rather than the whole set of combinations. Determining the most informative set of factors is done through a computer aided process that seeks to determine the optimal experimental design: The D-optimal approach [47].

7.2.1. The D-optimal approach

Selecting which combinations of factors yields the most information to fit the mathematical model (Equation 7.2) is done based on certain optimization criteria. D-optimality is a commonly used criterion, this approach is briefly explained in this section.

It starts with the candidate matrix, it contains all theoretically and practically possible combinations of the factors: each row in this matrix represents an experiment and each column one of the variables. In the current research there are four factors (T_1', T_c, h, v_{DU}) and each factor has three levels: the maximum, minimum and mean value of their respective range. So, in this case the candidate matrix has 3^4=81 rows and 4 columns. The order of the columns is based on the order of appearance in Equation 7.3; column 1 represents T_1', column 2 represents T_c, etc. To make this matrix simpler and easier to handle it is often rewritten in the extended notation, the entries are all scaled to have a range of 2 and values between -1 and 1. Table 7.2 shows the unscaled values of each of the factors and their corresponding value in the extended notation.

Table 7.2: *The extended notation of the factors and their unscaled values*

Extended notation	Unscaled factors			
	Ramp-up rate [°C/min]	Cure temperature [°C]	Sample thickness [mm]	DU content [vol%]
1	3.0	120	40	7.5
0	2.0	105	25	4.5
-1	1.0	90	10	1.5

The model matrix represents Equation 7.3 for each experiment, in this case it would be a 81 × 9 matrix because there are 9 β coefficients and 81 experiments. The first column of this matrix represents β_0, so it only contains ones. The next four columns are equal to the candidate matrix and the final four columns are equal to the square of the candidate matrix, in accordance to the order of appearance of the terms in Equation 7.3.

The model matrix needs to be reduced, the goal is to reduce the number of rows by getting rid of the rows that contain the 'least information'. The experimenter can choose the desired number of tests, the accuracy is increased by performing more tests but that also takes more time and resources. In theory the minimum number of tests to fit the quadratic model is equal to the number of coefficients β, which is 9.

If the desired number of test runs is defined as n, then the design matrix X is a $n \times 9$ matrix. The response y (the porosity) is equal to the product of the design matrix X and the coefficients vector β (Equation 7.2). An example of a design matrix for a purely quadratic model with 4 factors and 9 runs is given below:

$$X = \begin{bmatrix} 1 & 0 & 1 & 0 & 1 & 0 & 1 & 0 & 1 \\ 1 & -1 & -1 & 0 & 0 & 1 & 1 & 0 & 0 \\ 1 & 1 & 1 & -1 & 0 & 1 & 1 & 1 & 0 \\ 1 & 1 & -1 & 1 & 1 & 1 & 1 & 1 & 1 \\ 1 & 0 & 0 & 1 & 0 & 0 & 0 & 1 & 0 \\ 1 & -1 & 1 & 1 & -1 & 1 & 1 & 1 & 1 \\ 1 & 0 & -1 & -1 & -1 & 0 & 1 & 1 & 1 \\ 1 & 1 & 0 & 0 & -1 & 1 & 0 & 0 & 1 \\ 1 & -1 & 0 & -1 & 1 & 1 & 0 & 1 & 1 \end{bmatrix} \tag{7.4}$$

In order to use the D-optimality criterion for the selection of the best subset of experiments two other matrices need to be defined. Multiplying the transpose of the design matrix X with itself gives the information matrix $(X'X)$, the inverse of the information matrix is defined as the dispersion matrix $(X'X)^{-1}$. Applying the principle of least squares the model in Equation 7.2 has the following best set of coefficients $\hat{\beta}$ [47]:

$$\hat{\beta} = (X'X)^{-1} X' y \tag{7.5}$$

The D-optimal approach seeks to maximize the determinant of the information matrix. So, the optimal design matrix X^* is the matrix that contain those rows from the model matrix that maximize the determinant of the information matrix. These matrix manipulations can easily be performed by a computer, in fact MATLAB has a built-in toolbox for determining D-optimal designs and subsequently for performing curve fitting.

7.2.2. Number of test runs

Part of the designed experiment is deciding how many test runs are to be performed. In this case the minimum amount of runs is 9 and the maximum is 81. It is of course questionable whether the desired accuracy is achieved by performing the minimum amount of runs. Performing more experiments results in more data points to fill up the experiment design space, which gives more insight into the general trend of the data and whether it correlates well to the chosen mathematical model. Increasing the number of replications increases the accuracy of the fit of the mathematical model, provided the model is a good approximation of reality.

The DoE approach allows to expand the experiment space, data points can always be added or replications can be performed when desired. Therefore it is decided to start out small by assuming a quadratic model with just 18 experiment runs, which is twice the minimum number of test. For the porosity determination, each run contains eight measurements of the sample's surface. For mechanical testing 2 to 7 substrates can be extracted from a single sample, depending on the thickness h.

7.3. Test summary

In this section an overview is presented of what is to be tested in process window study 1. It includes a description of the process conditions and a brief summary of the experimental method. The general test-setup, tooling system (the tile mould) and porosity analysis are covered in Chapter 5, this section only covers procedures that are specific to this process window study.

The set-up for manufacturing samples for this study using the tile mould is drawn schematically in Figure 7.2. For clarification: the mould is filled with X-core up to the top surface of the mould walls, the top laminate is then placed over the core and the mould is closed. The excess core material does not flow out of the mould, it is simply compacted to accommodate the laminate thickness.

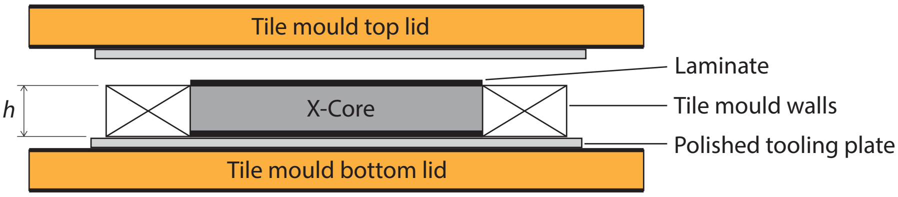


Figure 7.2: *The set-up for sample production in PW1*

The D-optimal approach is used to design the experiment for 18 runs, the resulting combinations of variables are provided in Table 7.3. It should be noted that the actual ramp-up rate T_1' is not exactly equal to the values that were stated in Table 7.2 (ranging from 1 to 3). A bug in the temperature controller software resulted in a error in the calculation of the slope of the ramp-up part of the cure cycle. This problem recurs in all experiments of this process window study; the ramp-up rate is lower than the set-point value in each test. The actual heat-up rate is recorded by the controller, this value is used as the reference value for the remainder of this study and is used in fitting the model. This does not affect the behaviour of the process, it only changes the range of the variable to 0.94-2.86°C/min.

For convenience, the last column in this table provides the cure time, it is solely dependent on the cure temperature and is therefore not an independent variable. The 270 mix is used for all samples, the constituents can be found in Table 6.5. All samples have a 3k/12k laminate, the description of this laminate can be found in Table 6.4. The samples are manufactured as described in Section 5.5. Eight photographs are taken of each sample and processed to determine the porosity content, using the optical inspection jig and procedure described in Section 5.3.

Table 7.3: *Description of the process variables for each sample in process window study 1.*

Test id	T_1' [°C/min]	Actual T_1' [°C/min]	T_c °C	h [mm]	v_{DU} [%]	Cure time [min]
PW1-TM-01	3	2.41	120	25	4.5	60
PW1-TM-02	3	2.42	120	10	4.5	60
PW1-TM-03	2	1.83	120	40	1.5	60
PW1-TM-04	2	1.80	120	10	7.5	60
PW1-TM-05	1	0.94	120	40	7.5	60
PW1-TM-06	1	0.96	120	25	1.5	60
PW1-TM-07	3	2.51	105	25	7.5	120
PW1-TM-08	3	2.76	105	40	1.5	120
PW1-TM-09	2	1.79	105	10	1.5	120
PW1-TM-10	2	1.81	105	25	4.5	120
PW1-TM-11	1	0.95	105	10	7.5	120
PW1-TM-12	1	0.94	105	40	4.5	120
PW1-TM-13	3	2.86	90	40	7.5	300
PW1-TM-14	3	2.75	90	10	1.5	300
PW1-TM-15	2	1.94	90	40	4.5	300
PW1-TM-16	2	1.93	90	25	7.5	300
PW1-TM-17	1	0.96	90	10	4.5	300
PW1-TM-18	1	0.96	90	25	1.5	300

7.4. Results

The porosity content of each sample, defined as the percentage of surface area covered by pores, is provided in the bar chart in Figure 7.3. The bars represent the mean porosity of the four measurement areas of either the top or the bottom facing of the sample. The standard deviation of the measurement data is included in each bar. An indication of the effect of surface porosity content on the surface finish of the sample can be obtained from the porosity reference chart in Appendix C.

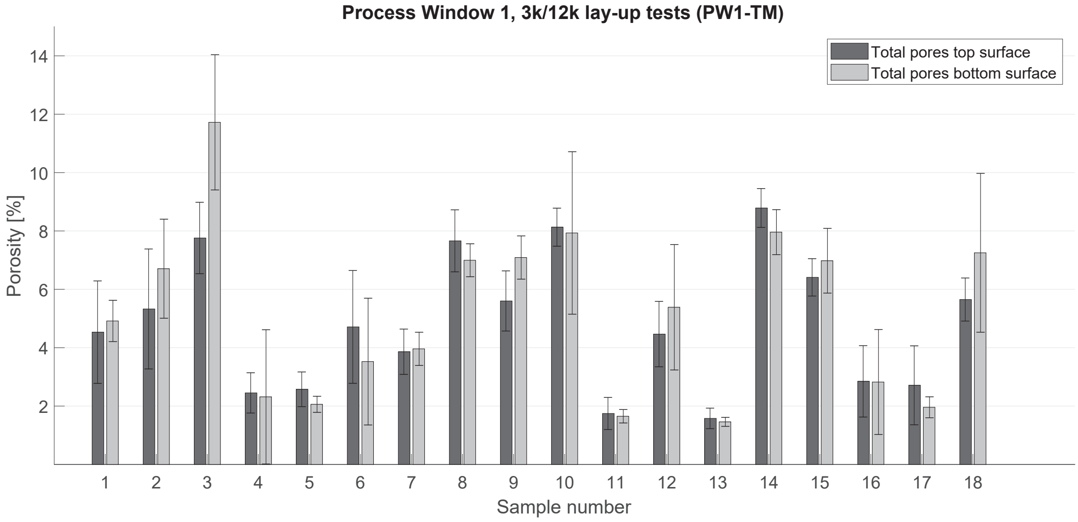


Figure 7.3: *Porosity content of each sample in PW1-TM, including one standard deviation/68% confidence bounds*

There are three important results from the porosity measurements:

1. There is a strong variation in porosity content, confirming that at least one of the four process variables or a combination of a number of variables that are intentionally varied in this study has a measurable effect on the surface porosity.
2. Not a single sample is below the 0.025% porosity benchmark. The lowest porosity content that is measured is 1.5% and is found in sample 13, it is much higher than the benchmark and the surface quality is found unacceptable upon visual inspection.
3. There is no statistically significant difference between the top and bottom facing: In all samples, with the exception of sample 3, the 68% confidence bounds of the mean porosity in both facings overlap. In addition, the mean porosity in either facing is not consistently higher or lower.

The last result is interesting because the opposite is observed in the floor panel sample mould, these samples consistently display a higher porosity content in the bottom facing. This problem can be related to a variable that was not previously identified, a process variation that is more pronounced in the floor panel sample mould than in the tile mould. It is hypothesized that the difference in porosity in the floor panel sample mould is related to air evacuation, which depends on the permeability of the parting lines in the mould. It is recommended that this hypothesis is tested in future research.

Which variables have the most pronounced effect on the surface porosity and what the trend is of the data can be determined by fitting the proposed mathematical model of the designed experiment (Equation 7.3) to the measured porosity. It is too early to conclude that the benchmark value cannot be achieved, the model may very well indicate that a lower porosity can be obtained for a certain set of parameters that has not been tested previously. From this point onward the porosity data is averaged over all eight measurement areas, which yields a single value for each sample. Based on the results it is not deemed necessary to make a distinction between the top and bottom facing, the process shows to be symmetric about the mid plane of the sample.

7.4.1. Fitting the model

The β coefficients are estimated using an iterative least squares estimation, MATLAB has a built-in toolbox for non-linear regressions using this method. The standard fit options in MATLAB are used, they are provided in Table 7.4. For non-robust estimation the Levenberg-Marquardt algorithm is employed by the software. With these options the fit is performed, keeping in mind the following quadratic model for the surface porosity α_p:

$$\alpha_p = \beta_0 + \beta_1 T_1' + \beta_2 T_c + \beta_3 h + \beta_4 \nu_{DU} + \beta_5 (T_1')^2 + \beta_6 (T_c)^2 + \beta_7 (h)^2 + \beta_8 (\nu_{DU})^2 \tag{7.6}$$

The initial estimate of the β coefficients in the iterative least squares estimation is set at $\beta_i = 1$. In every step of the iteration the coefficients are adjusted and the new model is compared to the experimental data. The iteration converges when the difference in the value of the coefficient of the current iteration with respect to the previous iteration is smaller than 'TolX'. It also converges when the change in Sum of Square due to Error (SSE) of the current iteration with respect to the previous iteration is smaller than 'TolFun'. Finally, the procedures stops when the maximum number of iterations is reached defined by 'MaxIter', this does not guarantee convergence.

The least squares estimation of the model in Equation 7.6 converged after 8 iterations because the change in the SSE was smaller than the specified tolerance. Decreasing the tolerance values or changing the initial values of β did not yield a different estimate of the coefficients. The β coefficients, the Mean Squared Error (MSE) of the fit and the error parameter of the error model in this fit are provided in Table 7.5. The plots in Figure 7.4 show the variation of surface porosity as a function of each variable individually, over the complete range of the respective variable. This is done to give an indication of the trend of the response as a result of a change in one variable.

Table 7.4: *Fit options of the non-linear fit using an iterative least squares estimation in MATLAB.*

Option	**Description**	**Value**
MaxIter	Maximum number of iterations [1]	200
TolFun	Termination tolerance for the residual sum of squares	$1 \cdot 10^{-8}$
TolX	Termination tolerance on the estimated β coefficients	$1 \cdot 10^{-8}$
Robust	Use robust weighting function, 'on' or 'off'	off (no function used)
ErrorModel	Form of the error term [2]	'constant' ($y = f + a \cdot e$)

[1] Iterations continue until the maximum number of iterations is reached, or until the estimates are within the convergence tolerance defined by TolFun and TolX.

[2] The error is defined as a mean-zero and unit-variance variable e, f is the function value and a is the error parameter.

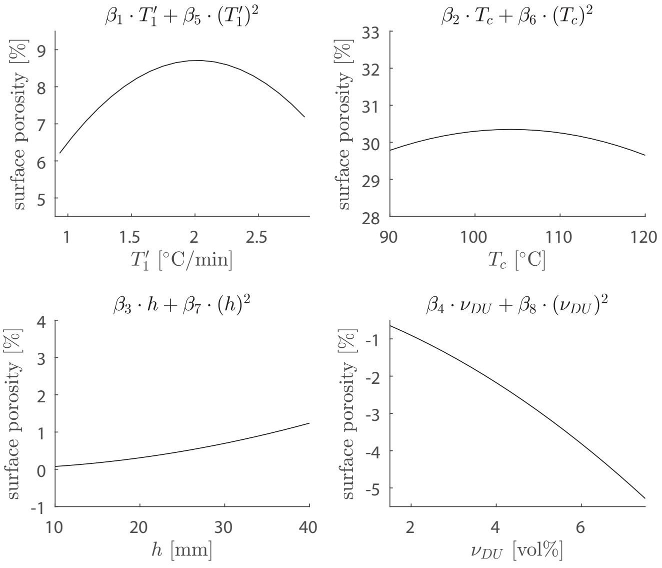


Figure 7.4: *The contribution of each variable to the variation of porosity content*

Table 7.5: *Coefficients β, error parameter 'a' and MSE of the full quadratic model*

β_0	-30.32
β_1	8.64
β_2	0.58
β_3	$-2.86 \cdot 10^{-5}$
β_4	-0.36
β_5	-2.14
β_6	-0.0028
β_7	$-7.75 \cdot 10^{-4}$
β_8	-0.0459
a	1.44
MSE	2.08

Figure 7.4 shows a quadratic relation between the heat-up rate and the cure temperature with a local optimum, whereas the relation to the sample thickness and the DU content is approximately linear. Taking into account the scale of the y-axis, the variation in surface porosity due to changes in cure temperature is relatively small compared to the other three variables: The total variation over the entire range of cure temperatures is 0.7%, whereas it is 1.2%, 2.5% and 4.6% for h, T_1' and ν_{DU} respectively. It is beneficial to simplify the mathematical model, it can help to reduce the Mean Square Error (MSE), which is defined as follows in regression analysis:

$$MSE = \frac{SSE}{v} \tag{7.7}$$

$$v = n - m \tag{7.8}$$

$$SSE = \sum_{i=1}^{n} (y_i - \hat{y}_i)^2 \tag{7.9}$$

Where v is the residual degree of freedom, n is the number of experiments, m the number of coefficients and SSE is the Sum of Squares due to the Error. So, reducing the number of coefficients for a fixed number of tests helps to increase v and hence reduce the MSE, provided that the change to the mathematical model does not lead to a high increase of the error. For instance, if the quadratic term $\beta_8(\nu_{DU})^2$ is removed the model can still be representative of the physical system, but there are now 18 data points to fit just 8 coefficients. Based on the trends in Figure 7.4 It is postulated that the effect of cure temperature is negligible and that the porosity changes linearly with a variation in DU content and sample thickness, which reduces the mathematical model to the following equation:

$$\alpha_p = \beta_0 + \beta_1 T_1' + \beta_2 h + \beta_3 \nu_{DU} + \beta_4 (T_1')^2 \tag{7.10}$$

The model is fitted to the porosity data using the same fit options as before (Table 7.4). The least squares estimation of the model in Equation 7.10 converged after 5 iterations; the change in the SSE was smaller than the specified tolerance. The new coefficients, MSE and error parameter a are provided in Table 7.6. The plots in Figure 7.5 show the variation of surface porosity as a function of the three remaining variables T_1', h and ν_{DU} over their respective ranges.

Table 7.6: *Coefficients β, error parameter 'a' and MSE of the reduced quadratic model*

β_0	β_1	β_2	β_3	β_4	a	MSE
-0.1631	8.7167	0.0389	-0.7734	-2.1657	1.28	1.63

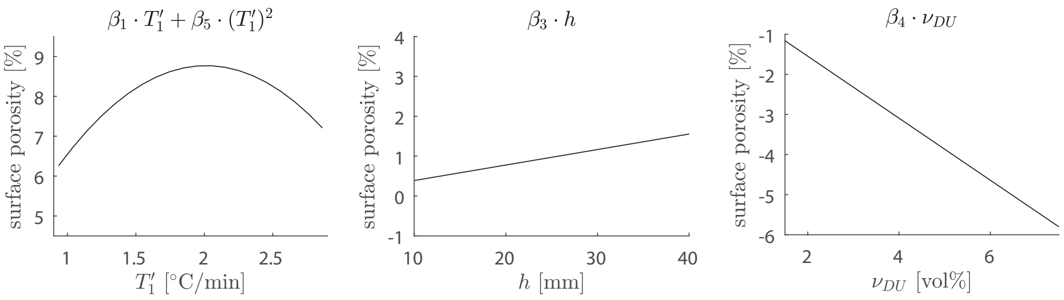


Figure 7.5: *The contribution of each variable to the variation of porosity content*

Fitting the newly proposed model in Equation 7.10, which is a reduced model that ignores the effect of cure temperature, yields a lower MSE than the fully quadratic model. This is desirable because the fit is more useful for prediction when the value of the MSE is closer to zero. The prediction of the model is compared to the measured mean porosity of each sample in Figure 7.6, the corresponding data is provided in Table 7.7.

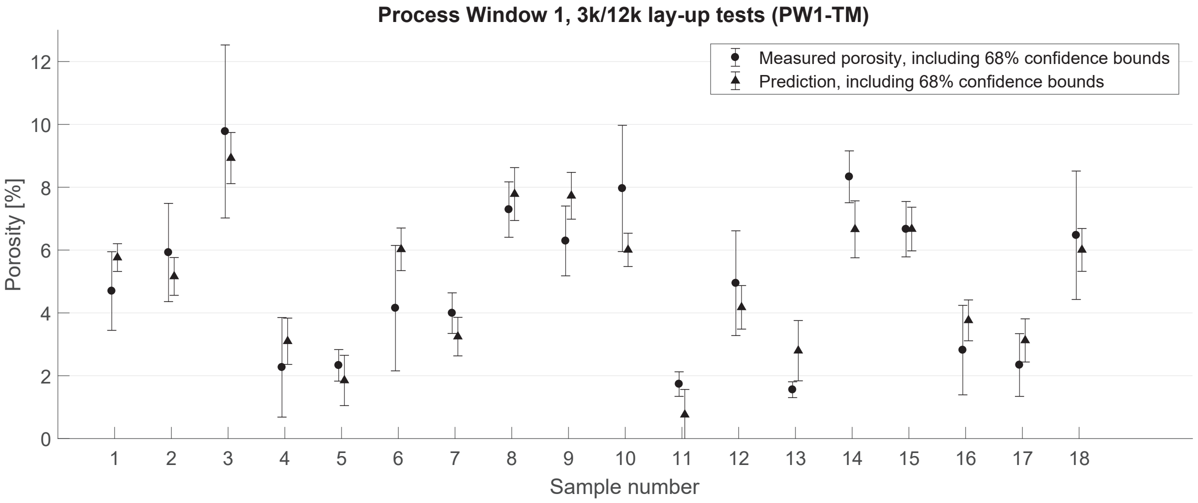


Figure 7.6: *Comparison of the mean measured porosity and the prediction including the 68% confidence interval of each.*

Table 7.7: *Porosity content of process window study 1 samples and the prediction of the mathematical model including standard deviation of the prediction.*

Sample ID	Measured porosity [%]	Predicted porosity α_p [%]	Error [%]	Standard deviation σ [%]
PW1-TM-01	4.70	5.76	-1.06	0.44
PW1-TM-02	5.92	5.16	0.76	0.60
PW1-TM-03	9.77	8.93	0.85	0.81
PW1-TM-04	2.27	3.10	-0.83	0.74
PW1-TM-05	2.33	1.85	0.48	0.80
PW1-TM-06	4.15	6.02	-1.87	0.68
PW1-TM-07	3.99	3.25	0.75	0.61
PW1-TM-08	7.29	7.78	-0.49	0.84
PW1-TM-09	6.29	7.73	-1.44	0.75
PW1-TM-10	7.96	6.01	1.95	0.53
PW1-TM-11	1.74	0.76	0.97	0.80
PW1-TM-12	4.95	4.18	0.77	0.69
PW1-TM-13	1.56	2.80	-1.24	0.96
PW1-TM-14	8.33	6.66	1.67	0.91
PW1-TM-15	6.66	6.67	0.01	0.69
PW1-TM-16	2.82	3.76	-0.95	0.65
PW1-TM-17	2.34	3.12	-0.78	0.69
PW1-TM-18	6.47	6.00	0.47	0.68

From the data in this table the adjusted coefficient of determination or adjusted R-square ($\bar{R}^2$) value is determined:

$$\bar{R}^2 = \frac{SSR \cdot (n-1)}{SST \cdot v} = \frac{\sum_{i=1}^{n}(\hat{y}_i - \bar{y})^2 \cdot (n-1)}{\sum_{i=1}^{n}(y_i - \bar{y})^2 \cdot v} = 0.74 \tag{7.11}$$

SSR is defined as the Sum of Squares of the Regression, SST is the Total Sum of Squares and $\bar{y}$ is the mean. This value means that this model explains 74% of the total variation in the data about the average. If required, the goodness of fit may be improved by adding more experiments or by changing the model. This can be done to get a more accurate prediction.

However, in this case there is no combination of process conditions that yields a porosity content that is comparable to the benchmark: a 0.025% porosity content. The model described by Equation 7.6 with the coefficients in Table 7.6 is as follows:

$$\alpha_p = -0.1631 + 8.7167 \cdot T_1' + 0.0389 \cdot h - 0.7734 \cdot v_{DU} - 2.1657 \cdot (T_1')^2 \tag{7.12}$$

The lowest possible predicted outcome of this equation is a surface porosity of 0.684%. This is achieved for the lowest heat-up rate that is measured, the thinnest sample and the highest DU content, which is 0.94°C/min, 10 mm and 7.5 vol% respectively. The model is not dependent on cure temperature, so it may be any temperature between 90°C and 120°C. This percentage is low in an absolute sense, but visually the difference between 0% and 0.68% is substantial, see Appendix C for an indication of the difference.

7.4.2. Implication of the results

In summary, 18 samples are manufactured that are subjected to a pre-defined set of process conditions that were determined through a designed experiment following the D-optimal approach. The porosity content of all these samples is measured and a quadratic mathematical model is fitted that relates the heat-up rate, DU content and sample thickness to the variation in surface porosity. In this study a process window could not be defined, but the regression model provides valuable information and helps to identify which measures help to reduce the porosity:

- Surface porosity decreases with increasing DU content.
- Surface porosity increases when the sample thickness increases.
- A heat-up rate of 2°C/min yields the highest porosity content, the best results are achieved for 1°C/min.
- The effect of cure temperature on surface porosity is negligible.
- Within the investigated range of the process variables and under the conditions of this experiment it is not possible to achieve a surface porosity content that is below 0.68%.

Comparison to the benchmark

It may be possible that a process window can be defined outside the range of variables, but the model cannot be extrapolated. Instead the focus is moved towards explaining why sample XC-TM3 (the benchmark) does yield a good surface quality whereas none of the samples in Process Window study 1 (PW1) can reproduce this result. Especially considering that XC-TM3 is just one data point, it is not necessarily on the boundary of a certain process window. If the conditions of XC-TM3 are substituted into Equation 7.12 the following surface porosity content is predicted (including the standard deviation σ):

$$\alpha_p = -0.1631 + 8.7167 \cdot (1.43) + 0.0389 \cdot (10) - 0.7734 \cdot (7.5) - 2.1657 \cdot (1.43)^2 = 2.46\% \quad (\sigma = 0.70\%) \tag{7.13}$$

This is a factor 100 more than the benchmark, which is made using the same tooling and same manufacturing procedure as all samples in PW1. This value is also supported by sample XC-TM4. It is manufactured with a 1.42°C/min heat-up rate, 110°C cure, 10 mm thickness and 7.5 vol% DU. The mean porosity of this sample is measured to be 1.60% with a standard deviation of 0.51%. The error with respect to the model is 0.86%, which is comparable to the errors in Table 7.7.

The only difference is the laminate: XC-TM3 has a 3k/3k laminate and sample XC-TM4 and the samples in PW1 have a 3k/12k laminate (see Table 6.4 for more detailed information). So, the surface ply is the same in both cases (3k), the difference resides in the second ply. The 3k and 12k weave differ in bundle size and aerial weight, this results in a difference in the tightness of the weave, see Figure 7.7.

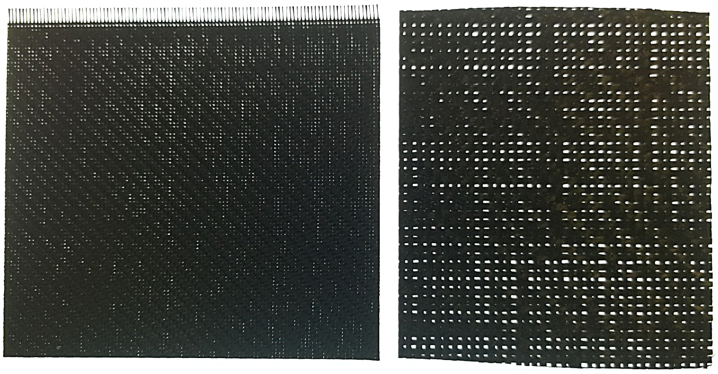


Figure 7.7: *Comparison of the tightness of the weave of the 245 g/m^2 3k weave (left) and the 420 g/m^2 12k weave (right).*

Recalling previous research it was suggested by both F. Minde and S. Vial [28, 29] that the use of a PET film between the core and laminate reduces the surface porosity. The PET film is unperforated, it effectively forms a barrier between core and laminate. Based on the results of XC-TM3, the results of PW1 and the difference in the weaves as observed in Figure 7.7 it is postulated that the 3k ply acts as a more effective barrier than the 12k ply. In order to support this hypothesis an investigation into the effect of barrier layers between the core and facings is carried out, it is covered in detail in Section 8.2.

Tile mould deflection

An additional problem that is encountered in this study is an insufficient stiffness of the lids of the tile mould. It does not necessarily explain the difference between samples XC-TM3 and the results of PW1, but it can still influence the results. The mould lids deflect when the pressure is applied, this results in an increase in sample thickness, which is dependent on the DU content. The thickness of each sample was measured at the centre of each of the four porosity measurement areas and averaged (see Figure 5.11 for dimensions), the data is provided in Appendix D.2. The increase in thickness is especially worrying for thin samples with a high DU volume content; the thickness of sample PW1-TM-04 increases by 40% at the centre of the porosity measurement area. Pressure is exerted on the laminate because the expansion of X-core is restricted by means of stiff tooling, it is likely that the pressure is lower when some expansion is allowed.

Therefore, when this study was completed, one of the two tile moulds was stiffened. The low stiffness was attributed to the MDF core which may have suffered from continuous use in high temperatures (120°C) combined with a high pressure (~6-8 bar). The facings of the mould were removed by manually delaminating them. All of the core material was removed and replaced by an aluminium honeycomb of equal thickness. The honeycomb core was bonded to the cured facings using the same SR1660/SD7820 epoxy as in the laminate, mixed with Q-cel to increases its viscosity. This repaired mould is used in the remainder of this study.

After the repair of the mould it was decided to directly manufacture the samples that needed to be subjected to the three-point flexure tests. The results show that the porosity contents of these samples no longer correspond to the prediction model. The measurements are in the order of 50% lower than the prediction, these results are presented in the next chapter.

7.5. Validation

A process window study has been carried out using a designed experiment, which means that not all combinations of process variables have been tested and that a model is fitted to predict their outcome. A few additional tests can be performed to validate that the fitted model can indeed predict the porosity content of these samples.

Initially three samples were going to be manufactured, but eventually only two samples are made because the process started to yield inconsistent results. More samples were manufactured in the same period as the validation samples and they all showed the same problem: the porosity contents are much higher than predictions and there are signs that indicate that the 3k prepreg has expired. This problem is covered in more detail in Section 8.1. For now only the results of these first two samples are covered, the processing conditions are as follows:

- **PW1-TM-val01:** $T_1' = 1.45$°C/min, $T_c = 110$°C, $h = 25$ mm, $v_{DU} = 4.5$ vol%
- **PW1-TM-val02:** $T_1' = 0.97$°C/min, $T_c = 90$°C, $h = 10$ mm, $v_{DU} = 7.5$ vol%

The measured porosity contents and the corresponding predictions are provided in Table 7.8.

Table 7.8: *Porosity content of PW1 validation samples and the corresponding prediction of the PW1 model.*

Sample ID	Measured porosity [%]	Std. deviation of measurement [%]	Predicted porosity α_p [%]	Std. deviation of prediction [%]	Error [%]
PW1-TM-val01	7.81	2.82	5.41	0.48	2.40
PW1-TM-val02	3.15	1.37	0.86	0.79	2.29

Both measurements are outside the 95% confidence bounds of the prediction, which means that the model cannot be validated with these results. It is however interesting to note that the error of both samples with respect to the prediction is comparable.

A new 3k prepreg was ordered to replace the one that is used in this experiment. However, this prepreg has an areal weight of $200\,\text{g/m}^2$ whereas the 3k prepreg in PW1 is $245\,\text{g/m}^2$. At first it was thought that this would yield no significant difference, but as will be proven in the next chapter the results from PW1 may no longer apply to this new prepreg. It is an unfortunate turn of events, but the fact that such a small change in areal weight can have a large impact on the surface porosity does introduce new and valuable insight in the origin of surface porosity in the X-core process.

8

Causes and mitigation strategies for surface porosity

In process window study 1 a benchmark sample was prepared with an excellent surface finish but subsequent tests with a different laminate failed to reproduce the result. It is found that the heat-up rate, sample thickness and DU volume content measurably influence the porosity content of the sample, but the consequence of changing the laminate was not considered. Its effect is however significant and requires further investigation to understand the origin of surface porosity in the X-core process.

In Appendix E the timeline during the production of samples is presented. This serves as an overview and is important in the discussion on ageing: At some moment during the study the 3k prepreg was no longer yielding consistent results and needed to be replaced. Ageing complicates the comparison of different measurements, quantifying its effect helps to understand the scale of the problem. The effect of ageing of the prepreg on the surface porosity is covered in Section 8.1. Section 8.2 covers the influence of a variety of barrier layers between the laminate and the core and evaluates their effectiveness. Results in this study and in previous years strongly suggest that reducing/preventing the out-of-plane transport of gas/material between the core and laminate helps to reduce surface porosity. In Section 8.3 the consequence of changing the composition of the laminate is assessed and related to the barrier layer effect. Section 8.4 covers the pigmentation of X-core, which is proposed as an attempt to reduce the visibility of the originally white X-core when it contaminates the surface of the carbon fibre product. Finally, the chapter is concluded in Section 8.5, where the investigated causes and mitigation strategies for surface porosity is summarized.

8.1. Ageing of the prepreg

A sudden increase in surface porosity and inconsistent results for five samples in a row indicated the presence of a problem. The porosity contents of these samples were ~3% higher than the expected value or an earlier produced reference sample. This problem occurred during the tests that are described in this chapter, it is not expected to have affected process window study 1 or previous studies. There were three main indicators that the ageing of the c-m-p 245 g/m^2 3k prepreg is the cause of this problem:

- The expiry date of the roll was actually in the first month of this study (PW1 was carried out in the fifth month). At first this was not considered to be a problem, mainly because it was still possible to manufacture a high quality sample after the expiry date, namely the benchmark sample XC-TM3 (Figure 7.1). It likely that the shelf-life of the prepreg had not been exceeded; according to c-m-p it is 2 weeks at room temperature, the recorded out time in this study is 6 days. The roll with the 3k prepreg is a sample roll, it had only been used once or twice to manufacture samples and is not used in the production cycle at Donkervoort.
- The tack of the 3k prepreg started to feel noticeably different from the tack of the 12k prepreg. The 3k could be handled with nitrile gloves without leaving residue, whereas the same c-m-p CP0093 resin in the 12k would tend to stick to the gloves. The lower tack relative to the fresh 12k prepreg indicates that the polymerization reaction has advanced to a higher degree of cure.

- Samples started to display dry spots, areas where there has been little to no flow of the resin. The type of porosity also changed: It was previously almost exclusively found at intersections between fibre bundles, which was no longer the case. Photographs of the samples that display these phenomena are presented in Figure 8.1.

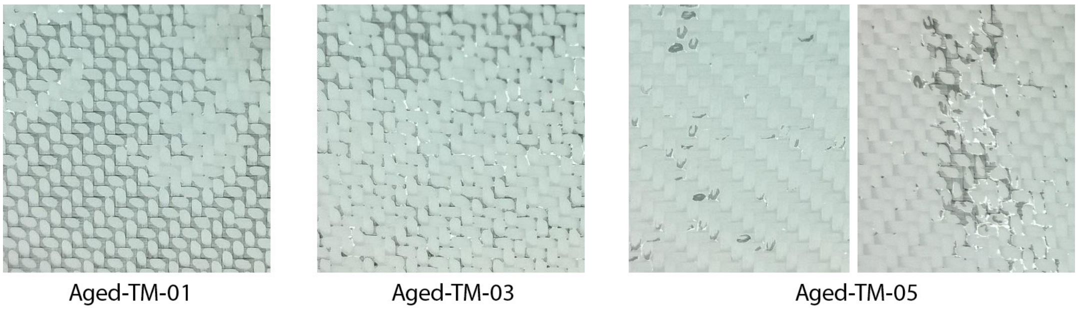

Aged-TM-01 Aged-TM-03 Aged-TM-05

Figure 8.1: *Photographs of areas of the specimens that show symptoms of an overaged prepreg*

The samples that are presented in this section were not part of a specific study into ageing, they are unintended results from other investigations. The samples were just relabelled as Aged-TM samples. The processing conditions of these samples are provided in Table 8.1. In Figure 8.2 the measured porosity of these samples is compared to the prediction of the process window 1 model (Equation 7.12) and, if applicable, a reference sample that was made with non-overaged prepreg.

Table 8.1: *The process variables and porosity contents for the samples that show symptoms of an overaged prepreg in Aged-TM.*

Sample ID	Laminate	T_1' [°C/min]	T_c [°C]	h [mm]	v_{DU} [vol%]	Mean porosity [%]	Standard deviation [%]
Aged-TM-01	3k/12k	1.79	105	10	1.5	10.65	8.20
Aged-TM-02	3k/12k	3.08	105	10	7.5	3.83	0.80
Aged-TM-03	3k/12k	1.46	110	25	4.5	7.81	2.82
Aged-TM-04	3k/12k	0.98	90	10	7.5	3.15	1.37
Aged-TM-05	3k/3k	0.91	120	10	7.5	0.87	0.56

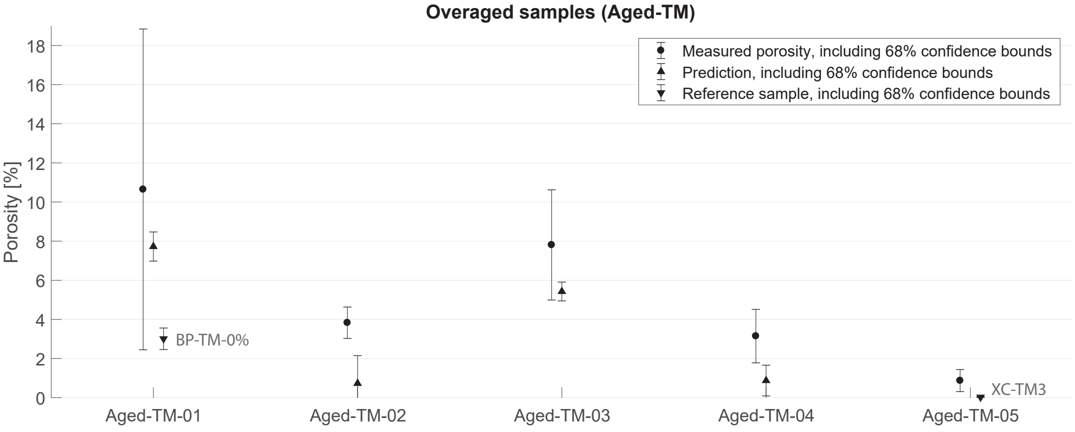


Figure 8.2: *Porosity contents and corresponding predictions of the specimens that were believed to be subject to ageing*

Sample Aged-TM-01 is compared to BP-TM-0%, which is a sample from the pigmentation tests (Section 8.4). It is subjected to the same cure cycle, has the same thickness and has an equal DU volume content. Samples Aged-TM-02 and Aged-TM-03 are validation samples for process window 1. There is no prediction for sample Aged-TM-05 because it is manufactured with a 3k/3k laminate, the conditions are however comparable to sample XC-TM3. Aged-TM-05 scores relatively well because the dry spot (Figure 8.1) is not captured within one of the porosity measurement areas.

The porosity content of BP-TM-0% is 61% lower than the prediction, this is the reason why an attempt was made at reproducing the result. It will be shown in Section 8.4 that it is not likely that this is an outlier. The difference is attributed to the fact that the mould is stiffened after process window study 1. The stiffened mould allows for less expansion and therefore the pressure may have been higher, which can explain the decrease in porosity: A higher DU content gives a higher pressure and from PW1 it was found that a higher DU content leads to a decrease in porosity. Samples 01, 04 and 05 are made in the stiffened mould, therefore their higher porosity than the predicted value is even more worrying.

8.1.1. Introduction of a new prepreg

In response to the ageing problem a new 3k prepreg was ordered, but it is not exactly the same as the one that was previously used. c-m-p did not have the 245 g/m^2 3k with CP0093 resin in stock, instead a 200 g/m^2 3k prepreg with the same resin was ordered. At the time it was not believed that it would yield a significant difference. However, at this stage it is clear that the lower density of the weave yields higher surface porosities and replicating the benchmark sample is no longer possible. The timeline in Appendix E shows at what stage of the research the switch of prepregs took place.

The prepregs differ in areal weight, but the bundle size is still the same (3k). This results in a different tightness of the weave, which is clearly visible from the photograph in Figure 8.3. The increased surface porosity may be explained by the higher permeability of the weave, based on the results of the barrier layer tests that are discussed in Section 8.2. According to c-m-p the weave also affects the impregnation of the prepreg, even though both prepregs are made through hot-melt impregnation [48]. Uncured pieces of both prepregs are photographed in the optical inspection set-up, the images are shown in Figure 8.4.

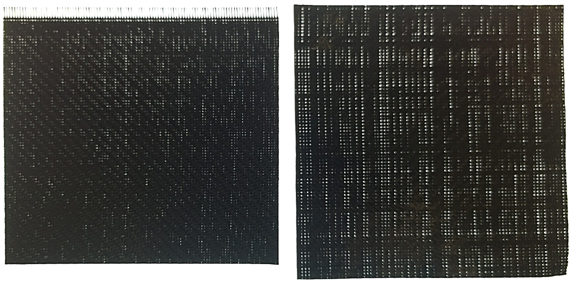


Figure 8.3: *Comparison of the tightness of the weave of the 245 g/m^2 3k weave (left) and the 200 g/m^2 3k weave (right).*

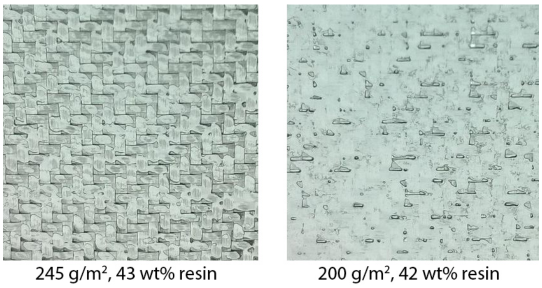


Figure 8.4: *Comparison of the impregnation of the 245 g/m^2 3k weave (left) and the 200 g/m^2 3k weave (right).*

Two new samples are made to compare to the benchmark sample. One sample has a 3k/3k laminate with the 245 g/m^2 prepreg that is overaged. The second sample has a 3k/3k laminate with the fresh 200 g/m^2 prepreg. These samples are labelled XC-TM5 and XC-TM6 respectively, their porosity contents and relevant process variables are provided in Table 8.2. Representative photographs of the samples that were used in the porosity measurement are presented in Figure 8.5.

Table 8.2: *Process variables and porosity contents of the X-core cycle reference test samples (XC-TM)*

Sample ID	T_1' [°C/min]	T_c [°C]	h [mm]	v_{DU} [vol%]	Mean porosity [%]	Standard dev. [%]	Description
XC-TM3	1.43	110	10	7.5	0.025	0.020	3k/3k 245/245 g/m^2 laminate
XC-TM4	1.42	110	10	7.5	1.61	0.51	3k/12k 245/420 g/m^2 laminate
XC-TM5	1.41	110	10	7.5	1.24	0.61	3k/3k 245/245 g/m^2 overaged
XC-TM6	1.41	110	10	7.5	3.28	1.44	3k/3k 200/200 g/m^2 laminate

The porosity in sample XC-TM5 also provides a strong indication that the 3k prepreg is overaged. The expired prepreg may be identified by localized areas that display poor wetting/resin flow and the porosity is not dispersed homogeneously over the surface. What is especially concerning is the high porosity content in sample XC-TM6. This result indicates that changing the weave of the prepreg can have a significant impact on the surface porosity. There can be other process variations at play, but repetitions with thicker laminates using the same prepreg did not provide any indication that a porosity content of 0.025% is achievable with this 200 g/m^2 weave at these conditions; see Section 8.3 for the results. There still is however one variable that could have a significant effect on surface porosity: the out-time.

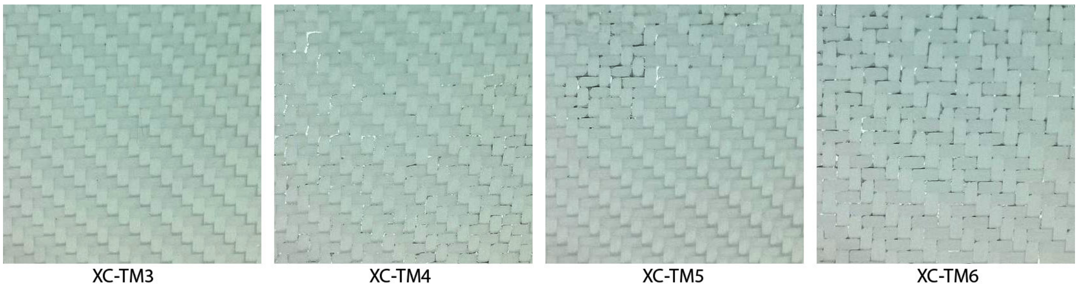


Figure 8.5: *Photographs of areas of XC-TM samples that are representative for the mean porosity content of the sample.*

8.1.2. Effect of out-time on surface porosity

A study by L. Hamill et al. [14] showed that the surface porosity decreased by 83% after just four days of out time and by as much as 99% after 14 days in VBO processing with a VBO prepreg. This was attributed to the decrease in tack which helps to increase the permeability of the ply-tool interface and provides a better path for the air to evacuate. The 200 g/m^2 prepreg is fresh and its out-time is less than 24 hours whereas the 245 g/m^2 prepreg was almost expired when it was used to manufacture sample XC-TM3. So, the out-time might play a significant role and is therefore investigated.

A test was performed whereby prepreg for two samples was removed from the freezer. After 7 days at room temperature the first sample was manufactured, the second sample is manufactured after 14 days. The reference sample is XC-TM6, which has a one day out-time. The results of this test are provided in Table 8.3.

Table 8.3: *Results of the out-time test*

Out-time	Mean porosity [%]	Standard deviation [%]
1 day	3.28	1.44
7 days	5.13	1.18
14 days	3.10	0.93

The results indicate that there is especially no strong decrease of surface porosity with increasing out-time. The behaviour is different from VBO processing, it is expected that the room temperature de-bulk in VBO is more effective when the tack is low at room temperature. In contrast, X-core delivers pressure above 80°C, where the viscosity of the prepreg is much lower and the permeability of the ply-tool interface is different compared to the room temperature situation. So, the discrepancy in results between samples XC-TM3 and XC-TM6 cannot be explained by the difference in age of the prepreg. Another theory is that the different weave style results in a larger out-of-plane permeability, how this affects surface porosity is the topic of the next section.

8.2. The effect of barrier layers

The barrier layer has been a returning topic in previous research and its concept is important to understand the origin of surface porosity in X-core products. In previous years the barrier layer was a non-permeable PET plastic film that was placed between the core and the facings. F. Minde [28] was the first to hypothesize the problem of degassing of X-core as the DU expands; the entrapped air in X-core is pushed out and finds its way into the prepreg. This hypothesis was however not tested, so it is revisited in Section 8.2.1. In this section a dry glass reinforcement and vacuum film are also proposed as an alternative barrier layer. The glass fibre is proposed because it does not seem necessary to introduce a PET film since the benchmark sample XC-TM3 has a very low porosity content and achieves that without the need for a PET film barrier layer. The vacuum film is used to identify whether the process is also viable if X-core is isolated.

8.2.1. PET film and its working principle

PolyEthylene Terephthalate (PET) is a thermoplastic with a glass transition around 75°C. In previous research PET film was effectively used as a barrier layer between X-core and the laminate to reduce the surface porosity. This material was chosen because it softens above the expansion temperature of X-core and it was believed that it would weld to the thermoplastic microspheres in X-core. The use of a barrier layer and further research into its effect was recommended by both F. Minde and S. Vial [28, 29].

In an exploratory study a passenger side floor panel is manufactured with a variety of prepregs and lay-ups. It is manufactured with the Floor panel 330 mix (see Table 4.6) and the standard cure cycle for X-core production (Figure 4.5). The panel is sectioned in three zones that contain different surface plies: zones A, B and C. These are, in turn, sectioned into four zones with a variation of lay-ups. The following four prepregs are used in this experiment:

- **3k visual:** 245 g/m^2, 2x2 twill weave, 3k carbon fibre, Cytec semi-preg with VTF261 resin, it has a glass fibre surface ply.
- **3k visual flipped:** same as the "3k visual", but placed inside out; the glass fibre surface ply faces away from the tooling surface.
- **12k visual:** 420 g/m^2, 2x2 twill weave, 12k carbon fibre, DeltaTech prepreg with DT806R resin.
- **12k:** 420 g/m^2, 2x2 twill weave, 12k carbon fibre, c-m-p prepreg with CP0093 resin.

These prepregs were covered in Section 6.2.1, the properties of the resins can be found in the same section in Table 6.1. The definition of the twelve zones of the floor panel is provided in Table 8.4. PET stands for a 0.04 mm PET film, the first ply in every lay-up is the surface ply, the last ply has an interface with the core. A photograph of the cured product after sectioning is shown in Figure 8.6.

Table 8.4: *Definition of the zones in the floor panel of test SP-FP1*

	1	**2**	**3**	**4**
A	3k vis	3k vis/PET	3k vis/12k/PET	3k vis/12k
B	3k vis flipped	3k vis flipped/PET	3k vis flipped/12k/PET	3k vis flipped/12k
C	12k vis	12k vis/PET	12k vis/12k/PET	12k vis/12k

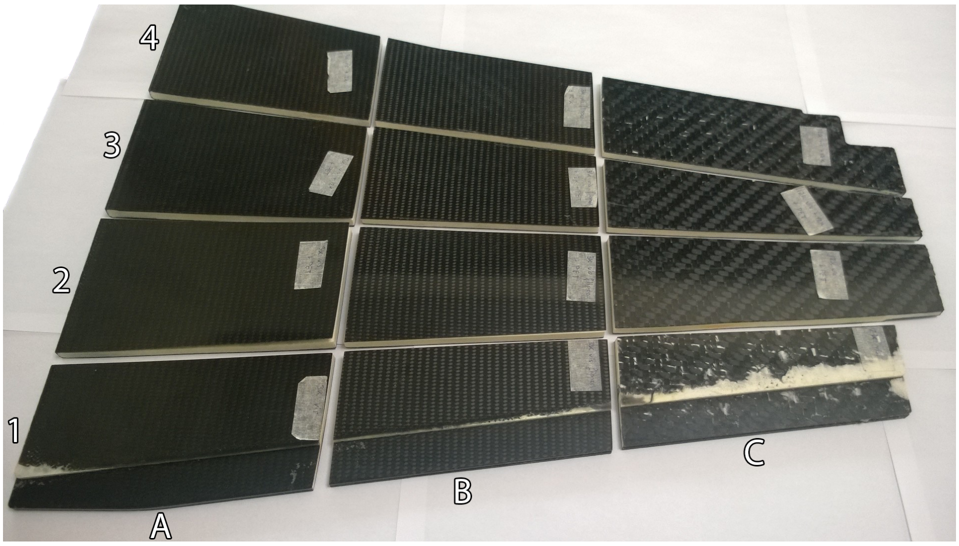


Figure 8.6: *The sectioned floor panel with varying lay-ups over its surface*

The samples are compared qualitatively by visual inspection with the naked eye. Upon inspection the surface porosity content of zones A and B is comparable. Within zones A and B it shows that the laminates containing a 12k ply have more porosity and that the PET film does not necessarily lead to a reduction of surface porosity. The use of PET film does however make a significant difference in zone C, where the 12k visual is used. The 12k visual is not as tightly woven as the 3k visual and as a result the X-core is able to find its way to the surface, even when two layers of 12k are placed (section C4). In this case the use of PET also results in a visible reduction of porosity.

There is however a major drawback associated with the use of PET film. All sections containing PET readily delaminated when the floor panel was sectioned using a FEIN oscillating tool. Figure 8.7 shows section A2, there is no evidence of a mechanical bond between the PET film and X-core. So, under these conditions there is no welding of the PET film to the microspheres in X-core and there is insufficient adhesion of the epoxy to the PET film.

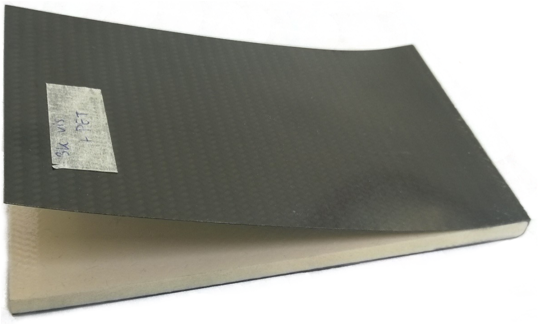


Figure 8.7: *Delamination of section A2 of test SP-FP1.*

In a follow-up test the working principle of PET film is investigated; PET cannot be used because it causes delaminations, understanding its working principle can help to find a more suitable replacement. Two hypotheses were formulated for the working principle of the barrier layer:

- **hypothesis 1:** PET film prevents the resin of the prepreg from being absorbed by X-core
- **hypothesis 2:** PET film prevents entrapped air in X-core from venting into the laminate as it is pressurized by the expanding DU.

Four samples are manufactured in the floor panel sample mould, see Appendix B.3 for a description of the mould. The test is designated as test SP-FP2, the samples have the following stacking sequence:

1. Cytec 3k visual / X-core / Cytec 3k visual
2. Cytec 3k visual / glue film / X-core / glue film / Cytec 3k visual
3. Cytec 3k visual / glue film / X-core / 1 mm Airex C71-55 / glue film / Cytec 3k visual
4. Cytec 3k visual / X-core / 1 mm Airex C71-55 / Cytec 3k visual

The glue film is a 263 g/m^2 film of Cytec VTA$^{®}$260 epoxy resin on a 13 g/m^2 knit polyester carrier, it is used for bonding core materials (foams and honeycombs) to prepreg laminates. So, if hypothesis 1 is true the glue film should help to reduce the surface porosity. The 1 mm layer of Airex C71-55 foam acts as a non-permeable barrier that can absorb some resin. In this experiment the low pressure door mix X-core (Table 4.6) is used and the samples are cured using the standard production cycle for floor panels (Figure 4.5). A photo of the cured samples is shown in Figure 8.8.

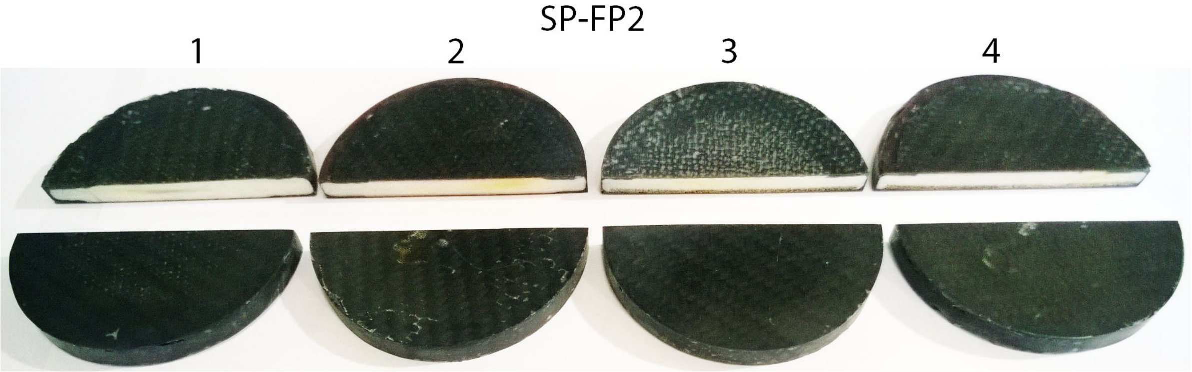


Figure 8.8: *The samples of test SP-FP2, the top halves in the photo show the top facings of the samples.*

More detailed photographs of the samples in test SP-FP2 can be found in Appendix D.3. Sample 2 shows a clear improvement over sample 1 but there is still porosity present. In addition the porosity changed from small pinholes in the fabric to large interconnected pores. In the bottom facings of samples 3 and 4 however, where the Airex foam is present, nearly all of the surface porosity has been mitigated. Even more interesting is the dramatic increase of porosity in the top facing of sample 3. In line with the second hypothesis this seems to indicate that the air entrapped in the core is forced out as it is being pressurized and the only way out is through the prepreg on the opposing side of the specimen. The same is true for sample 4 but the difference is less pronounced. It is postulated that the glue film may act as a barrier as well, which could also explain the difference between samples 1 and 2.

It may be true that both resin absorption and degassing are a major cause for the surface porosity in X-core samples. At the moment however it seems that the latter has a more pronounced effect. This experiment is also carried out with just a single ply at the facing, it is expected that the effect of absorption is less pronounced for thicker laminates. Therefore the focus is moved towards the effect of having a barrier that reduces the flow of air between the core and facings, which is covered in the remainder of this section.

8.2.2. Dry glass fibre reinforcement

Using a plastic film such as PET can help to reduce surface porosity, but it also yields a poor adhesion between the core and facings. Instead of a plastic film it is therefore suggested to use a low areal weight and tightly woven glass fibre fabric as boundary layer. The reasoning is that two layers of the tightly woven 245 g/m^2 carbon fibre prepreg can also yield high quality surfaces under the right conditions, as was demonstrated by sample XC-TM3 (Section 7.1). It is argued that it may not be necessary to completely obstruct the out-of-plane airflow with an impermeable layer, a layer with reduced permeability may already yield the desired result. A layer of dry glass fabric is chosen because:

- It is relatively cheap compared to carbon fibre and is available in a wide variety of weave densities.
- It has good adhesion to epoxy and allows for mechanical interlocking at the core/laminate interface.
- fabrics, unlike plastic films, can conform to double curved shapes without needing to be cut or folded

The weave that was opted for is a 160 g/m^2 twill weave, it has a density of 12 ends per centimetre. A photograph of the weave, adhered to the facing inside the tile mould is shown in Figure 8.9. In comparison, the 245 g/m^2 carbon fibre 3k prepreg has 6 bundles per centimetre and the 200 g/m^2 prepreg has 5 bundles per centimetre.

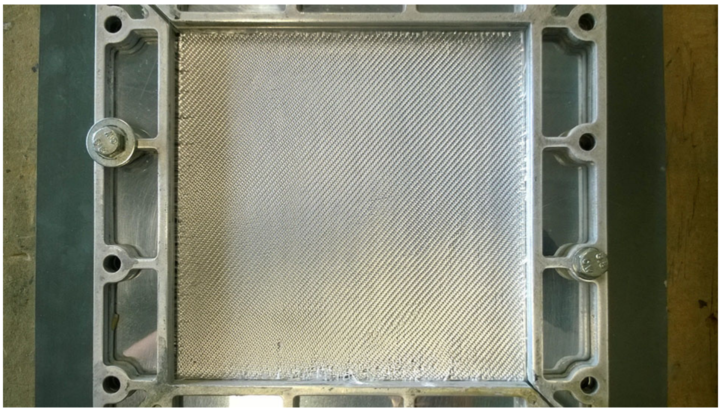


Figure 8.9: *The 160 g/m^2 dry glass fibre barrier layer.*

Porosity measurement

Samples were prepared in the tile sample mould in order to quantify the impact of the dry fibre boundary layer on the surface porosity content. Two laminates are tested and for each laminate a reference sample and a sample containing the glass fibre barrier layers is manufactured. The laminate of sample DF-TM-01 is a 3k/12k laminate with a 245 g/m^2 3k and 420 g/m^2 12k carbon fibre fabric. Sample DF-TM-02 has a 3k/3k laminate with a 200 g/m^2 carbon fibre fabric (see Appendix D.1 for an explanation of sample labels). The processing conditions of the dry fibre barrier layer tests and the corresponding porosity contents are provided in Table 8.5. The mean porosity contents of both facings and the mean porosity content of the entire sample is shown graphically in Figure 8.10.

Table 8.5: *Process variables and porosity contents of the dry fibre barrier layer test samples (DF-TM)*

Sample ID	T_1' [°C/min]	T_c [°C]	h [mm]	v_{DU} [vol%]	Mean porosity [%]	Standard deviation [%]
DF-TM-01-ref	2.72	105	10	1.5	3.46	1.53
DF-TM-01	2.81	105	10	1.5	1.34	0.83
DF-TM-02-ref	1.40	110	10	7.5	3.28	1.44
DF-TM-02	1.44	110	10	7.5	1.35	1.05

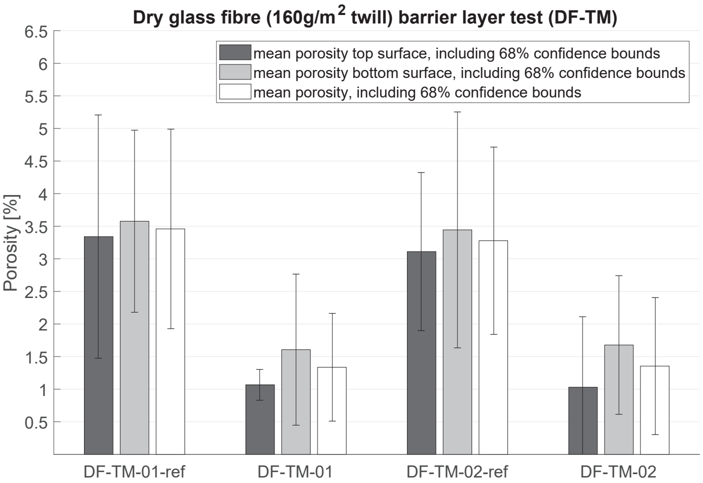


Figure 8.10: *Chart of porosity contents of the dry glass fibre barrier layer test samples (DF-TM)*

The addition of the glass fibre barrier layer yields a 61% reduction in surface porosity in sample 01 and a 56% reduction in sample 02. It is also striking that the laminates and the processing conditions are very different in both test runs, but the reference porosity content and the reduction in porosity are almost equal. It is clear that the use of a glass fibre barrier layer helps to reduce the surface porosity, but it is not as effective as the 245 g/m^2 3k/3k laminate. The benchmark sample for high quality surfaces (sample XC-TM3) had a porosity content of 0.025% under the same conditions as sample DF-TM-02. This difference may be attributed to the fact that introducing dry fibres introduces entrapped air between the core and facings or that a dry fabric is not as effective as an impregnated fabric at forming a barrier. These results do however still support the hypothesis that a cause of surface porosity in X-core products is venting of entrapped air out of the core into the laminate: The 160 g/m^2 glass fabric between the core and facings does not prevent absorption of the epoxy in the prepregs, but it reduces surface porosity by more than 50% in the tested samples.

Sample DF-TM-01-ref has a 3k/12k laminate just like all samples in process window 1, which means that its porosity may be compared to the prediction using Equation 7.12:

$$\alpha_p = -0.1631 + 8.7167 \cdot (2.72) + 0.0389 \cdot (10) - 0.7734 \cdot (1.5) - 2.1657 \cdot (2.72)^2 = 6.75\% \quad (\sigma = 0.88\%) \tag{8.1}$$

The measured porosity is 49% lower than the prediction, which cannot be explained with the variation in the model. This is attributed to the repair of the tile mould, it has been stiffened which means that the expansion of X-core is more restricted and this is expected to result in a higher compaction pressure on the laminate for the same DU content. The difference is comparable to the difference between BP-TM-0% and the corresponding prediction (see the prediction for sample Aged-TM-01 in Figure 8.2), in this case the measurement is 61% lower than the prediction.

Adhesive bond strength

It has been shown that introducing dry fibres between the core and the laminate may help to reduce surface porosity, but it is also likely to affect the adhesive bond strength between the core and facing. Mechanical tests are conducted to establish whether the flexural strength is affected by the addition of a dry 160 g/m^2 glass fabric. Five flexure specimens are extracted from samples DF-TM-01-ref and DF-TM-01, they are cut to be 25×175 mm. The test procedure and set-up are explained in detail in Section 5.2.2.

All samples from both panels failed in core shear. This indicates that cohesive failure of the core precedes adhesive failure of the core-to-facing bond, irrespective of the use of the glass fibre barrier layer. Photographs of the failed specimens and the corresponding load-deflection curves are provided in Appendix D.4. The calculated shear strength of both sandwich panels, including 95% confidence bounds, is presented in the bar chart in Figure 8.11.

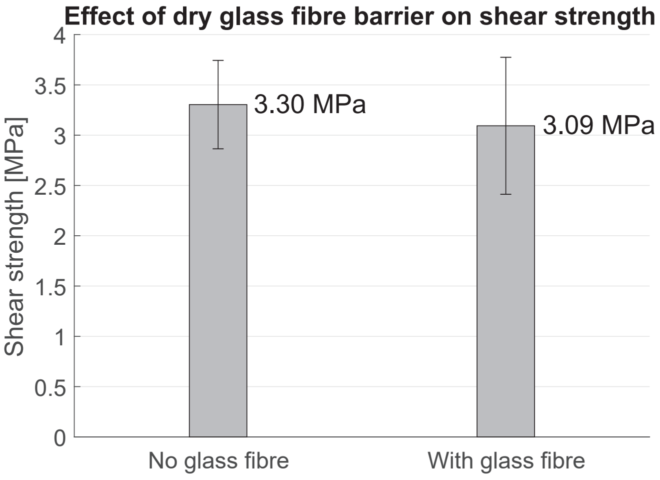


Figure 8.11: *Shear strength of X-core, with and without a dry fibre barrier layer. The chart includes 95% confidence bounds of the measurement.*

The test indicates that the shear strength of the sample with a 160 g/m^2 glass fabric barrier layer is on average 6% lower. Based on the measurements the B-basis value of the shear strength is 2.86 MPa and 2.41 MPa for the reference sample and the glass fibre barrier layer sample respectively. This is the value that should be used for structural design and it indicates that the addition of glass fibre yields a 16% reduction in strength.

It is expected that the reduction in shear strength results from resin absorption by the unimpregnated glass fibres. It has been proven by S.Vial [29] that the epoxy in X-core gives the main contribution to the compressive strength of the foam, it is expected that it has the same effect on the shear strength. The cured X-core has a lower volume fraction of epoxy if some of the resin is absorbed, as a result it is expected to have a lower shear strength.

The results indicate that it helps to reduce surface porosity, but that is at the cost of a shear strength reduction. This may however be circumvented by increasing the volume fraction of resin in the uncured X-core or by adding a resin rich layer between the core and barrier layer (e.g. a glue film). The dry fibres are impregnated with the excess resin and become part of the facing of the sandwich. In addition, when carbon fibre is used to replace glass fibre the barrier layer is no longer 'added mass', instead it contributes to the load carrying capability of the facings. This can allow for the replacement of a layer of prepreg by comparatively cheap dry fibres. It is however recommended that this suggested approach should be investigated further to assess its feasibility.

8.2.3. Vacuum film

Applying a vacuum film as a barrier layer is principally the same as using PET film, but it is used because this film is more representative of autoclave processing. The idea is to completely isolate X-core from the laminate and subsequently to assess whether the laminate is processable at all within the set processing limits for ramp-up rate, temperature and DU content (pressure). In this set-up X-core provides compaction pressure on the laminate, no vacuum is applied but the film between the core and laminate allows the air to vent into the atmosphere. The vacuum film that is used in all samples is the Scabro Vacfilm® 450V, it is suitable for high pressure autoclave curing up to 170°C.

In this test the same reference samples are used as in the dry fibre barrier layer test DF-TM. So, DF-TM-01-ref and DF-TM-02-ref are the same as VB-TM-01-ref and VB-TM-02-ref respectively. This is done such that the effectiveness of the vacuum film can be directly compared to the dry fibre barrier layer. This means that sample 01 has the 3k/12k laminate with the 245 g/m^2 3k and 420 g/m^2 12k fabric and sample 02 has the 3k/3k laminate with the 200 g/m^2 3k fabric. The vacuum film is only placed on the bottom facing of the sample. Two samples were made to compare to the second reference in order to have eight measurement areas to determine the mean porosity content. This is necessary for process window study 2, which is covered in Chapter 9. The process conditions and the porosity contents of the samples are given in Table 8.6. A bar chart showing the porosity contents with 68% confidence bounds is presented in Figure 8.12.

Table 8.6: *Process variables and porosity contents of the vacuum film barrier layer tests (VB-TM)*

Sample ID	T_1' [°C/min]	T_c [°C]	h [mm]	v_{DU} [vol%]	Top surface porosity [%]	Standard dev. [%]	Bottom surface porosity [%]	Standard dev. [%]
VB-TM-01-ref	2.72	105	10	1.5	3.34	1.87	3.58	1.40
VB-TM-01	2.81	105	10	1.5	4.95	1.48	0.67	0.088
VB-TM-02-ref	1.40	110	10	7.5	3.11	1.21	3.45	1.81
VB-TM-02A	1.44	110	10	7.5	3.05	0.36	0.040	0.013
VB-TM-02B	1.45	110	10	7.5	4.67	0.53	0.16	0.080

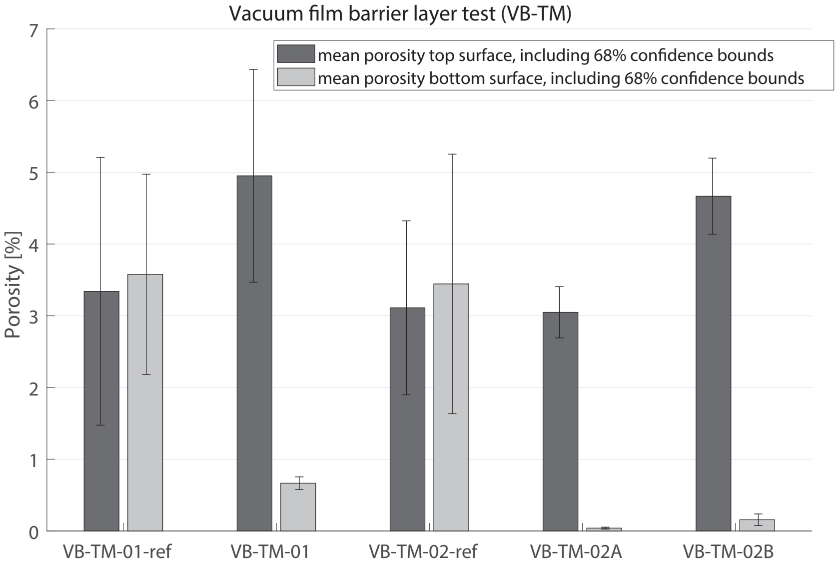


Figure 8.12: *Chart of porosity contents of the vacuum film barrier layer test samples (VB-TM)*

The reduction in porosity is much larger compared to the dry fibre barrier layer, it is 81% for sample 01 and 97% for sample 02. These results show that the process is viable for the new 245 g/m^2 prepreg as well, provided that there is an effective barrier layer. The results also show that the porosity in the top surface increases by 43% in sample 01 and on average by 18% in sample 02 when a vacuum film is placed at the bottom surface. This phenomenon was also observed in test SP-FP2 (Section 8.2.1). It is postulated that this is a result of blocking the path for air evacuation on one side of the core, resulting in increased air evacuation on the opposing side.

In sample VB-TM-02B the vacuum film is placed such that it does not cover the entire sample, see Figure 8.13. The areas that are photographed for porosity measurement are within this area, only the outer 15 mm of the laminate is left uncovered. Figure 8.14 shows a close-up of the edge of the cured sample. The photograph shows how local the effect of the barrier layer can be and how effective it is in reducing surface porosity.

Low density X-core

The barrier layer effectively isolates X-core from the laminate, therefore it was hypothesized that the amount of entrapped air in X-core would no longer be a factor when a barrier layer is used. This is beneficial, because achieving lower density cores requires lower volume fractions of binder material. Less binder material results in more interstitial voids; there is not enough material to fill all the gaps between the microspheres. A 110 g/L foam was developed in a concurrent research that focusses on using the X-core process for the manufacture of the lower door piece of the Donkervoort D8 GTO RS. In this door piece the weight is a primary requirement, the core is needed to provide some stability to the facings and to enable the one-shot process. The volume fractions of this mixture are provided in Table 8.7.

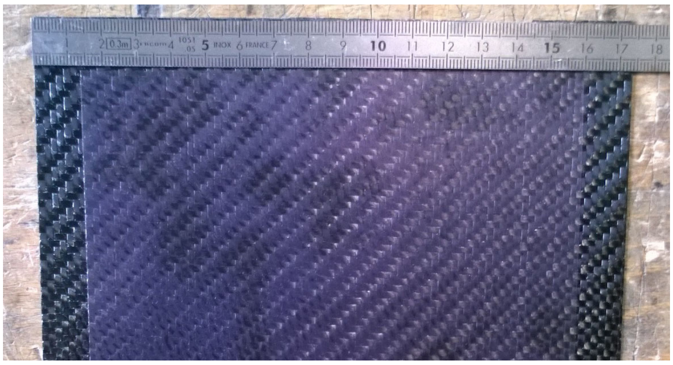


Figure 8.13: *Placement of the vacuum film barrier in sample VB-TM-02B: a gap of 15mm is left all around the sample*

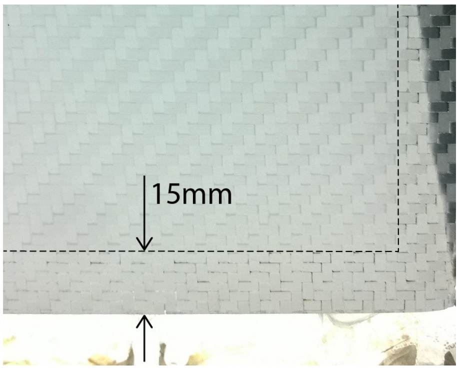


Figure 8.14: *The edge of sample VB-TM-02B containing a large amount of porosity.*

Table 8.7: *Volume percentages of the constituents of the 110 g/L mix.*

	Resin	Q-cel	DE
110 g/L mix	4.8%	0%	95.2%

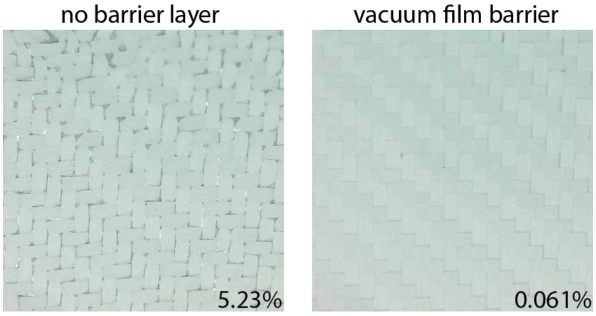


Figure 8.15: *The effect of having an impermeable barrier layer on sample containing a 110 g/L X-core mixture*

Two samples are prepared, both are 10 mm thick and are manufactured with the same cure cycle and laminate as sample VB-TM-02. The sample without barrier layer contains 7.5 vol% DU, the one with the vacuum film barrier 6.75%. Photographs of representative areas of the samples are shown in Figure 8.15.

The mean porosity content of the sample without the barrier layer is 5.25%, the use of the vacuum film helps to reduce the surface porosity to 0.10%, even with a lower DU volume content. In addition, the reference sample porosity is 2% higher than the reference sample with the 270 mix (sample VB-TM-02-ref). This can be expected since the 110 g/L mixture should contain more entrapped air. The result of the vacuum film barrier sample is comparable to the porosity contents of samples VB-TM-02A and VB-TM-02B. This shows that the use of an impermeable barrier layer can allow for using a core material with a much lower density. There was however a problem with the use of the vacuum film: blisters were formed in the laminate by large air pockets in the interface between X-core and the vacuum film. The air is not being dispersed and does not find its way out of the product. So, the use of a vacuum film barrier layer can eliminate almost all surface porosity, but it also tends to entrap air and it leads to a poor core-to-facing adhesion.

8.3. Laminate stacking sequence

Up to this point three variations of laminates have been tested: the 3k/3k laminate with a 245 g/m^2 carbon fibre weave, the 3k/12k laminate with a 245 g/m^2 and 420 g/m^2 weave and finally the replacement 3k/3k laminate with the 200 g/m^2 fabric. The results indicate that the 245 g/m^2 3k/3k yields a lower porosity content than the other two laminates, which is attributed to the lower out-of-plane permeability of that particular weave. If reducing the permeability helps to lower the surface porosity content it may help to add more plies to the laminate. In addition, a similar effect is also expected when the plies are rotated with respect to each other. In order to test these hypotheses two samples were prepared with the new 200 g/m^2 3k prepreg and compared to the 3k/3k reference (sample XC-TM6), their lay-ups are as follows:

- **reference:** $[(0/90)_2/\text{X-core}/(0/90)_2]$
- **laminate 1:** $[(0/90)_4/\text{X-core}/(0/90)_4]$
- **laminate 2:** $[(\pm45)/(0/90)/(\pm45)/(0/90)/\text{X-core}/(0/90)/(\pm45)/(0/90)/(\pm45)]$

The process variables and the porosity contents of the samples are provided in Table 8.8, the porosity content is also shown in the bar chart in Figure 8.16.

Table 8.8: *Process variables and porosity contents of the stacking sequence variation test.*

Sample ID	T_1' [°C/min]	T_c [°C]	h [mm]	v_{DU} [vol%]	Mean porosity [%]	Standard deviation [%]
Reference (XC-TM6)	1.41	110	10	7.5	3.28	1.44
Laminate 1	1.44	110	10	7.5	2.86	0.96
Laminate 2	1.45	110	10	7.5	2.31	0.65

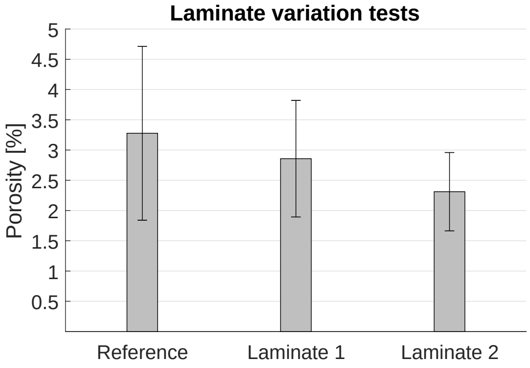


Figure 8.16: *The porosity content including 68% confidence bounds for different laminates*

Increasing the thickness of the lay-up (laminate 1) shows a 13% reduction in porosity. Alternatively, increasing the thickness and alternating the ply orientation between (0/90) and (±45) yields a 30% reduction in porosity. So, increasing the number of plies and changing the ply orientation can help to reduce surface porosity. However, the reduction is relatively small compared to the use of a 160 g/m^2 dry glass fabric (56%) or a vacuum film barrier (97%).

8.4. Pigmentation of X-core

Pigmentation of X-core is not a strategy to reduce surface porosity, it is a means of masking manufacturing flaws. X-core is white, which is clearly visible on the black carbon fibre (e.g. see Figure 8.6). Therefore, if X-core would be dark grey/black (colour of carbon fibre) the product looks aesthetically less flawed when X-core penetrates the weave or when the worker accidentally drops X-core between the tooling surface and the laminate. The colour of X-core is a result of the combination of the colours of the constituents. The 270 mix with 0 vol% DU contains 14.9 vol% resin, the remainder of the volume is Q-cel and DE, which are both white. The Ancarez 4010 resin in the epoxy is transparent, the Ancamide 3399 hardener is dark yellow and as a result the true colour of X-core is an off-white that tends to cream.

It is proposed to pigment X-core by making use of a black epoxy pigment. The main advantage is that the pigment can be added to replace a part of the base resin, the Ancarez 4010. So, there is no addition of mass and the density of X-core remains unaffected. The pigment is chemically reactive and partakes in the cross-linking reaction. To this end Poly-Pox pigment paste is used, the supplier recommends a dosage of up to 10 wt% in epoxies. With this dosage the impact on the mechanical properties of the resin should be minimal.

Three X-core mixtures with a varying pigment content were formulated for this test, all based on the 270 mix with 1.5 vol% DU. One sample serves as the reference and does not contain any pigment. In the other two samples 5 wt% and 10 wt% of the Ancarez 4010 is replaced by black pigment, the samples are labelled as follows (see Appendix D.1 for an explanation of sample labels):

- **BP-TM-0%** 0 wt% black pigment in the epoxy
- **BP-TM-5%** 5 wt% black pigment in the epoxy
- **BP-TM-10%** 10 wt% black pigment in the epoxy

The density of the pigment was measured to be 1730 g/L, so this pigment content corresponds to just 0.3 vol% and 0.6 vol% in X-core respectively. In the remainder of this section it is shown what the impact of such a small addition of pigment is on the appearance of X-core, the surface porosity and the mechanical properties of the core.

8.4.1. Visual inspection

In terms of visual inspection both the porosity content and the colour of X-core are considered. During preparation of the X-core of both samples BP-TM-5% and BP-TM-10% the mixture of resin, DU and pigment appears completely black, see Figure 8.17a. However, after adding Q-cel and DE, which amounts to 83.8 vol%, the colour changes to dark grey, see Figure 8.17b. In Figure 8.17c it can be seen that sample BP-TM-10% is slightly darker than sample BP-TM-5%. So, increasing the pigment content helps to make X-core darker, but realistically the 270 mix X-core can never be completely black since there is always less than 14.7 vol% resin.

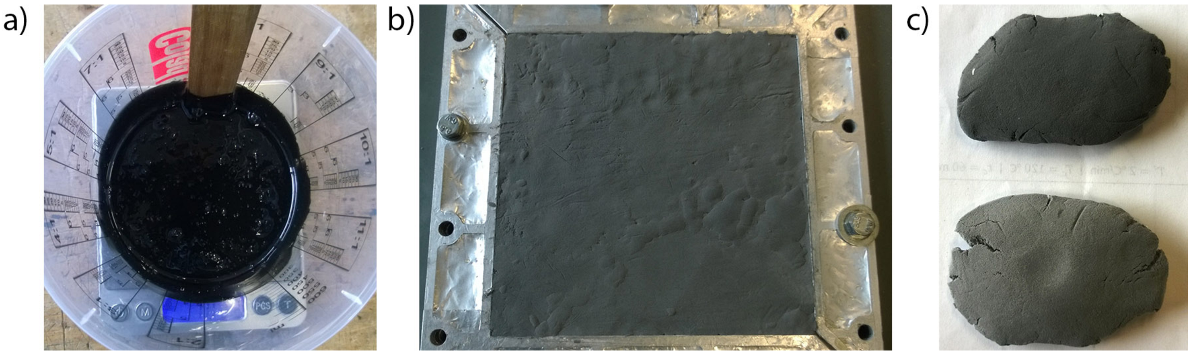


Figure 8.17: *a) Ancarez 4010/Ancamide 3399 including DU and 5 wt% black pigment. b) The moulded X-core with 10 wt% black pigment. c) Comparison of the shades of grey of cured leftovers of samples BP-TM-5% (bottom) and BP-TM-10% (top).*

The samples are subjected to the same processing conditions as sample PW1-TM-09. This was decided because out of all previously manufactured samples this sample displayed the most penetration of X-core through the weave. It was assumed that this might have been a result of the particular combination of processing conditions. The process conditions and the resulting porosity contents of all three samples are provided in Table 8.9

Table 8.9: *Process variables and porosity contents of the black pigment test samples (BP-TM)*

Sample ID	T_1' [°C/min]	T_c [°C]	h [mm]	v_{DU} [vol%]	Mean porosity [%]	Standard deviation [%]
BP-TM-0%	1.78	105	10	1.5	3.01	0.55
BP-TM-5%	1.79	105	10	1.5	2.23	0.87
BP-TM-10%	1.79	105	10	1.5	1.83	0.65

The result indicate that the surface porosities of the 0%, 5% and 10% pigment samples are comparable. There seems to be a trend in the results that surface porosity decreases with increasing pigment content. This can however be coincidental considering the standard deviation (0.55-0.87%) of the samples and the absolute difference in porosity (1.18%). In addition, the reference sample was not manufactured at the same time as the pigmented samples, it was postponed and may have been subjected to ageing of the prepreg. A second sample was manufactured in order to determine whether it was an outlier, but it displayed a surface porosity content of 10.56% (see Figure 8.2, sample Aged-TM-01). In all three samples there was also a comparatively small amount of X-core penetrating through te weave, so this phenomenon is not necessarily related to the DU content, ramp-up rate and sample thickness.

Finally, it should be noted that the porosity content falls outside of the bounds of the prediction of the process window 1 model, which indicates that a porosity content of 7.73% should be expected. There are three repetitions of this sample, all indicating a porosity content in the order of 2-3%. The only intended process variation between these samples and the samples in PW1 is the stiffness of the mould lids. After PW1 the mould was repaired by replacing the MDF core by an aluminium honeycomb, see Section 7.4.2. The expansion of the DU is more restricted for the BP-TM samples and can therefore result in more pressure on the laminate, which can explain the difference in porosity.

8.4.2. Three-point Flexure tests

Three-point flexure tests are performed in order to determine whether the addition of pigment affects the shear strength of X-core. This was expected since the epoxy in X-core is the binder material and it delivers the main contribution to the mechanical properties of the core. Five 25x175 mm samples are extracted out of each sandwich panel and subjected to a three-point flexure test, as described in Section 5.2.2. Photographs of the failed specimens and the corresponding load-deflection curves are provided in Appendix D.4. The calculated shear strength, including 95% confidence bounds, is presented in the bar chart in Figure 8.18.

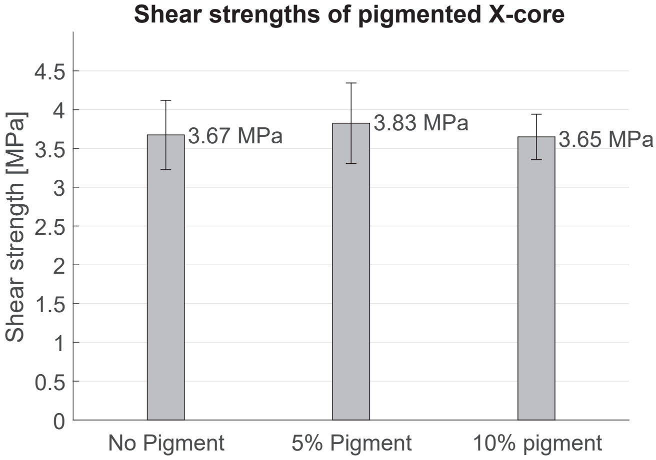


Figure 8.18: *The effect of black pigment on the shear strength of X-core. The chart includes 95% confidence bounds of the measurement.*

The B-basis shear strength values are 3.22 MPa, 3.31 MPa and 3.36 MPa for the 0%, 5% and 10% black pigment samples respectively. This difference is not considered to be significant and it is concluded that the addition of up to 10 wt% black pigment in the epoxy of X-core does not impair the shear strength of the foam or lead to an increase in surface porosity in composite sandwich panels.

8.5. Conclusion

The 245 g/m^2 carbon fibre prepreg with c-m-p CP0093 resin that was used in process window study 1 was used in subsequent studies as well, but it was no longer delivering consistent results. The age of the prepreg, the low tack and the existence of dry spots in the cured products were three main indicators that the prepreg had expired. As a replacement a new 200 g/m^2 carbon fibre prepreg with the same resin was ordered, it was however found that it also yields a higher surface porosity. Reproducing the results of benchmark sample XC-TM3 (0.025% porosity) was not possible with this prepreg, under the same conditions it is measured to have a 3.28% surface porosity content. Out-time tests indicate that the problem is not related to the difference in age of the prepregs, it is expected to be related to the higher out-of-plane permeability of the weave.

It was postulated that entrapped air in X-core is forced out as it expands during the cure cycle and that preventing or reducing the flow of air to the surface ply will then help to reduce surface porosity. Tests wherein a vacuum film was placed between the core and laminate yield a reduction in surface porosity of up to 97%, thus supporting this hypothesis. The vacuum film does however impair the core-to-face bond. Therefore, the use of a 160 g/m^2 dry glass fibre twill weave was proposed as an alternative. It does not impair the core-to-face bond, but it also yields a 16% reduction in the core shear strength and a smaller surface porosity reduction of up to 61%. Increasing the number of plies in the laminate and changing the stacking sequence also helps to reduce surface porosity, but both measures are less effective than the glass fibre barrier layer.

Finally, tests were done with pigmented X-core; pigmentation is done to mask manufacturing flaws and X-core penetrating through the weave. Black pigment is added to the epoxy in X-core in an attempt to change the colour of the foam from white to dark grey. The addition of up to 10 wt% pigment does not negatively affect the surface porosity or the core shear strength, but it does help to make the colour of X-core more comparable to that of the carbon fibre in the laminate.

9

Process window study 2

Chapter 7 covered a process window study, but within the set limits of the process variables and the chosen prepreg/X-core system no samples could be manufactured with a high surface quality. Therefore follow-up studies were performed to identify the cause of the surface porosity in the X-core process and to find strategies to mitigate it, which is covered in Chapter 8. The results of the studies that have been treated in this report all point towards an undesirable interaction between X-core and the laminate: Entrapped air in the core is vented into the laminate, causing an increase in surface porosity. It was found that isolating core and laminate with an impermeable barrier layers can yield a significant reduction in surface porosity.

Therefore, a second process window study is proposed wherein a vacuum film is used as a barrier layer to isolate X-core from the laminate. This is done to identify whether a process window exists for the prepreg if the interaction with X-core is not a factor. The vacuum film is not a suitable barrier layer for structural applications, it impairs the core-to-facing bond. It is however a very effective measure to reduce surface porosity, which is why further research into a suitable replacement for this barrier layer is part of the recommendations.

A description of the test set-up and which process variables are researched in process window study 2 is covered in Section 9.1. The results of this study and its implications are covered in 9.2

9.1. Test description

Process Window study 2 (PW2) is not as extensive as Process Window study 1 (PW1), mainly because up to this point a lot of information has already been gathered and the research can be narrowed down. This section describes which process conditions are different with respect to PW1 and which variables are to be investigated in this study. The test set-up is mostly the same, any differences are highlighted in this section.

For the manufacture of the samples the tile mould is used, the setup is the same as in PW1 (Figure 7.2). The samples are manufactured by the same procedure as in test PW1, the only difference being the preparation of the laminate. For all samples in PW2 a two ply 3k/3k laminate is used containing the $200\,\mathrm{g/m^2}$ 3k carbon fibre twill weave prepreg with c-m-p CP0093 resin. Laminates for all samples in this study have been cut from the roll simultaneously, they are packed individually in a sealed plastic bag and stored in the freezer until they are needed. The prepreg laminates are thawed about 90 minutes before they are unpacked and collated. One layer of vacuum film is adhered to the side of the prepreg that directly faces the core, the vacuum film is applied on both facings.

The 270 mix is used for all samples, the constituents can be found in Table 6.5. Eight photographs are taken of each sample and processed to determine the porosity content, using the optical inspection jig and procedure described in Section 5.3.

In PW1 the effect of four process variables on the surface porosity content is investigated: ramp-up rate of the cure cycle T_1', cure temperature T_c, sample thickness h and DU volume content v_{DU}. Within the range of these variables a surface porosity content of 0.025%, which was set as the benchmark in Section 7.1, is the lowest that has been achieved with the X-core process. Two other samples came relatively close, namely the bottom facings of samples VB-TM-02A and VB-TM-02B (Section 8.2.3), which are manufactured with a vacuum film between the core and laminate. All three samples were subjected to the same process conditions: heat-up rate of ~1.5°C/min, a cure temperature of 110°C and all samples were 10 mm thick and contained 7.5 vol% DU. In PW2 this is defined as the reference condition.

This reference condition was found coincidentally: The cure cycle is a simplification of the standard X-core production cycle, the DU content was capped at 7.5% because it was thought that a higher percentage would deliver too much pressure for the tile mould to handle and the height was simply chosen to be the smallest possible to save preparation time. Based on the results from PW1 the reference condition is desirable to minimize surface porosity, with the exception of the heat-up rate which may preferably be lower. The goal of PW2 is to find out whether this condition is at the border of a process window or that it is a robust point that ensures consistent results, even when there is a slight variation in process conditions.

In PW2 the variable of interest is the DU volume content; according to the fitted model in PW1 the surface porosity is most sensitive to changes in DU content. The other three variables that were tested in PW1 are fixed, PW2 starts from the reference condition and samples are prepared to find a relation between DU content and surface porosity. The fixed conditions in PW2 are therefore:

$$T_1' = 1.5\text{°C/min} \qquad T_c = 110\text{°C} \qquad h = 10\,\text{mm}$$

These variables are fixed because it is more efficient to first explore whether such a window could exist under the set conditions with just a few samples, rather than making a large number of samples to explore all processing conditions.

Data is already available for the 7.5 vol% DU content, namely from samples VB-TM-02A and VB-TM-02B. PW2 procedes by making a sample with half the DU content, which is 4.5%, and measuring its porosity. Based on the available data it was already expected that this is too low to yield a porosity content that is near the benchmark. Subsequently three more samples are made with 5.25%, 6.0% and 6.75% DU respectively. With these results a relation between DU content and surface porosity is determined for these process conditions and this 3k/3k laminate with a vacuum film barrier layer.

9.2. Results

In PW2 the effect of DU content on the surface porosity is quantified for a process wherein X-core is isolated from the laminate by means of a vacuum film. The process conditions and porosity contents of each of the samples in PW2 is provided in Table 9.1. Photographs of representative areas of the samples are provided in Figure 9.1.

Table 9.1: *Process variables and porosity contents of the samples in process window study 2 (PW2)*

Sample ID	T_1' [°C/min]	T_c [°C]	h [mm]	v_{DU} [vol%]	Mean porosity [%]	Standard deviation [%]
PW2-TM-4.5	1.44	110	10	4.5	1.79	0.58
PW2-TM-5.25	1.45	110	10	5.25	0.33	0.19
PW2-TM-6.0	1.44	110	10	6.0	0.24	0.20
PW2-TM-6.75	1.45	110	10	6.75	0.030	0.037
PW2-TM-7.5	1.45	110	10	7.5	0.043	0.043

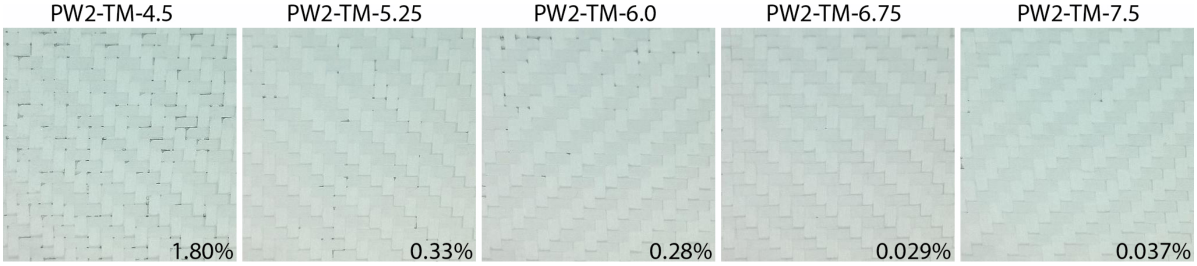


Figure 9.1: *Photographs of representative areas of the samples in PW2, including porosity content.*

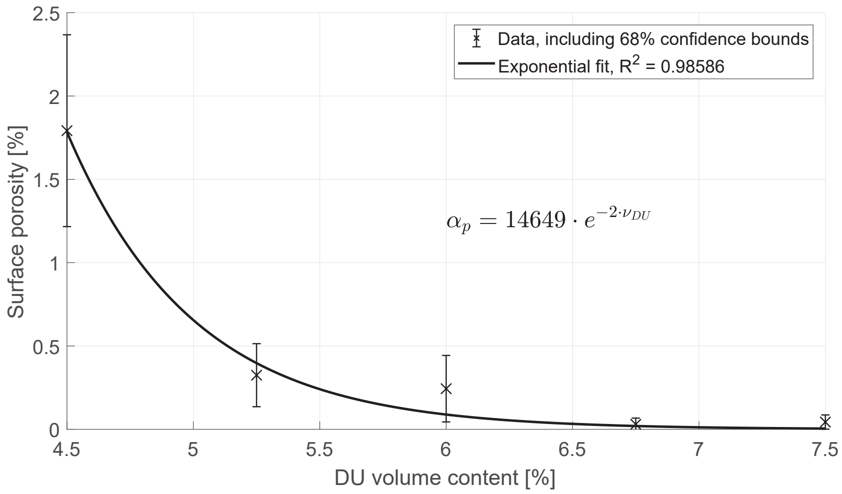


Figure 9.2: *Exponential curve fit on the porosity data of PW2*

The results show that there is a strong decrease in surface porosity between 4.5% and 5.25% DU. After this point the decrease is more moderate as the surface porosity approaches 0%. The trend is better visible when the data is plotted, see Figure 9.2. An exponential curve is fitted to the data, an exponential decay is chosen because it is representative of the problem; the surface porosity can only be expected to approach zero as the pressure increases, it cannot be negative. In addition, the R^2 value of the regression is equal to 0.986, indicating that it is an accurate fit. The equation of the exponential fit is as follows:

$$\alpha_p = 14649 \cdot e^{-2 \cdot \nu_{DU}} \tag{9.1}$$

According to this fit the benchmark value of 0.025% for a high quality surface is achieved at a DU content of 6.64 vol%. Samples PW2-TM-6.75 and PW2-TM-7.5 have a porosity content of 0.030 and 0.043 respectively, both values are comparable to the benchmark and are still found to be of high quality, see Figure 9.1. The porosity in sample PW2-TM-6.0 on the other hand is considered to be too high and it needs rework (application of a transparent filler). So, the threshold value for rework lies somewhere between 0.04% and 0.28%, which corresponds to DU volume contents in the range of 6.0% to 6.75%.

9.2.1. The effect of the surface ply on surface porosity

The results suggest that the laminate, and especially the surface ply may significantly influence the surface porosity. The effect of barrier layers was investigated in Section 8.2, the results confirmed a decrease in surface porosity when an impermeable barrier layer is used. So, a cause of surface porosity in the X-core process was identified as the degassing of entrapped air in the core into the laminate as the DU expands. This theory was then used to explain the difference in surface porosity between the 245 g/m^2 3k and the 200 g/m^2 3k.

However, the results of PW2 suggest that there may be another phenomenon at work that can partly explain the difference between the 245 g/m^2 3k and the 200 g/m^2 3k as well. To that end the results from the vacuum bag barrier layer test (VB-TM) need to be recalled. Sample VB-TM-01 in the vacuum bag barrier layer test has 1.5 vol% DU and a mean surface porosity content of 0.67%, which is lower than sample PW2-TM-4.5, which contains 4.5 vol% DU and has a mean surface porosity content of 1.79%. In both tests the core and facings are separated by a vacuum film, so out-gassing of X-core is no longer affecting the surface porosity. But, as stated in Section 8.1.1 the change in weave also affects the impregnation of the prepreg, which is shown in Figure 8.4. c-m-p uses the same hot-melt procedure for both weaves, the difference in impregnation is not fabricated intentionally.

The pinholes that are visible at the surface is a direct consequence of air that is trapped between the mould surface and the first ply [20]. There is a clear difference between the impregnation of the 245 g/m^2 prepreg and the 200 g/m^2 prepreg, see Figure 8.4, page 67. The 200 g/m^2 pepreg contains small unimpregnated islands at the surface of the prepreg, whereas the 245 g/m^2 perpreg has large interconnected pores. This affects air entrapment at the ply-tool interface and is therefore expected to affect the surface porosity. A recommended solution to this problem is making use of a surfacing film or surface ply. This is however not investigated in this study, it is part of the recommendations for future research (Chapter 13).

9.2.2. Tile mould deformation

At the end of process window study 1 the lack of stiffness of the tile mould and how it could affect the results was discussed. After PW1 the mould was stiffened and subsequent manufacture of samples confirmed that the deflection was reduced. However, between PW1 and PW2 25-30 samples are manufactured in the repaired tile mould and especially the lower lid of the mould started to deform permanently. So, besides the elastic deflection that is present due to the pressure there was also some plastic deformation. The mould is still stiff, but there is a dent in the mould lid, see Figure 9.3.

The problem may be related to the epoxy that is used in making the tile mould. When postcured, the SR1660/SD7820 resin has a glass transition temperature of 141°C. The T_g is not a well-defined event, the material may also start softening at some lower temperature, depending on the interpretation of the T_g by the supplier of the epoxy. The deformation may have been the result of high stress ($7.5\,\text{vol\%}$ DU $\approxeq 8.5\,\text{bar}$) at 120°C, which has lead to permanent deformation of the epoxy in the facings or in the adhesive between the core and facing.

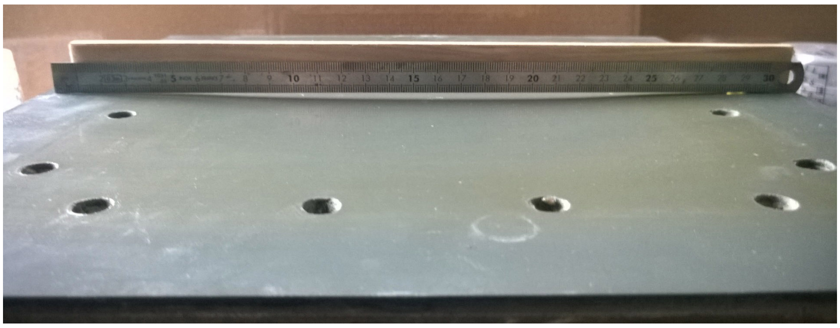


Figure 9.3: *Permanent deformation in the lower lid of the tile mould, the deflection at the centre is 3 mm.*

The dent is not filled when the X-core is moulded because the aluminium tooling plates between the mould lid and the sample are still flat. So, the mould allows for some expansion of X-core before it is restricted, which will affect the pressure that is exerted on the laminate. In order to show this effect a sample is made with a 3k/12k laminate containing an X-core mixture with just 0.82 vol% DU, which is the same as the floor panel mix. A vacuum film is placed between the core and the facing, this allows for delaminating the core and facing without damaging the substrates. A photograph of the surface of the core is shown in Figure 9.4.

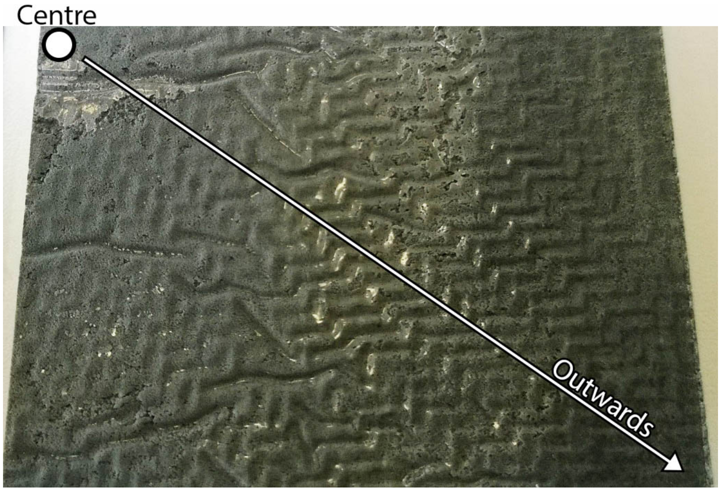


Figure 9.4: *Non-homogeneous pressure on the laminate due to expansion of the sample in the deformed tile mould cavity.*

The top left corner in the photo is the centre of the sample, going away from the centre the texture of the core changes. Away from the centre it is visible that X-core has conformed to the weave of the 12k ply, whereas at the centre there is almost no evidence of compaction. Near the edges of the specimen the features of the weave become sharper and folds in the vacuum film are no longer observed. This photo indicates the presence of a pressure gradient, which may be linked to the deformation of the tile mould lid that is seen in Figure 9.3. Based on this behaviour it is hypothesized that the pressure is not redistributed over the complete surface as X-core expands, that it is more of a local phenomenon that depends on the DU content, the local degree of mould filling and the allowable expansion of the sample. The behaviour of X-core during expansion and the resulting pressure is researched by M.P.J. Eversdijk [30].

Conclusion and recommendations

Based on the result it is concluded that a process window can exist within the suggested range of process variables, it does however require the use of an impermeable barrier between X-core and the laminate. It is strongly recommended that a suitable replacement for the vacuum film is found. This new barrier layer should prevent the entrapped air in X-core from entering the laminate and it should not allow adhesive failure of the core-to-facing bond to precede cohesive failure of the core. More tests are also required to identify whether changes in the heat-up rate, sample thickness or cure temperature affects the porosity content within this range of DU contents. Furthermore, it is recommended that the tile mould is redesigned and replaced by a stiffer and stronger mould with a higher temperature resistance. Eventually, with these changes implemented, a process window may be developed for this particular laminate within the range of cure temperatures, heat-up rates, DU contents and sample thicknesses defined in Chapter 6.

10

Processing guidelines for the X-core process

In this chapter processing guidelines are formulated based on the results that are obtained in this research. Formulating these guidelines is the goal that was formulated at the start of this research. At that moment it was assumed, based on preliminary results, that a process window could exist wherein X-core structures could be manufactured repeatedly with consistent results and a high quality surface finish. Subsequent studies into the effect of processing conditions on the surface porosity however revealed that this may be more challenging and that more variables are involved than was initially expected. The guidelines are formulated for the process variables that were identified in Chapter 6. The results from Chapters 7, 8 and 9 are used to quantify the desirable value of these variables.

The type of prepreg and laminate

For the X-core process the prepreg format should be suitable for autoclave processing (no semi-pregs), the advised resin system is c-m-p CP0093 epoxy. A different epoxy can be opted for, it is advised that the viscosity profile, gel time and cure time at different temperatures is comparable to that of the CP0093 resin (see Section 6.2.2, pages 42-43). A list of the important cure related properties of the resin is provided below:

- The resin can be cured at temperatures between 80°C and 170°C.
- At a heat-up rate of 1-2°C/min it has a minimum viscosity of 350-450 mPa·s, it achieves that a temperature of 105-110°C
- The time to reach 98% degree of cure is 300 minutes at 90°C, 120 minutes at 105°C and 60 minutes at 120°C.
- The gel time is 110 minutes at 90°C, 36 minutes at 105°C and 14 minutes at 120°C

The weaves that are covered in this study are a 245 g/m^2 3k, a 200 g/m^2 3k and a 420 g/m^2 12k fabric, all are 2x2 twill weaves. The use of the 12k as a surface ply is not recommended, in terms of reducing the surface porosity content the 245 g/m^2 3k is preferred over the 200 g/m^2 3k. The best results are obtained with two 245 g/m^2 3k plies at the surface of the product. Additional plies may be added to the laminate because, generally, the surface porosity is lower when the out-of-plane permeability of the laminate decreases.

The 200 g/m^2 3k can only be used as a surface ply and yield high quality surfaces when there is an impermeable barrier layer between the core and facings. In order to mitigate nearly all surface porosity it is necessary to prevent out-of-plane gas transport from the core to the laminate when the X-core expands.

X-core formulation

The recommended X-core foam is the 270 mix: in combination with the two-ply 245 g/m^2 3k laminate this formulation can yield products where nearly all porosity is mitigated. Using this foam in the X-core process is extensively researched, the guidelines in this chapter are formulated based on results from samples containing this foam. The use of 10 wt% black pigment in the epoxy of X-core is also recommended, it helps to mask X-core penetration or manufacturing errors and does not affect the shear strength of the core or the surface porosity of the laminate. If a different X-core mixture is desired the following should be taking into account for either a lower density or higher density foam:

- **Lower density foams:** When lower density X-core mixtures with lower resin contents are desired it is strongly recommended to use an impermeable barrier layer between the core and laminate. The lower density mixes contain more entrapped air in the form of interstitial voids, the barrier prevents it from venting into the laminate as the X-core expands.
- **Higher density foams:** In terms of surface porosity a higher density mix with a higher resin content may perform better than the 270 mix due to the reduced amount of interstitial voids, but this has not yet been researched. A higher resin content can also impose problems related to the exothermic reaction of the epoxy; the temperature inside the core can easily exceed the maximum expansion temperature of the DU microspheres (125°C). For more information see the transient heat transfer model for X-core of M.P.J. Eversdijk [30].

Expancel DU volume content

Generally, increasing the DU content leads to a higher pressure on the laminate and helps to reduce surface porosity. DU contents between 1.5 and 7.5 vol% are sufficient to compact the laminate, provided the mould is completely filled with X-core (degree of mould filling is covered at the end of this chapter). However, very high volume contents are required to achieve a surface porosity of 0.025%, which is set as the benchmark for high quality surfaces. For the 245 g/m^2 3k/3k laminate this amounts to a 7.5 vol% DU content without barrier layer and for the 200 g/m^2 3k/3k laminate at least 6.75 vol% when an impermeable barrier layer is used. A 7.5% DU content corresponds to a pressure of approximately 8.5 bar, working with such an X-core mix should be considered in the design of the tooling.

Cure cycle profile

The product can be cured in a simple two-step cycle, the heat-up rate is preferably between 1°C/min and 1.5°C/min and the cure temperature between 90°C and 120°C. The window wherein the cure can be performed is displayed graphically in Figure 10.1. The dashed line in the figure represents the boundary that guarantees a 98% degree of cure of the prepreg resin. The cure cycle may be extended beyond this line, but this is not necessary.

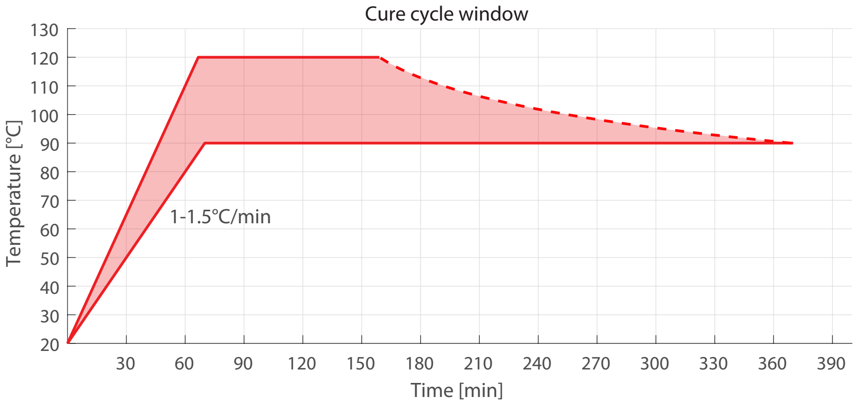


Figure 10.1: *The cure cycle window, the temperatures inside the mould should fall within these boundaries. The dashed line represent the time it takes to guarantee 98% degree of cure of the prepreg resin.*

Degree of mould filling with X-core

It is required that the mould cavity is 100% filled with X-core prior to the placement of the top laminate. For complex geometry components it may be required to make a dedicated X-core mould to determine when a 100% filling is achieved (see Appendix A.2). Local under-filling of the mould leads to a local decrease in pressure, excessive under-filling can even result in insufficient laminate compaction. In addition, applying an insufficient amount of X-core effectively introduces entrapped air between the core and facing, which will result in an increase in surface porosity.

11

Floor panel production

The Floor panel is the first X-core product that is taken into production at Donkervoort, it served as a demonstration of the X-core process. These floor panels are installed in the Donkervoort D8 GTO RS.

The manufacturing process of the floor panels has however not been developed any further since the first assembly-worthy components came out of the mould. This study has provided new insights into the X-core process, based upon which processing guidelines have been formulated. In the tests described in the Chapter these guidelines are applied as much as possible to the floor panel production process with the aim to make the process more robust.

First of all, Section 11.1 explains how the variables/conditions of the current production process need to be changed such that it is more in accordance with the guidelines formulated in Chapter 10. Small scale tests using the tile mould are then conducted with these changed conditions, this way some process variations can be quickly introduced and assessed. These reference tests are covered in Section 11.2. Subsequently the process is carried out on full-scale; two sets of floor panels are manufactured and the changes with respect to the old production process are compared one-to-one. This is described in Section 11.3, which concludes with photographs of the newly manufactured floor panels after painting.

11.1. Changing the process conditions

The process for floor panel production was previously established through an iterative procedure, but now the guidelines can be used to establish which conditions may yield high quality floor panels. This section starts out by describing the current production process, which is followed by an explanation of the changes that are going to be made, keeping in mind the guidelines that are formulated in Chapter 10.

Current process conditions

The floor panels are currently made using a VBO prepreg as surface ply: A 245 g/m^2 3k carbon fibre twill weave with Cytec VTF-261 resin and a glass fibre surface veil. The second ply is the 420 g/m^2 12K carbon fibre twill weave with c-m-p CP0093 resin. The X-core in the panels is a 330 mix with 0.82 vol% DU. The cure cycle is the standard cycle for X-core production (see Figure 4.5). The required volume of X-core is determined beforehand and mixed, after which all of the foam is spread out manually over the mould cavity. It is known that there are variations in the density of X-core due to variations in the weighing and mixing procedure, so the volume does not necessarily correspond to the desired quantity. In addition, the foam is spread out manually and it is gauged by visual inspection whether the entire cavity is filled homogeneously, which is also subject to variation. The degree of mould filling is therefore not necessarily 100% and is not uniform in the manufacture of floor panels.

New process conditions

A constraining factor in floor panel production is the mould: It lacks stiffness which may result in a thickness increase of the floor panels. The pressure, and hence DU content, should be kept as low as possible to prevent excessive deformation or damage to the mould. It is therefore decided to opt for 1.5 vol% DU, which is estimated to correspond to a 2.3 bar pressure. First order calculations for a stiffened plate using 2D Euler-Bernoulli beam theory indicate a thickness increase of the panel in the order of 0.4 mm at this pressure.

In order to get the lowest possible porosity content the 3k/3k laminate with the 245 g/m^2 carbon fabric is used. The use of a non-permeable barrier layer like a vacuum film can help to reduce the surface porosity content, but a suitable barrier layer that does not impair the core-to-facing bond has not yet been found. In these conditions the floor panels will not be free of porosity, but this can be acceptable if it is easily removed with a filler.

The cure cycle is changed to a 1°C/min ramp-up to 120°C. The highest temperature is chosen because it leads to the shortest cycle time. The low ramp-up rate is opted for to improve temperature homogeneity in the mould as the temperature progresses through the onset of expansion temperature of X-core. For the core material the X-core 270 mix will be used, which also benefits from being 36 g/L lighter than the 330 mix. The procedure of moulding X-core needs to be changed, which brings forward additional changes in the process. The goal is to have a 100% degree of mould filling, how this is achieved is covered in more detail in Section 11.3. 10 wt% Black pigment is added to the epoxy to make the X-core dark grey, this is also required for the new production procedure of the floor panels. The process conditions are summarized in the Table 11.1.

Table 11.1: *New process conditions, type of laminate and X-core composition for the floor panels*

Laminate	2 plies of 245 g/m^2 3k carbon fibre twill weave with c-m-p CP0093 resin
X-core	270 mix with 10 wt% black pigment in the epoxy, 1.5 vol% Expancel DU
Cure cycle	1°C/min ramp-up, 120°C cure temperature, 120 minutes cure time

11.2. Tile mould reference tests

Samples are prepared in the tile mould to see if the changes in processing conditions yield acceptable results. It is known beforehand that there will be surface porosity and that it will need to be masked/filled afterwards. The focus has moved towards making this porosity as low as possible and ensuring that the overall finish of the product is good: no distortions in the weave, X-core penetrating the weave, waviness of the surface ply or large air pockets at the surface.

In total three samples are made, the process conditions are the same as for the floor panel, which are provided in Table 11.1. The only difference is the thickness; the floor panel is 8 mm thick whereas the tile mould samples are 10 mm thick. In the Process Window 1 regression model the thickness is multiplied by coefficient β_2, which is equal to 0.0389 (Equation 7.10 and Table 7.6). So, a 2 mm difference only gives an 0.078% change in porosity for the 3k/12k laminate. It is expected that the difference for the 3k/3k laminate will be of the same order of magnitude. The samples are defined as follows (see Appendix D.1 for information on sample labelling):

- **FP-TM-01:** Standard conditions as described in Table 11.1.
- **FP-TM-02:** A 160 g/m^2 dry glass fibre weave is added as a barrier layer between the core and laminate.
- **FP-TM-03:** An intermediate dwell of 30 minutes is added to the cure cycle at 70°C.

The glass fibre barrier layer is investigated because it may help to reduce porosity. The intermediate dwell is introduced because it helps to homogenize the temperature in the floor panel mould just before the expansion starts, the test is performed to see whether this affects the surface porosity. The process variables and porosity contents of the samples are provided in Table 11.2.

Table 11.2: *Process variables and porosity contents of the floor panel reference test samples (FP-TM)*

Sample ID	T_1' [°C/min]	T_c [°C]	h [mm]	v_{DU} [vol%]	Mean porosity [%]	Standard deviation [%]
FP-TM-01	1.01	120	10	1.5	1.52	0.37
FP-TM-02	0.98	120	10	1.5	2.43	1.88
FP-TM-03	0.96	120	10	1.5	2.41	1.05

The porosity content of sample 02 is disturbing, it indicates that the addition of the glass fibre barrier layer leads to an increase in surface porosity. It also shows a large scatter in the data ($\sigma = 1.88$), which is not seen in sample 01. The results of the test could not be repeated because the prepreg had expired shortly after. With the fresh 200 g/m^2 prepreg and the 3k/3k lay-up a reduction of porosity was observed with the glass fibre barrier layer (test DF-TM-02). It is recommended that sample FP-TM-02 is manufactured with a fresh prepreg to support these findings. The possibility that the results could have been affected by ageing of the prepreg should also be considered. Appendix E shows the timeline of sample production, it shows that the FP-TM samples were made right before the 'Aged' samples. The latter refers to the samples that display signs of an expired prepreg. Nevertheless, the results of all three samples are comparable: sample 01 falls within the 68% confidence bounds of both samples 02 and 03.

Based on these results it was decided not to use the glass fibre barrier layer. It takes more time to prepare the laminate and there is no decrease in surface porosity. Sample FP-TM-03, which was cured with a 30 minute dwell at 70°C, also shows an increase in surface porosity. It was however still decided to make use of the dwell. This is mainly done because the floor panel mould does not heat up as homogeneously as the tile mould, measurements indicate a 35°C difference when the setpoint temperature of the cure cycle is at 80°C. The dwell allows for the temperature to homogenize before the expansion temperature. It was expected that this is necessary to to achieve a homogeneous pressure application on the laminate and a consistent finish over the entire surface of the floor panel. It was decided to first cure one set of floor panels with a 30 minute dwell and subsequently the second set with a 15 minute dwell, upon which the effect of changing the dwell time can be determined through comparison.

11.3. Full-scale floor panel tests

Some changes to the current floor panel production process were required in order to ensure a 100% degree of mould filling, as per the process guidelines. In this section the changes with respect to the original production process are explained and shown. This is followed by an analysis of the porosity content of the floor panels and finally the quality of the finished product, after painting, is reflected upon. A description of the floor panel mould can be found in Appendix B.2.

11.3.1. Changes in the manufacturing process

The way in which the manufacturing process is changed is presented schematically in Figure 11.1. The process of laying down the laminate and moulding the X-core is summarized in four steps:

1. The two plies of the top laminate are placed; the floor panels are manufactured upside-down because the top surface is visible from the cockpit.
2. X-core is added to the mould and spread out to fill the mould cavity.
3. The two-ply top laminate is correctly positioned over the core.
4. The mould is closed.

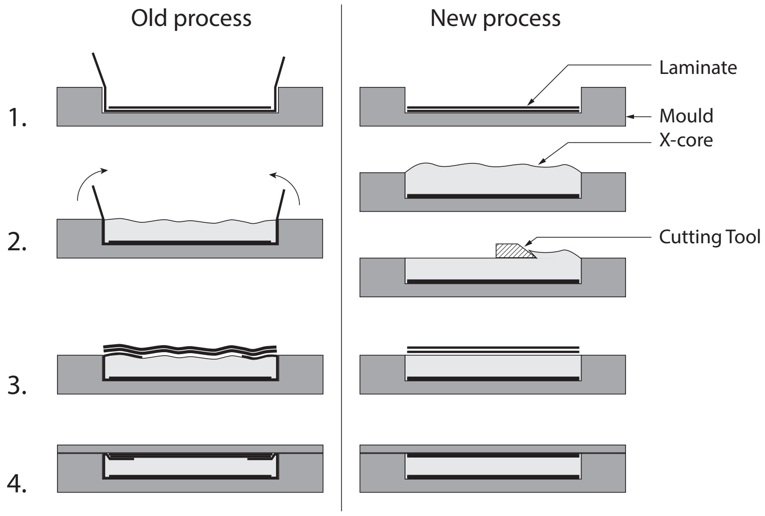


Figure 11.1: *Comparison of the old and the newly proposed manufacturing process of the floor panels.*

The changes are made to simplify the process and mostly to make it easier to ensure a consistent 100% degree of mould filling with X-core. Previously, the floor panel was completely enclosed by the laminate. It was done to demonstrate the capabilities of X-core: sharp corners and mould defined features along the edges of a sandwich panel. Leaving the edges uncovered should not be a problem; all of the samples that have been made in the tile mould with prepreg facings were made following the 'new process'. None of these samples had problems with X-core penetrating between the first ply and the tooling surface. X-core is also closed cell and will not absorb water, so it may be exposed to the environment. Finally, black pigment can be used to mask these foam core edges, the difference will be hardly observable as soon as the panels are installed in the car: The outer 25 mm of the panel is covered by the steel beams of the chassis.

The new process offers some important benefits. First of all laying the prepreg has been simplified as it is no longer required to work the prepreg into the corners. Secondly, the overlapping fibres are no longer present which simplifies the process of filling the mould cavity with X-core. In the new process an excess amount of X-core is moulded into the cavity, which is subsequently removed by cutting it away using a stiff steel tool that is moved along the flanges of the mould. This was not possible in the old process and the main benefit is that it is now much easier to ensure a consistent degree of mould filling for every new set of floor panels. It also helps to get a smooth surface on which the laminate is to be placed. The extent of the difference may be clearer when comparing photographs of the mould filling in the old process and in the new process, see Figure 11.2. It is also expected that the dimples in the core material promote air entrapment, which in turn leads to an increase in surface porosity. The quantity of X-core constituents that is prepared for the manufacture of both floor panels is provided in Table 11.3.

Figure 11.2: *Difference in mould filling between the old and the new manufacturing process of the floor panels.*

Table 11.3: *The quantity of the constituents and estimated volume of the floor panel X-core mixture.*

	Estimated volume [mL]	Ancarez 4010 [g]	Ancamide 3399 [g]	Polypox black pigment [g]	Q-cel [g]	Expancel DU [g]	Expancel DE [mL]
Floor panel X-core mixture	3900	371.0	247.3	41.2	324.2	21.6	1242

11.3.2. Results

Two sets of floor panels are manufactured in accordance with the conditions that are provided in Table 11.1. There was however not enough of the 245 $\mathrm{g/m^2}$ 3k prepreg to completely cover both sets of floor panels on both sides. Therefore in each set only one panel is made with the 3k/3k laminate on the top surface. The other panel has a 3k/12k laminate on the top surface, the bottom surfaces of both panels are made with a different prepreg and their surface porosity content is not of interest in this study.

The floor panel does not fit inside the sample bed of the optical inspection set-up, instead the panel is rested against the bed, ensuring that all angles are still the same. The only differences are the decreased distance from the lens of the camera to the surface of the panel and the decreased distance from the LED panel light source to the surface of the panel. This needed to be corrected for in the brightness filter of the digital image processor, all other settings remain unaffected.

The optical inspection set-up can also be used as a tool for quality control in the manufacturing process. To that end a new set-up can be manufactured for the inspection of cured floor panels or other panels of any size. It is then recommended that the light source and camera are suspended above the surface that is to be inspected. The fixture that holds the light source and camera does not need to be adjustable, the correct alignment can be obtained from Figure 5.5. Different areas can be inspected by translating the fixture over the surface of the component, see Figure 11.3 for a sketch of the proposed set-up.

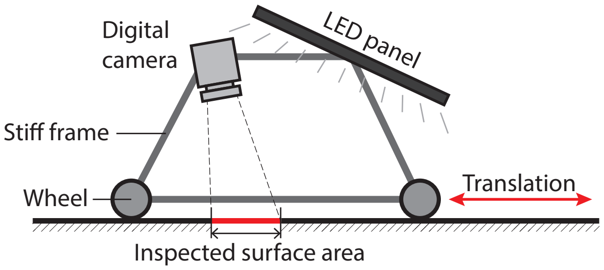


Figure 11.3: *Proposed set-up for porosity detection in fibre composite surfaces.*

For the determination of the surface porosity content five photos are taken of the top surface of each floor panel, using the fixture that is described in Chapter 5. The sample IDs are defined as follows (see Figure 3.9 for drawings of the floor panels):

- **FP-01L:** Driver side floor panel (large panel), 3k/3k laminate
- **FP-01S:** Passenger side floor panel (small panel), 3k/12k laminate
- **FP-02L:** Driver side floor panel (large panel), 3k/12k laminate
- **FP-02S:** Passenger side floor panel (small panel), 3k/3k laminate

In FP-01 a 30 minute intermediate dwell is carried out at 70°C, in FP-02 this dwell is shortened to 15 minutes. The measured surface porosity contents of the top surfaces of the floor panels is shown in Figure 11.4. The results show an excellent consistency of the results for both floor panels. The 3k/3k laminate has a surface porosity content of 4.5%, both in the passenger side floor panel of the first set and in the driver side floor panel of the second set. The same holds true for the 3k/12k laminate, which has a 2.5% surface porosity content in both floor panels. From the results it is also concluded that the difference in dwell time does not affect the surface porosity in the floor panels.

The result is also contradicting earlier studies, namely that the 3k/3k laminate yields more porosity than the 3k/12k laminate. It also performs worse than the reference samples in the tile mould, see Table 11.2.

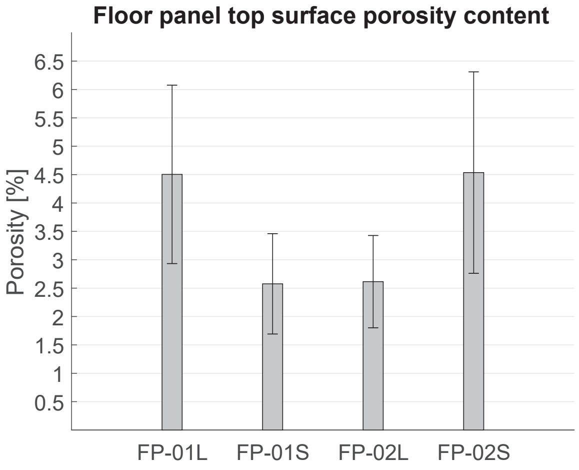


Figure 11.4: *The porosity content including 68% confidence bounds of the top surfaces of the floor panels.*

Figure 11.5: *Photograph of floor panel FP-01L right after demoulding*

Due to the consistency of the results it is not believed that it is an outlier. The problem may be related to the larger size of the floor panel or the different characteristics of the floor panel mould. Additional research is recommended to identify what the cause may be.

11.3.3. Synthesis

The mean surface porosity content of the floor panels is in the worst case 4.5%, which needs to be remedied; see Appendix C for an indication of the severity of the porosity. However, other surface defects like large air inclusions, waviness of the surface ply and contamination of the surface by X-core is not observed. These defects are not easily remedied, but were present in the floor panels that were manufactured following the 'old procedure'. With the porosity eliminated the panel looks visually flawless: See floor panel FP-01L in Figure 11.5, where the porosity is hardly visible due to the angle at which the photo is taken.

The panels are spray-painted with a transparent lacquer, like all other components inside the cockpit the carbon fibre weave needs to be visible. The surface porosity was easily eliminated by sanding the panels and spraying on one layer of a transparent filler. This was then sanded, followed by the application of a single layer of lacquer. The result of this process is shown in Figure 11.6. The procedure is not different from any other 'visual' carbon fibre component that is installed in the car.

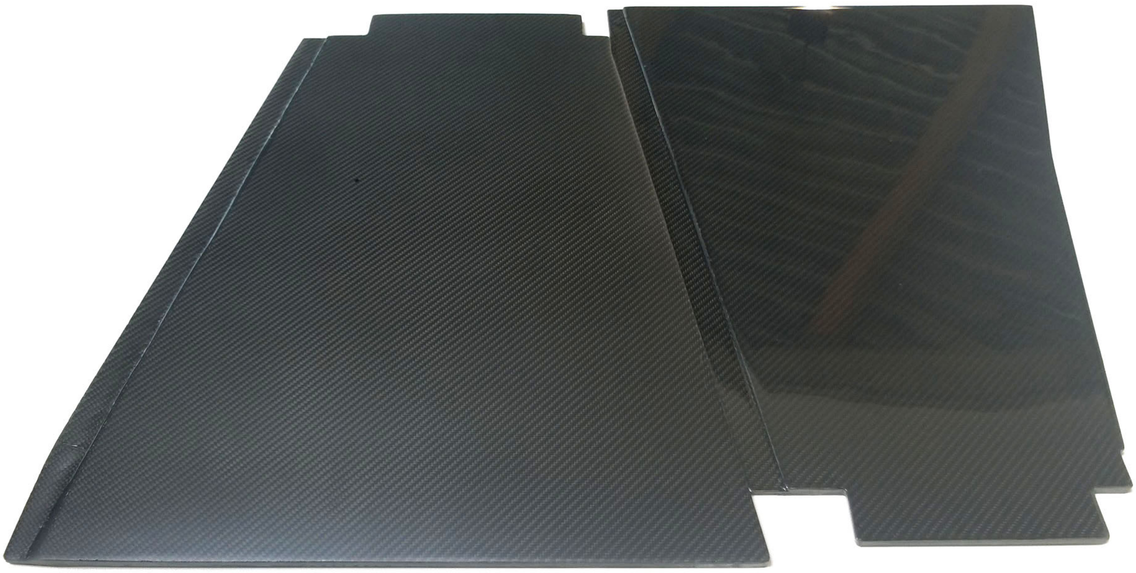


Figure 11.6: *Photograph of floor panels after painting, semi-gloss finish on the left (panel FP-01L) and high-gloss finish on the right (panel FP-02S).*

This is an interesting result, because it seems to undermine the importance of eliminating all surface porosity. However, the floor panels are flat, which makes the application of a thick layer of filler easier. According to the painter the same procedure is more challenging on vertical surfaces. A thick layer of filler also results in additional mass that does not contribute to the strength and stiffness of the panel. In addition, a high amount of surface porosity may also be indicative of a high amount of bulk porosity, which affects the mechanical properties of the laminate [15]. So, for the floor panels it shows that a surface porosity content of 4.5% can still be acceptable if the goal is to achieve a high quality surface finish after painting, provided that extra work is not required to remove the porosity prior to painting, but this may not hold true for other components.

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Conclusion

The objective of this study was to formulate processing guidelines for repeated one-shot production of X-core sandwich structures and to increase the understanding of the relationship between process conditions and surface defects in these structures. The surface defects are a concern in the X-core process: The door window frames and floor panels of the Donkervoort D8 GTO RS are X-core products and have a 'bare carbon' finish, which means that the surface flaws are exposed. A defect like surface porosity may be masked by sanding the component and applying a transparent filler, but it introduces extra processing steps and is therefore not desirable. Other flaws like insufficient compaction, fibre distortions, waviness of the surface ply and penetration of X-core through the weave will always remain visible and will therefore need to be prevented.

The research mainly focusses on surface porosity, it is the most prevalent flaw in X-core structures. An approach is suggested wherein flat rectangular samples are prepared that can be subjected to optical inspection and three-point flexure tests. A new integrally heated mould was designed and manufactured for this purpose, it allows for direct temperature control by means of a PI controller that takes feedback from thermocouples in the mould. For the optical inspection a fixture is built that aligns a digital camera, a light source and the sample. Eight photographs are taken of each sample, the fixture helps to achieve the same brightness in each photograph for each sample. This effectively eliminated the tedious process of changing threshold values for detection of the surface porosity in the sample, which is highly dependent on the light intensity of the pores with respect to the pristine surface.

In the next stage of the research preliminary tests were carried out to determine the effect of a variety of process variables/conditions on the surface porosity content. The results pointed out that the use of a different prepreg, a 245 g/m^2 3k carbon fibre twill weave with c-m-p CP0093 resin, is more promising for reducing surface porosity than the traditionally used Cytec VTF261 VBO prepreg with the same weave. The results also stressed the importance of having a 100% degree of mould filling to ensure consistent results; underfilling can result in insufficient compaction and a sharp increase in surface porosity. Under the same processing conditions an overfilled sample (~102% filling) displayed just 0.1% surface porosity whereas an underfilled sample (~97% filling) displayed 4.5% surface porosity. It was also shown that increasing the Expancel DU volume content in X-core, which is directly related to the compaction pressure, is an effective measure to reduce surface porosity. A reference sample was made with a 2 ply laminate using the proposed 245 g/m^2 3k prepreg, its surface porosity content was just 0.025% for 7.5 vol% DU, 1.5°C/min ramp-up, 110°C cure and a core thickness of 10 mm. This was considered to be the benchmark for a 'high quality' surface finish.

In the first process window study the effect of DU volume content, cure temperature, heat-up rate and sample thickness were investigated. The number of tests was minimized through a designed experiment following the D-optimal approach. A quadratic response surface was fitted to the data, showing that varying the cure temperature between 90°C to 120°C has a negligible effect on surface porosity. Furthermore, increasing the Expancel DU volume content has the most profound effect on reducing the surface porosity. The investigated range of DU contents was 1.5-7.5 vol%, it was capped to prevent damage to the tooling because the highest DU content delivers an estimated pressure of 8.5 bar. Finally, the lowest ramp-up rate of 1°C/min was also more beneficial than 2 or 3°C/min and thinner samples lead to less surface porosity. However, even in the most optimal conditions of this study a sample cannot not be produced that yields a surface porosity lower than 0.68%. This is an interesting result, because the only difference with respect to the benchmark sample is the laminate: the samples in this process window study have the same surface ply, but the second ply is a 420 g/m^2 12k carbon fibre twill weave.

The results of the first process window study suggested that the composition of the laminate affects the surface porosity. It was hypothesized that the problem was related to out-gassing of the interstitial air that is entrapped in the core, which is forced out as the DU expands during the cure cycle. Tests were performed with a vacuum film between the core and facings, which showed a surface porosity reduction of up to 97% compared to a reference case without the vacuum film. The 420 g/m^2 12k has a larger out-of-plane permeability than the 245 g/m^2 3k weave because it is woven less tightly, which may explain the higher porosity content of the samples in the process window study.

During this research the 245 g/m^2 prepreg had expired, it was subsequently replaced by a new 200 g/m^2 prepreg with the same resin. In contrast to initial beliefs this relatively small change in the weave density significantly impacts the surface porosity content as well: The benchmark sample could no longer be replicated, the same two ply laminate with this new weave yielded a surface porosity content of 3.3%. To rule out the effect of ageing the prepreg was conditioned at room temperature for two weeks (the maximum shelf life), which still yielded a 3.10% porosity content.

For the laminates with 200 g/m^2 weaves it is a necessity to make use of a vacuum film barrier layer between the core and laminate to achieve porosity contents that are comparable to the 0.025% benchmark. This does require DU contents in excess of 6.75 vol% and leads to practically no delamination resistance of the samples due to the vacuum film. The use of a different barrier layer like a 160 g/m^2 dry glass fabric prevents this delamination problem, but it reduces the core shear strength by 16% and it yields a 61% surface porosity reduction, which is much smaller than the 97% offered by the vacuum film.

When X-core expands it may penetrate through the gaps in the weave of the laminate. X-core is naturally white, which leaves white stains that impair the surface finish of 'bare carbon' components. To reduce the aesthetic impact of this problem 10 wt% of black pigment is added to the epoxy in X-core, which gives it a dark grey colour. By doing this the mechanical properties of the core and the surface porosity content of the laminate are unaffected.

Finally, two sets of floor panels are manufactured following newly formulated processing guidelines. The surface porosity content of the panels is 2.5-4.5%, which requires to be remedied. However, the painter experienced no extra effort in achieving a class-A finish for these products. It is standard practice to sand all components and to apply one layer of transparent filler. Because the floor panels are flat a relatively thick layer is easily applied and all the pores are filled in one step. Unexpectedly, it shows that 4.5% surface porosity can be acceptable in some cases, even if a high quality finish is required for a bare carbon component.

From this research a more profound understanding of the relation between processing conditions and surface defects is obtained. The most important findings are:

- A primary source of surface porosity in the X-core process is entrapped air in the core.
- The lamina weave density and laminate composition has a large impact on the surface porosity, for a 3k bundle size a higher density weave yields lower surface porosity contents.
- High DU contents (≥6.75 vol%) are a necessity to mitigate surface porosity
- The degree of mould filling with X-core has a strong impact on the surface finish, underfilling should be prevented at all times

During the research the process has demonstrated to be capable of manufacturing sandwich panels with nearly defect-free surfaces whilst eliminating the use of an autoclave and a vacuum bag. The process is promising, but further research into its capabilities is needed to make it more competitive with respect to conventional manufacturing processes for sandwich composites.

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Recommendations

The thesis research that is presented in this report has shown that sandwich structures can be made with minimal surface defects in the one-shot X-core process. Carbon fibre/epoxy prepregs were co-cured to the core in a process that solely relies on the expansion of the foam to provide pressure on the laminate. The capabilities of X-core have been demonstrated and there is a clear potential for it to grow into a commercial product. It should however be realized that the development of this process is still at its early stages and that further research into this novelty is needed to fully capitalize on its potential. In this chapter recommendations for further research are formulated based on the results of the thesis that is presented in this report and the author's experience with X-core.

13.1. X-core

The development of X-core itself was outside the scope of this study, but there are still a number of unknowns related to the properties and processing of the material. The recommendations in this section are related to further research into X-core and its effect on the surface finish of the laminate and into processability of the foam.

13.1.1. Entrapped air

In this study it became apparent that there is entrapped air in X-core that is being vented into the laminate when it expands. Placing an impermeable barrier like a vacuum film between X-core and laminate prevents this problem, but this also impairs the core-to-face bond of the sandwich. Another suggestion could be to solve the problem at its source: Reducing air entrapment in the core. The entrapped air is partly introduced during mixing and is partly a result of the low resin content. In the 270 X-core mix that is used in this study there is not enough resin to fill up all the interstitial voids between the fillers. Three suggestions are proposed to reduce these interstitial voids:

1. **Increase microsphere packing density:** Theoretically the maximum packing density of randomly packed spheres of the same size is in the order of 60%. Q-cel 5028 and Expancel 920 DE 80 d30 are almost equal in size, their mean diameters are 65 μm and 70 μm respectively. By changing their size and relative volume fractions a denser packing of spheres can be achieved. A denser packing leaves less interstitial volume and therefore less voids for the same resin content.
2. **Increase resin content:** Instead of increasing the packing factor the amount of resin may be increased to fill up the space. It would lead to higher density foams, but it could still be a viable option when this is considered acceptable for non weight-critical applications.
3. **Pre-compact X-core:** Entrapped air may be removed partly by pre-compressing the core material in the mould. All samples in this study and the floor panels were filled 100%, but overfilling may offer lower surface porosity contents by forcing out more air that is initially entrapped in the core.

13.1.2. Pressure generation

The pressure that is generated by X-core is estimated in this report based on a relation between DU volume content and pressure that was developed in previous research. The relation was however formulated for a ~95% degree of mould filling, whereas in this study a guideline is formulated dictating that a 100% mould filling should be aspired. A new study is recommended such that the pressure can be estimated more accurately, which is important for mould design. In addition, it is suggested that the study should also consider the effect of temperature on pressure. The temperature in the core may depend on the epoxy content, ramp-up rate, cure temperature and core thickness. These variables affect the exothermic reaction and hence the temperature inside the core.

13.1.3. Variations in the mixture

When preparing different batches of X-core for this study it became apparent that the volume is not always the same, even if the exact same quantities of the constituents were to be mixed. The step of the X-core preparation process that is most prone to error is measuring the correct quantity of Expancel DE. The volume of DE in the measuring cylinder is sensitive to the duration of tapping and the time that is allowed for the microspheres to settle. It is recommended that a new standardized method for measuring DE is developed that yields more consistent results.

In addition, it was experienced that the X-core formulations with a higher DU content would always yield less volume of the uncured mixture. So, it is suggested that the determination of the volume of X-core based on the volume fractions and densities of the constituents should be revisited.

13.1.4. Shelf life and pot life

A study into shelf life and pot life of X-core at different temperatures is recommended. For production purposes it could save man hours to make a large batch of X-core and subsequently store it in the freezer at -23°C. It is thereby however important to know for how many days the X-core may be stored (shelf life). Similarly, X-core can be prepared at the start of the day for multiple components that are to be processed on that same day. It should then be investigated whether an ~8 hour pot life is feasible and whether that affects the quality of the cured products.

13.2. Surface porosity mitigation

This report covered mitigation strategies for surface porosity, but improvements can still be made and some options have not yet been explored. The use of barrier layers, surfacing films and higher weave densities is covered in this section.

13.2.1. Barrier layers

The vacuum film barrier layer can help to mitigate nearly all surface porosity, but it impairs the core-to-facing bond. The dry glass fibre barrier layer has minimal impact on the flexural strength of the tested specimens, but it does not yield the same porosity reduction as the vacuum film. It is desired to have a barrier layer with the impermeability of the vacuum film and the adhesion of the glass fibres. To that end two routes are recommended for further research into barrier layers:

- **Application of a coating:** A polymer film can be coated or sprayed onto the interface between the core and laminate and left to cure to form the impermeable barrier. This material should allow for good adhesion with the epoxy in X-core.
- **Higher density woven fabric:** The dry glass fibre weave in this research was a $160\,\mathrm{g/m^2}$ twill weave with 12 ends per centimetre, higher density weaves are available that may offer a lower out-of-plane permeability. In addition, a carbon fibre weave may also be used. In that case the added mass is partly offset because the barrier layer is a stiff and strong load carrying element of the sandwich. Doing this may require the addition of extra resin to wet these fibres and requires new mechanical tests to assess the core-to-facing adhesion.

13.2.2. Surfacing films

Surface porosity is also caused by air entrapment between the first ply and the tooling. Specialized surfacing films have been development for prepreg processing to help achieve class A finishes in carbon fibre products. They may be suitable for the X-core process as well and could yield significant porosity reductions.

13.2.3. Prepreg weave density

In this study it became apparent that the 245 g/m^2 3k carbon fibre twill weave yields lower surface porosity contents than the 200 g/m^2 3k twill weave prepreg containing the same resin. The higher aerial weight of the fibres for the same bundle size (3k) yields a more tightly woven fabric, which is expected to cause this difference. A 285 g/m^2 3k carbon fibre twill weave is also available on the market, this may prove to be even more effective in reducing surface porosity and should therefore be investigated.

However, in the production of the floor panels it was found that the laminate containing two 3k plies (245 g/m^2) consistently yielded a higher surface porosity content than the 3k/12k laminate (245/420 g/m^2). It was expected that this would be the other way around because the 3k ply has a lower out-of-plane permeability, which helps to reduce surface porosity that originates from out-gassing of entrapped air in X-core. This was confirmed by measurements in the tile mould, but it seems that this may not generally be true for all processing conditions. When a new process window study is conducted it is recommended to consider the effect of laminate composition as a dependent variable.

Finally, all samples prepared in this study were flat panels whereas X-core is especially suitable for manufacturing complex shapes. On a curved mould surface the fabric can conform to the shape of the product by a shearing deformation of the fibre bundles in the weave. This allows the weave to locally stretch or contract, but it may also affect the local out-of-plane permeability of the weave and hence the surface porosity content. Flat panels are easier to manufacture, but for subsequent research it is recommended to also make samples with more complex shapes to assess how that affects surface porosity.

13.3. Bulk porosity

This research mainly focusses on surface porosity, but it is not known how the processing conditions affect the bulk porosity (void volume fraction) of the laminate. The bulk porosity affects the mechanical properties of the laminate and is therefore important to consider. It is expected that, without the application of a barrier layer, the plies closest to the core may suffer from a large amount of bulk porosity. It is recommended to visually inspect the laminates of the different samples that were made in this study with an optical microscope or Scanning Electron Microscope (SEM). As such the bulk porosity can be correlated to the processing conditions, similar to how this research is conducted.

13.4. Integrally heated tooling

Two major points of concern in the Integrally Heated Tooling (IHT) were identified in this research: temperature homogeneity and dimensional stability. The latter is actually more related to the materials that were used to manufacture the mould. Continued research using the tile mould that was manufactured for this study is not recommended. A new mould needs to be developed that uses a resin system with a higher thermal stability for continuous use at 120°C.

The temperature homogeneity problem in the tile mould was partly solved by using 1 mm thick aluminium tooling plates between the mould lids and the sample. This works well for flat panels but becomes problematic for complex products that are to be manufactured in a laminated composite mould. Therefore, it is recommended that different strategies are explored to homogenize mould temperature:

- **Multiple heating zones:** Make use of multiple loops of resistance wire (carbon braids) combined with multiple temperature controllers. As such the mould is separated into smaller zones that are independently heated, enabling more accurate temperature control.
- **Increased thermal conductivity:** A higher thermal conductivity of the mould material helps to homogenize the temperature at the mould surface. Metals are strong conductors (aluminium, copper), the use of fibre metal laminates can offer the solution for relatively simple components; components with a single curved surface or a double curved surface with large radii. For more complex shapes a woven copper mesh can be embedded into the laminate.

13.5. Permeability of the mould flanges

Every X-core product is manufactured in a completely enclosed mould, which needs one or more parting lines to allow the product to be removed. There is no vacuum in the X-core process, the only driving force to remove air from the prepreg is the pressure that is exerted by the expanding core. This air needs to be removed from the laminate, but this also raises an important question: where does the entrapped air go? It is expected that it leaves through the parting lines of the mould, which means that the location and permeability of the parting lines may affect surface porosity. A study is recommended whereby the permeability of the gap between the tile mould lids and walls is varied, this may then be related to changes in surface porosity. The gap can be sealed completely with a gasket, or it can be made more permeable by placing dry fibres or peelply at the mould lid/wall interface.

13.6. Optical inspection set-up

The fixture for positioning the camera, LED panel light source and the sample bed worked well for repeatedly determining the porosity content of the tile mould samples, but it was not as easily used for inspecting floor panels. It does offer great potential for quality control in the manufacturing process, but that requires a redesign of the fixture. A new design is proposed whereby a frame that only holds the LED panel and the camera can be moved over the surface of the sample/product. The main benefit is that the correct alignment is already known from the set-up that is described in this report, so the new jig does not need to be adjustable. Simply translating the jig over the product allows for photographing different parts of the surface, the size of the component is no longer a constricting factor.

13.7. Floor panel production

The floor panel production process has been altered in this research for the purpose of improving the surface finish of the panels. However, mitigating all surface porosity is not possible with the current tooling system because the DU content needs to be kept as low as possible to prevent excessive deflection of the mould lids. A redesign of the mould is suggested whereby the two floor panels are stacked on top of each other, which reduces the area of the mould by a factor two. It is also recommended to use a sandwich construction for the mould lids rather than a stiffened plate. These changes can help to achieve a stiffer mould design for the same weight (~30 kg), which allows for using a higher DU content.

Additionally, the process of achieving a 100% degree of mould filling turned out to be a time consuming procedure for the floor panels. Overfilling the mould cavity and removing excess material by scraping it off was very difficult, for the floor panels scraping tends to remove the material that is inside the cavity as well. It is recommended that a different procedure is devised for the manufacture of the floor panels. This is new procedure should save processing time and help to achieve a consistent 100% degree of mould filling.

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A

X-core processing procedures

A.1. Weighing, metering and mixing of the constituents

In this section a step-by-step procedure is provided for the fabrication of an uncured mouldable X-core foam. The mixing is done manually, it requires a mixing cup, spatula, measuring cylinder and a digital balance with a 0.1 g or smaller accuracy. In terms of personal protection it is advised to use nitril or vinyl disposable gloves and respiratory protection with a filter for hydrocarbon based volatiles. The procedure for making X-core is as follows:

1. Determine the amount of constituents that is required based on the desired volume fractions and the total required volume of X-core. The density of the resin, DU and Q-cel are 1150 g/L, 370 g/L and 160 g/L respectively. DE is measured in millilitres, this has been standard practice because it has a very low density. For the resin a weight ratio of 100:60 of 4010 to 3399 is required.
2. Weigh the correct amount of 4010 resin and 3399 hardener using the digital balance and mix the two.
3. Zero the balance and add the required amount of DU microspheres to the epoxy, mix them thoroughly; the DU tends to lump together but it needs to be dispersed homogeneously.
4. Add the required weight of Q-cel to the mix. After mixing for some time it becomes a highly viscous paste that may be kneaded.
5. Measure the required volume of DE in a measuring cylinder, see Figure A.1. This part of the process is most prone to error, care must be taken to do this correctly. DE has a very low density, it easily goes airborne and it takes a long time for the microballoons to settle. Careful handling, respiratory protection and proper ventilation are advised. When adding the DE to the measuring cylinder it is necessary to 'tap' the cylinder, this helps to evacuate the air and settle the microballoons. The cylinder is firmly tapped with the spatula until the DE level in the cylinder has stabilized - there is no visible change in volume after tapping a few more times. This process is then repeated until the desired volume is reached. The first time the tapping may take ~30 seconds because of the large amount of volume that is added.
6. Carefully add the DE to the X-core mixture and slowly knead it into the X-core paste to prevent losses. Once all of the DE is sticking to the X-core paste it is thoroughly kneaded manually, making sure that all of the components are adequately mixed.

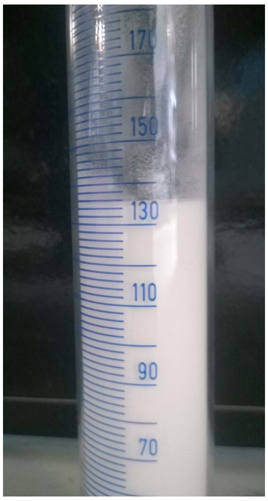


Figure A.1: *Measuring DE in a glass measuring cylinder.*

At this stage the X-core is ready for moulding. The pot life has not yet been investigated, but from experience in experiments and in production it is known that X-core is at least still workable after up to 3 hours at room temperature.

A.2. Moulding of X-core

X-core kan be kneaded and shaped manually to conform to the shape of the structure. In the current process it is desirable that X-core fills the entire mould cavity, which is challenging when the shape is complex. An example of this is the door window frame; having one part of the mould does not define the entire contour, it is only when all mould parts are combined that the product is defined. But, combining all parts leaves no room to add the X-core to the mould. The solution to this problem is to use a transparent mould for X-core, the concept is illustrated in Figure A.2.

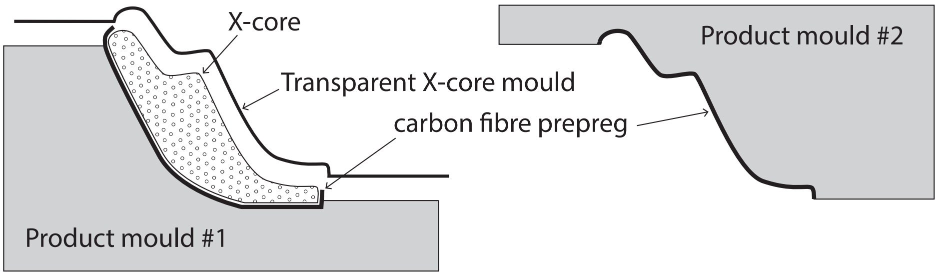


Figure A.2: *Schematic of the moulding process of complex X-core structures*

In this procedure the carbon fibre prepregs are first placed on the mould surfaces. Excess prepreg is placed around the contours of the product in the mould halve where the X-core is to be applied, this allows the prepregs from both halves to overlap and to be co-cured. An excess amount of X-core is added manually, it is placed such that it has a coarse resemblance to its final shape. Subsequently the transparent X-core mould is placed on top and tapped in place. The tapping allows the excess X-core to flow out and this can be checked visually because the mould is transparent; it is simply a thin glass-fibre epoxy cover. The X-core mould is then removed, leaving the correctly shaped core. The other mould part that contains the prepreg is placed on top, which finalizes the moulding process.

B

Tooling for sample production

B.1. X-core sample mould

The X-core sample mould is used to manufacture 130x60x30 mm X-core samples, it has been used extensively in previous years to make samples for compression tests and density measurements. A technical drawing of the mould is show in Figure B.1. It consists of 15 mm thick aluminium walls sandwiched between two 3 mm thick steel plates (only one is shown in the drawing), it is bolted shut with 40 steel M6 bolts and nuts. It has six compartments and there are two of these moulds, allowing for the simultaneous production of 12 samples. The heating is done in an industrial oven. This mould has been used in the past for pressure measurements, it has proven to be at least capable of coping with a 10 bar pressure without resulting in a visible deformation of the samples.

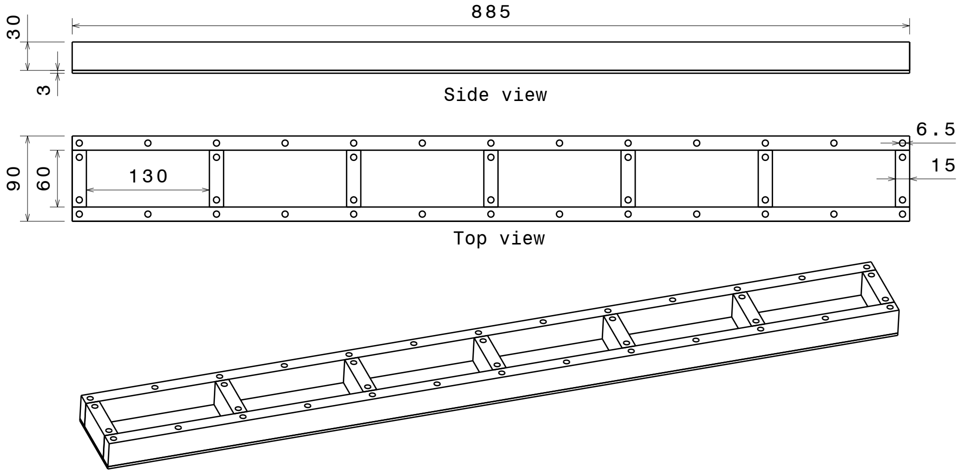


Figure B.1: *Side view, top view and isometric view of the X-core sample mould, dimensions are in millimetres.*

B.2. Floor panel mould

The floor panel mould is a 100x65 cm sandwich of steel and aluminium plates that define the shape of the passenger and driver side floor panels, see Figure B.2 for an exploded view of the CAD model. This figure shows the original design of the mould, nowadays there are two heating elements that are sandwiched between the mould lids and the floor panels. The lids of the mould are locked down by means of eight steel beams and eight M12 bolts, see Figure B.3.

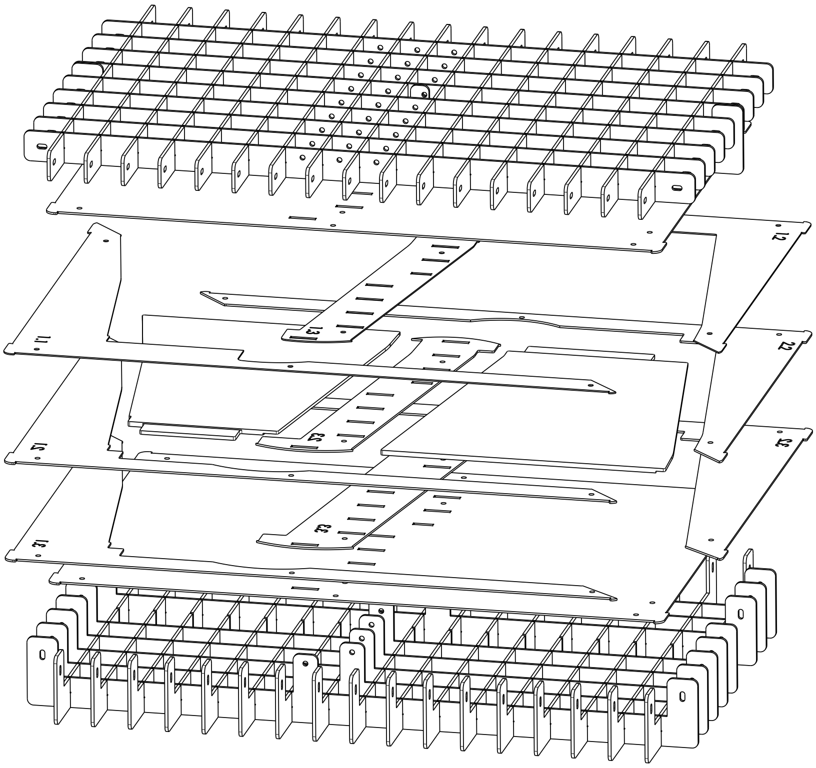


Figure B.2: *Exploded view of the floor panel mould CAD model.*

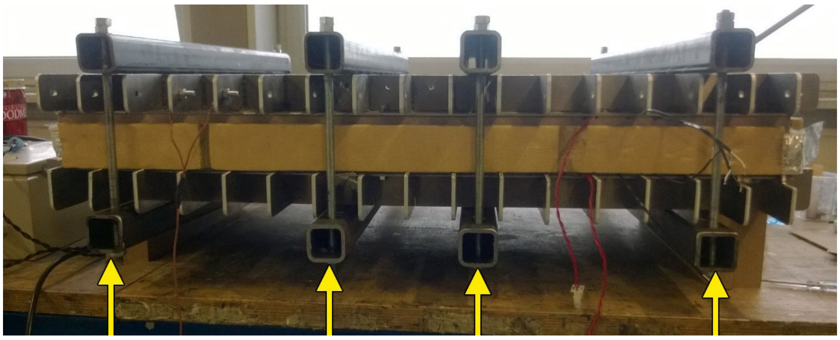


Figure B.3: *Photo of the floor panel mould in use, the arrow indicate the positions of the reinforcing bars that lock down the mould lids with eight M12 Bolts.*

B.3. Floor panel sample mould

For the manufacture of small samples it was decided to use a discarded driver side floor panel in which pockets can be made that can fit the samples. This allows for the use of the Twenco PI temperature controller combined with the integrally heated floor panel mould to control the cure cycle of the samples. The driver side was chosen because this side of the tooling contains the thermocouples for temperature control. A photograph of the modified floor panel is shown in Figure B.4.

Figure B.4: *The floor panel sample mould, a discarded driver side floor panel with four 67 mm diameter holes in which ~8 mm thick cylindrical samples can be manufactured. The numbers in the figure correspond to the numbering system used for the samples.*

This mould allows for the simultaneous production of four samples. The number is limited to four to limit the size of the area that is covered by the pockets. Temperature measurements in the floor panel mould have indicated that the temperature difference between the hottest and coldest location may be as high as 30°C [34]. So keeping the area that is covered by the samples small provides more certainty that they each experience a similar temperature and a similar cure cycle. Their location is chosen such that the centre of these four holes coincides with the location of the thermocouples in the integrally heated mould.

The diameter of each cavity is 67 mm, the thickness is approximately 7.5 mm. The floor panel is not perfectly flat, the pressure is exerted by X-core from inside the mould, the surface area of the panel is relatively large ~0.2 m^2 and the lids of the mould were not stiff enough to cope with the force during production of this panel. The consequence is that the samples vary in thickness: sample 1 is 8.0 mm thick, sample 2 is 7.25 mm thick, sample 3 is 7.6 mm thick and sample 4 is 7.25 mm thick.

C

Porosity reference chart

This appendix serves as a visual reference for surface porosity. Eight photographs of a variety of samples that were manufacture in the tile mould are presented, their surface porosity contents range from 0% to 8%. The binary image that is used to determine the porosity content is also included.

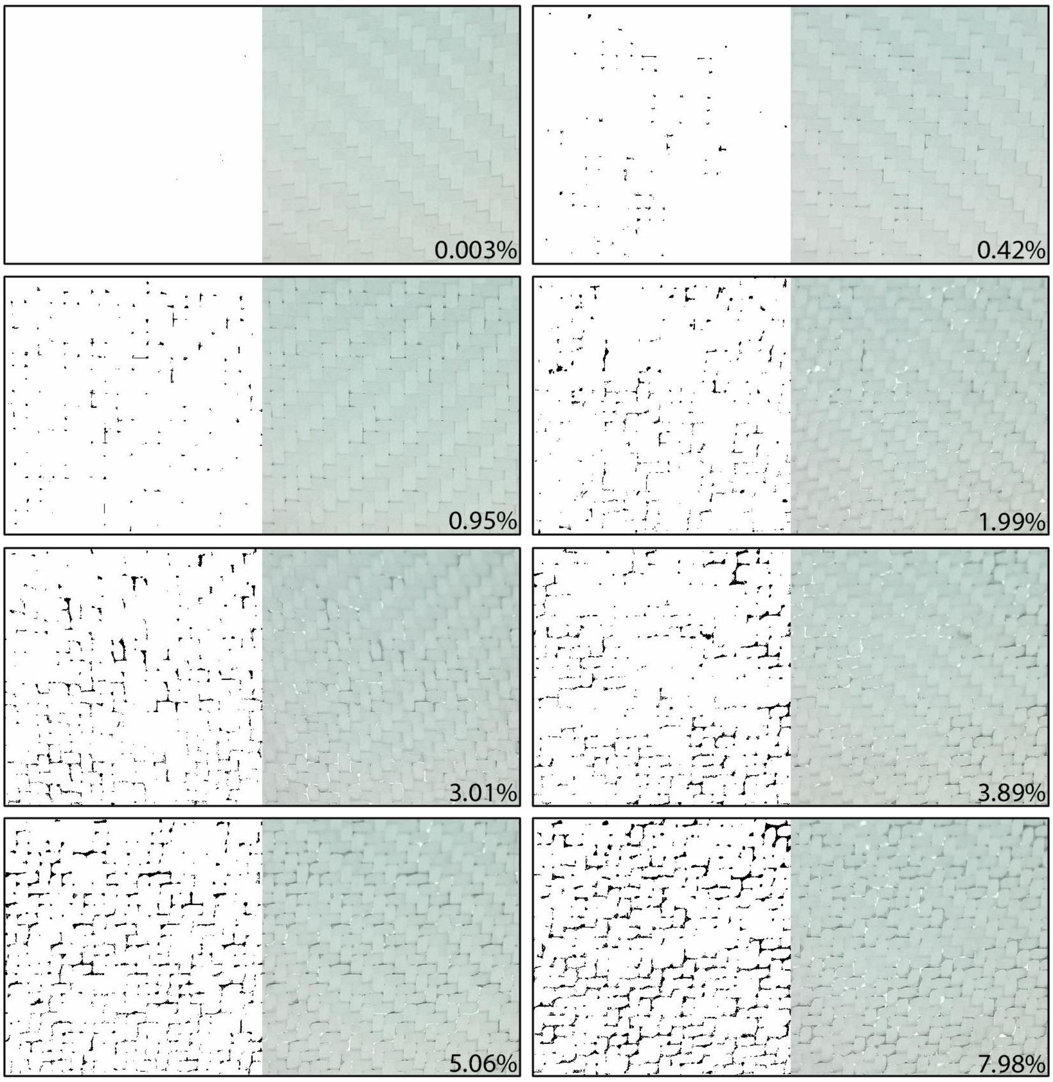


Figure C.1: *Photographs of samples and binary images including the corresponding surface porosity content.*

D

Test results

D.1. Naming convention of samples

In this report the samples are identified by a certain test code. Most often it is a three-part name containing information on the test series, the mould that is used and the sample number in the test series. Figure D.1 shows two examples of sample names and their explanation.

The first measurement series were all designated with ’SP’, these tests are covered in Chapter 6. In all following Chapters the naming convention for samples is changed: The test number is incorporated into the test name. A description of the different test names is provided in Table D.1. The mould names that are used are ’FP’, ’SM’ and ’TM’, they refer to the Floor Panel (sample) mould, the X-core Sample Mould and Tile mould. These tooling systems are covered in Appendix B, with the exception of the tile mould, which is covered in Section 5.4.

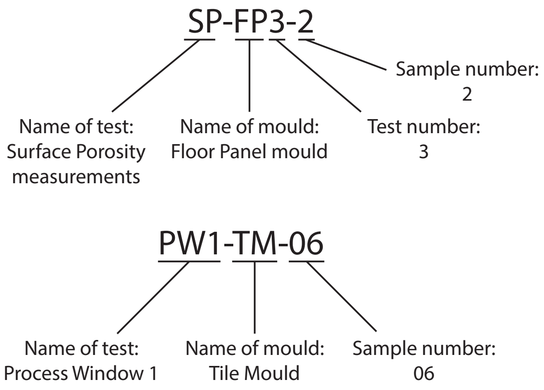


Figure D.1: *Explanation of naming convention for samples*

Table D.1: *The different test names and the corresponding description*

Test name	**Description**
SP	Surface Porosity, exploratory tests into the effect of processing conditions on surface porosity
XC	X-core Cycle tests, benchmark test following the standard X-core cycle
PW1	Process Window study 1
BP	Black Pigment, pigmentation of X-core
DF	Dry Fibre, tests with a dry glass fibre fabric between the core and laminate
VB	Vacuum film Barrier, tests with a vacuum film between the core and laminate
FP	Floor Panel, either small scale in the Tile Mould (TM) or full scale in the Floor Panel mould (FP)
Aged	Aged, samples that show symptoms of ageing
PW2	Process Window study 2

D.2. Thickness measurements PW1 samples

In process window study 1 (PW1) the samples are all thicker than the intended value (*h*) due to deflection of the lids of the tile mould. The thickness at the centre of each of the four inspection regions is measured, these four values are then averaged and are provided in the table below.

Table D.2: *Thickness measurements of process window 1 samples.*

Sample id	h [mm]	DU content [vol%]	Measured thickness [mm]	Difference [mm]
PW1-TM-01	25	4.5	28.85	3.85
PW1-TM-02	10	4.5	12.35	2.35
PW1-TM-03	40	1.5	42.35	2.35
PW1-TM-04	10	7.5	14.30	4.30
PW1-TM-05	40	7.5	45.08	5.08
PW1-TM-06	25	1.5	27.53	2.53
PW1-TM-07	25	7.5	29.50	4.50
PW1-TM-08	40	1.5	42.03	2.03
PW1-TM-09	10	1.5	10.98	0.98
PW1-TM-10	25	4.5	28.33	3.33
PW1-TM-11	10	7.5	13.33	3.33
PW1-TM-12	40	4.5	42.80	2.80
PW1-TM-13	40	7.5	45.13	5.13
PW1-TM-14	10	1.5	11.98	1.98
PW1-TM-15	40	4.5	42.83	2.83
PW1-TM-16	25	7.5	29.80	4.80
PW1-TM-17	10	4.5	11.70	1.70
PW1-TM-18	25	1.5	26.55	1.55

D.3. Detailed photos of test SP-FP2

In test SP-FP2 four samples are made in the floor panel sample mould. The goal of the test is to assess what the effect is of introducing a barrier layer between X-core and the laminate. The samples are compared qualitatively through visual inspection. This appendix provides detailed photographs of the samples to support the observations.

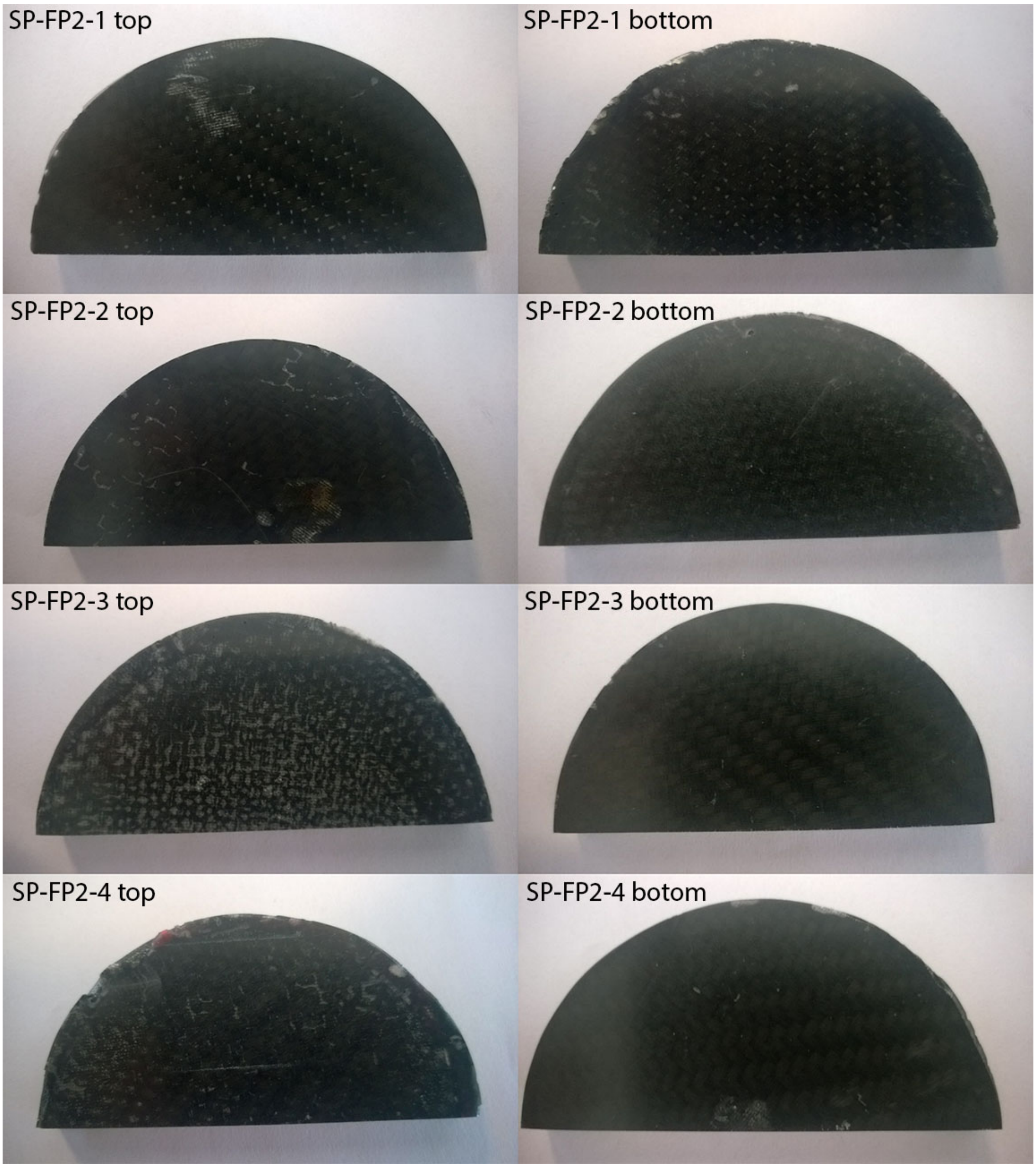


Figure D.2: *Photographs of the samples in test SP-FP2*

D.4. Three-point flexure test results

In this appendix the load-displacement curves and photographs of the failed specimens of the three-point flexure tests are presented. The description of the test set-up is provided in Section 5.2.2. The samples that are tested are samples DF-TM-01-ref, DF-TM-01, BP-TM-0%, BP-TM-5% and BP-TM-10%. These samples are described in more detail in Sections 8.2.2 and 8.4 respectively. Five test samples were extracted out of each of the parent samples, they are name based on the definition in Table D.3. This table also provides the calculated shear strengths of each sample.

Table D.3: *Shear strengths of the three-point flexure test samples.*

Flexure test series	DFR	DF	NP	5P	10P
Parent sample	DF-TM-01-ref	DF-TM-01	BP-TM-0%	BP-TM-5%	BP-TM-10%
Shear strength #1 [MPa]	3.11	2.55	3.36	3.44	3.47
Shear strength #2 [MPa]	3.03	3.00	3.68	4.03	3.85
Shear strength #3 [MPa]	3.48	3.21	3.57	4.10	3.73
Shear strength #4 [MPa]	3.53	3.30	3.90	3.81	3.59
Shear strength #5 [MPa]	3.36	3.41	3.87	3.75	3.60

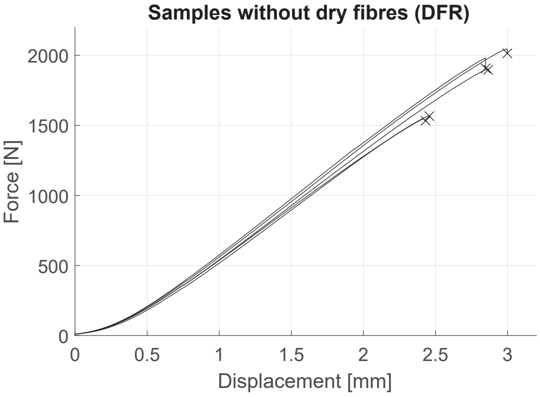


Figure D.3: *Force displacement curves of reference samples without dry glass fibre barrier layer.*

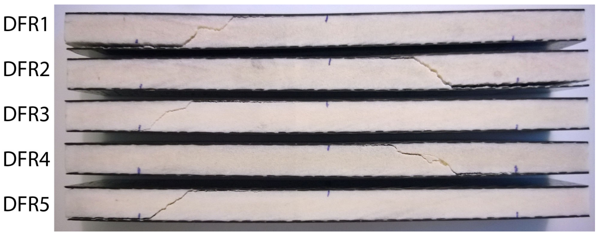


Figure D.4: *Photographs of tested reference samples without dry glass fibre barrier layer.*

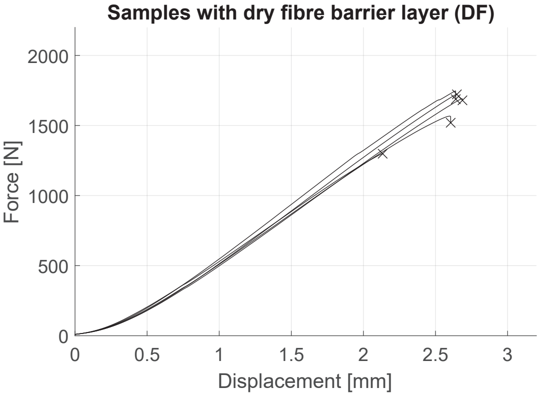


Figure D.5: *Force displacement curves of samples containing a dry glass fibre barrier layer.*

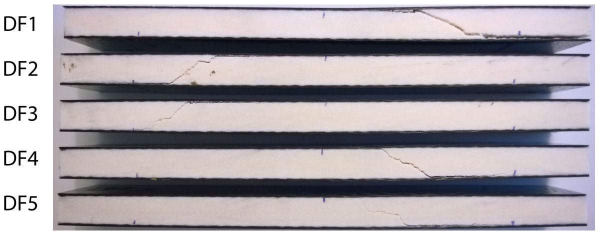


Figure D.6: *Photographs of tested samples containing a dry glass fibre barrier layer.*

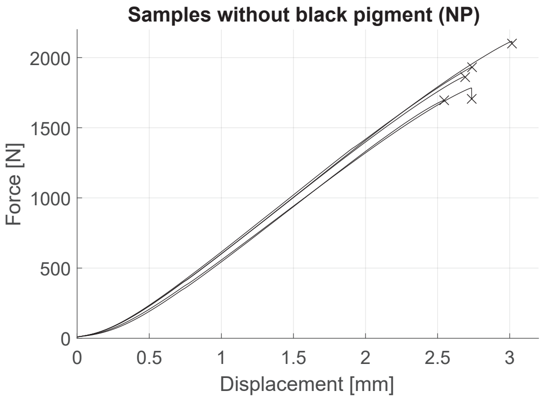


Figure D.7: *Force displacement curves of reference samples without black pigment of X-core.*

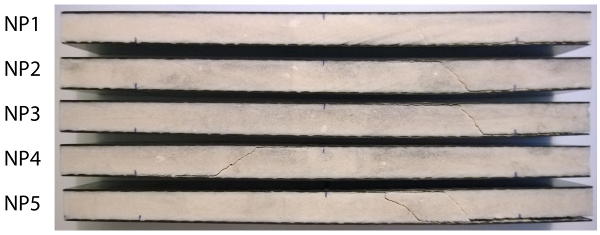


Figure D.8: *Photographs of tested reference samples without black pigment of X-core.*

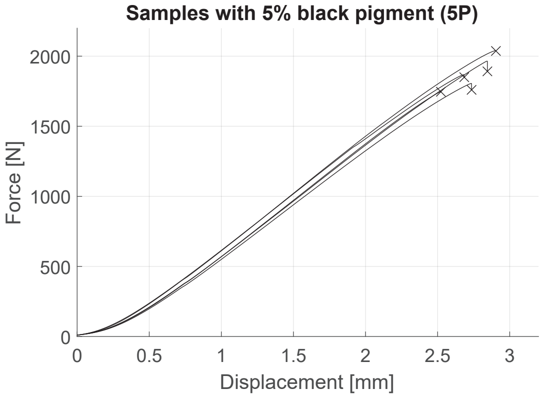


Figure D.9: *Force displacement curves of samples containing 5 wt% black pigment in the epoxy of X-core.*

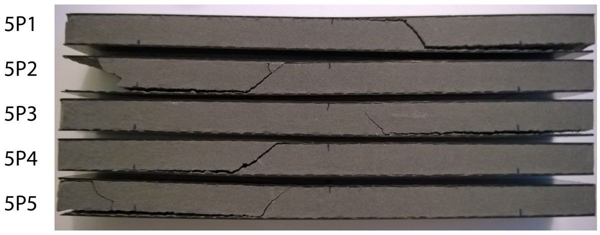


Figure D.10: *Photographs of tested reference samples containing 5 wt% black pigment in the epoxy of X-core.*

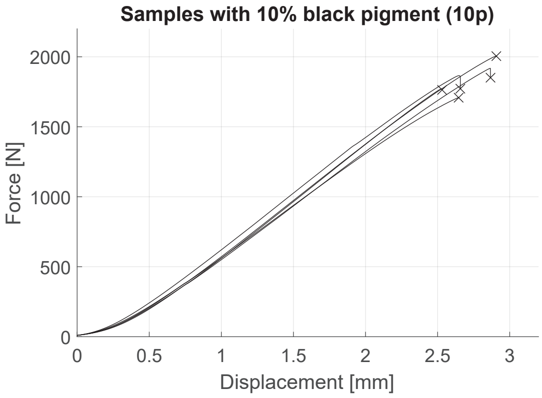


Figure D.11: *Force displacement curves of samples containing 10 wt% black pigment in the epoxy of X-core.*

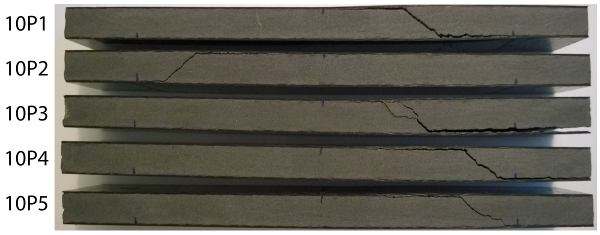


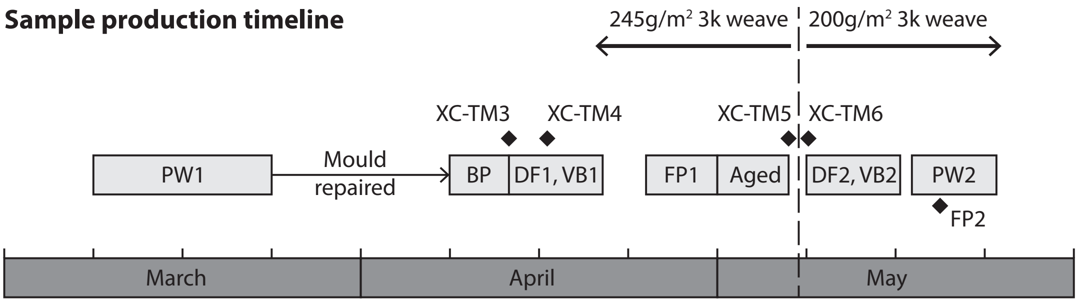
Figure D.12: *Photographs of tested samples containing 10 wt% black pigment in the epoxy of X-core.*

E

Timeline of sample production

Figure E.1 shows the timeline of events during the production of samples. It starts in March 2017 with Process window study 1 (PW1), which is covered in Chapter 7. After PW1 one of the tile moulds was repaired because it had become apparent that there was a lack of stiffness. It was then decided to postpone the validation of PW1 and to first work on the pigmentation (BP, see Section 8.4) and dry fibre barrier layer tests (DF, see Section 8.2.2). These samples are required to be flat because they are to be subjected to three-point flexure tests. During this stage samples XC-TM3 and XC-TM4 are also manufactured, these were part of the discussion in Chapter 7. XC-TM3 is the benchmark sample for 'good surface quality'.

By the time the first vacuum film barrier layer samples (VB, see Section 8.2.3) were finished Donkervoort needed a new set of floor panels for a car in production. Therefore by the end of April the focus moved to making floor panel samples in the tile mould (FP-TM tests) followed by a set of floor panels, which is discussed in Chapter 11.



Name	Description	Sample ID
PW1	Process window study 1	PW1-TM (18 samples)
BP	Black pigmentation tests	BP-TM-0%,5%,10%
DF1	Dry fibre barrier layer	DF-TM-01-ref & DF-TM-01
VB2	Vacuum film barrier layer	VB-TM-01-ref & VB-TM-01
FP1	Floor panel samples and floor panel production	FP-TM-01,02,03 & FP-01
Aged	Overaged prepreg samples	Aged-TM-01,02,03,04,05
DF2	Dry fibre barrier layer	DF-TM-02-ref & DF-TM-02
VB2	Vacuum film barrier layer	VB-TM-02-ref & VB-TM-02
FP2	Floor panel production	FP-02
PW2	Process window study 2	PW2-TM (5 samples)

Figure E.1: *A timeline of the sample production for different studies*

After the production of floor panels the validation of the model of process window 1 had priority. After two samples it started to become apparent that there was a problem: the porosity is much higher than predicted by the model. Three more samples were prepared with varying processing conditions but the porosity was consistently higher than expected. At this stage of the research it was identified that the problem was related to an overaged prepreg: the 3K surface layer. It was therefore replaced, but only a $200\,\mathrm{g/m^2}$ variant with C-M-P CP0093 was readily available. With this new prepreg additional barrier layer tests are performed (DF2 and VB2) and a new process window study (PW2, see Chapter 9).