

Characterization of high-pressure resin transfer molding process variants for manufacturing high-performance composites

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Fraunhofer-Institut für Chemische Technologie ICT
Joseph-von-Fraunhofer-Straße 7
76327 Pfinztal (Berghausen)
Telefon 0721 4640-0
Fax 0721 4640-111

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Fraunhofer-Informationszentrum Raum und Bau IRB
Postfach 80 04 69, 70504 Stuttgart
Nobelstraße 12, 70569 Stuttgart
Telefon 07 11 9 70-25 00
Telefax 07 11 9 70-25 08
E-Mail verlag@fraunhofer.de
URL <http://verlag.fraunhofer.de>

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Characterization of high-pressure resin transfer molding process variants for manufacturing high-performance composites

Submitted to the Faculty of Mechanical Engineering
of the Karlsruhe Institute of Technology
for the award of Ph.D. (Dr.-Ing.)

Dissertation

by

M. Sc. Raman Chaudhari

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Second reviewer:	Prof. Dr.-Ing. Andreas Bueter
Third reviewer:	Prof. Dr.-Ing. Frank Henning
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Preamble

ॐ गणेशाय नमः



वक्रतुण्ड महाकाय सूर्यकोटि समप्रभ

निर्विघ्नं कुरु मे देव सर्वकार्येषु सर्वदा

Dedicated to Shri Ganesha and my family

I am thankful to the god Ganesha for giving me enough strength and motivation.

This doctoral thesis was completed during my work as a research employee at the Fraunhofer Institute for Chemical Technology (ICT) in Pfinztal, Germany.

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Karlsruhe, October 2013

Raman Chaudhari

Abstract

Abstract in English

The current developments in the Resin Transfer Molding (RTM) process for the automotive industry are strongly driven by the need for automotive light-weight design and high-volume manufacturing capacity. The classical RTM process in its current status, however, is limited by the low volume production capacity of the preforming processes, long impregnation time and lack of robust injection equipment. In order to adapt the RTM process for industrial scale manufacturing of the automotive components it is essential to implement newly developed fast curing resin systems. If such resin systems shall be implemented in the RTM process, then it raises the necessity to achieve shorter resin injection time in this process which also requires use of modern equipment technology such as precise compression press and high-pressure RTM equipment. The possible resin injection time is further determined by the component geometry and requirements.

In this thesis different variants of the RTM process namely compression RTM (CRTM), high-pressure compression RTM (HP-CRTM) and high-pressure injection RTM (HP-IRTM) were studied to understand their effects on the manufacturing of high-performance composites with a goal to reduce the resin injection time. A predefined mold gap is used in the CRTM and HP-CRTM processes to obtain non compacted textiles and, if mold gap is sufficiently high, an active gap between mold surface and preform may also be obtained. Thus, the permeability inside the mold cavity is not reduced after mold closure step and resin can be injected in the mold cavity at high flow rate. In the conventional RTM and HP-IRTM processes the mold is completely closed before starting the resin injection step. This may lead to strong compaction of the textile reinforcement thereby reducing the permeability of textile reinforcements significantly and thus not allowing resin injection in the mold cavity at high resin flow rate.

The investigation of RTM process variants in this thesis was carried out using newly developed thermoset resins, and glass and carbon fiber based textile reinforcements. The important parameters such as mold gap created during the resin injection step, laminate layup, resin viscosity, resin injection rates and mold geometry were investigated through different process studies. The studies carried out using glass fiber reinforcements showed that the selected laminate layups and mold geometry affected significantly the choice of mold gap for the manufacturing of laminates of higher impregnation quality. The different studies concluded a clear advantage of using a film gate geometry, low viscosity resin (impregnation viscosity ≤ 20 mPa.s) and use of an effective gap (gap value ≤ 0.3 mm) to obtain carbon fiber

reinforced plastics (CFRP) of high quality indicating relevance for these process parameters for high-volume manufacturing of automotive components.

Abstract in German

Die Notwendigkeit zum automobilen Leichtbau und der Bedarf an hohen Stückzahlen haben neue Entwicklungsziele im Bereich der Resin Transfer Molding (RTM)-Verfahren definiert. Das klassische RTM-Verfahren ist derzeit durch geringe Produktionskapazitäten bei der Preformherstellung, lange Injektionszeiten und dem Einsatz von nicht großserientauglichen Injektionsanlagen geprägt. Um das RTM Verfahren im industriellen Maßstab für die Herstellung von Automobilkomponenten einzusetzen, ist die Verwendung neuentwickelter, schnellaushärtender Harzsysteme notwendig. Durch den Einsatz solcher Harzsysteme im RTM Verfahren müssen kurze Injektionszeiten erzielt werden, was den Bedarf an moderner Anlagentechnik, wie z.B. hochpräzisen Pressen und Hochdruck RTM Anlagen, erfordert. Die realisierbare Harzinjektionszeit wird hauptsächlich durch die Bauteilgeometrie und – anforderungen bestimmt.

In der vorliegenden Arbeit wurden verschiedene RTM-Verfahrensvarianten - compression RTM (CRTM), high-pressure compression RTM (HP-CRTM) und high-pressure injection RTM (HP-IRTM) – untersucht, um deren Einflüsse bei der Herstellung von Hochleistungsfaserverbunden zu evaluieren. Ein übergeordnetes Ziel war dabei die Reduktion der Harzinjektionszeit. Bei den CRTM und HP-CRTM Verfahrensvarianten wird ein definierter Werkzeugspalt eingestellt (je nach Spalthöhe kann auch ein Spalt zwischen Kavitätsoberfläche und textiler Verstärkungsstruktur vorhanden sein), um eine Kompaktierung der textilen Verstärkungsstrukturen zu vermeiden. Dadurch wird die Permeabilität der textilen Verstärkungsstrukturen nach dem Schließen des Werkzeuges nicht reduziert und das Harz kann mit einer hohen Injektionsgeschwindigkeit in die Kavität injiziert werden. Bei den herkömmlichen RTM und HP-IRTM Verfahren wird das Werkzeug vollständig vor Beginn der Harzinjektion geschlossen. Dies kann zu einer starken Kompaktierung der textilen Verstärkungsstrukturen und einer signifikanten Reduzierung der Permeabilität der textilen Verstärkungsstrukturen führen, was eine Harzinjektion mit einer hohen Injektionsgeschwindigkeit erschwert.

Die Untersuchungen der RTM Verfahrensvarianten in der vorliegenden Arbeit wurden mit neuentwickelten, schnellaushärtenden Duromerharzen und textilen Verstärkungen auf Basis von Glas- und Kohlenstofffasern durchgeführt. Die wichtigsten Parameter, wie Werkzeugspalt während der Harzinjektion, Lagenaufbau, Harzviskosität, Injektionsgeschwindigkeit und Werkzeuggeometrie wurden mittels unterschiedlicher Prozessstudien

untersucht. Die Ergebnisse der Studien zur Herstellung glasfaserverstärkter Lamine zeigten, dass der Lagenaufbau, die Werkzeuggeometrie und der Werkzeugspalt die Imprägnierungsgüte signifikant beeinflussen. Die Untersuchungen mit kohlenstofffaserverstärkten Kunststoffen zeigten klare Vorteile bei der Verwendung einer Filmangussgeometrie, niederviskosem Harz (Imprägnierviskosität $\leq 20 \text{ mPa}\cdot\text{s}$) und dem Einsatz eines Spaltes (Spalt $\leq 0,3 \text{ mm}$) hinsichtlich der Lamineigenschaften. Zusammenfassend kann festgehalten werden, dass alle untersuchten Prozessparameter für die großserienfähige Herstellung von Automobilkomponenten wichtig sind.

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List of Abbreviations

Terminology	Long form
AIP	away from injection point
CF	carbon fiber
CFRP	carbon fiber reinforced plastic
CRTM	compression RTM
DGEBA	diglycidyl ether of bisphenol A
DP-RTM	differential pressure RTM
EV	electric vehicle
FEA	finite element analysis
FRP	fiber reinforced plastic
HEV	hybrid electric vehicle
HP-RTM	high-pressure RTM
HP-CRTM	high-pressure compression RTM
HP-IRTM	high-pressure injection RTM
HP-T-RTM	high-pressure thermoplastic RTM
ICE	internal combustion engine
ILSS	inter-laminar shear strength
IP	injection point
LCM	liquid composite molding
L-RTM	light-RTM
NIP	near injection point
PAN	polyacrylonitrile
PES	polyether sulfone
ROP	ring-opening polymerization
ROMP	ring-opening metathesis polymerization
RTM	resin transfer molding

SEM	scanning electron microscope
SRIM	structural reaction injection molding
TE-RTM	thermal expansion RTM
TPC	thermoplastic composite
UD	unidirectional
VARTM	vacuum assisted RTM

List of formulas

Symbol	Meaning	Unit
A	cross sectional area of the sample	mm ²
A_{flow}	total cross sectional area for resin flow	m ²
A_{mold}	surface area of the mold	mm ²
A_{pores}	area of all the detected pores	mm ²
A_{sample}	specific sample area	mm ²
B	porosity ($1 - V_f$)	%
b	sample width	mm
C	constant dependent on gating choice	-
C_1	constant	
d	fiber diameter	mm
E_{CO_2}	CO ₂ discharge	CO ₂ /km
E_f	flexural modulus	MPa
E_t	tensile modulus	MPa
F	maximum tensile force	N
$F_{flexural}$	maximum flexural force	N
F_{shear}	maximum measured shear force	N
h	sample thickness	mm
K	permeability of the textile preform	m ²
L	sample support length	mm
l	characteristic length of the mold	mm
M	vehicle mass	kg
m_{fibers}	reinforcement fiber material weight	kg
m_{sample}	composite sample weight	kg
P	injection pressure	bar

Q	total amount of flow	kg
T	mold filling time	s
V	resin flow front velocity	m/s
V_{cavity}	cavity volume	mm ³
V_f	fiber volume fraction	%
$V_{fibers\ in\ cavity}$	volume of fibers in cavity	mm ³
$Void\ content_{areal}$	areal void content	%
η	resin viscosity	mPa.s
$\frac{dp}{dx}$	pressure gradient	bar/m
ψ	fiber weight content	%
ρ_{fibers}	fiber material density	kg/m ³
ρ_{matrix}	matrix material density	kg/m ³
σ	tensile strength	MPa
σ_f	flexural strength	MPa
τ	inter-laminar shear strength	MPa
$\rho_{fabrics}$	areal density of fabric	g/m ²
ρ_{fibers}	density of fiber material	g/m ³
η	resin viscosity	mPa.s

1 Introduction

The transport sector in its current status is one of the largest sources for greenhouse gas emissions. The European Commission has proposed to set a CO₂ emission limit on vehicle manufacturers for new automobiles registered in the European Union in order to reduce the CO₂ emissions from running automobiles. If this is exceeded, the concerned manufacturer will be liable for financial penalties. The core of this legislation is the so-called limit value curve, whose slope is such that manufacturers of heavier vehicles must achieve higher percentage reductions in emissions than manufacturers of lighter vehicles.

$$E_{CO_2} = 130 + 0.457(M - 1289)$$

Equation 1.1

According to equation 1.1 the CO₂ discharge (E_{CO_2}) of a new car with a mass (M) of 1289 kg must not be more than 130 g CO₂/km by 2012. A target value of 95 g/km of CO₂ is also set for 2020 for the new passenger vehicle fleet which is 26 % reduction compared to 2012 norms [1]. The weight of automotive structures if reduced can significantly improve the fuel efficiency and more importantly reduce the environmental impact by reducing CO₂ emissions. The automotive weight reduction by 100 kg leads to a fuel savings of 0.35 l/100 km and 8.4 g CO₂/km which is a huge environmental and economic gain [2]. The fuel efficiency of the conventional vehicles can be improved by developing further the efficiency of internal combustion engines (ICE) and implementing vehicle design strategies using light-weight materials thus leading to less fuel consumption. Apart from legislations demanding for lower emissions and also considering that the oil as a finite resource, the global automobile manufacturers are also developing electric vehicle (EV) and hybrid electric vehicle (HEV) concepts to meet the requirements of the transportation industry in future. The EV though has been invented since 19th Century was limited in applications due to short distance travel capacity of the vehicle [3]. Hence, in order to achieve longer drive distance with a defined battery module capacity the implementation of light-weight materials and concepts for automotive components is highly essential in case of EV.

The research on different materials indicate that the potential of automotive body light-weighting can reach 7 % after structure optimization, high strength steel use can allow further weight reduction, full aluminum body can lead to 30-50 % weight reduction and by using fiber reinforced composites further weight reduction can be achieved [4]. The strategic roadmap for light-weighting using different materials as published by one of the German automotive manufacturer is shown in Figure 1.1 [5]. As can be seen, continuous fiber reinforced composites, especially carbon fiber reinforced plastics (CFRP), can be used for achieving highest possible light-weighting which further can be optimized by adapting suitable fiber

orientation (quasi-isotropic or unidirectional) depending on component performance requirements. Also, the recent research activities are focused on multi-material vehicle design concepts which allow using right material at the right place to get the best material properties for the given requirement of the respective automotive component [5, 6].

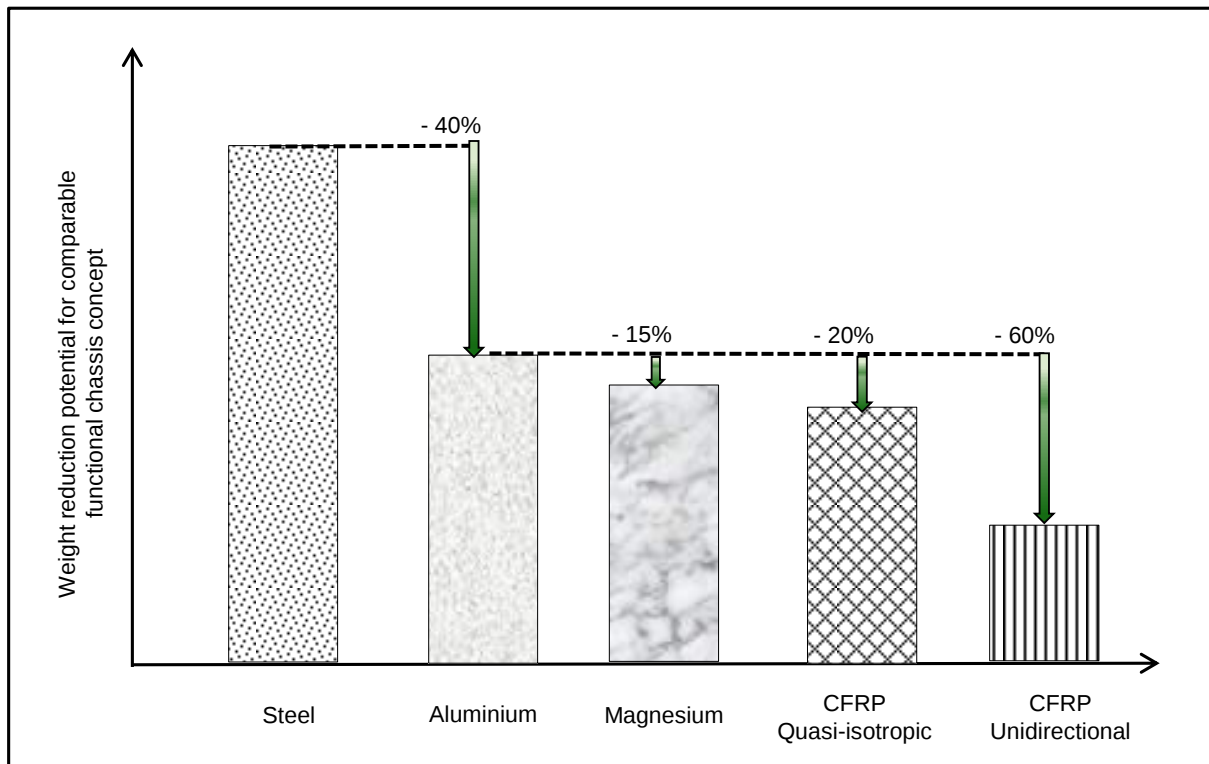


Figure 1.1: Weight reduction potential for comparable functional chassis concept by using different materials [5]

The continuous fiber reinforced composites can be manufactured by either infusion process in which the textile reinforcement is impregnated by using suitable resin during component manufacturing or by prepreg process in which the reinforcements are pre-impregnated prior to component manufacturing. The resin transfer molding process (RTM), one type of infusion processes, has been considered and evaluated for manufacturing structural automotive components based on continuous fiber reinforced composites [7]. However the established RTM processes are still limited to low-volume manufacturing capacity. In order to adapt the RTM process for high-volume manufacturing, it is essential to develop new process variants allowing reduction of the process cycle time. The work in this thesis is investigating alternative in-mold process variants for the RTM process with the goal of establishing high-volume manufacturing concept for continuous fiber reinforced components.

2 Research motivation and goals

For manufacturing of the high-performance composites for automotive applications various manufacturing routes have been extensively studied which may utilize either thermoplastic or thermoset based matrix materials [8 - 10]. The selection of a particular manufacturing route for high-performance composites is strongly dominated by various requirements such as component geometry, physical and mechanical properties of the component, costs and required production volume per year [11]. In recent years thermoset matrix based high- pressure resin transfer molding process (HP-RTM) has gained enormous attention of automotive industry for manufacturing structural automotive components with cycle times ≤ 5 minutes [12]. This doctoral thesis aims at characterization of alternative RTM process variants for high-volume manufacturing of high-performance composites having fiber volume content between 50 – 60 %. The automotive components manufacturing by RTM process is associated with different technological barriers, when compared to metal based manufacturing of the same component, and these technological barriers need to be conquered to implement this process successfully for high-volume manufacturing [13]. One of the technological barriers is to achieve impregnation of the textile reinforcements in shorter time in RTM mold. Recently a variety of fast curing resin systems for manufacturing automotive components have been developed. However, in order to use these resin systems, alternative process concepts shall be studied and developed to achieve short resin injection time while manufacturing such high-performance components. This technological challenge formed the main motivational background for development and optimization through evaluation of new generation of RTM process variants to correlate their applicability for high-volume manufacturing of high-performance composites in this doctoral thesis. The research work was executed by using modern generation of equipment technology to create a know-how basis for achieving a link between basic research and industrialization of the RTM process chain. The scientific and technological work in this thesis is associated with following goals:

1. Selection and adaption of modern equipment and mold technology for large scale manufacturing
2. Characterization of different newly developed thermoset resins for their use in the selected RTM process variants
3. Implementation of highly reactive resin systems (cure time ≤ 5 minutes) especially to target need for high-volume manufacturing of automotive components
4. Variation of the RTM processes to correlate their applicability for obtaining quick resin injection in the mold cavity

5. Investigating the effect of materials and process parameters on the morphology and mechanical properties of the manufactured high-performance composites
6. Use of different mold geometries to correlate their influence on the RTM process parameter selection and parameter optimization
7. Development of suitable resin injection gate geometry for the RTM process and investigation of their influence on component quality
8. Correlation of process concepts for high-volume manufacturing

3 State of the art resin transfer molding process

In this chapter basics of the resin transfer molding (RTM) process are described. The typically used reinforcement materials and preform manufacturing technologies, infiltration process variants, matrix materials, equipment technology and aspect affecting mold design for the RTM process are discussed in detail.

3.1 Reinforcement materials

For RTM processes a variety of reinforcements can be used. The commonly used fiber types and their properties are mentioned in Table 3.1. Typically for manufacturing of continuous fiber reinforced composites by RTM processes glass, carbon and aramid fibers are used due to their properties, availability and processing viability.

Table 3.1: Types of commonly used fibers and their basic mechanical properties [14]

Fiber type	Density [g/cm ³]	Tensile modulus (GPa)	Tensile strength (MPa)	Elongation at break [%]
Glass fibers				
E-Glass	2.54	80	3500	4.0
S-Glass	2.46	90	4500	5.7
Carbon fibers				
HT- carbon fibers	1.78	240	3750	1.6
HM- carbon fibers	1.85	400	2450	0.7
UHM- carbon fibers	2.00	540	1850	0.4
Aramid fibers				
Kevlar 49	1.45	135	3500	2.8
Kevlar 149	1.47	185	3400	2.0

Most of the composite applications are based on use of glass fibers (E-glass or S-Glass) as reinforcement due to their desirable price-performance ratio. As mentioned in Table 3.1 S-glass offers higher tensile strength and modulus than E-Glass. Carbon fibers are less widely used than glass fibers due to their higher cost. Different types of carbon fibers produced using polyacrylonitrile (PAN) as a precursor are available on the market. These carbon fibers differ from each other in terms of their production conditions and thus resulting tensile strength and modulus. Due to their excellent properties carbon fibers are often used for manufacturing of light-weight components in aerospace or high-end applications. Aramid fibers, if compared to glass and carbon fibers, offer very high toughness. Thus aramid fibers are often used for applications where high energy absorption is required. Glass or carbon fiber based reinforcements can be used in different forms (chopped fibers, continuous roving,

random mat or uni-/bidirectional fabrics etc.) for RTM applications. For a complex RTM component it might be necessary to manufacture an intermediate fiber preform instead of using the reinforcements directly in the mold for composite manufacturing.

3.2 Preform manufacturing technologies

RTM processes belong to the class of liquid composite molding processes. The process generally involves two important process sub cycles, namely *preform manufacturing* and *infiltration process* (also referred as *infusion process*). For preform manufacturing various state of the art technologies have been established and their selection in the RTM process chain is determined by the component (part to be manufactured) parameters such as required fiber orientation, required fiber volume content, geometrical complexity and performance of the component. Two methods are commonly used for the preform manufacturing namely, *direct preforming* and *sequential preforming*. The methods for *direct preforming* include standard processes such as fiber spraying, programmable Powdered Preforming Process (P4) and water slurry method, and three dimensional (3D) textile processes such as 3D weaving, 3D braiding and knitting processes [15-16].

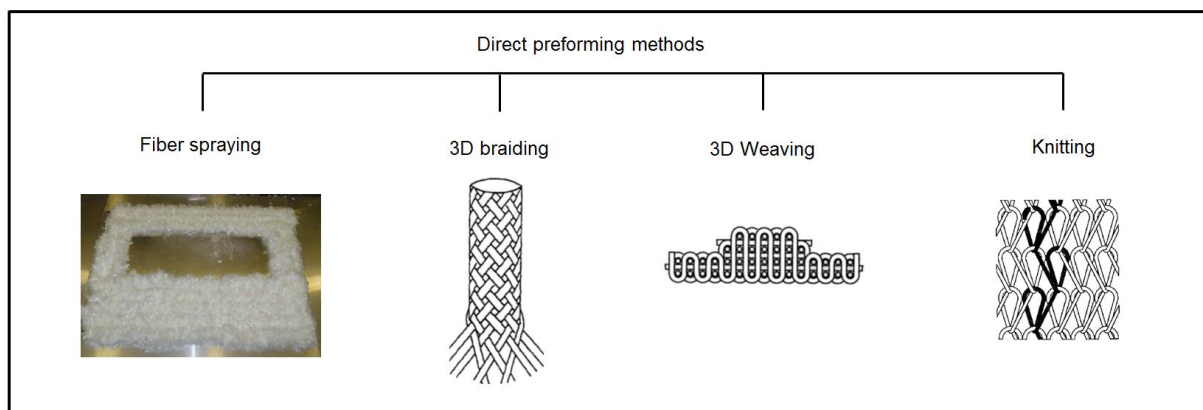


Figure 3.1: Structure of reinforcements obtained by direct preforming methods [14, 17]

The methods for *sequential preforming* are based on forming of textile semi-finished products in which binder based or textile based forming methods are implemented. The commonly used semi-finished textile reinforcements for the sequential preform manufacturing are shown in Figure 3.2. As can be seen in this figure, fiber ondulation and draping ability of the woven textile reinforcements is determined by their structure itself. The non-crimp fabrics if compared to the woven fabrics show comparatively lower ondulation [17]. Researchers are also investigating development of new generation of textile reinforcements to assist the preform manufacturing process while considering the process economy. Such new generation of textile reinforcements includes development of hybrid reinforcements in which different fiber materials (e.g. glass, carbon and/or aramid fibers) are combined to optimize

the performance and cost of the reinforcement to the required component. Integration of a metal mesh in the textile structure during its manufacturing by weaving process has been also evaluated as an alternative to classical preforming (adhesive based). The metallic mesh in such textiles can be used to assist the preforming process for inducing permanent plastic deformation of the mesh [18, 19].

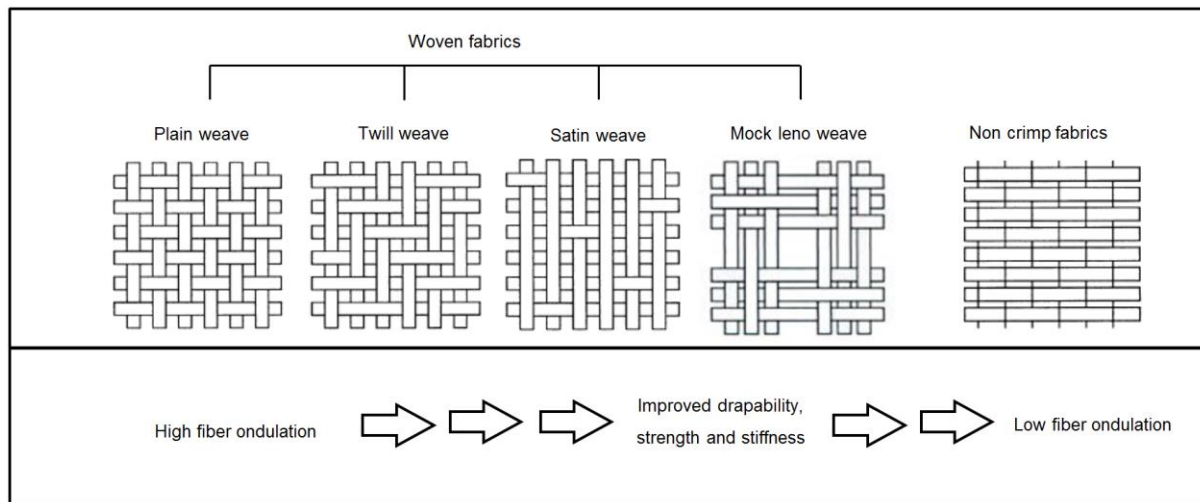


Figure 3.2: Commonly used semi-finished textile reinforcements for sequential preform manufacturing [17]

The detailed process steps in the RTM process chain based on *sequential preforming* are shown in Figure 3.3. For contour cutting of the textile semi-finished products various solutions based on either mechanical cutting operations or thermal cutting operations can be implemented whereas their selection is strongly determined by the geometry of the semi-finished cut to be obtained. For cutting large number of constant and non-varying contours punching can be an economical process, whereas for constantly varying contours CNC based mechanical cutting operations using knives are often favored [20, 21].

Implementation of automated handling techniques is essential to transport the textile layers using suitable grippers, placement of textile layers to prepare a stack for preforming and handling of the manufactured preform to the infiltration mold. The selection of a particular handling technology in the RTM process chain is determined by ability of the handling system to pick and place textile layers without disturbing the fiber orientation. The investigations of handling technologies by different researchers conclude needle grippers, clenching grippers and vacuum grippers as most suitable for the automated handling of textile layers and these techniques are associated with respective advantages and disadvantages [20, 21].

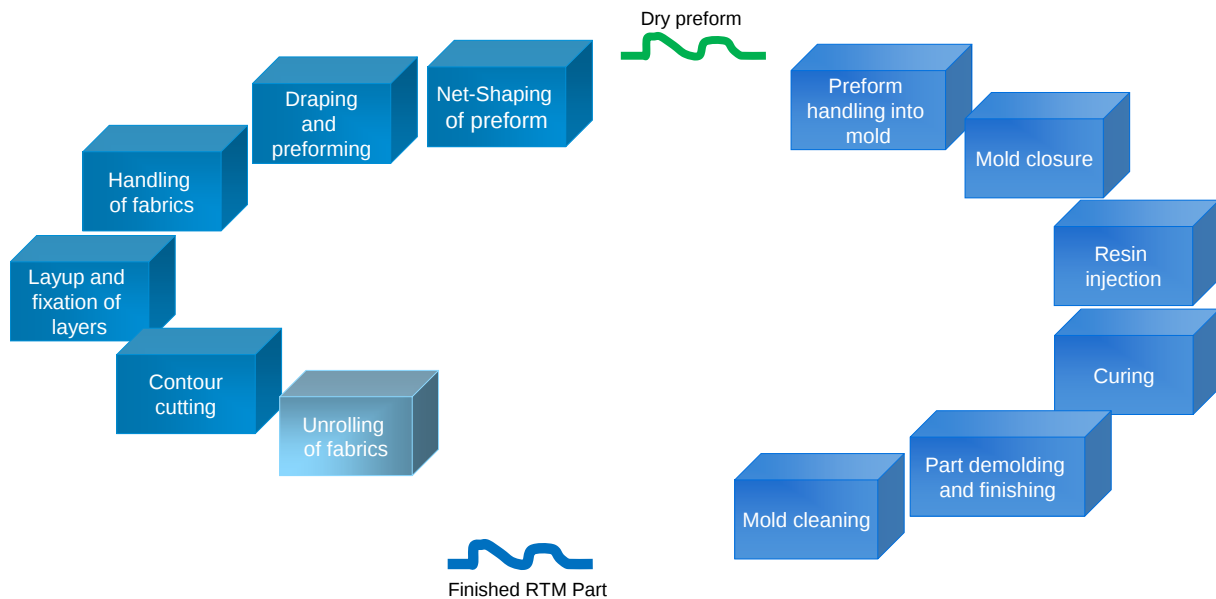


Figure 3.3: The resin transfer molding process based on sequential preforming [22]

For fixation of textile reinforcement layers after contour cutting and as an aid for preform manufacturing, different types of fixation techniques are investigated. Suitable binders can be used for stabilizing the textile reinforcements and these binders may act as a physical binder or chemical binder depending on its formulations. Investigations have shown the binders, depending on their type and formulation may affect the mechanical properties of the final component [20, 23]. Hence, new concepts for fixation of textile layers based on local injection of binders or adhesives are under investigation. If used in small quantities, such local adhesive do not affect the properties of the final composite component [24]. Stitching technologies provide an alternative to binder based fixation of the textile reinforcements. The stitching process can be used as an aid to draping, assembling of fabric stacks, fixing of contour, integration of structural elements (e.g. inserts) and for optimizing structural performance if desired [25-28].

In the draping and preforming process step the stack of textile reinforcement layers is placed into a preforming tool using suitable grippers and the layers are draped to the component geometry to be manufactured. Technologists around the globe are involved in development of robust equipment technology where different draping and preforming methods for high-volume preform manufacturing of complex components are investigated. Such complex components included development of automotive floor pan, front bumper and B-pillar for automotive applications [29-31]. For the implementation of a particular preform manufacturing technology special attention must be given for the design optimization of the component concept to achieve maximum possible light-weighting while utilizing anisotropic

laminate properties. Often for implementing continuous fiber reinforced composites a balance must be found between performance and efforts (cost and labor) associated to the resulting composite structure [32]. Further for the cost-efficient preforming of complex components, it's highly essential to implement suitable quality assurance techniques [33].

3.3 Infiltration techniques for dry preforms

3.3.1 Basics of resin flow through preform

The resin flow through the preform during infiltration process step is governed by the Darcy's law:

$$V = Q/A_{flow} = \frac{K}{\eta} \cdot \frac{dp}{dx} \quad \text{Equation 3.1}$$

Where, V is the resin flow front velocity, Q is total amount of flow, A_{flow} is total cross sectional area for resin flow, K is the permeability of the textile preform, dp/dx is the pressure gradient and η is the viscosity of the resin. For arbitrary reinforcement orthogonal directions x , y and z can be considered such that:

$$V_x = \frac{K_x}{\eta} \cdot \frac{dp}{dx}, \quad V_y = \frac{K_y}{\eta} \cdot \frac{dp}{dy}, \quad V_z = \frac{K_z}{\eta} \cdot \frac{dp}{dz} \quad \text{Equation 3.2}$$

If the permeability of the preform is known in all the orthogonal directions, then the resin flow behavior in all these respective directions can be predicted using simpler finite element analysis (FEA) methods. Often, in reality the Z-direction permeability is neglected even for thick components. As per the Darcy's law at a constant pressure gradient for a selected resin of particular viscosity, the resin flow front velocity and thus the resin injection time is strongly dependent on the permeability of the textile reinforcement.

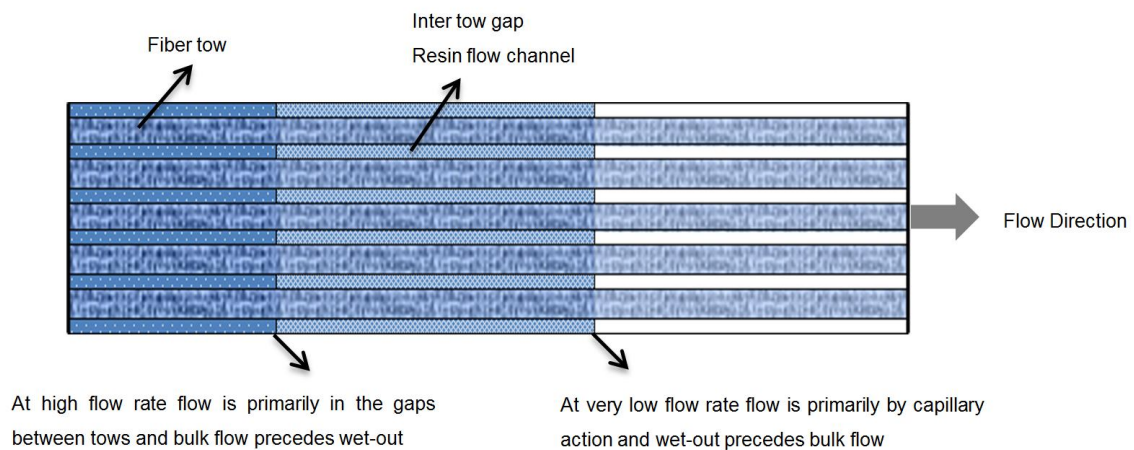


Figure 3.4: Bulk flow and wet-out position as a function of resin flow rate in the textile reinforcement [34]

In order to understand the resin flow through the textile reinforcements and defect generation (voids) during the RTM process it is highly essential to understand the bulk flow behavior, referred as macroscopic resin flow, and wet-out, referred as microscopic flow through the textile reinforcements as shown in Figure 3.4. The macroscopic resin flow is strongly determined by the applied pressure, resin viscosity and inter-roving spaces, whereas, the microscopic resin flow is affected by the capillary flow and wetting behavior of the used textiles [34].

Taking into consideration the large variety of applications of RTM processes various modifications in the process flow, process conditions and equipment characteristics are proposed so as to overcome the limitations of RTM processes. These RTM process variants include modification in flow front and flow characteristics by altering the pressure gradient and other pressure characteristics in mold.

3.3.2 RTM process variants

The common infiltration variants of the liquid composite molding (LCM), especially RTM relevant variants, are shown in Figure 3.5. All these processes involve pressure gradient driven resin infusion in static dry preform. Two widespread techniques in this process are vacuum assisted infusion processing, also referred in literature as vacuum assisted RTM (VARTM) and resin transfer molding (RTM). There are several other processes of interest, such as gap impregnation RTM and compression RTM (CRTM) as shown in the same figure which schematically compares these processes [35]. In vacuum infusion process as shown in Figure 3.5 (a), one side of the mold is rigid and second side of the mold is generally a vacuum bag. During vacuum infusion process vacuum is drawn which compacts the preform and also causes the resin to flow through the preform. Tooling costs for VARTM are generally low and large components can be manufactured. As the injection is driven by atmospheric pressure, it is usually necessary to help the resin flow by introducing a flow enhancement media which is a highly permeable layer that distributes the resin over the component surface. Even with this assistance, the filling of large structures with resin may take many hours. After the component is cured; the vacuum bag, the flow enhancement media and the tubings, etc., need to be separated from the component and discarded which is time consuming, labor intensive and generates additional production waste [36, 37].

An advancement of the VARTM process include the variants such as Light-RTM (L-RTM) and differential pressure RTM (DP-RTM) and. The L-RTM process differs from vacuum infusion process only in terms of upper mold half which generally is made up of a semi-flexible male mold. The semi-flexible mold is supported by steel frame to obtain sufficient stiffness. Like in the vacuum infusion process a rigid female mold is used as a lower mold

half. The mold closure occurs by using simple clamping mechanism. After mold closure mold is evacuated and the resin is injected up to atmospheric pressure in the mold cavity [38].

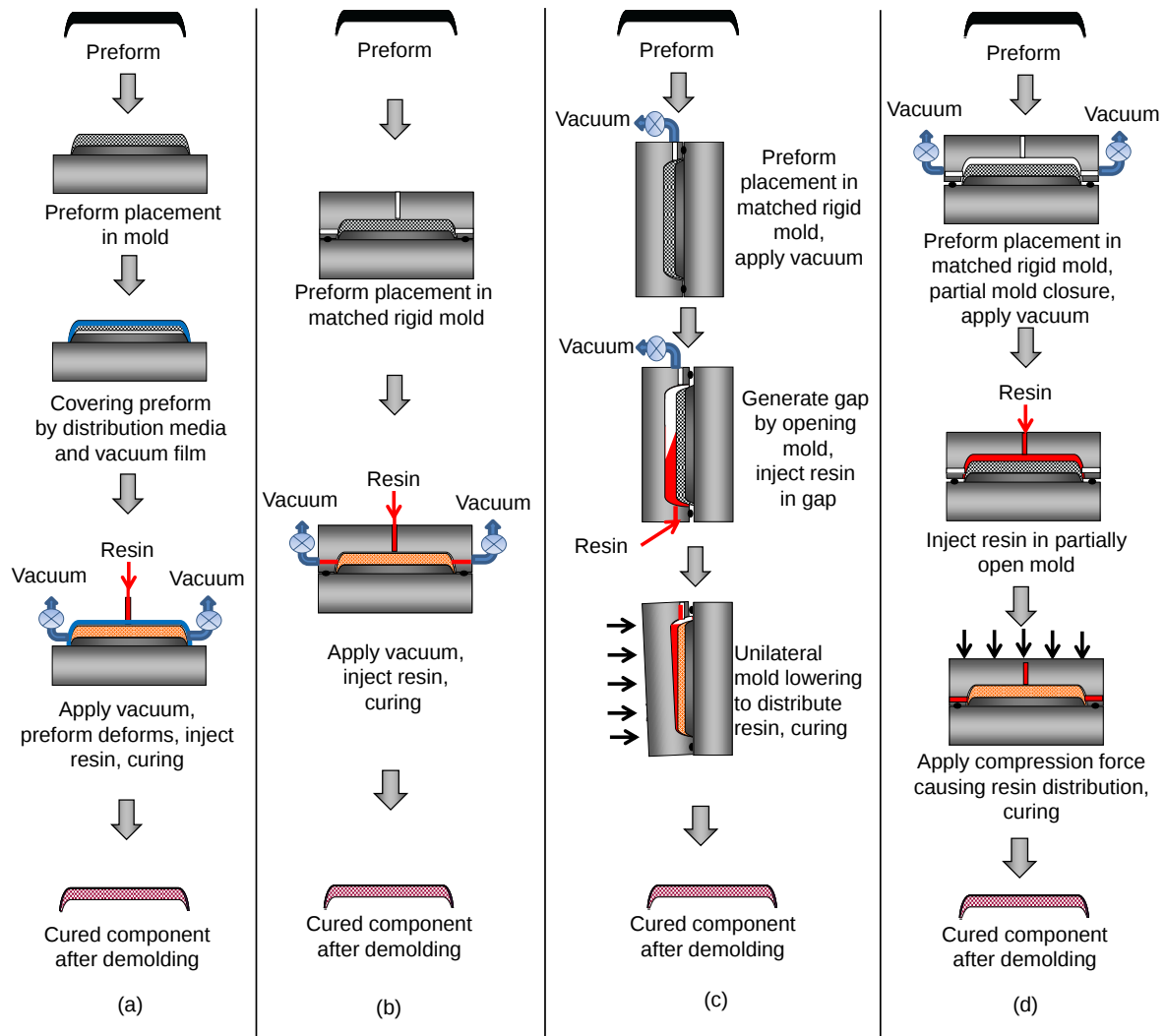


Figure 3.5: Variants of the liquid composite molding process; (a) vacuum infusion process, (b) RTM, (c) gap impregnation RTM, (d) compression RTM [35, 40]

In DP-RTM process, like vacuum infusion process, a rigid lower mold half made up of sheet metal is used and upper side of the preform is covered by a flexible material. A vacuum pump evacuates the preform which placed in an autoclave. Resin is infused into the preform under the pressure gradient created by applying vacuum. After impregnation of the preform, pressure is applied through the autoclave and the impregnated preform is held under pressure till completion of the resin cure reaction. Application of pressure helps to minimize voids in the final component. This process has an advantage over conventional RTM as

higher viscosity resins can also be used for manufacturing high-performance composites [39].

As shown in Figure 3.5 (b) in the RTM process two sided rigid mold is used. The process is thus used for manufacturing complex components with very good surface quality and excellent dimensional tolerances. The process rate is limited by the need to force the viscous resin through the compacted preform which resists the resin flow due to reduced permeability. Consequently, the injection process may take significant time [34-35]. The advanced variants such as gap impregnation RTM and compression RTM are often considered as an alternative to RTM in order to reduce the resin injection time by facilitating the flow through non compacted textile preform. As the preform is not compacted, the permeability of the preform is not reduced thereby allowing fast resin injection in these process variants.

The gap impregnation RTM, as shown in Figure 3.5 (c), consists of a fixed lower mold half (generally cavity) and movable upper mold half. Mold in this process is mounted differently (mold length oriented in vertical direction) than the conventional RTM process. The resin injection point is generally at the lower side and vacuum port is located at the highest point of the mold. A temporary flow gap is created before resin injection step and defined amount of resin is then injected in partially open mold at low flow resistance. In the next step the gap is closed by tapered closing of the upper mold half from the injection point to the other end of the mold. Due to tapered closing of mold, within few seconds the impregnation takes place which is exceptionally fast and thus this process is suitable for use of fast curing resins for component manufacturing [40, 41]. However due to complex mold closure mechanism such a process shall lead to limitations in design freedom for mold technology for manufacturing complex shaped components.

The CRTM process combines the dimensional stability and good surface quality of RTM with a fast processing cycle of compression molding. This is achieved by resin injection into a partially open mold as shown in Figure 3.5 (d). The gap between the mold and the preform is highly permeable and distributes the resin very quickly over a significant region of the surface of the component. Then the mold is closed by applying compression force, compact the component and squeezing the resin into the remaining non-impregnated regions. Obviously, the tooling requirements are higher for the CRTM process than those for other LCM processes, but the CRTM process can be used for achieving higher production rates due to a faster processing cycle [35].

Further variants of the RTM process include thermal expansion RTM (TE-RTM) and core expansion RTM. TE-RTM is a method of forming a composite sandwich core molded article, using thermo-elastic rigid foam core. As shown in Figure 3.6 (a) the core is wrapped with the

reinforcing fabric and this assembly is placed in a mold whose inner confining surfaces form the shape of the final article. In the next step resin is injected into the mold to impregnate the fabric. The mold is then heated to sufficient temperature so that the rigid foam core can expand to compress the fabric wrapped surface and final shape of the component is achieved. After resin curing the mold is cooled and component is demolded. In the mold significant pressures can be created during this process which leads to high quality, complex shaped and void free molded components [42].

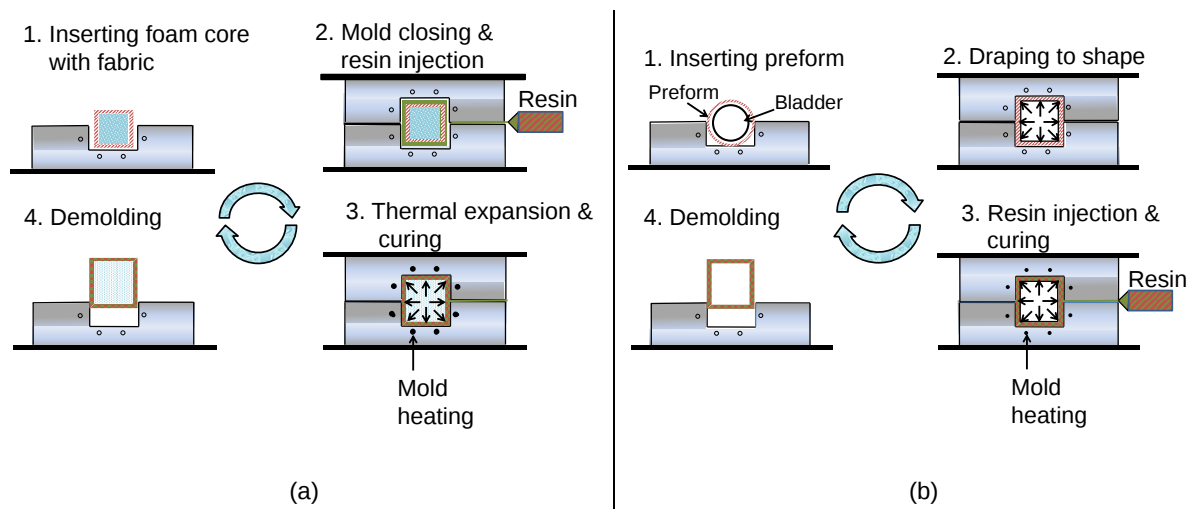


Figure 3.6: Variants of the liquid composite molding process based on in-mold expansion technique; (a) Thermal expansion RTM, (b) Core expansion RTM [40]

In the core expansion RTM process, as shown in Figure 3.6 (b), the dry reinforcement plies are layered to form the general component shape around an inexpensive (single use) thermoplastic or elastomeric (reusable) bladder. The dry preform and bladder are then placed in a female mold defining the component shape. The mold is closed and the bladder is inflated so that the preform takes the shape of the mold cavity. In the next step resin is introduced under vacuum or positive pressure through strategically located injection gate. The component is cured at room temperature or at elevated temperature by heating the mold. After demolding of component the bladder is removed or left in the component [43].

Recently, new variants of the LCM processes are also being investigated to develop an alternative to the RTM manufacturing route which are very close to the classical prepreg manufacturing process. In such processes, a pre-impregnated preform is obtained in first step and the curing of the pre-impregnated preform is carried out in second step using a press mold. The high degree of automation in these processes may lead to shorter cycle time [44, 45].

3.4 Resin systems for RTM process

A variety of the resin systems can be used for manufacturing components by the RTM process. The selection of the final resin system type depends predominantly on the requirement of the component to be produced. The RTM processes utilize polyesters, vinylester and (for aerospace applications) epoxies and bismaleimides. Polyurethane resins are used exclusively in the structural reaction injection molding (SRIM) process, which in principle is very similar to the RTM process [46]. While considering the requirements of the automotive sector, epoxies and polyurethanes are being consistently developed to meet the high-volume manufacturing requirements. In addition, thermoplastic based matrix systems are also under development to their use in the RTM process [47-49].

3.4.1 Epoxy resins

The most common epoxy resins are based on diglycidyl ether of bisphenol A (DGEBA). State of the art epoxy resins include approximately 95 % pure DGEBA. The curing of epoxies can be carried out by three different chemical routes, which involve either amine-epoxy reaction or anhydride-epoxy reaction or lewis acid catalyzed epoxy homo-polymerization.

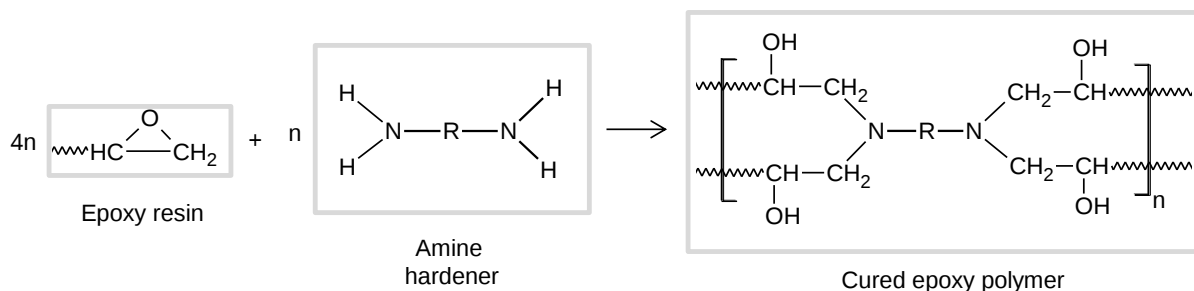


Figure 3.7: Typical amine-epoxy curing reaction [47]

A typical amine-epoxy reaction as shown in Figure 3.7 is a polyaddition reaction of an amine group with the epoxide group to form a hydroxyl group. A secondary amine may be formed and can further react with an epoxide to form a tertiary amine and an additional hydroxyl group. The continuation of the polyaddition reaction leads to formation of a cured cross-linked epoxy polymer. Such a reaction is very flexible and enables the resin formulation to be adjusted to give a wide range of properties. The fast-cure epoxy resin systems for RTM applications are typically based on amine-epoxy formulations. The latest developments in epoxy resins include the use of latent catalysts to offer long resin impregnation time and short resin curing time [47, 50, 51].

3.4.2 Polyurethane resins

Similar to the epoxy resins polyurethane resins can be obtained by polyaddition reaction of bi-functional monomers. The bi-functional monomers are based on diols and diisocyanates. During the polyurethane reaction, as shown in Figure 3.8, the functional groups react with each other and the hydrogen atom of the hydroxyl group acts with the N-atom of the isocyanate group leading to formation of low molecular weight chains. The continuation of the reactions leads to low molecular weight polymers. Polyurethanes produced exclusively using bi-functional monomers are thermoplastic whereas using tri-functional monomers (e.g. alcohols with three hydroxyl groups) creates three-dimensional cross-linked polyurethane network [47, 52].

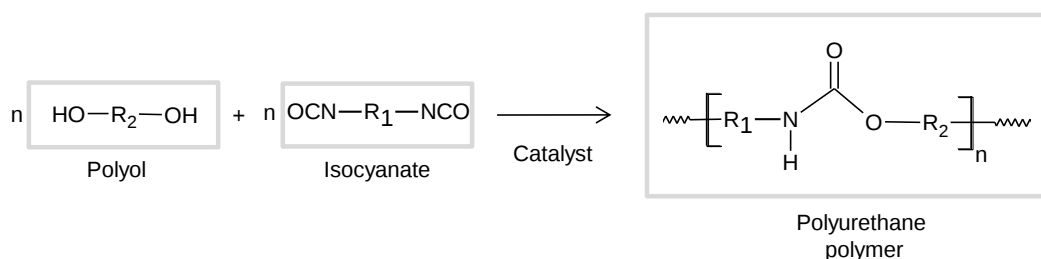


Figure 3.8: Polyol-isocyanate reaction to produce polyurethane [47]

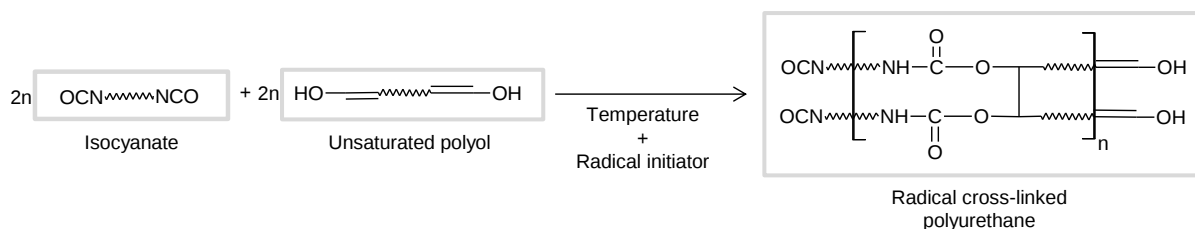


Figure 3.9: Thermoset polyurethane based on radical cross-linking reaction [53]

Further advancements in the polyurethane resins include the combination of polyurethane reaction and radical polymerization (through use of peroxides) for manufacturing structural composites as shown in Figure 3.9. The processing speed of such a resin is significantly high (due to combination of two curing reactions) and hence it is relevant for manufacturing high number of parts per year [53].

3.4.3 Reactive thermoplastic resins

For manufacturing of the textile reinforced thermoplastic composites (TPC) generally melt processing routes are used. The main disadvantage of TPCs manufacturing by melt processing is the need for high processing temperatures and pressures, because of the high melt viscosity of the matrix. An alternative solution to melt processing is reactive processing

of TPCs. In such a process, after impregnating the fibers with a low viscosity mono- or oligomeric precursor, polymerization of the thermoplastic matrix is conducted *in-situ* either by vinyl polymerization or ring-opening polymerization (ROP). Polymerization can be initiated by heat or UV radiation and might require the addition of a catalyst system, which can be added to the precursor prior to impregnation. Due to their low molecular weight, precursors have extremely low melt viscosities and proper fiber impregnation is therefore achieved without the need for high processing pressures. Moreover, through reactive processing, textile fiber reinforced TPCs can be even manufactured through low pressure infusion processes such as resin transfer molding (RTM), structural reaction injection molding (SRIM) and vacuum infusion. Anionic polymerization of lactams, shown in Figure 3.10, is the oldest and up to now the most developed way for reactive processing of thermoplastics through ROP [54].

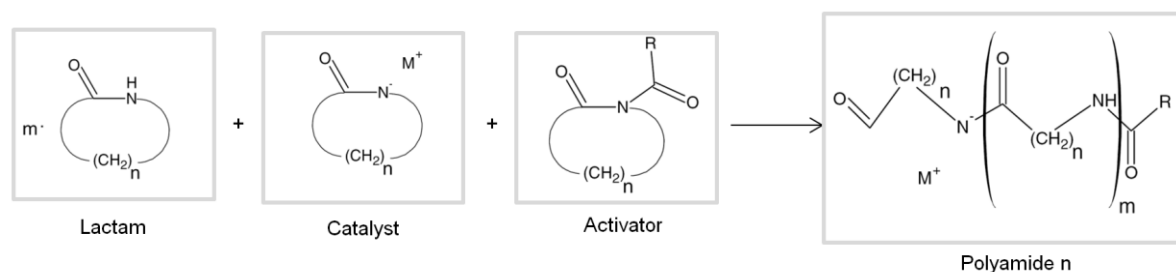


Figure 3.10: Anionic ring-opening polymerization of polyamides [54]

In the last two decades, the anionic ROP of ϵ -caprolactam to polyamide-6 (PA-6), and synthesis of macrocyclic polyesters and their ring-opening metathesis polymerization (ROMP) have been studied intensively for materials and process development to manufacture TPCs for automotive applications [47, 55-57].

3.5 Equipment for resin processing

For processing of the above mentioned resins different equipment technologies can be used to transfer the resin from the RTM equipment to the mold. Some of the most prominently used resin processing equipment and their working is explained below.

3.5.1 Pressure pot system

The pressure pot is the simplest method for processing single component resin systems in which the resin is premixed with hardener. The important elements of a pressure pot system are shown in Figure 3.11. Vacuum may be used to fill the resin via the pressure differential into the pot. After resin filling in the pot, the resin can then be degassed, and then pressurized for injection. An alternative type of pressure pot system works on the

basis that the operator places a resin manufacturer's original container directly into the degassing chamber, the resin is then degassed and pressurized for injection.

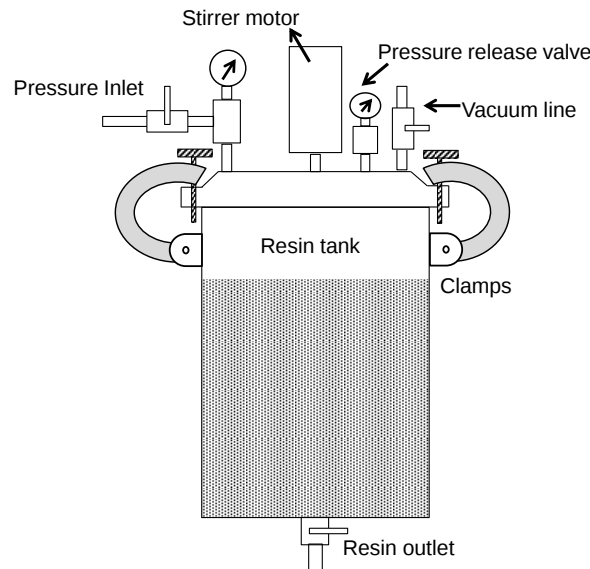


Figure 3.11: Schematic of simple pressure pot resin injection system [46]

The major limitation to the pressure pot system is that the flow rate cannot be directly controlled. Flow rate can only be controlled by adjusting the pressure in the pot and balancing it against the resistance to flow presented by the fiber preform in the mold [46, 58].

3.5.2 Two component equipment

For processing of two component resin systems, different equipment technologies have been developed for dosing, mixing and metering of the resin and hardener. A typical two component RTM equipment is shown in Figure 3.12. As can be seen, resin and hardener are stored separately in either heated or non-heated reservoirs or the reservoirs are placed in an heated cabin. Using suitable pump technology the resin and hardener fed from the reservoirs in required stoichiometric ratio to the static mixer, where resin and hardener are mixed with each other to form reactive resin mixture. The resin mixture is then injected at a predefined injection pressure in the mold cavity. One particular disadvantage of such a device is the necessity to clean the static mixer after each injection cycle by using appropriate solvent in order to avoid the reaction of resin and hardener in the static mixer which may otherwise lead to its blockage.

The single acting or double acting pneumatic piston pumps, a class of reciprocating type pumps, may be used in the inexpensive two component RTM equipment. A limitation of

single acting pump is that pumping cannot be continuous as the pump must be refilled after each injection process. Cleaning such a system automatically can present difficulties, and therefore trying to automate a manufacturing process using this transfer method would present challenges. On the other hand, double acting piston pump can pump on both the upstroke and the down stroke allowing the output to be continuous. In composite manufacturing where two components must be meter-mixed, these pumps are highly efficient and, hence, are a preferred option [46, 58, 59].

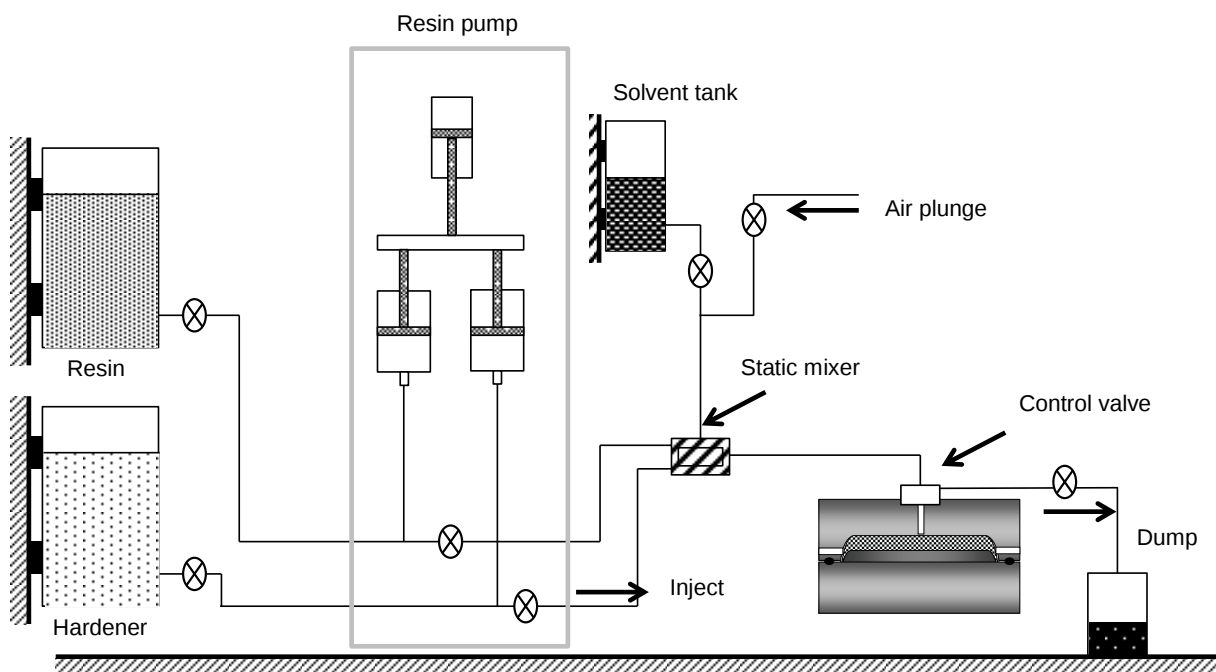


Figure 3.12: Schematic of two component RTM injection system [46]

An alternative two component RTM equipment may use a gear pump, a class of rotary positive displacement pump, instead of reciprocating piston pump system. The fluid is transferred around by toothed gears. The gears mesh forcing the fluid out of the discharge port. Although the internal geometry is highly tolerant, it is possible for material to 'slip' back through the gap between the outer casing of the pump and the gear surface. Due to its simplicity the pump can be easily flushed, however, the pump is susceptible to solid contaminants within resin systems which can result in wear, reducing the tolerance between the gears and the housing, and in turn reducing the efficiency of the pump. With simple and complete control over flow, combined with the ability to provide continuous flow and a flushable system, the gear pump provides a solution that is suitable for processing any size of composite component. Although having a lower efficiency than the other two pumping systems, the use of flow meters can permit tight closed-loop control of flow [58].

3.5.3 High-pressure RTM equipment

A further modification of two component RTM equipment for processing of highly reactive two component resin system based on impingement mixing principle is referred in the literature as structural reaction injection molding (SRIM) and high-pressure RTM (HP-RTM) process equipment. The SRIM equipment is used for processing of polyol and isocyanate, as shown in Figure 3.13, and the SRIM process is typically used for manufacturing glass fiber reinforced composites. The HP-RTM equipment technology is identical to the SRIM equipment and it is used for processing of fast-cure resin systems based on epoxy and amine hardener. The central feature of this process equipment is rapid delivery of low viscosity reactant on a metered basis through some form of mixing device, which is usually an impingement mixing head which is mounted directly in the heated mold [59-61]. In this type of resin mixing and injection equipment, the resin and hardener components are pumped separately using an axial piston pump system from the resin container to the mixing head of the equipment. Using a specially designed needle plug valve which is mounted in the mixing head of the equipment a preferred pressure from 50 to 230 bar can be set on the resin and hardener components which is maintained between dosing pumps to the mixing head of the equipment.

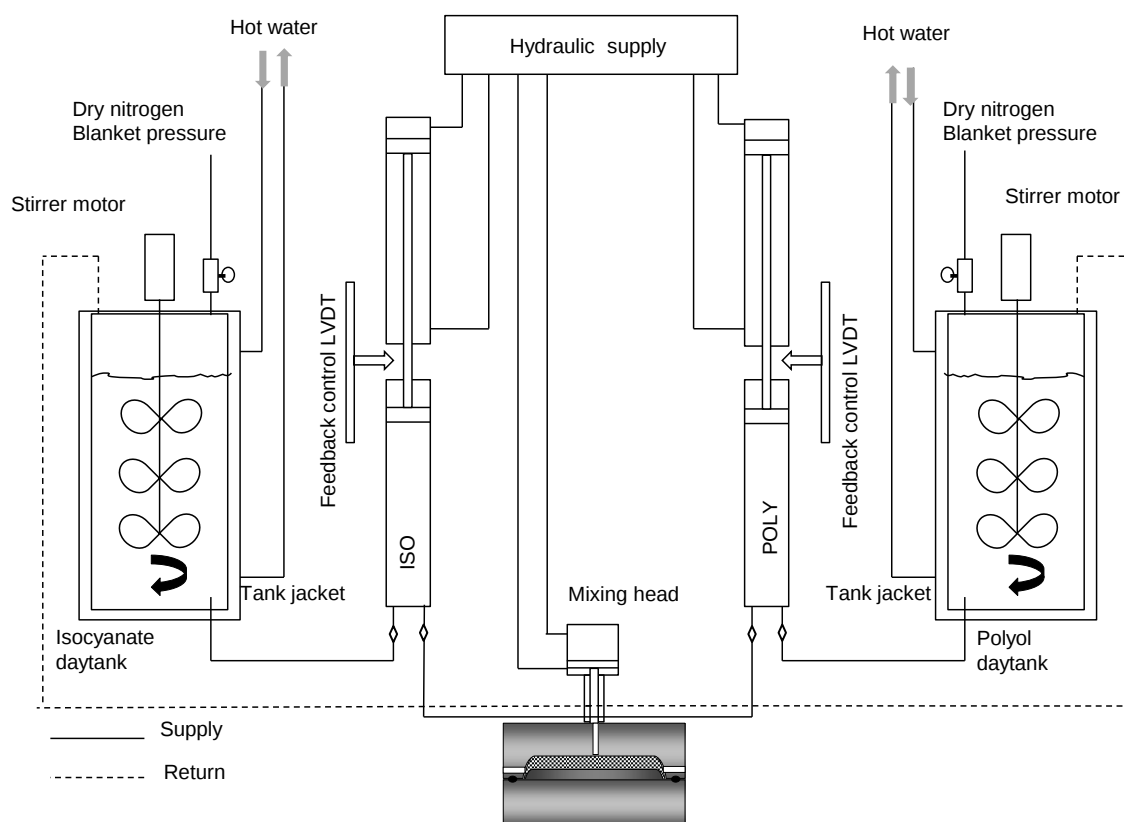


Figure 3.13: Schematic of SRIM injection system [46]

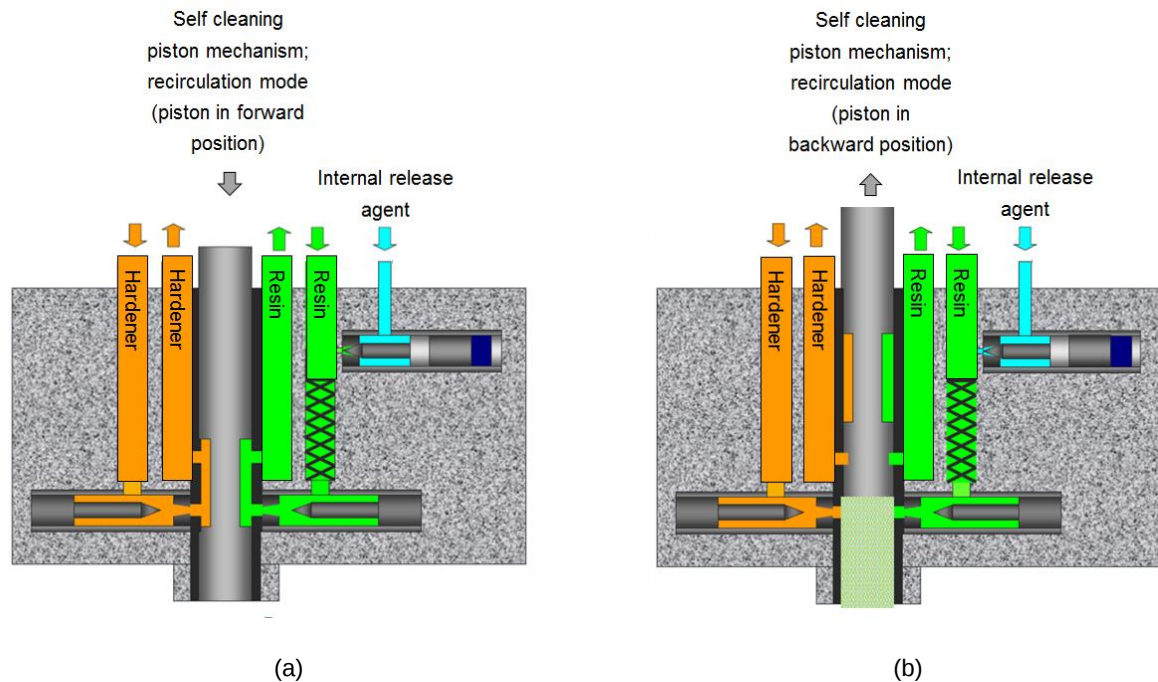


Figure 3.14: Principle of a high-pressure mixing head for impingement mixing;

(a) mixing head in recirculation mode; (b) mixing head during resin mixing and injection step [59]

The working principle of the impingement mixing head is shown in Figure 3.14. In the mixing head of the equipment a piston is constructed for re-circulating the reactive materials to the mixing head and back to the tanks periodically in order to maintain the entire system at uniform temperature. If the resin shall not be injected into the mold cavity then the piston is in forward position as shown in Figure 3.14 (a), and resin and hardener are only re-circulated. During the resin injection step the piston is moved back in backward position as shown in Figure 3.14 (b) and the circulation of the resin components is stopped. The resin components are mixed with each other into the free room of the mixing head which is otherwise accommodated by the piston if it is in the front position. After the defined amount of the resin and hardener is injected into the mold cavity over the defined resin injection time, the piston of the mixing head is moved again in forward position by hydraulic pressure thereby pushing the reactive resin and hardener mixture into the mold and the piston in the mixing head is brought back to the position as shown in Figure 3.14 (a). Thus after each resin injection cycle the mixing head is cleaned by itself and the circulation of the resin components is restarted. Also, this kind of equipment does not generate any reactive resin waste or does not require any solvents for purging during a manufacturing mode [59].

Also as it can be seen in Figure 3.14, the HP-RTM equipment allows dosing of an internal release agent if required during the resin injection step. If the internal release agent shall be dosed during resin injection step, then the pump of the internal release agent doses the required amount of release agent at the required flow rate into the epoxy component of the

resin. The epoxy resin and internal release agent are mixed homogeneously by using the static mixing element. If the equipment is circulating the resin components, the pump for internal release agent does not dose any release agent into the epoxy resin but the pump is only activated during the resin injection step to avoid any concentration increase of the release agent into the epoxy resin over the period of time. Due to the ability of the HP-RTM/SRIM equipment technology to handle fast cure resin systems, such equipment are often combined with fast acting hydraulic presses for mounting the RTM molds to reduce the overall component molding time [22].

For processing of reactive monomers for thermoplastic resins (e.g. caprolactam material system) new generation of equipment technology has been investigated and developed. Such an equipment technology is referred as reactive injection molding or high-pressure thermoplastic RTM (HP-T-RTM). In the HP-T-RTM process equipment (Figure 3.15) the caprolactam reactive material is processed as two component material system.

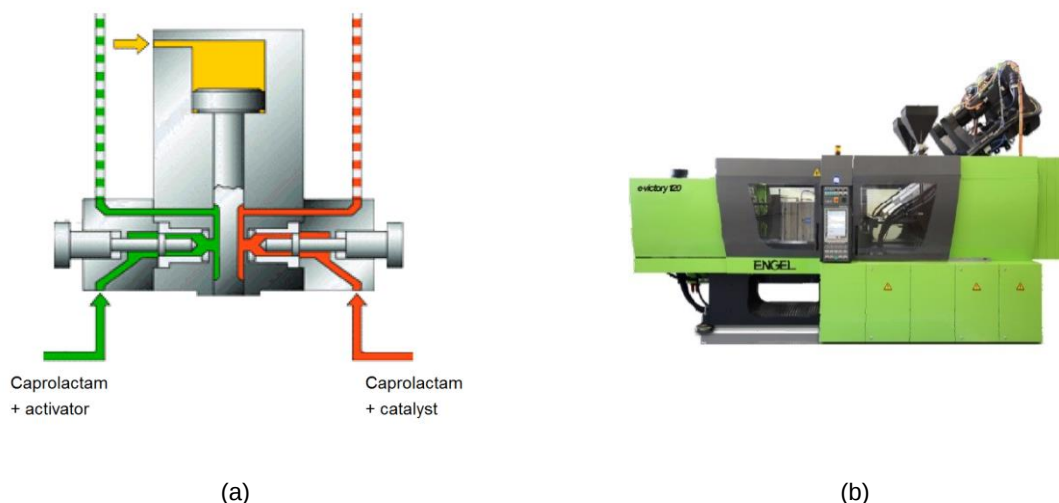


Figure 3.15: HP-T-RTM process; (a) mixing head for processing caprolactam material system; (b) ENGEL prototype equipment for in-situ processing of reactive caprolactam thermoplastic resin [63]

The first component is a mixture of caprolactam and catalyst, whereas, the second component is a mixture of caprolactam and activator. These two components are plasticized at temperature of 110-115 °C using a specially designed screw-barrel configuration. The screw is designed with a special non-return valve which allows precise dosing of low viscosity material components through a non-recirculating high-pressure mixing head. The dotted materials line in Figure 3.15 (a) indicates inactive flow of materials back to the plasticizing unit. The two components are mixed in the mixing-head using impingement

mixing principle and are injected in a mold which is mounted in a hydraulic horizontal acting clamping mechanism [62-64].

3.6 Mold technology for RTM process

The mold design for the RTM process is strongly affected by various factors related to the manufacturing route, component geometry and requirements, resin type, production volume, etc. For high-volume manufacturing of the RTM components typically matched metal molds are used. The mold design plays a significant role in determining the component impregnation quality and hence special attention must be given during mold design to decide the position of the resin injection gate, vacuum ports, fiber clamping (also referred as pinch-off) and gaskets/sealing [46, 65]. The pinch-off is simply trapped reinforcement in the mold cavity between the closed mold halves, which creates an edge restriction to the advancing resin flow.

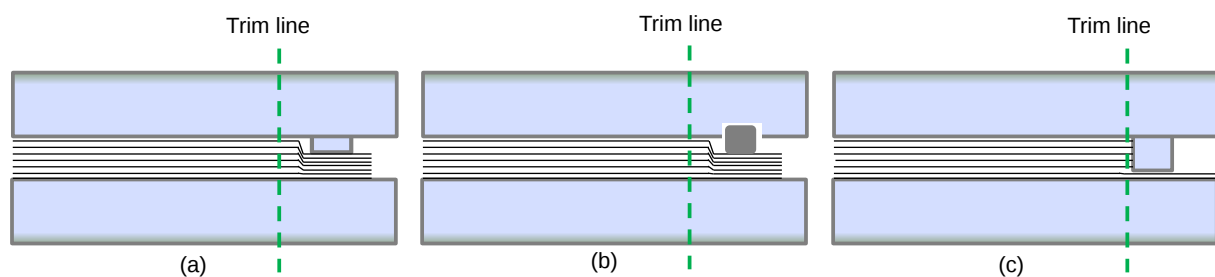


Figure 3.16: Pinch-off techniques in RTM molds; (a) Conventional pinch-off, (b) Rubber insert, (c) Partial pinch-off [46]

The different types of pinch-off mechanisms are shown in Figure 3.16. The conventional pinch-off uses machined recess in the upper mold half which causes the clamping of the fiber preform along the edges of the mold cavity. As an alternative to recess machining, a polymeric rubber based material as shown in Figure 3.16 (b) may be used in one of the mold halves to clamp the fibers. For the partial pinch-off a machined recess is used as shown in Figure 3.16 (c), however, only few predefined textile layers of the preform are clamped in this case below the recess [46].

A further important aspect of RTM mold design is the decision of sealing concept. The sealing in an RTM mold can take many shapes as shown in Figure 3.17. A very important principle that is recommended is to keep the sealing in one plane because it's very difficult to seal the mold on a curved surface in a reliable way [65].

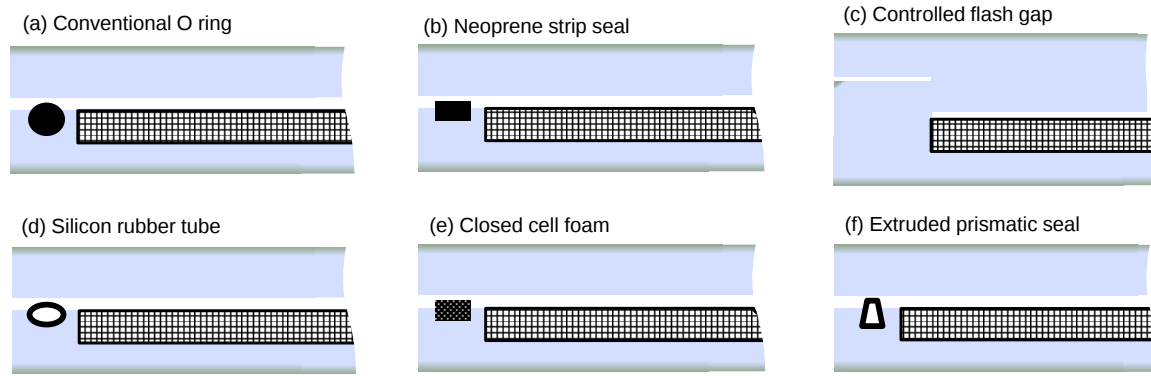


Figure 3.17: RTM mold sealing alternatives [46]

The mold-filling time and the part quality are affected by the mold-filling strategy i.e. the design of injection gate. The mold filling strategies can be subdivided into three main types: point injection, edge injection and peripheral injection. In point gate injection resin is introduced through a port in the center of the part, the resin flows essentially radially into the reinforcements, and air is vented at the periphery of the part. Edge gate injection is accomplished by injection through a film inlet at one edge of the part, the flow is more or less unidirectional over the part, and air is vented at the opposite side. Finally, in peripheral gate injection resin is introduced in a resin distribution channel. Around the periphery of the part, the flow is radially inward and air is vented at the center of the part. The relationship of mold filling time and injection gate strategy is given by following equation [34].

$$T = \frac{C\eta l^2(1-V_f)}{PK} \quad \text{Equation 3.3}$$

Where T is the mold filling time, η is the resin viscosity, C is a constant dependent on gating choice, l is characteristic length of the mold, V_f is the volume fraction in the component, P is injection pressure and K is permeability of the preform. For flow time calculations, dependent on chosen gating choice, values of C differ: for an edge gate in a constant width mold C can be taken as $1/4$, for a peripheral gate on a circular panel C can be taken as $1/16$, and for a center ported gate on a circular panel C can be taken as

$$C = 0.25(X^2 - 1 + 2 \ln 1/X) \quad \text{Equation 3.4}$$

Where, X is the injection point diameter divided by L with L being the flow length. For center ported tools, X^2 term is generally neglected as it is very small. The equation 3.3 thus indicates that the mold filling time differs considerably between the different gate strategies, with peripheral injection gate being much faster than the other two. In practice several other

phenomena may occur invalidating the equation 3.3. The selection of the gate geometry thus may also be affected by further parameters such as process safety, materials behavior, fiber-washout, race tracking, etc. [46, 65].

3.7 Prospects of carbon fiber reinforced plastics as automotive light-weight construction material

As the vast majority of carbon fiber (CF) produced, over 98 %, is processed into composite materials of all types, the carbon composite market is currently developing at the same pace as the carbon fiber market.

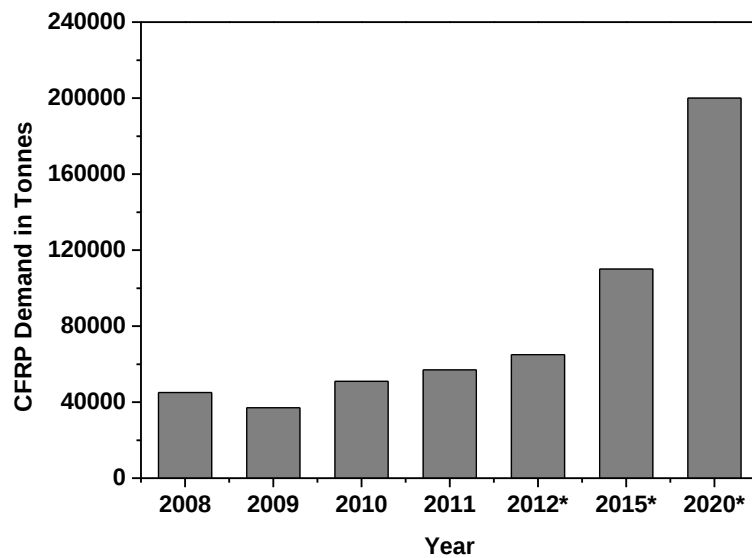


Figure 3.18: Global demand for CFRP in tonnes 2008-2020 (*estimates) [66]

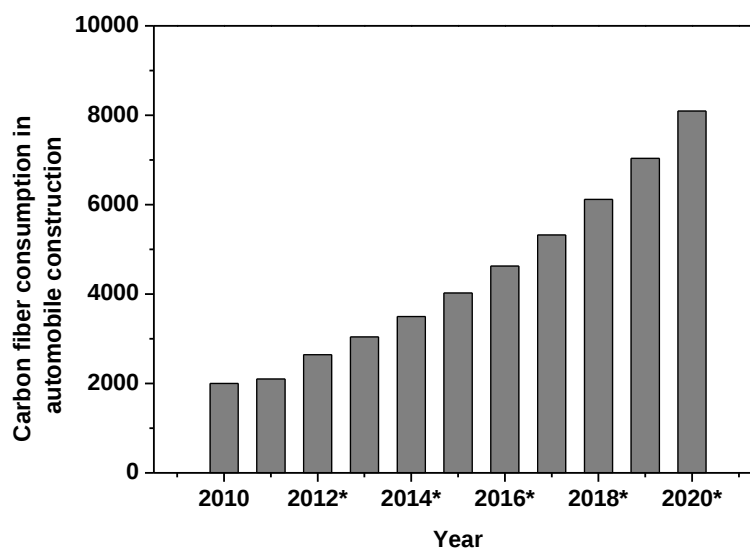


Figure 3.19: CF consumption in automobile construction (*estimates) [66]

From the worldwide produced carbon fibers, 95 % carbon fibers are used for manufacturing fiber reinforced plastic (FRP) materials. The tonnage of carbon fiber reinforced plastics (CFRP) is naturally much higher, however, due to the addition of the matrix component. The global demand of the CFRP is shown in Figure 3.18.

A variety of different production processes are used in the manufacture of CFRP materials/components. Around 54 % of the carbon fiber produced world-wide is used for manufacturing prepregs, of which 42 % are based on unidirectional fabrics and 12 % on woven fabrics. 5 % of carbon fiber is used to make semi-finished products such as fabrics, braids etc., which are in turn used to make CFRP parts via an infiltration process (e.g. RTM). The winding (approx. 15 %) and pultrusion (approx. 8 %) processes are also important techniques in CFRP production. Here the fibers are used in the form of yarns.

As can be seen from Figure 3.19 the automobile industry is considered as one of the most important drivers for the CFRP market growth in the future. Taking consumption of approx. 2000 tonnes of carbon fibers in 2010 as their base, conservative analysts are forecasting annual growth of 15 % for the sector. However, from 2013 onwards, this could be much higher than predicted depending on the success of the electric vehicles scheduled for launch [66].

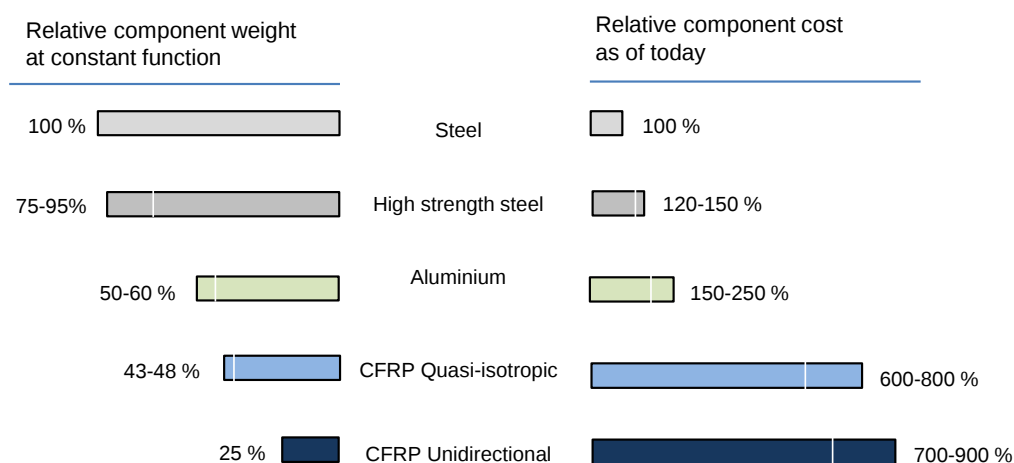


Figure 3.20: Comparison of the component weight saving and manufacturing costs using different construction materials [67]

Most of the CFRP automotive components are currently manufactured using the RTM process. As can be seen in Figure 3.20, though CFRP as a construction material offers highest weight saving potential for a particular automotive component, the component costs can increase up to 600-800 % if compared to steel as a standard construction material. In the

current status both materials and manufacturing contribute to approximately 50 % of the CFRP component costs. The carbon fibers alone are responsible for 45 % of the component costs [67].

In order to be able to implement the CFRP constructions materials economically for the automotive applications it is essential to reduce the materials and production costs significantly as shown in Figure 3.21. As a vision, the automobile manufacturers aim at reducing material costs by 50 % and production costs by 90 % for the CFRP materials. If the production costs of the thermoset based CFRP materials by RTM process shall be reduced by 90 % then it is essential to manufacture the preforms and infiltrated parts in cycle time ≤ 6 min. Intense research is vital to develop new and alternative processing strategies for manufacturing the high-performance composites for automotive applications [68].

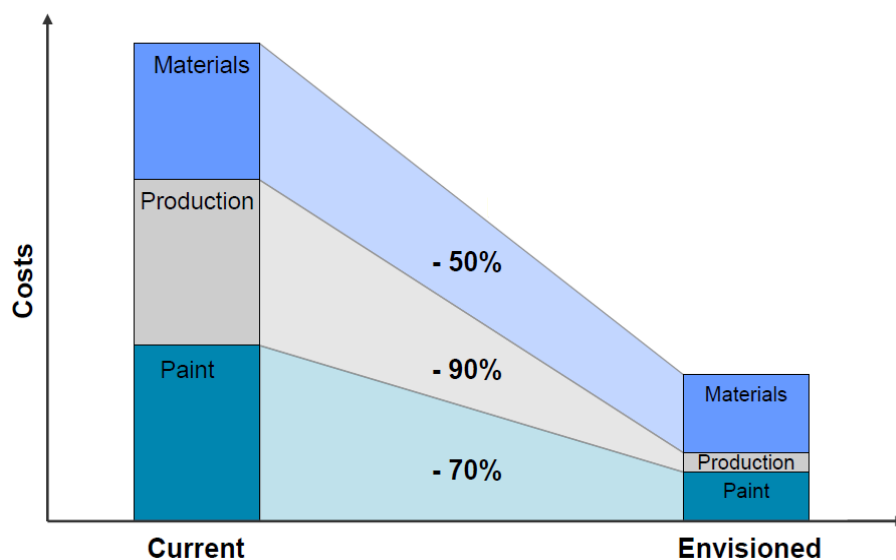


Figure 3.21: Cost reduction vision for the CFRP construction materials [68]

The development and investigation of selected RTM process variants in this work thus targeted on studying the parameters affecting the resin injection time. The reduction of resin injection time shall enable the use of fast curing resins to reduce the in-mold process time for the RTM components thereby reducing the overall manufacturing time. The selected process variants and important process parameters for studying these variants are defined in the next chapter.

4 Selection of RTM process variants and parameters

The work in this doctoral thesis is oriented towards studying the RTM process variants for understanding the effects of selected materials and process parameters on the resin injection time in the mold cavity. The Darcy's law (equation 3.1) formed main basis for selecting the process parameters for studying the selected RTM process variants. The Darcy's law forms basis for understanding fluid flow through a porous media and generally it is applied also to the RTM process in which a liquid resin is used to infiltrate / infuse through selected textile reinforcements having a particular porosity. As per Darcy's law resin flow velocity (V) is directly proportional to resin flow rate (Q) and these two parameters for a specific mold geometry determine the mold filling time (T). The mold filling time is further affected by materials and process parameters thus leading to following interdependencies for the RTM process.

- **Hypothesis 1: Interdependency of permeability (K) and resin flow velocity (V)**

Provided that specific mold geometry and process configuration is selected, resin viscosity (η) and injection pressure of resin are defined as constants, then the differences in porosity and thus differences in permeability of textile reinforcements (K_x , K_y , and K_z) in a particular mold geometry may lead to different resin flow velocities (V_x , V_y , and V_z)

- **Hypothesis 2: Interdependency of permeability (K) and resin flow rate (Q)**

Provided that specific mold geometry is selected, resin viscosity (η) and injection pressure of resin in selected RTM equipment are defined as constants, then the resin flow rate (Q) and thus the resulting mold filling time (T) shall be purely affected by permeability (K) of reinforcements in selected process configuration

- **Hypothesis 3: Interdependency of permeability (K) and pressure gradient (dp/dx)**

Provided that specific mold geometry is selected, and resin viscosity (η), resin flow rate (Q) and thus mold filling time (T) are defined as constants, then the differences in permeability (K) shall result in different pressure gradients (dp/dx)

- **Hypothesis 4: Interdependency of cross sectional area for resin flow (A_{flow}) and pressure gradient (dp/dx)**

Provided that specific mold geometry is selected, and resin viscosity (η), permeability (K), resin flow rate (Q) and thus mold filling time (T) are kept constant, then the differences in cross sectional area for resin flow (A_{flow}) resulted due to selection of different gate geometries shall influence different pressure gradients (dp/dx)

These hypotheses made on basis of Darcy's law lead to selection of specific RTM process variants, materials and process parameters for their investigations. The permeability of textile reinforcement, as one of the important factors affecting mold filling time, formed the basis for selecting different RTM process variants namely: compression RTM (CRTM), high-pressure compression RTM (HP-CRTM) and high-pressure injection RTM (HP-IRTM).

4.1 Compression RTM and high-pressure compression RTM processes

The Compression RTM process is a combination of resin transfer molding (RTM) and compression molding. In the CRTM process, as shown in Figure 4.1, the preform is placed into the mold cavity and then the mold is closed partially, leaving a small gap between the mold surface and the fiber preform. The resin is introduced into the gap, flows easily over the preform and may partially impregnate it. Once the required amount of resin has been injected into the gap and the injection point is closed, the mold closes further and applies compression pressure to squeeze the resin into the preform. In this step, the preform is compacted to achieve the desired part thickness and fiber volume fraction. The part can be demolded after the resin has cured [35]. In CRTM process, due to partially open mold cavity, the permeability of the textile reinforcement is not reduced and thus it is assumed that the resin injection time in CRTM can be reduced significantly if compared to the classical RTM process.

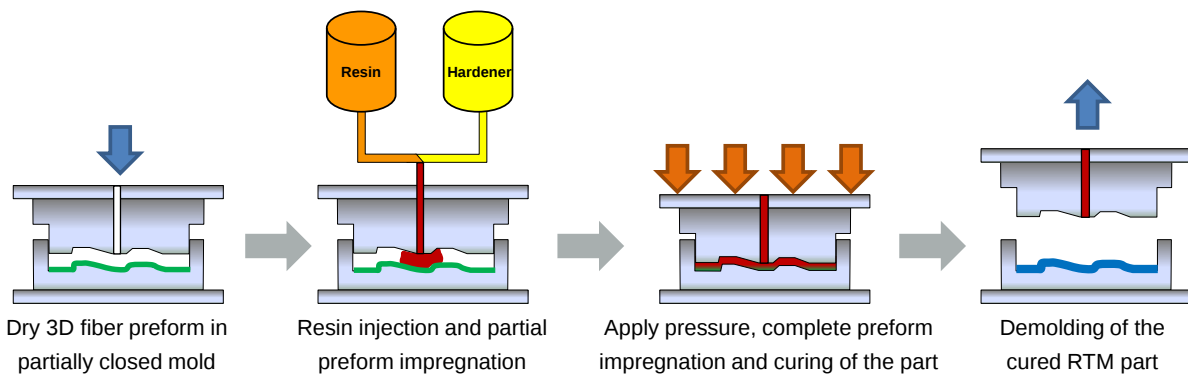


Figure 4.1: Resin injection sequence in the CRTM and HP-CRTM processes

The working principle of the HP-CRTM process is equivalent to the CRTM process. High-pressure RTM (HP-RTM) equipment is used for injecting highly reactive resin into the mold under high resin throughput rate (20 - 200 g/s) in the HP-CRTM process [69]. As described in chapter 3, selecting a small mold gap on CRTM process and HP-CRTM processes leads to increased permeability of textile reinforcements thus theoretically fulfilling hypotheses 2 & 4 where a combination of higher permeability and increased cross sectional flow area is targeted to be achieved.

4.2 High-pressure injection RTM process

The HP-IRTM process differs from the classical RTM process only in terms of the processing equipment for the resin system. In the classical RTM process, low pressure mixing and dosing equipment is used for processing resins, whereas in the HP-IRTM process resin mixing and dosing is carried out using high-pressure RTM equipment. Similar to the classical RTM process, as shown in Figure 4.2, in the HP-IRTM process the preform is placed into the mold cavity and then the mold is completely closed to compact the preform to final part thickness. Thus in this process step the permeability of the reinforcement is reduced significantly due to compaction. Once the mold is closed, the resin and hardener mixture is injected into the cavity under high injection speed.

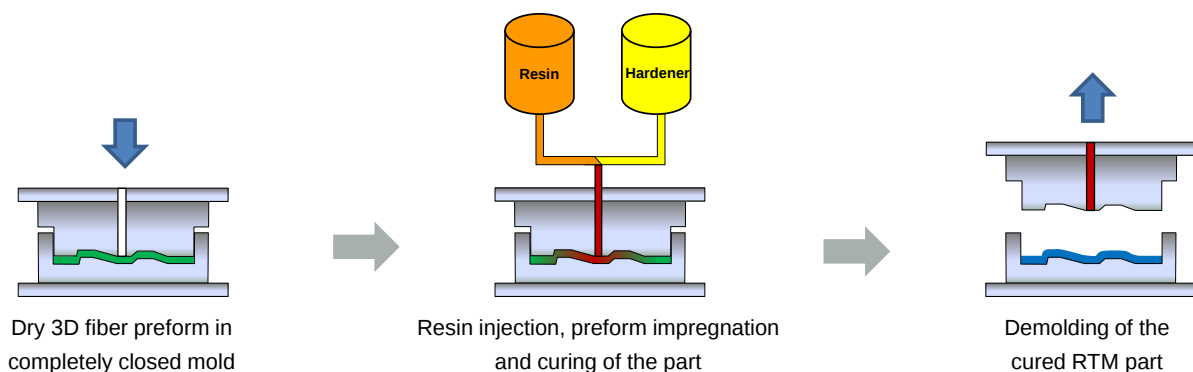


Figure 4.2: Resin injection sequence in the HP-IRTM processes

The high throughput rate allows the cavity to fill quickly, significantly reducing the resin injection time. As the resin can be injected into the cavity in a shorter time, this process variant allows the use of resins with relatively high reactivity. After the resin curing reaction is completed the part can be demolded [69].

4.3 Definition of parameters for investigating RTM process variants

Various studies have been conducted by different researchers to investigate the effect of certain process parameters on the quality of manufactured laminates in the CRTM process and summary of used process parameters for these studies is mentioned in Table 4.1. The studies conducted by Chang et al indicated that *compression pressure* and *resin viscosity* affect the mechanical properties of the laminates significantly. The effect of pre-heated mold temperature appeared to be trivial and *vacuum assistance* in CRTM helped to reduce the voids in the reinforcement thereby improving the part quality significantly [70]. From the investigations conducted by Ikegawa et al, along with the mold gap value, the gap closure rate was detected as an important process parameter affecting the void formation in the laminates. The formation of the voids in the structural CRTM process at different gap closure

rate was determined by a balance of permeability of the textile reinforcement in different directions, i.e. *longitudinal and transverse permeability* of the reinforcement. It is concluded in this study that using compression step better impregnation was obtained in the structural CRTM process if compared to the standard structural RTM process in which no mold gap is used [71].

Table 4.1: Overview of literature parameters for the CRTM process [70-74]

Process parameter	Chang et al.	Ikegawa et al.	Rosenberg et al.	Haspel et al.
Reinforcement type	Biaxial woven glass fabric	Random fiber mat	Unidirectional woven fabric	Unidirectional woven fabric
Resin type	Epoxy	Epoxy	Epoxy	Epoxy
Fiber vol. content [Vol %]	40.5	33	53 - 57	63 - 66
Mold gap [mm]	1, 5, 10	0, 3, 6, 12	2	2
Gap closure speed [mm/min]	not available	5, 100	0, 12, 24	24, 48
Mold temperature [°C]	25, 50, 75	130	100	100
Curing temperature [°C]	80, 100, 120	130	100	100
Resin temperature [°C]	25, 32, 40	90	25, 45	25, 42
Injection time [sec]	not available	not available	13, 60	13, 60
Injection pressure [bar]	1, 1.5, 2	5	6	6
Compression pressure [bar]	1, 1.5, 2	13	60	60

The CRTM process studies conducted by Rosenberg et al using a complex shaped component (V-shaped geometry) also concluded compression pressure and resin viscosity as important parameters affecting the CRTM process [72]. In the extended study conducted by Haspel et al using the same geometry (V-shaped geometry) a drastically high fiber volume content in the range of 63 - 66 % was obtained in the laminates. In this study as well the resin temperature (i.e. resin viscosity), press force, gap closure speed and laminate layup were concluded as important parameters affecting void content in the CRTM process [74].

From the available literature and considering Darcy's law as a basis the most important parameters affecting the CRTM, HP-CRTM and HP-IRTM process can be concluded as follows:

1. Mold parameters:

mold gap (for HP-IRTM process, mold gap = 0), mold geometry, gate design, gasket concept

2. Resin parameters:

impregnation viscosity, gel time, curing temperature, demolding time

3. Textile parameters:

textile architecture, fiber type, laminate layup, longitudinal and transverse permeability of reinforcements at different fiber volume content

4. Injection equipment parameters

Resin amount, resin flow rate

5. Compression press parameters

compression force, gap closure speed (relevant for CRTM and HP-CRTM)

Table 4.2: Summary of varied parameters for process studies

Chapter	Process	Fiber type	Investigated paramters
Chapter 6.1	CRTM, classical RTM	glass fibers	mold gap/permeability, laminate layup
Chapter 6.2	HP-CRTM	glass fibers	mold gap/permeability, laminate layup, different mold geometries, compression force
Chapter 6.3	HP-IRTM, HP-CRTM	carbon fibers	fast cure resins of different viscosity, gate geometry, mold gap, fiber volume content, resin flow rate

The important parameters were studied, derived from literature and Darcy's law as summarized in Table 4.2, for their influence on CRTM, HP-CRTM an HP-IRTM processes. The investigations in this thesis work started with a feasibility study for the CRTM process to validate hypotheses 1 & 2 using suitable mold gap as described in chapter 6.1. The study was extended from CRTM feasibility study to HP-CRTM process, as described in chapter 6.2, in which especially high resin flow rate was used to maintain the resin injection time at lower level (resin injection time < 10 s). The detailed HP-CRTM process studies

included understanding the effect of permeability, laminate layup, mold geometry and compression force on the HP-CRTM process. The investigation of CRTM and HP-CRTM processes in chapters 6.1 and chapter 6.2 was accompanied by use of glass fibers. A comparison of the HP-CRTM to the HP-IRTM process was carried out using carbon fibers, different resins and gate geometries to validate hypotheses 3 & 4 and to understand effect of these parameters on the selected process variants as described in chapter 6.3.

During study of the selected RTM process variants using selected materials and equipment technology, in all the process studies, at least 2 laminates were manufactured per selected process parameter to validate the process reproducibility. Using these laminates, at least five samples were tested per selected process parameter and per materials test group to evaluate the influence of selected parameters on the physical and mechanical properties of the laminates (e.g. fiber volume content, and tensile, flexural and ILSS properties). Samples were prepared for all the process studies from the laminates by water jet cutting. All the samples in this thesis work were tested according to respective DIN EN ISO norms [90-93] and the samples were prepared from the laminates by the water jet cutting method. The specifications of the used materials and hardware for conducting different process studies are given in chapter 5.

5 Experimental materials and equipment technology

This chapter describes in detail the used materials, molds, process equipment and characterization methods which were used for studying the resin transfer molding (RTM) process variants.

5.1 Materials

The study of the RTM process variants in this thesis work was associated mainly with the use of various thermoset matrix materials, glass and carbon fiber based reinforcements and three different mold geometries. The matrix materials were processed using two different resin injection equipment. The goal behind the use of various epoxy resins was to evaluate the potential of such resins for high-volume manufacturing of high-performance composites by the selected RTM process variants.

Characterization of thermoset resins

Table 5.1: Process parameters of the selected epoxy resins for studying RTM process variants [75-78]

Material provider	Momentive Specialty Chemicals	Huntsman Advanced Materials	Huntsman Advanced Materials	Confidential	Confidential
Epoxy resin	EPIKOTE™ Resin MGS® RIMR 935	Araldite® LY 564	Resin XB 3585	Resin EA	Resin EB
Curing agent	EPIKURE™ MGS® RIMH 936	Hardener XB 3458	Hardener XB 3458	Hardener HA	Hardener HB
Mixing ratio [parts by weight]	100:29	100:20	100:19	100:24	100:13
Preheating temperature* [°C]	room temp	60	80	80	80
Impregnation viscosity** [mPa.s]	10	100	70	40	20
Impregnation time** [s]	140	60	18	55	45
Mold temperature [°C]	100	100	100	120	120
Demolding time*** [s]	1500	110/140	40/70	580/610	100/130
Practically used Demolding time [s]	1200	240	240	300	300

* Preheating temperature resembles to the temperature of the epoxy resin components in the RTM equipment (hardener always at room temperature)

*** Impregnation viscosity and impregnation time were measured using chemo-rheological characterization; a temperature ramp program was selected to heat the resin system from preheating temperature to curing temperature/mold temperature*

**** Demolding time measured using chemo-rheological characterization at a temperature equivalent to the selected mold temperature in the RTM process studies (second value includes additional 30 s time which was required to transfer the sample in the rheometer and to start the measurement)*

For characterization of RTM process variants five different epoxy resins were used. The important parameters for processing of these resins are mentioned in Table 5.1. As can be seen, these epoxy resins were associated with different processing properties especially in terms of impregnation viscosity, impregnation time and demolding time at selected mold temperature. The detailed test parameters and curves obtained from the chemo-rheological characterization of the used resin systems are given in annex 9.3.

The investigations in this thesis work started by studying the compression RTM process. For characterizing the CRTM process a resin system from Momentive Specialty Chemicals was used and it was a slow reacting epoxy resin system based on Resin MGS® RIMR 935 and hardener MGS® RIMH 936. Glass fibers were used as reinforcements. The selected resin system exhibited 10 mPa.s impregnation viscosity, 140 s impregnation time and relatively long demolding time of 1500 s. Detailed investigation of HP-RTM process variants was conducted using glass fibers as reinforcements and two different fast curing resin systems from Huntsman Advanced Materials. These resin systems constituted epoxy resins Resin XB 3585/Araldite® LY 564 and a common Hardener XB 3458 which can be used for curing both the epoxy resins. The resin systems based on Resin XB 3585/Araldite® LY 564 and Hardener XB 3458 exhibited impregnation viscosity of 100 mPa.s and 70 mPa.s, impregnation time of 60 s and 18 s, and demolding time of 140 s and 70 s respectively. The chemo-rheological characterization indicated that the combination of Resin XB 3583 and Hardener XB 3585 was the fastest curing resin system among the selected resin systems. The last two characterized resin systems, namely Resin A constituting epoxy resin EA and hardener HA, and Resin B constituting epoxy resin EB and hardener HB, are given imaginary names due to confidentiality of these resins and as these resins are still at development stage. These resins were used for investigating HP-RTM process variants for manufacturing carbon fiber reinforced composites. Resin A exhibited 40 mPa.s impregnation viscosity, 55 s impregnation time and demolding time of 610 s, and Resin B exhibited 20 mPa.s impregnation viscosity, 45 s impregnation time and demolding time of 130 s.

The characterization of the selected resin systems shows that, if shorter curing time shall be achieved in the RTM process by using a particular resin, then it is essential to impregnate the textile reinforcements within the available impregnation time of the respective resin system.

As all the fast curing resin systems were associated with an impregnation time of 18 s to 60 s, for a particular resin it is essential to adapt the resin injection rate into the mold cavity in accordance with the required weight of the resin that is needed for complete impregnation of the component to be manufactured by the RTM process. These epoxy resins were combined with below described reinforcement materials and molds for studying the RTM process variants in detail.

Reinforcements

Non-crimp glass fabric

A non-crimp unidirectional (UD) fabric made of E-glass from SAERTEX GmbH & Co. KG was used as one of the reinforcements for studying the selected RTM process variants. The chosen glass fabric is commercially available with product name S14EU960-01210-01300-487000. The E-glass fiber product used for manufacturing of the non-crimp fabric is a commercial product from PPG Industries and it is available as Hybon® 2002. The Hybon® 2002 fiber product used by SAERTEX GmbH & Co. KG for manufacturing the above mentioned UD fabric has fiber diameter of 17 μm , 1200 g/km roving Tex and multi-compatible sizing. The UD fabric has surface area weight of 1218 g/m². The construction of the fabric includes 95 wt % fibers in 0° orientation and 4.5 wt % fibers in 90° orientation. The fabric is stabilized using polyether sulfone (PES) based stitching fiber which contributes to 0.5 wt % of the total surface area weight [79, 80].

Non-crimp carbon fabric

A non-crimp fabric made of carbon fiber was used as second reinforcement material for studying the RTM process variants. This non-crimp fabric is manufactured using Toray carbon fiber type T620S-24K-50C. The used non-crimp fabric had a surface area weight of 300 g/m² and was available in three different orientations (0°, 0°/90° and 45°/-45°) [81, 82].

5.2 Molds

Plate mold 1

A steel mold with plate geometry was used to conduct feasibility study for the CRTM process and detailed investigation of the HP-CRTM process. Figure 5.1 shows cross sectional view of both the assembled mold halves to illustrate the mold concept. This plate mold is referred here after as plate mold 1.

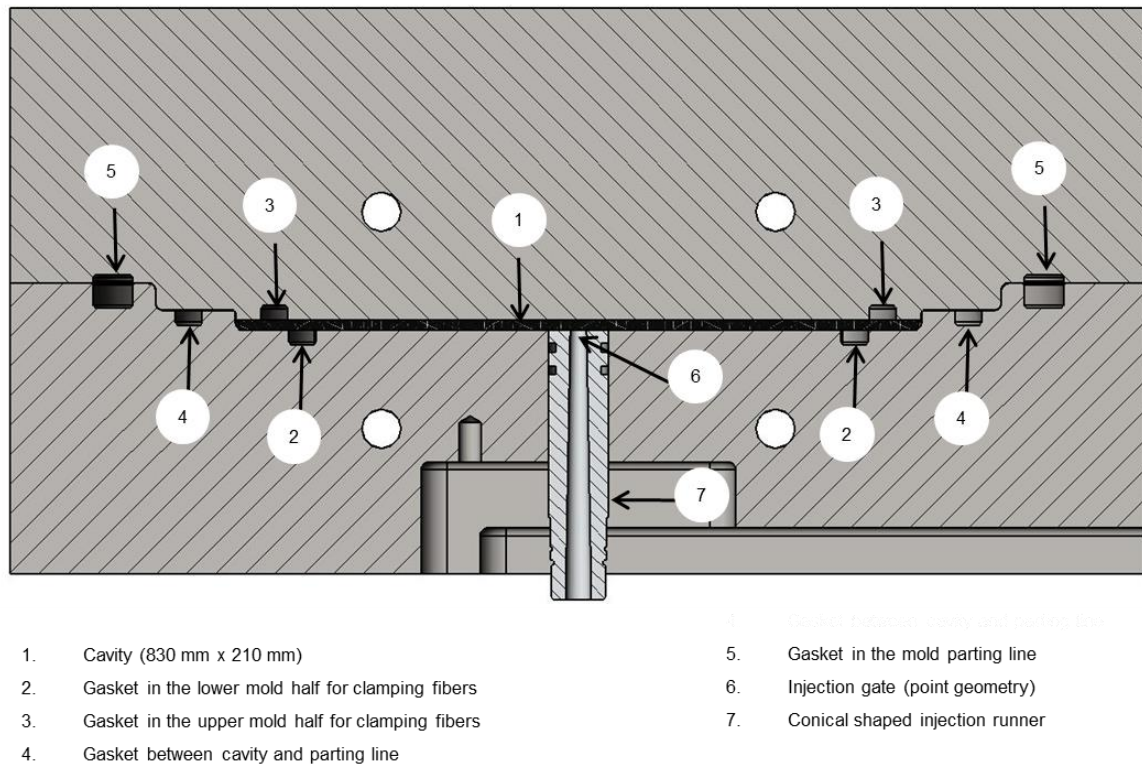


Figure 5.1: Cross sectional view of plate mold 1 (mold cavity in closed condition)

The cavity of this mold measured 830 mm x 210 mm and the cavity height varied between 2.87 mm to 2.9 mm over the complete surface of the mold if the mold was completely closed. This plate mold was designed with four different gaskets located at different positions. The first and second gaskets were located in the lower and upper mold halves at position 2 and position 3 respectively as shown in Figure 5.1. These both the gaskets were designed to clamp the fibers of the textile reinforcement (pinch-off) into the cavity. These pinch-offs were designed to trap the reinforcement in the mold cavity between the closed mold halves to create an edge restriction to the advancing resin flow. The third gasket was located between the cavity and the parting line of the mold, as shown at position 4, to avoid resin flow out of the cavity and towards the mold parting line. A cord shaped silicon gasket was used in the cavity in the lower mold half at position 2, and between the cavity and the parting line at position 4 while manufacturing laminates using this mold. The fourth gasket was located in the parting line of the mold as shown at the position 5 and this gasket served an additional safety to avoid the resin flow out of the mold parting line, in case, if the inner gaskets were not enough to sustain the created cavity pressure during the laminate manufacturing. As a gasket a hollow silicon tube was used in the parting line of the mold.

The resin injection gate of point geometry was located in the middle of the lower mold half. The runner for resin injection was designed to have a conical shape in such a way that the cured resin in the injection runner could be removed from the lower side of the mold after manufacturing the laminates. For applying vacuum, holes were available at both the ends of the upper mold half. However for all the process studies these holes were closed using metallic stoppers. For centering of the lower and upper mold halves while opening and closing of the mold, cylindrical holes were designed at four corners of the lower mold half and guide pillars were designed in the upper mold half resembling to the position of the cylindrical holes. The effective plate geometry was defined to 700 mm x 130 mm with the help of small groove which was designed in the lower mold half. This area of the plate was used as a test area to characterize the laminates manufactured using various process parameters. The upper and lower mold halves were designed to offer mold heating to obtain required resin curing temperature using oil as heating medium.

Hat shaped mold

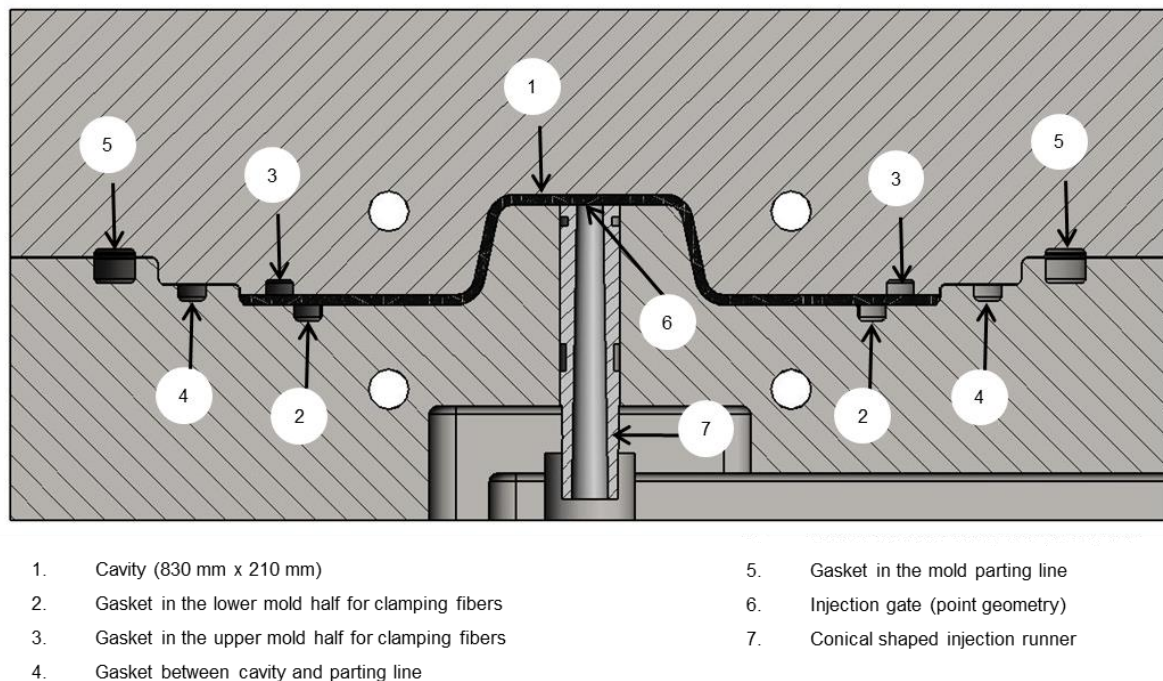


Figure 5.2: Cross sectional view of the closed hat shaped mold (mold cavity in closed condition)

A second steel mold designed to manufacture hat shaped components (Hutprofilwerkzeug) was used for studying the HP-CRTM process. This mold geometry was used to correlate the

effect of process parameters from the flat plate geometry (plate mold 1) to the complex hat shaped geometry. As seen in Figure 5.2, the design and construction of the hat shaped mold is similar to plate mold 1 in terms of resin injection system, gasket concept and mold centering concept. In this mold the resin injection gate of point geometry was located as well in the middle of the lower mold half. The runner for resin injection was designed with conical shape to offer easy removal of the cured resin from lower side of the mold. Similar to plate mold, the hat shaped mold had two holes for applying vacuum at both the ends of the upper mold half and for all the process studies these holes were closed using metallic stoppers.

The hat shaped mold varied from plate mold 1 only in terms of the cavity geometry. The cavity surface area (2-dimensional) of the hat shaped mold also measured 830 mm x 210 mm. However the dry textile reinforcement of 250 mm width was used to obtain the hat shaped cavity due to the hat shaped geometry of the component. The cavity height of this mold varied between 2.9 mm to 3.2 mm if the mold was completely closed. In order to compare the effect of the HP-CRTM process parameters on the manufacturing and properties of the flat plate and hat shaped components, gaskets of equivalent diameter were used at positions 2, 4 and 5 in both of the molds. Similar to plate mold 1, the effective geometry of hat shaped component was also defined to 700 mm x 130 mm with the help of small groove which was designed in the lower mold half.

The injection runners of plate mold 1 and hat shaped mold were connected to the used resin injection equipment through a metal tube of external diameter of 10 mm and internal diameter of 7 mm. The free volume of the metal tube was considered during resin injection step and equivalent resin weight was added to calculated amount of resin to be injected into the mold cavity while manufacturing test laminates.

Plate mold 2

Plate mold 1 and hat shaped mold were used for detailed investigation of CRTM and HP-CRTM process variants. After finishing these investigations a new plate mold, specially designed and constructed for studying HP-RTM process variants, was constructed and used for comparing the HP-CRTM process with HP-IRTM process. Figure 5.3 shows the conceptual sketch of this steel plate mold, here after referred as plate mold 2. The injection point of gate geometry was also designed in the middle of the lower mold half for this mold. The mold was designed with oil based heating to obtain required temperature for resin curing.

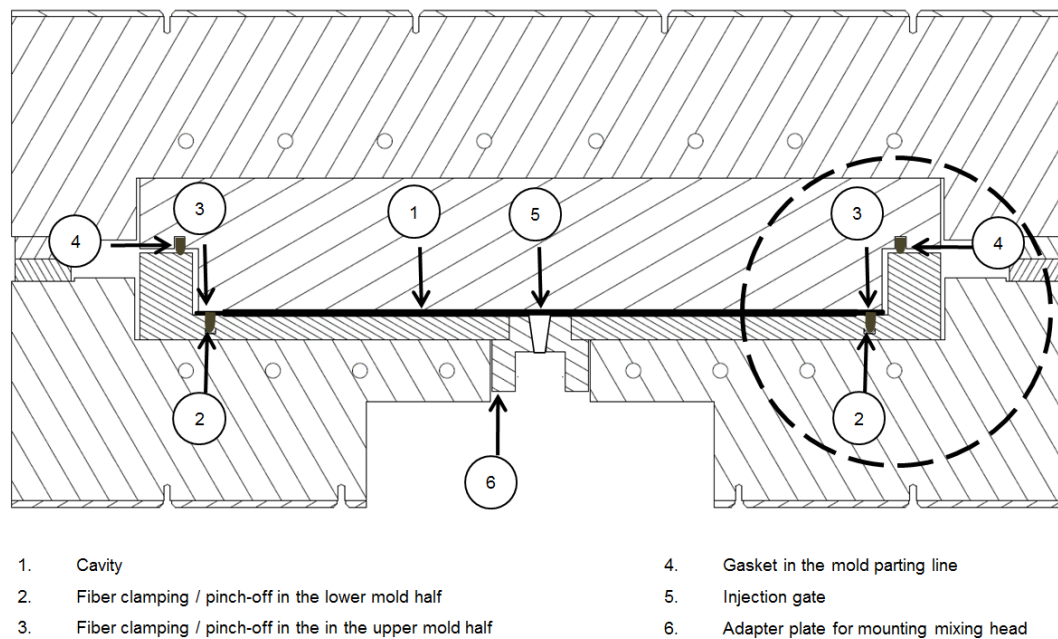


Figure 5.3: Cross sectional view of plate mold 2 (mold cavity in closed condition)

The centering of this mold was facilitated also by using guide pillars as for plate mold 1 and hat shaped mold. The mixing head was mounted into the mold from the lower side into an adapter plate at position 6. The opening of the mixing head was connected to the cavity surface by this adapter plate via an injection runner of conical shape. The injection runner was designed in the adapter plate in such a way that the cured resin rest in the injection runner could be removed together with the manufactured laminate during its demolding.

Plate mold 2, as shown in Figure 5.4, was designed with different cavity inlays to offer different process configurations (i.e. fiber clamping, gate geometry and pressure sensors) to study the effect of important process parameters of the HP-RTM process variants.

Configuration 1: cavity size 910 mm x 560 mm x 1.65 mm, point gate, fiber clamping using metal edge and Vulkollan® gasket

Configuration 2: cavity size 910 mm x 560 mm x 1.65 mm, film gate, fiber clamping using metal edge in upper mold half and Vulkollan® gasket in lower mold half

Configuration 3: cavity size 900 mm x 500 mm x 1.8 mm, film gate, fiber clamping using silicon gasket in upper and lower mold half

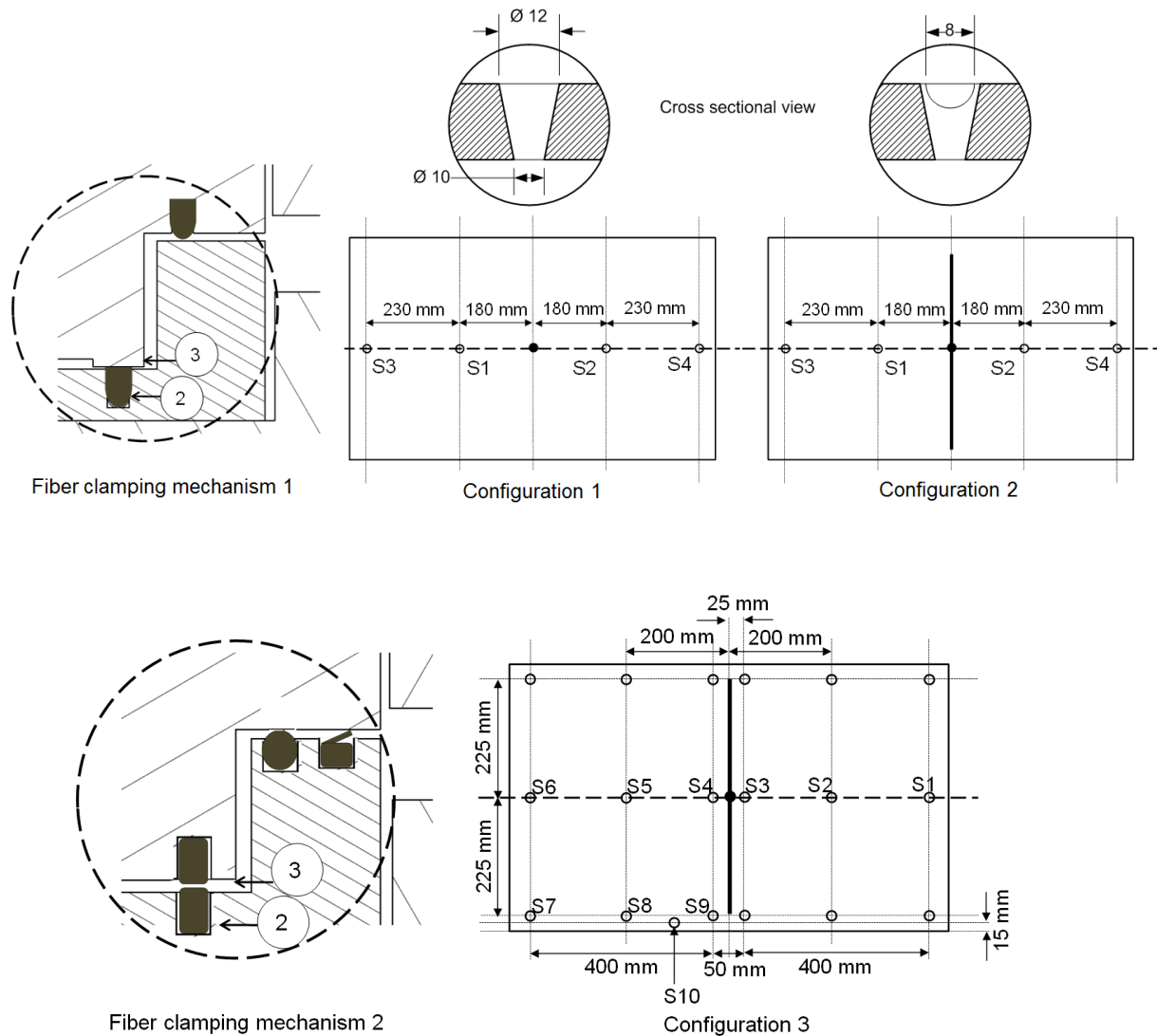


Figure 5.4: Fiber clamping mechanisms, gate geometries and sensor positions for different design configurations of plate mold 2

In case of configurations 1 & 2, the width of the metallic clamping edge and Vulkollan® gasket was 20 mm and 10 mm respectively. The metallic clamping edge had a height of 0.75 mm and it was designed in the upper mold half. The Vulkollan® gasket was placed in the lower mold half by fixing it in a channel of defined depth to obtain a height of 0.2 mm above this channel. So the total height of fiber clamping for configuration 1 and 2 was 0.95 mm. In case of configuration 3, the width of the silicon gasket for fiber clamping was 7 mm. This gasket was also placed in upper and lower mold halves by fixing it in a channel of defined depth. By adjusting the gasket height, it was possible to adjust the fiber clamping as per the requirement.

5.3 Process equipment

Compression press

The above mentioned molds were mounted in a hydraulic press of type DYL630/500 from company Dieffenbacher GmbH & Co.KG. for manufacturing of different test samples using defined materials and process parameters. This compression press is designed for applying a maximum compression force of 6300 kN and a compression force of 5000 kN if the parallel control mechanism for mold closure is active. Using this compression press a defined mold gap was obtained for all the CRTM and HP-CRTM process studies. After the resin was injected in the partially closed mold, the press closed the mold to obtain the defined compression force value (e.g. 1060 kN for plate mold 1) which was maintained during the defined laminate curing time. In case of HP-IRTM process studies this compression press was used for closing the mold with defined force value which was maintained during resin injection as well as defined laminate curing time [83].

Resin injection equipment

A 2-component resin mixing and injection system of type 100/120/25/17 from Wolfangel GmbH was used for processing of the resin system based on Resin MGS® RIMR 935 and MGS® RIMH 936 for all the CRTM process studies. The working principle of such equipment is already explained in chapter 3.5.2. The materials in this equipment are pumped separately through pneumatically driven vertical piston pumps system. During the resin injection step, the resin components are mixed using a static mixer assembly which is mounted in the heat-able cabin of the resin mixing equipment itself. During rest of the time, when the resin is not injected into the mold cavity, the resin components are circulated between the resin containers to avoid contact with each other. While conducting CRTM experiments using this equipment, after each resin injection cycle, it was essential to clean the static mixing element and injection hose of the equipment by acetone as it contained reactive mixture of the resin and hardener. A two way ball valve was used between the metal tube which was mounted on plate mold 1 and resin injection hose of the equipment to avoid back flow of resin from the mold cavity towards resin injection equipment after resin injection step ended. After each resin injection cycle and demolding of the laminates, the set of metal tube and two way ball valve was replaced by either new or cleaned set [84]. For processing of other chosen fast curing resin systems a high-pressure RTM (HP-RTM) equipment of type RTM 8/3,2K from company KraussMaffei Technologies GmbH was used. In this type of resin mixing and injection equipment, the resin and hardener components are pumped separately using an axial piston pump system from the resin

container to the mixing head of the equipment. Similar to the resin injection equipment system from Wolfangel GmbH, the resin components are circulated in the HP-RTM equipment between the resin component containers to the mixing head of the equipment. The mixing head function of the HP-RTM equipment already explained in chapter 4.2 [85].

6 Characterization of RTM process variants

This chapter describes the results of the studies conducted for the development and characterization of the RTM process variants CRTM, HP-CRTM and HP-IRTM.

6.1 Feasibility study for CRTM process

The feasibility study for the CRTM process was carried out using plate mold 1 and a resin system based on an epoxy resin and an amine hardener commercially available as EPIKOTE™ Resin MGS® RIMR 935 and EPIKURE™ MGS® RIMH 936 respectively. The resin components were processed at room temperature using piston pump based injection equipment. As reinforcement the non-crimp glass fabric was used (details given in 5.1.1). Laminate layups for studying CRTM process were based on two different orientations: four layers in unidirectional orientation, [0/0/0/0] referred hereafter as [0₄], and four layers in bidirectional symmetric orientation, [0/90/90/0] referred hereafter as [0/90]_s. At the start of the feasibility study a mold fill study was carried out to understand the resin flow behavior in the mold with and without application of a compression force in correlation to the injected resin amount in the mold cavity. Additionally, laminates were manufactured using mold gaps of 1 mm and 2 mm for each laminate layup which resulted in the effective gaps of 0.9 mm and 1.9 mm respectively. The basis for calculation of the effective mold gap is given in annex 9.1. For each experiment a resin amount of 270 g, which was slightly higher than the theoretically calculated resin amount (235 g) required for laminate impregnation, was injected in approximately 30 to 33 s into the partially open mold cavity using the selected RTM equipment. The resin amount of 270 g also contained the additional amount of resin which is required for filling the injection runner and the connecting tubes from the injection equipment to the mold (20 g). After the resin was injected into the partially open cavity the mold was closed completely with a closure speed of 0.2 mm/s to apply a compression force of 1060 kN. The mold closure compact the preform and led to its impregnation by the resin. The mold was heated to 100 °C to cure the chosen epoxy resin to a laminate for 20 min. Further details of the process parameters applied are given in annex 9.1 [86, 87].

6.1.1 Mold fill study for CRTM process

A mold fill study was conducted as shown in Figure 6.1 through two different series of experiments. In the first series of experiments, defined amount of resin was injected into the partially open mold cavity and after the resin injection was completed the mold was immediately opened to determine the flow front progression before application of compression force. In the second series of experiments, a defined amount of resin was injected into the partially open mold cavity and after the resin injection was completed the

mold was immediately closed using the defined compression force. The laminates were then cured for the defined resin curing time of 20 min. After the mold was opened the flow front progression after compression was then compared with the flow front progression before compression. In both the experimental series the flow front progression was compared by taking pictures with a digital camera. For both laminate layups the resin amounts of 70 g, 140 g, 210 g and 270 g were used for the mold fill study and these resin amounts were injected into the partially open mold with a 2 mm mold gap.

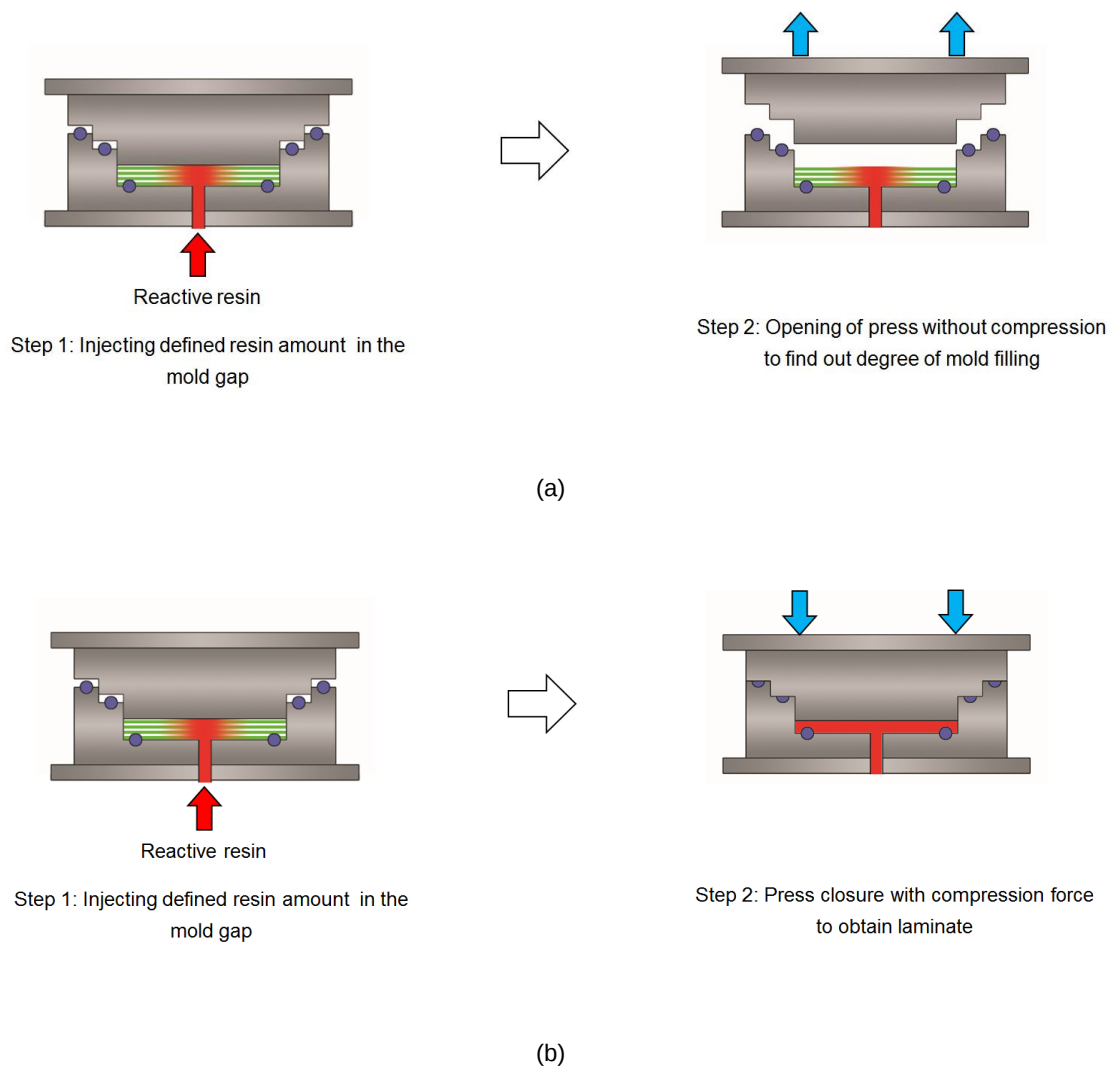
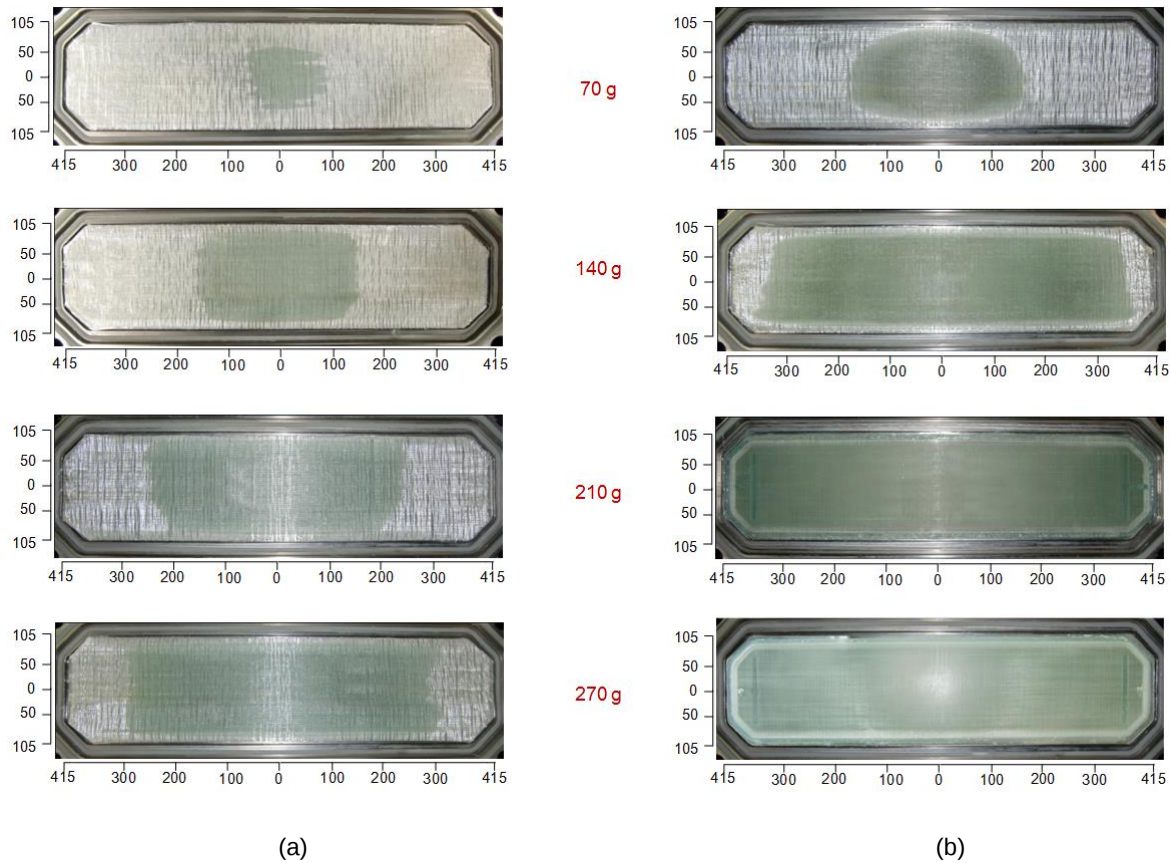


Figure 6.1: Mold fill study in the CRTM process; (a) Resin injection in partially open mold cavity and no application of compression force, (b) Resin injection in partially open mold cavity and immediate mold closure with compression force

Results of mold fill study for unidirectional laminates

Picture 6.1 shows the results of the mold fill study for unidirectional laminates as a function of the injected amount of resin. The flow front progression after injection and before

compression is shown in Picture 6.1 (a) and the flow front progression after injection and compression is shown in Picture 6.1 (b).



Picture 6.1: Flow front progression in CRTM process as a function of injected resin amount for a unidirectional laminate layup; (a) before compression, (b) after compression (mold dimension indicated in centimeters on the scale)

Flow front progression for unidirectional laminates at 70 g resin amount:

For the unidirectional laminates at an injected resin amount of 70 g a flow front progression of approximately 140 mm in length and 130 mm in width was measured in the top layer of the laminate (injection point from the bottom side of the mold) before compression. Along the lengthwise orientation and across the width of the mold, the formation of “local resin rich areas” shaped like a human tongue was observed. After application of compression force and laminate curing oval shaped resin flow front progression of 300 mm length and 160 mm width was observed. An explanation for the formation of such “local resin rich areas” only along the length before application of compression force, and the formation of an oval shaped flow front after compression and curing is the unidirectional laminate layup and circular shaped point injection runner located in the middle of the mold. Such an

unidirectional laminate layup based on an unidirectional non-crimp fabric has higher permeability parallel to the roving orientation due to inter-tow spaces (free passage between rovings) and lower permeability perpendicular to the roving orientation. Hence during resin injection it must have led to higher macroscopic resin flow between the free passages of rovings which were oriented along the length of the mold, thereby leading to the appearance of tongue-shaped resin rich areas. The application of compression force, which caused the resin to squeeze and flow through fabric layers, led to a higher macroscopic as well as microscopic resin flow along the length of the rovings (330 mm) due to higher permeability, compared to lower resin flow perpendicular to the rovings (160 mm) due to lower permeability. In the literature for a common type of unidirectional reinforcement longitudinal permeability of $7.1 \times 10^{-11} \text{ m}^2$ and transverse permeability of $1.2 \times 10^{-11} \text{ m}^2$ has been reported indicating their relevance on the resin flow through the reinforcement material [65, 88, 89].

Flow front progression for unidirectional laminates at a resin amount of 140 g:

For an injected resin amount of 140 g a relatively linear flow front progression of approximately 310 mm (maximum) length and 160 mm width was observed before compression. In this case as well the formation of “local resin rich areas” was observed along the length and across the width of the mold. However, such resin rich areas had very little length and the progression of resin flow front appeared more homogenous. After compression and curing of the laminate the resin flow front progression of approximately 750 mm (maximum) length was observed. The resin flow front remained at 160 mm width after compression and curing, which was same as before compression. This effect can be explained again on the basis of the difference in permeability along the length (higher permeability) and perpendicular to the length (lower permeability) of unidirectional laminate layup based on unidirectional non-crimp fabric. Also, the lower half of the mold was designed to have a gasket on which the textile reinforcement was placed. The free width of the textiles between the gasket edges in the cavity was 160 mm. If the mold was closed to a 2 mm mold gap in the presence of four layers of textiles and the gasket, the initial contact with the gasket was already established and the textiles were already slightly compacted over the area of the gasket, which reduced the permeability of the textiles at the compaction area. The combination of a cavity height of 3 mm, a mold gap of 2 mm vs. the combination of a gasket height of 2 mm in a cavity and perform thickness of 4 mm led to a total compaction (gasket and preform) of 1 mm of gasket and textile layers together. Hence the resin flow front progression was only possible up to 160 mm across the width of the mold before compression due to the higher permeability of unidirectional laminate layup in lengthwise orientation. During the compression step, as the cavity was not completely filled by resin, and as the permeability of the textiles in the area of the gasket reduced due to mold closure, the

flow front width of 160 mm remained as before the compression. Thus the resin flow front progression resulted only along the length of the mold as unidirectional laminate layup had a higher permeability along the mold length

Flow front progression for unidirectional laminates at a resin amount of 210 g:

At an injected resin amount of 210 g a resin flow front progression of 480 mm length and 180 mm width was observed before application of compression force. Slightly increased length of resin flow front progression across the width of the cavity was observed with increasing resin amount, indicating that some resin could flow between the slightly compressed preform - gasket assembly. After compression nearly complete mold filling was achieved, however optical examination of the laminates revealed the presence of air entrapments.

Flow front progression for unidirectional laminates at a resin amount of 270 g:

For an injected resin amount of 270 g the flow front progression of 580 mm in length and 200 mm in width was observed before application of compression force. In this case as well, similar to an injected resin amount of 210 g, the increased length of resin flow front progression across the width of the cavity was observed. At this injected resin amount, complete mold filling was achieved after the compression step.

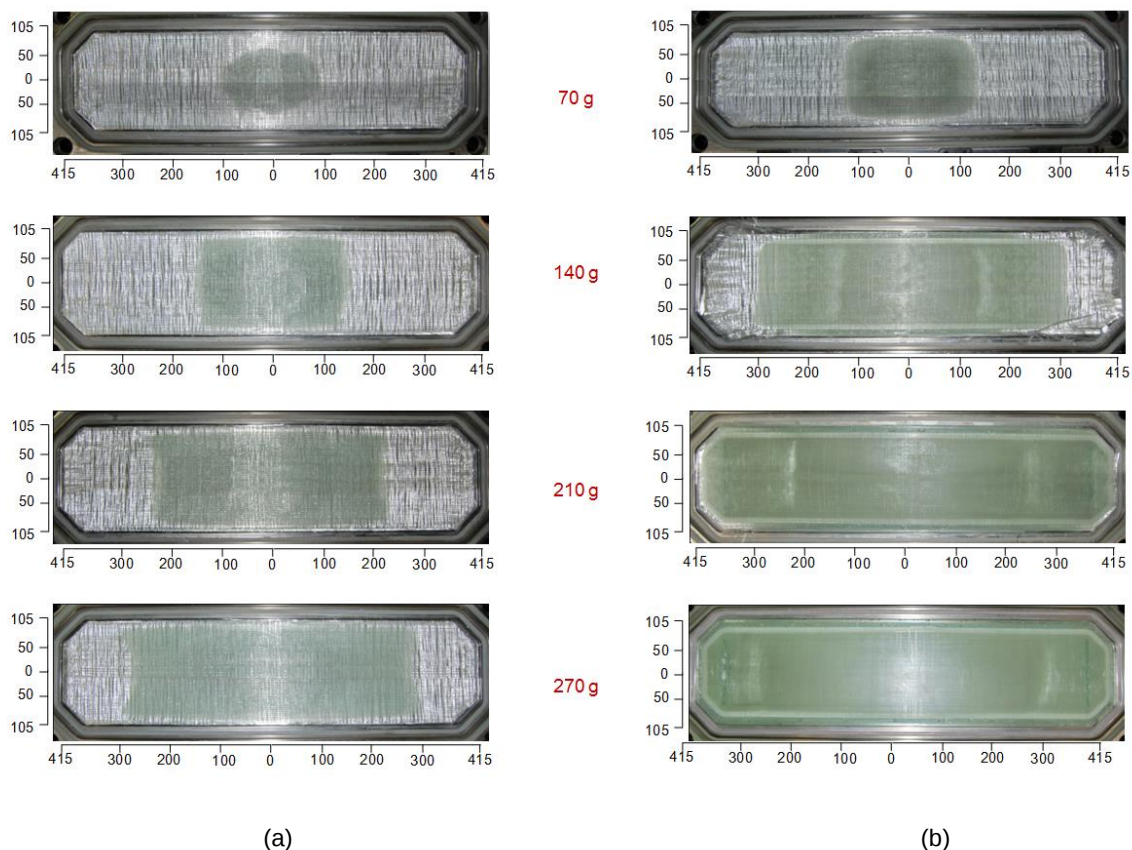
Results of mold fill study for bidirectional laminates

The flow front progression for bidirectional laminates after injection and before compression is shown in Picture 6.2 (a) and the flow front progression after injection and compression is shown in Picture 6.2 (b).

Flow front progression for bidirectional laminates at a resin amount of 70 g:

In case of bidirectional laminates, after injecting 70 g of resin and before application of compression force an oval shaped resin flow front progression with 180 mm length and 120 mm width was observed. In comparison to unidirectional laminates, formation of “local resin rich areas” shaped like a human tongue was not observed. The formation of the oval shaped geometry and the absence of local resin rich areas can be explained on the basis of the bidirectional laminate layup $[0/90]_s$. The chosen bidirectional laminate layup had a top and bottom layer based on the unidirectional layer which was oriented along the length of the mold. One of these unidirectional layers (bottom layer) was in direct contact with the resin, as the resin was injected into the mold cavity from the bottom side which led to a more dominant

flow of resin in lengthwise orientation to the mold. The presence of two layers in the middle of laminate layup, with a 90° orientation to the mold length, resulted in an overall increased permeability of laminate layup across the mold width than the entire unidirectional laminate layup studied earlier. The resin penetrating through the unidirectional layer from the bottom side of the mold was therefore distributed across the width of the mold due to the higher permeability of the two layers with 90° orientation to the mold length, thereby leading to homogenous distribution of resin along the width, and a consequent absence of “local resin rich areas” along the length of the mold. After compression and curing of the laminates, a rectangular shaped flow front progression of approximately 270 mm length and 160 mm width was observed. Due to a rectangular shaped flow front progression in bidirectional laminates the maximum resin flow length was only 270 mm compared to a maximum flow length of 310 mm for oval shaped flow front progression in unidirectional laminates.



Picture 6.2: Flow front progression in CRTM process as a function of injected resin amount for bidirectional laminate layup; (a) before compression, (b) after compression (mold dimension indicated in centimeters on the scale)

Flow front progression for bidirectional laminates at a resin amount of 140 g:

For an injected resin amount of 140 g a flow front progression of 300 mm length (approximately 150 mm on either side of the injection point) and 160 mm width was observed before compression. After application of compression force a rectangular shaped flow front progression of 600 mm length and 210 mm width was observed. As seen in Picture 6.2 (b) in the cured laminate, the formation of areas with displacement of rovings and poor quality impregnation on either side of the mold was observed, at a distance of approximately 150 mm from the injection point. This roving displacement was not observed after the end of the resin injection and before compression. The occurrence of roving displacement in the laminate coincides with the observed resin flow front progression at the end of resin injection and before application of compression force. This indicates that the application of compression force led to the displacement of rovings in the middle layers of the laminate with a 90° orientation to the mold length at the flow front progression obtained after completion of the resin injection step. The occurrence of roving displacement must have led to a local reduction of the permeability (due to a local increase of roving content) thereby leading to poor quality impregnation of the layers in such areas. Obtaining a 210 mm flow front width in bidirectional laminates at an injected resin amount of 140 g, compared to 160 mm flow front width in unidirectional laminates, can again be explained on the basis of the bidirectional laminate layup ([0/90]_s). The presence of two layers with a 90° orientation to the mold length led to a comparative increase in resin flow along the width of the mold compared to unidirectional laminates, due to the increased permeability of the bidirectional laminate layup across the width of the mold.

Flow front progression for bidirectional laminates at a resin amount of 210 g:

At an injected resin amount of 210 g a flow front progression of 450 mm length (approximately 225 mm on either side of the injection point) and 210 mm width was measured before application of a compression force for bidirectional laminates. After application of compression force and laminate a front progression of 820 mm length and 210 mm width was observed. In this case as well, in the cured laminates roving displacement and poor quality impregnation was observed at a distance of 225 mm from the injection point, which again coincides with the flow front progression length obtained after completion of the resin injection step and before application of compression force.

Flow front progression for bidirectional laminates at a resin amount of 270 g:

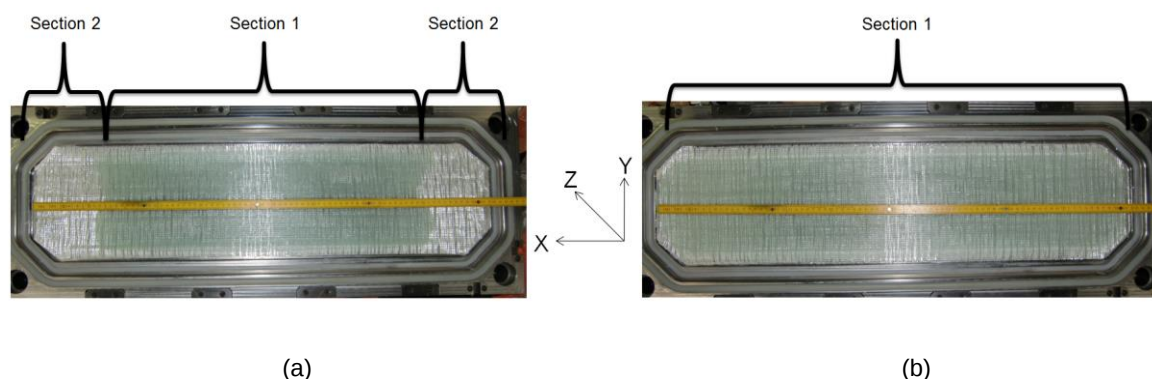
For bidirectional laminates at an injected resin amount of 270 g, flow front progression of 580 mm length (approximately 290 mm from the injection point) and 210 mm width was

observed before application of compression force. After application of compression force bidirectional laminates were completely impregnated; however, the occurrence of roving displacement and thus the formation of areas of poor impregnation quality were still observed in the laminates. The roving displacement was observed at a distance of 290 mm from the injection point, coinciding with the flow front progression length obtained after resin injection and before compression.

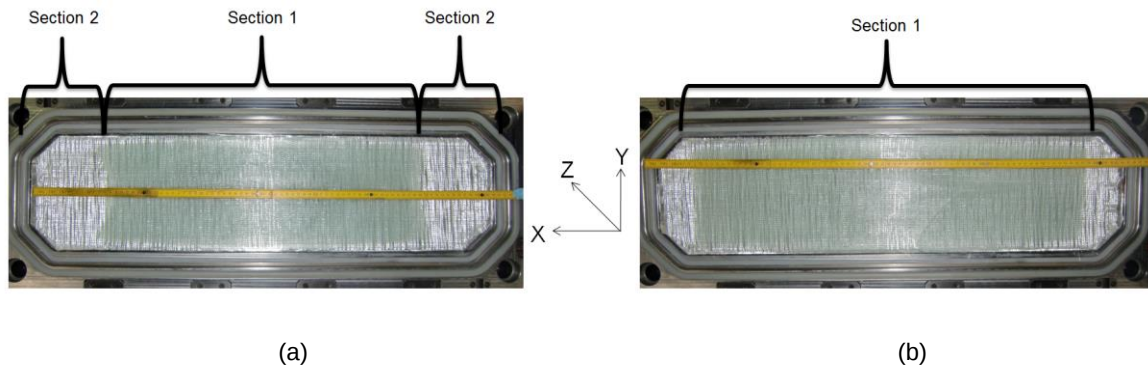
The conducted mold fill study indicated that at 2 mm mold gap value, depending on laminate layup, a different resin flow front progression was observed before and after application of compression force at a particular injected resin amount. Also, the problem of roving displacement was observed using bidirectional laminates which was absent using unidirectional laminates. In the next step of the feasibility study for the CRTM process, the degree of mold filling after resin injection in a partially open mold gap and before application of compression force was determined for the defined laminate layups at a mold gap of 1 mm, in order to compare the degree of mold filling with a 2 mm mold gap.

Results of mold fill study as a function of selected mold gap value

The study of mold fill as a function of selected mold gap values was conducted using an injected resin amount of 270 g. The degree of mold filling can be seen in Picture 6.3 and Picture 6.4 at 1 mm and 2 mm mold gaps for unidirectional and bidirectional laminates respectively. As it can be seen from the pictures, at 2 mm mold gap, the resin covers approx. 70 % of the mold surface and also impregnates the fabrics partially over this area for both fabric layups. At 1 mm mold gap the resin covers almost the entire mold surface and partially impregnates the fabrics.



Picture 6.3: Flow front progression after resin injection and before compression in the CRTM process for unidirectional laminates; (a) at 2 mm mold gap, (b) at 1 mm mold gap



Picture 6.4: Flow front progression after resin injection and before compression in the CRTM process for bidirectional laminates; (a) at 2 mm mold gap, (b) at 1 mm mold gap

From the analysis of the degree of mold filling before the compression step, as shown in Picture 6.3 and Picture 6.4, two different sections of the CRTM laminates can be identified in terms of their impregnation behavior. At 1 mm and 2 mm mold gap, section 1 is partially impregnated after the resin injection step and prior to the compression step. In case of 1 mm mold gap, the resin covers almost the entire surface of the mold after resin injection for unidirectional laminate layup. Only a small section in case of bidirectional laminates is not covered by resin at 1 mm mold gap. However this non-impregnated area is outside the effective plate geometry (700 mm x 130 mm) which was used for testing of laminate properties. In case of 2 mm mold gap for unidirectional as well as bidirectional laminates the non-impregnated area was observed to be inside the effective plate geometry to be used for materials testing. The application of compression pressure at 1 mm mold gap therefore leads to a compaction of fabrics as well as further impregnation of fabrics in Z direction in section 1, due to almost no flow of resin in X and Y directions for unidirectional laminates and very little amount of resin flow in X any Y directions for bidirectional laminates. In case of a 2 mm mold gap, the application of compression pressure leads to compaction of the fabrics as well as further impregnation in section 1. It also leads to the impregnation of dry fabrics in section 2 due to the resin flow in the X and Y directions resulting from additional resin which is squeezed out from section 1.

For characterization of CRTM laminates the differences in the impregnation behavior were taken into account and the laminates produced using 1 mm mold gap and 2 mm mold gap were characterized by testing ILSS properties in section 1 and section 2. In the case of the 2 mm mold gap, the samples identified as “near injection point (NIP)” were located in section 1 which is partially impregnated during the resin injection step and further impregnated by application of compression force. The samples identified as “away from injection point (AIP)” were located in section 2 which is purely impregnated by the resin flow

during the compression step. In case of 1 mm mold gap, as concluded, only section 1 was identified and section 2 did not exist within the effective plate geometry to be used for materials testing. Hence in case of the laminates manufactured using 1 mm mold gap the samples identified as near and away from injection point resemble only the respective location of ILSS samples taken from laminates manufactured using 2 mm mold gap.



Picture 6.5: Partially impregnated RTM laminate - unidirectional laminate layup (bright area showing partial impregnation, dark area showing dry fibers)

Also, as a part of the feasibility study for the CRTM process it was planned to compare the properties of laminates manufactured using the CRTM process with laminates manufactured by the classical RTM process in which no mold gap was used. The classical RTM experiments were also conducted using plate mold 1, selected resin, reinforcing glass fabric and processing equipment to manufacture reference RTM laminates in order to compare properties. However, it was found that it was not possible to obtain a fully impregnated laminate after a significantly longer resin injection time (approx. 6 min) during manufacturing of the laminates using the conventional RTM process. The chemo-rheological characterization of the resin at 100 °C showed that the resin starts to gel after approximately 140 seconds (see Table 5.1) and hence even after 6 min resin injection time complete filling of the mold was not achieved. The manufactured RTM laminate with partial and bad quality impregnation is shown in Picture 6.5. As the manufacturing of the RTM laminates was not possible, only CRTM laminates were characterized in the next step.

6.1.2 Characterization of CRTM laminates

The sample preparation plan for testing the CRTM laminates and the testing parameters are given in annex 9.2.

Fiber volume content in laminates

The measurement of the fiber volume fraction in this thesis work was been carried out by conducting ashing experiments. In these experiments the composite sample weight (m_{sample}) was taken before ashing experiment and the weight of the reinforcement fiber material (m_{fibers}) was obtained after ashing of the matrix resin. The fiber weight content (ψ) in the sample was obtained using following equation:

$$\psi = \frac{m_{\text{fibers}}}{m_{\text{sample}}} \cdot 100 \quad \text{Equation 6.1}$$

The fiber volume content (V_f) of the samples was obtained using following equation

$$V_f = \left[1 / \left(1 + \frac{(100 - \psi) \cdot \rho_{\text{fibers}}}{\psi \cdot \rho_{\text{matrix}}} \right) \right] \cdot 100 \quad \text{Equation 6.2}$$

Where, ρ_{fibers} is the density of fiber material and ρ_{matrix} is the density of the matrix material.

Figure 6.2 shows the fiber volume content and part thickness of the laminates manufactured using unidirectional and bidirectional laminate layups and 1 mm and 2 mm mold gaps. The fiber volume content values shown in Figure 6.2 for each laminate layup and mold gap combination is an average value of the fiber volume content (each average value based on at least 5 samples) measured in two predefined sections of the laminate, namely near injection point and away from injection point.

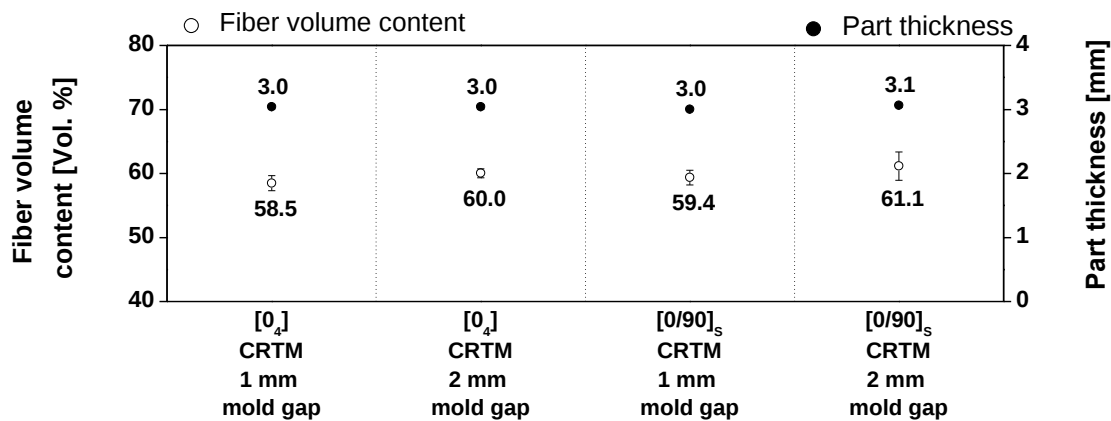


Figure 6.2: Effect of mold gap on the fiber volume content and thickness of the laminates in the CRTM process feasibility study

As can be observed, laminates manufactured using different layup and mold gap combinations exhibited nearly equivalent part thickness in the range of 3 mm to 3.1 mm. The fiber volume content in laminates varied between 58.5 - 61 %. However for each laminate layup laminates manufactured using a 2 mm mold gap exhibited slightly higher fiber volume content compared to laminates manufactured using a 1 mm mold gap at equivalent laminate layups.

Mechanical properties of laminates

Tensile properties of samples in this thesis work were characterized according to the DIN EN ISO 527 norm. The used sample geometries and test parameters are given in annex 9.3. Tensile strength of samples was obtained by using following equation:

$$\sigma = \frac{F_{tensile}}{A} \quad \text{Equation 6.3}$$

Where, σ is tensile strength [MPa], $F_{tensile}$ is measured maximum tensile force [N] and A is the cross sectional area of the sample [mm²]. The secant tensile modulus (E_t) of samples was measured in the linear elastic range between 0.05 % and 0.25 % strain (ε) using following equation:

$$E_t = \frac{\sigma_{0.25\%} - \sigma_{0.05\%}}{\varepsilon_{0.25\%} - \varepsilon_{0.05\%}} \quad \text{Equation 6.4}$$

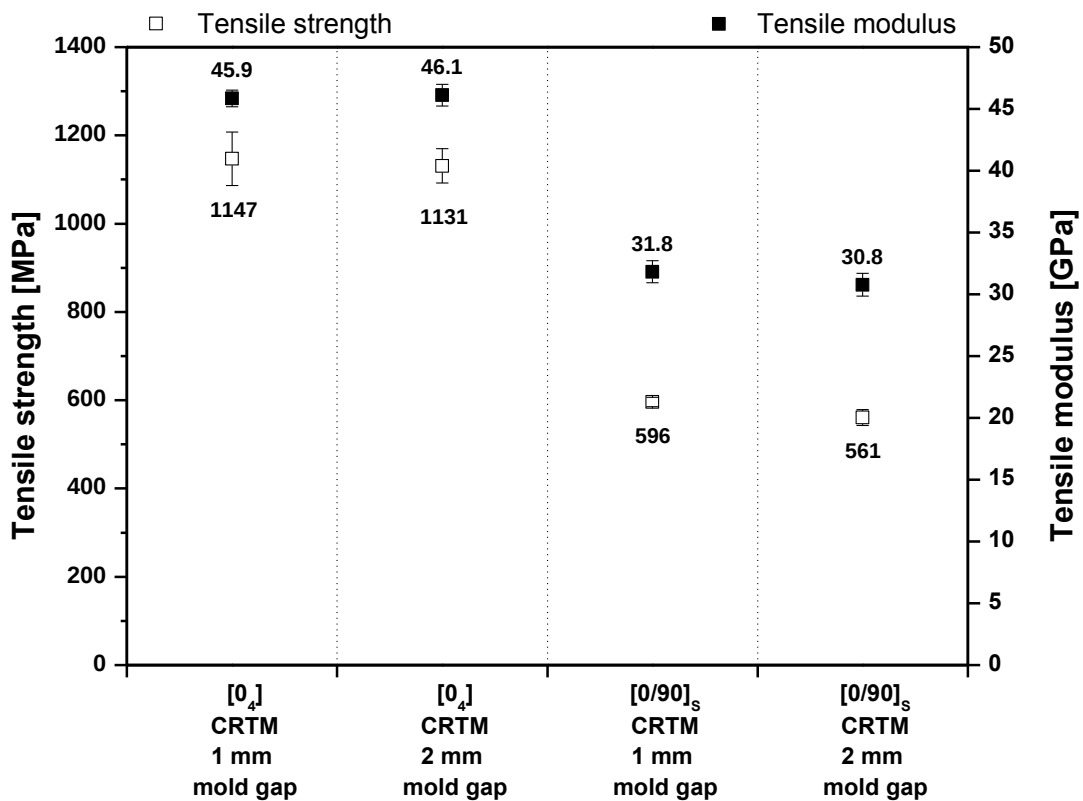


Figure 6.3: Effect of mold gap on tensile properties of the laminates in the CRTM process feasibility study

Figure 6.3 shows tensile properties of the CRTM laminates. The selection of a 1 mm or 2 mm mold gap exhibited nearly equivalent tensile properties at the respective laminate layup. For unidirectional laminates the average tensile strength values varied between 1147 - 1131 MPa and average tensile modulus values varied between 46.1 - 45.9 GPa,

whereas for bidirectional laminates the average tensile strength values varied between 596 - 561 MPa and average tensile modulus values varied between 31.8 - 30.8 GPa respectively for the selected 1 mm and 2 mm mold gap. The consistency of tensile properties at the equivalent laminate layup shows that during the CRTM process the application of compression pressure did not lead to any major displacement of the textile layers or fiber rovings in the final cured laminates, at least in the area from which tensile testing specimen was prepared. Also, the mold gap did not affect tensile properties of the laminates.

Flexural properties of samples were tested according to the DIN EN ISO 14125 norm. The used sample geometries and test parameters for flexural testing are given in annex 9.3. Flexural strength (σ_f) of the samples was obtained by using following equation:

$$\sigma_f = \frac{3 F_{flexural} L}{2 b h^2} \quad \text{Equation 6.5}$$

Where, $F_{flexural}$ is the measured maximum flexural force, L is the sample support length, b is the sample width, h is the sample thickness. Flexural modulus (E_f) of the samples was obtained using following equation:

$$E_f = \frac{L^3}{4 b h^3} \left(\frac{\Delta F}{\Delta s} \right) \quad \text{Equation 6.6}$$

Where, ΔF is the difference of flexural force measured at respective strain difference of Δs . Flexural modulus was measured at strain values of 0.05 % and 0.25 %.

Figure 6.4 shows the effect of the mold gap on flexural properties of the CRTM laminates. As can be seen for unidirectional laminates the measured flexural properties with a 1 mm and 2 mm mold gap were almost identical to each other as the average flexural strength varied between 1270 - 1228 MPa and the average flexural modulus varied between 37.3 - 37.5 GPa respectively. Flexural properties of bidirectional laminates were significantly higher though such a laminate layup consisted of two textile layers in 90° orientation to the lengthwise orientation of the test specimen. The explanation for the higher flexural properties can be given on the basis of the used laminate layup. The selected bidirectional laminate layup ($[0/90]_s$) consisted of a 0° layer on the top and the bottom of laminate layup. The presence of the 0° layer as an outer layer resulted in significantly higher flexural properties for the bidirectional laminate layup. For bidirectional laminates, however, a slight drop in flexural strength was observed with a 2 mm mold gap compared to flexural strength measured for the laminates produced using a 1 mm mold gap. Flexural strength value decreased to 1037 MPa with a 2 mm mold gap compared to flexural strength value of 1139 MPa for a 1 mm mold

gap. The explanation for this decrease in flexural strength at a 2 mm mold gap is given on the basis of Picture 6.6.

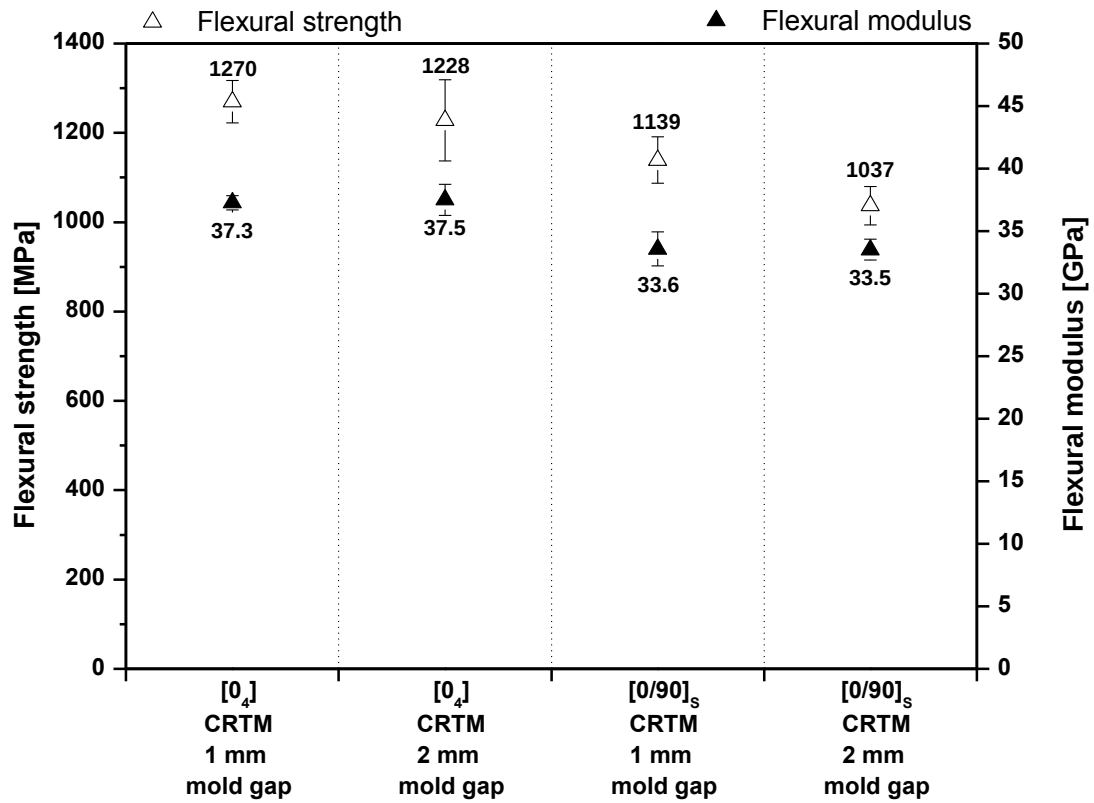


Figure 6.4: Effect of mold gap on flexural properties of the laminates in the CRTM process feasibility study

Inter-laminar shear strength (ILSS) samples were tested according to the DIN EN ISO 14130 norm. The used sample geometries and test parameters for ILSS testing are given in annex 9.3. ILSS (τ) of the samples was obtained using following equation:

$$\tau = \frac{3 F_{shear}}{4 b h} \quad \text{Equation 6.7}$$

Where, F_{shear} is the maximum measured shear force, b is the sample width and h is the sample thickness.

Figure 6.5 shows the inter-laminar shear strength (ILSS) of the unidirectional and bidirectional laminates in correlation to the fiber volume content and the laminate thickness. As can be seen, ILSS properties were measured at two different locations within the laminates. For characterization of the laminate impregnation quality by ILSS testing, samples were taken from the sections near and away from injection point. As can be seen in Figure 6.5, similar to tensile and flexural properties, ILSS properties of unidirectional laminates were not influenced by the selected mold gap value. The fiber volume content and ILSS properties of unidirectional laminates in the sections near and away from injection point

were almost identical to each other for the selected mold gap of 1 mm and 2 mm. ILSS values varied between 57.6 - 59.4 MPa and the fiber volume content values varied between 58.3 - 60.4 % for unidirectional laminates for these two mold gaps.

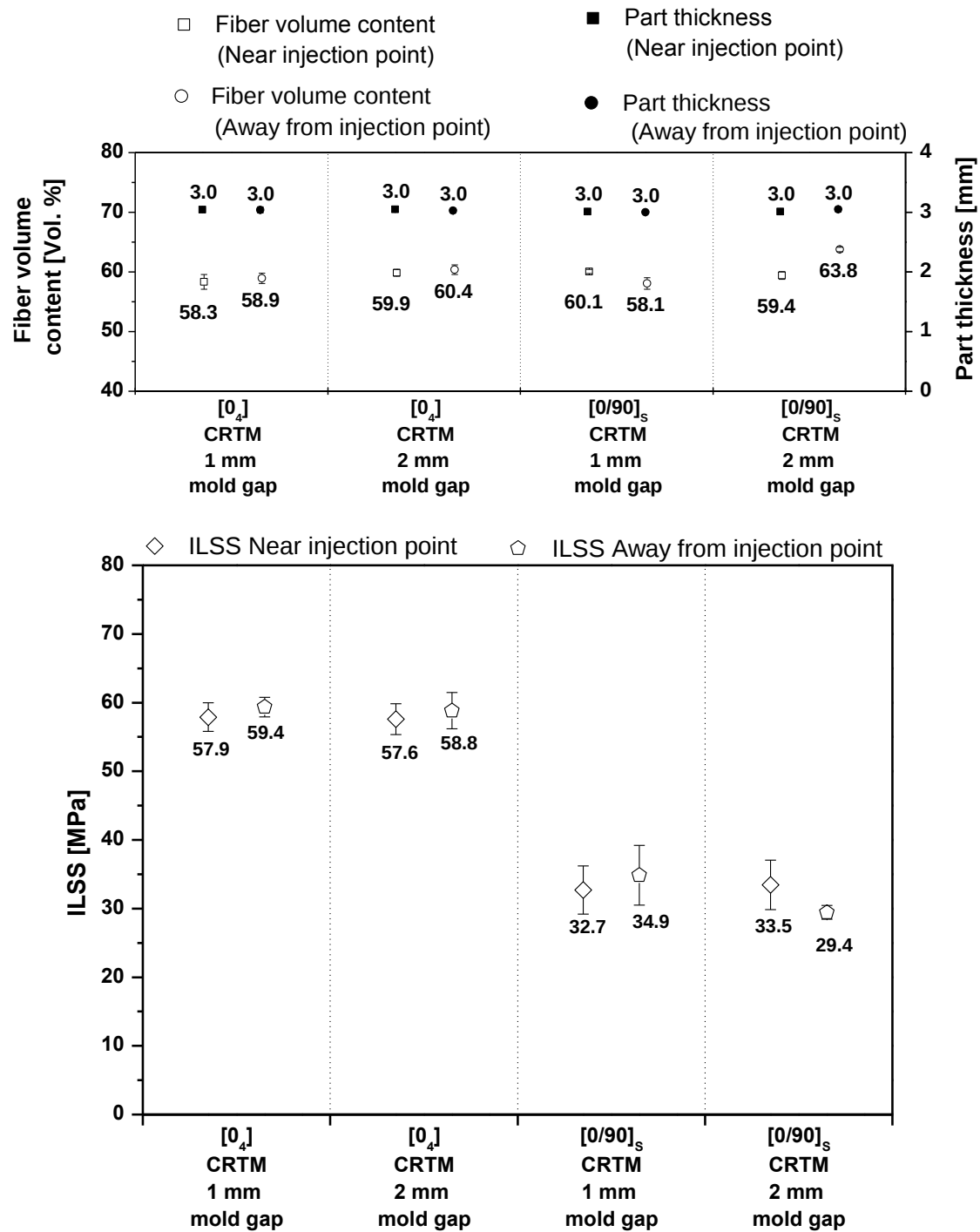
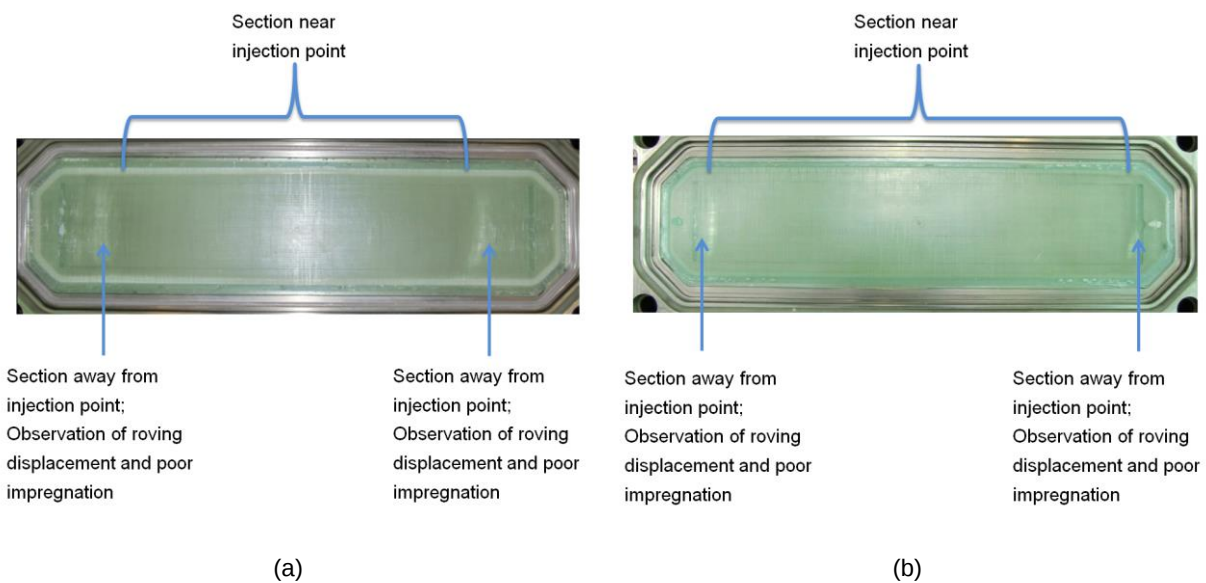


Figure 6.5: Effect of mold gap on ILSS properties of the laminates in the CRTM process feasibility study

For bidirectional laminates with a 2 mm mold gap ILSS value dropped to 29.4 MPa in the section away from injection point compared to ILSS value of 33.5 MPa in the section near injection point. Also for bidirectional laminates the measured fiber volume content in the section away from injection point was significantly higher (63.8 %) than in the section near injection point (59.4 %) with a 2 mm mold gap. The fiber volume content and ILSS of bidirectional laminates in the sections away from injection point and near injection point were identical to each other with a 1 mm mold gap as ILSS values varied between 32.7 - 34.9 MPa and the fiber volume content values varied between 58.1 - 60.1 %.

Picture 6.6 explains the probable reason for the drop in ILSS properties and flexural strength in bidirectional laminates in the section away from injection point for a 2 mm mold gap in comparison to a 1 mm mold gap. As can be seen from Picture 6.6 the section near injection point was well impregnated for 1 mm and 2 mm mold gaps. However in the section away from injection point the 90° layers showed roving displacement due to the resin flow under compression pressure leading to poor quality impregnation.



Picture 6.6: Difference in impregnation quality of CRTM laminates; (a) 2 mm mold gap, (b) 1 mm mold gap

In case of a 1 mm mold gap the section away from injection point, where the poor quality impregnation and roving displacement was observed, was outside the test area of the laminate and no samples were taken from this section. However, in the case of a 2 mm mold gap the section away from injection point, where poor quality impregnation and roving displacement was observed, coincided with the test area of the laminate, and from this section samples were taken for testing. The poor quality impregnation and roving displacement resulted in measurement of higher fiber volume content (63.8 %) compared to the fiber volume content measured in the section near injection point (59.4 %) for a 2 mm

mold gap. This also caused a drop in ILSS properties in the section away from injection point compared to ILSS properties close to injection point for a 2 mm mold gap.

6.1.3 Conclusion of the feasibility study

Using the feasibility study hypotheses 1 & 2 were validated. The mold fill study validated hypothesis 1 according to which differences in permeability of textile reinforcements (K_x , K_y , and K_z) in a particular mold geometry may lead to different resin flow velocities (V_x , V_y , and V_z). The differences in resin flow velocities which shall lead to different resin distribution in textile reinforcements were characterized by studying the resin flow front progression in the mold cavity along two different directions (x and y). The mold fill study concluded that the permeability of laminate layup plays a significant role in the flow front progression (i.e. resin flow velocity) of resin before and after application of compression force, depending on the selected resin injection amount. Also, the mold gap significantly affected the resin flow front progression before and after compression for both laminate layups. Especially the impregnation quality of bidirectional laminates was affected by the resin flow front progression. For bidirectional laminates, depending on the degree of mold filling after resin injection and before application of compression force (which is determined by the selected mold gap), strong roving displacement was observed at the flow front in the textile layers with 90° orientation to the mold length. For unidirectional laminates, however, such an effect was not observed due to dominant unidirectional resin flow. The conducted feasibility study showed that the CRTM process can be used for obtaining quick resin injection due to the higher permeability of the non-compacted textiles and also quick impregnation of the laminates due to the shorter time required for the compression step. In the CRTM process the resin injection was completed in 30 - 33 seconds and the mold was closed in approximately 5 - 10 seconds (depending on the selected mold gap), leading to completion of impregnation of the reinforcing fibers by the resin. Using the selected materials, mold concept and injection equipment it was not possible to manufacture the laminates by the conventional RTM process as the permeability of the textiles was strongly reduced in the conventional RTM process and low injection pressure and low flow rate of the selected resin injection equipment could not lead to complete impregnation of the laminate. These investigations of CRTM process and conventional RTM processes validated hypothesis 2 according to which for a selected mold geometry, resin viscosity (η) and resin injection pressure as being constants, the resin flow rate (Q) and thus the resulting mold filling time (T) shall be purely affected by permeability (K) of reinforcements in selected process configuration. The resin injection time of 30 - 30 seconds in the CRTM process resulted from the slow injection speed / flow rate of the used equipment. It indicates that the

resin injection time for the CRTM process can be further reduced if alternative resin injection equipment is used which allows higher flow rate of the resin. If such equipment is used then it could also allow the use of resins with higher reactivity to reduce the curing time of the laminates in the CRTM process. Hence, new experiments were conducted to investigate the CRTM process using a high-pressure RTM equipment (hereafter referred as HP-CRTM process) which allows a higher resin injection speed and is capable of using resins of higher reactivity. Also, the mold geometry was varied to study the effect of the component geometry on the HP-CRTM process.

6.2 Detailed investigation of HP-CRTM process for manufacturing glass fiber reinforced composites

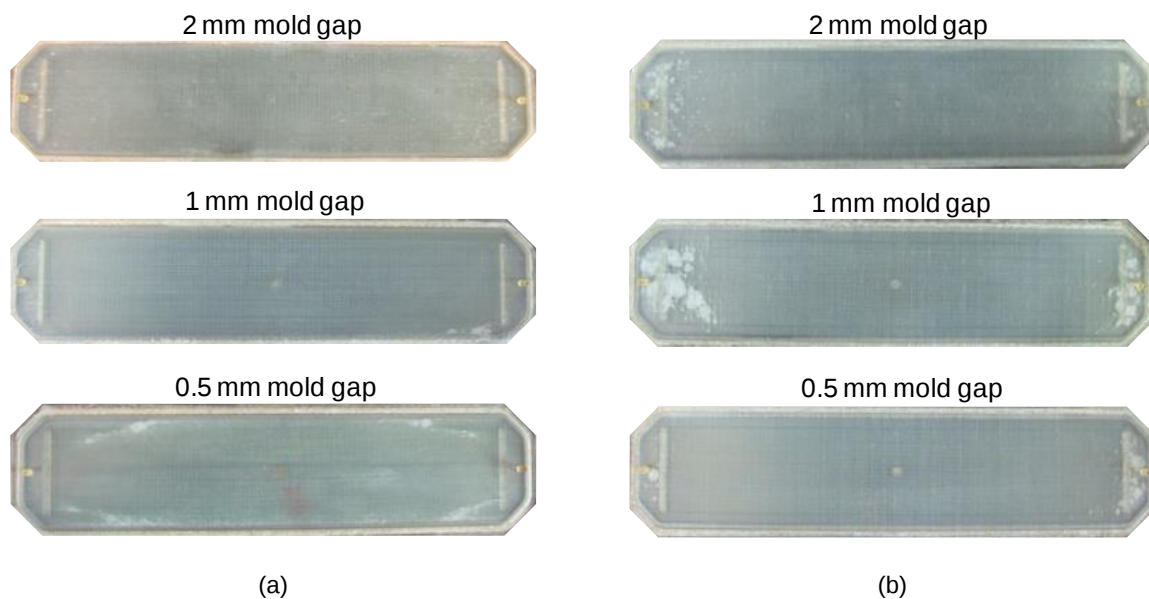
The detailed investigations of the HP-CRTM process included evaluating the effects of mold gap, mold geometry and laminate layup in the process. For these investigations a fast curing resin system based on an epoxy resin commercially available as Araldite® LY 564, and an amine hardener commercially available as Hardener XB 3458, was used. For mixing and dosing of the selected resin system a high-pressure RTM equipment was used to obtain a relatively high resin flow rate compared to the feasibility study for the CRTM process. The same non-crimp glass fabric as in the feasibility study was used as reinforcement. The details of the conducted investigations are discussed below.

6.2.1 Effect of mold gap on laminates manufactured by HP-CRTM process

6.2.1.1 Laminates manufactured by HP-CRTM process using plate mold 1

The effect of the mold gap on the HP-CRTM process was investigated based on the results of the feasibility study. From the feasibility study a conclusion was made that unidirectional laminates of equivalent optical impregnation quality and nearly constant mechanical properties were obtained for 1 mm and 2 mm mold gaps. In the case of bidirectional laminates, however, the effect of fiber roving displacement was observed at both the mold gap values. The goal of investigating the effect of the mold gap in the HP-CRTM process was to use a new resin system, new resin processing equipment and to understand the effect of an even smaller mold gap on the laminate quality. In this study, similar to the feasibility study, two different fiber orientations ($[0_4]$ and $[0/90]_s$) were therefore used to study the effect of the mold gap selection on the HP-CRTM process. The mold gaps of 0.5 mm, 1 mm and 2 mm were used, resulting in the effective gaps of 0.2 mm, 0.7 mm and 1.7 mm respectively. The required amount of the resin, 410 g and 420 g resin for unidirectional and bidirectional laminate layup respectively, was injected into the partially open mold cavity in 7.5 seconds using high-pressure RTM equipment. The resin amounts of 410 g and 420 g for injection were defined on the basis of a theoretical calculation of the resin amount required

for laminate impregnation (235 g), the required amount of resin for filling the free volume of the injection tubes (110 g) and an additional amount of resin of 65 g and 75 g for unidirectional and bidirectional laminates respectively. This additional amount of resin was not considered in the feasibility study. The purpose of injecting an additional amount of resin into the cavity was to evaluate whether by increasing the resin amount laminates with better impregnation quality can be obtained (probably due to higher compression pressure build-up on the materials than in the feasibility study). The epoxy resin was preheated to 60°C and the hardener was held at room temperature in high-pressure RTM equipment while manufacturing the laminates. As the defined amount of the resin was injected into the partially open cavity, immediately after that the mold was closed completely at closure speed of 0.2 mm/s in the case of a 1 mm and 2 mm mold gap and 0.1 mm/s in the case of a 0.5 mm mold gap, to apply a compression force of 1060 kN. The mold temperature was 100 °C and the laminates were cured in the mold at this temperature for 4 min. Further details of the process parameters applied are given in annex 9.1 [94, 95].



Picture 6.7: Impregnation quality of the HP-CRTM laminates manufactured using different mold gaps;
(a) Laminate layup $[0_4]$, (b) Laminate layup $- [0/90]_s$

The optical comparison of the laminates manufactured using different mold gaps is shown in Picture 6.7. As can be seen, unidirectional laminates manufactured using a 2 mm mold gap showed the presence of partially impregnated rovings and air entrapment, especially on the surface of the laminates. The laminates manufactured using a 1 mm mold gap showed the best impregnation quality of the laminates. The laminates manufactured using a 0.5 mm mold gap showed the presence of dry areas in the laminates. For bidirectional laminates, air entrapment was observed in all the laminates in the farthest areas of the laminates on either

side of the injection point, irrespective of the selected mold gap values. The air entrapment was significantly higher in the laminates manufactured using 2 mm and 1 mm mold gaps, and only slight air entrapment was observed in the laminates manufactured using a 0.5 mm mold gap. This shows that depending on laminate layup, the selection of the mold gap plays a significant role in the HP-CRTM process to obtain laminates with good impregnation quality. Also, the selection of a new resin system and resin injection equipment resulted in a very significant change in the impregnation behavior of the laminates compared to the feasibility study conducted using same mold gap values.

In the HP-CRTM mold gap study the problem of roving displacement for bidirectional laminates, which was observed in the feasibility study, was not observed. The resin system used in the feasibility study - Resin MGS® RIMR 935 and hardener MGS® RIMH 936 - was held at room temperature in the resin injection equipment and then injected into the mold cavity at a mold temperature of 100 °C. Such a resin system at room temperature has a resin - hardener mixture viscosity of 340 - 400 mPa.s. The epoxy resin Araldite® LY 564 in the HP-CRTM process was preheated to 60°C, the Hardener XB 3485 was held at room temperature in the resin injection equipment and the resin mixture was injected in the mold cavity at a mold temperature of 100 °C. The resin system used in the HP-CRTM process has a resin - hardener mixture viscosity of 60 - 90 mPa.s at 60 °C. Hence selection of the resin system with higher viscosity in the feasibility study, compared to the low viscosity resin in the current study, must have led to the formation of higher pressure gradient at the resin flow front obtained at the end of resin injection and at the start of the compression step, thereby leading to roving displacement at the flow front which was visible in the manufactured laminates exactly at the same flow front position after the compression and curing steps were completed. The manufactured HP-CRTM laminates were characterized in order to understand the effect of the mold gap selection on the mechanical and physical properties of the laminates. The results of these characterizations are given in the next sub chapter.

6.2.1.2 Characterization of HP-CRTM laminates manufactured using plate mold 1

Fiber volume content in laminates

The sample preparation plan used for testing the HP-CRTM laminates and the testing parameters are given in annex 9.2. As the mold geometry and laminate layups used in the current study were similar to the feasibility study, the additional 65 g to 75 g resin injected into the mold cavity in the current study must have led to a slightly longer flow front progression at the end of resin injection and before compression for these laminates at mold gaps of 1 mm and 2 mm. In order to compare the properties of the samples in the feasibility

study and current study at their respective sample locations, the samples were again characterized near injection point and away from injection point. Hence the samples from the sections near and away from injection point in the current study have an identical location in the test area of the laminate as in the feasibility study. Figure 6.6 shows the effect of the mold gap on the fiber volume content and thickness of the unidirectional and bidirectional laminates manufactured using 0.5 mm, 1 mm and 2 mm mold gaps.

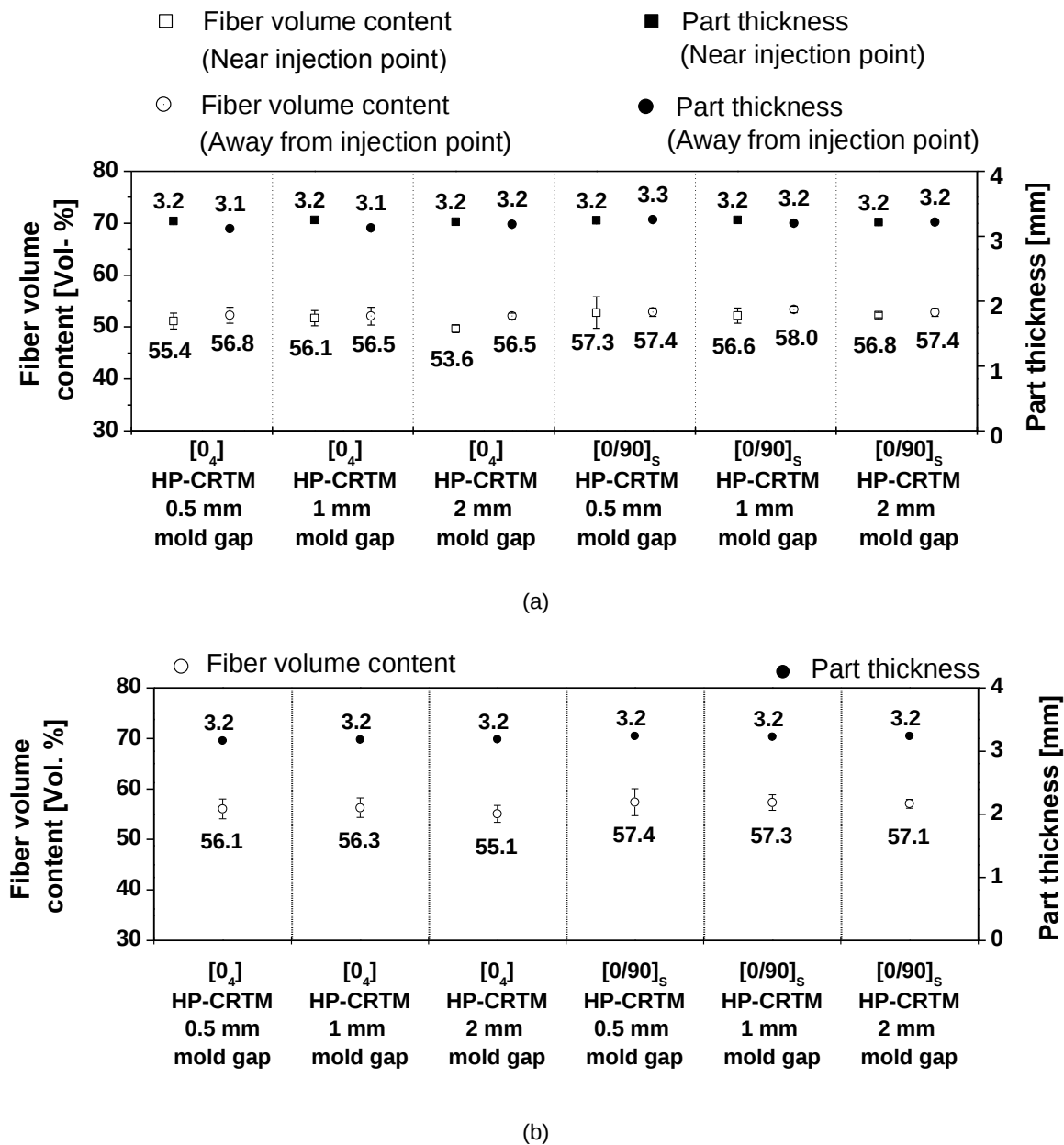


Figure 6.6: Effect of mold gap on the fiber volume content and thickness of laminates manufactured by the HP-CRTM process using plate mold 1; (a) in the sections near and away from injection point (b) average over the entire laminate

Figure 6.6 (a) shows the fiber volume content measured in the sections near injection point and away from injection point for each material - process combination. The average fiber volume content and laminate thickness of the entire laminate is shown in Figure 6.6 (b). As can be observed for all the materials and process combinations the laminates exhibited a nearly equivalent thickness of 3.1 to 3.2 mm in both sections. For unidirectional laminates manufactured using a 1 mm mold gap and bidirectional laminates manufactured using a 0.5 mm mold gap, almost constant fiber volume content was measured in the sections near and away from injection point. For the rest of the laminates slightly higher fiber volume content was measured in the section away from injection point compared to the fiber volume content in the section near injection point measured within the same laminate. For unidirectional laminates manufacturing using different mold gaps, the average fiber volume content over the entire laminate varied in the range of 55.1 % to 56.3 %.

Bidirectional laminates manufactured using different mold gaps exhibited nearly constant average fiber volume content over the entire laminate, with a variation between 57.1 % and 57.4 %. The laminates manufactured in the current study showed slightly (approx. 2-5 %) lower fiber volume content compared to the feasibility study. A probable explanation for this change in the fiber volume content is that the laminates in the current study had a slightly higher average thickness (3.2 mm) than the average laminate thickness of 3.0 mm obtained in the feasibility study. The higher laminate thickness in the current study must have resulted from the additional amount of injected resin which was 65 g for unidirectional laminates and 75 g for bidirectional laminates.

Mechanical properties of laminates

Figure 6.7 shows tensile properties of the HP-CRTM laminates. For each laminate layup, the selection of a mold gap of 0.5 mm, 1 mm or 2 mm led to similar tensile properties. In the case of unidirectional laminates an average tensile strength of 1013 - 1043 MPa and an average tensile modulus of 42.0 - 42.9 GPa were measured. For bidirectional laminates an average tensile strength of 476 - 531 MPa and an average tensile modulus of 29.3 - 30.0 GPa were measured. These measurements indicate that tensile properties of the laminates were consistent at different mold gaps. From tensile results it can be seen that for the current HP-CRTM process investigations the resin injection into the cavity at a relatively high flow rate (55 g/s) and the application of a high compression force on the laminates led to no displacement of the reinforcement layers or fiber rovings in the reinforcement layers, and consequently showed no adverse influence on tensile properties.

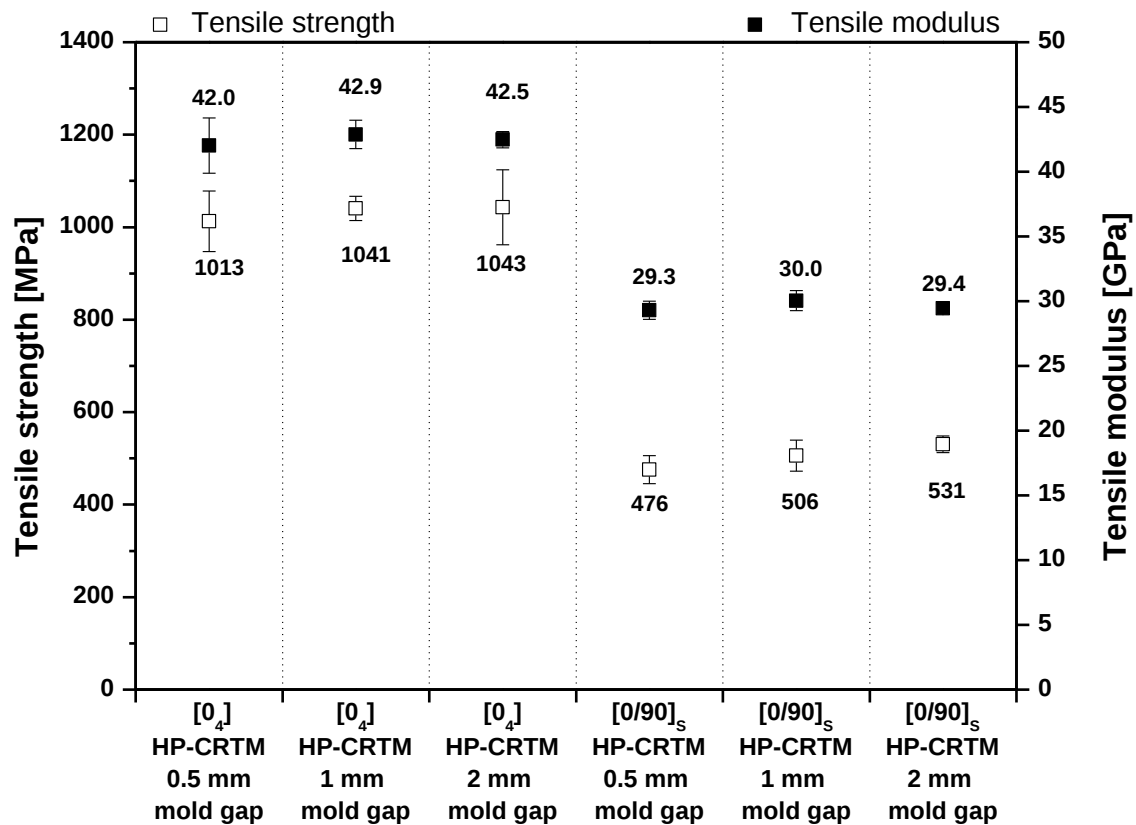
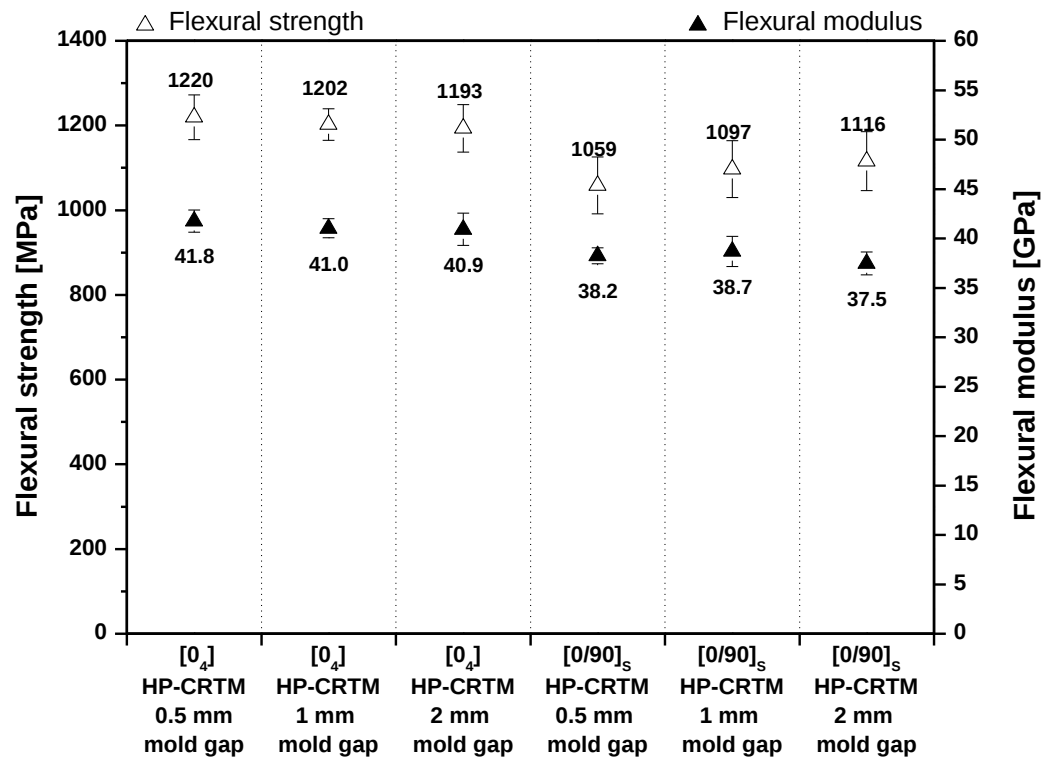
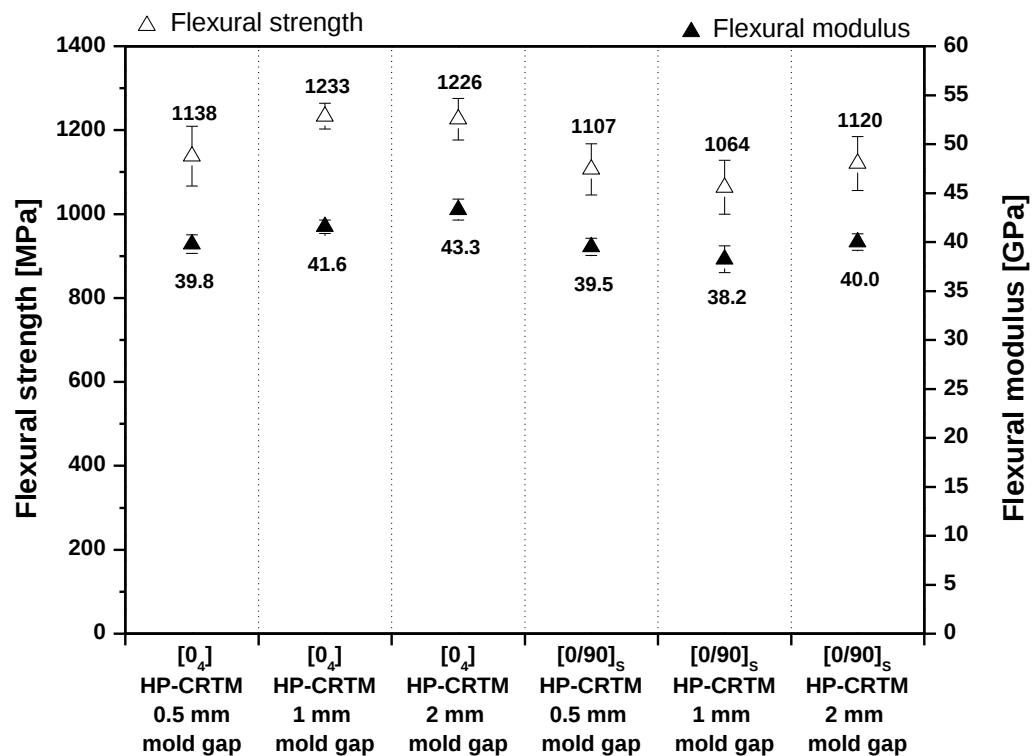


Figure 6.7: Effect of mold gap on tensile properties of the HP-CRTM laminates manufactured using plate mold 1

Figure 6.8 shows the effect of the mold gap on flexural properties of HP-CRTM laminates tested in lengthwise orientation (samples prepared along the mold length). Flexural properties of these laminates were measured near injection point and away from injection point. Flexural properties of unidirectional laminates in the section near injection point were almost constant. For unidirectional laminates the average flexural strength values between 1193 - 1220 MPa, and average flexural modulus values between 40.9 - 41.8 GPa were measured in the section near injection point. In the section away from injection point flexural properties of unidirectional laminates fluctuated slightly as average flexural strength values between 1138 - 1233 MPa and average flexural modulus values between 39.8 - 43.3 GPa were measured for unidirectional laminates. As can be seen, flexural properties of unidirectional laminates manufactured using 1 mm and 2 mm mold gaps were almost constant and equivalent in the sections near and away from injection point. It shall be noted that unidirectional laminates manufactured using a mold gap of 0.5 mm exhibited significant dry areas compared to unidirectional laminates manufactured using 1 mm and 2 mm mold gaps.



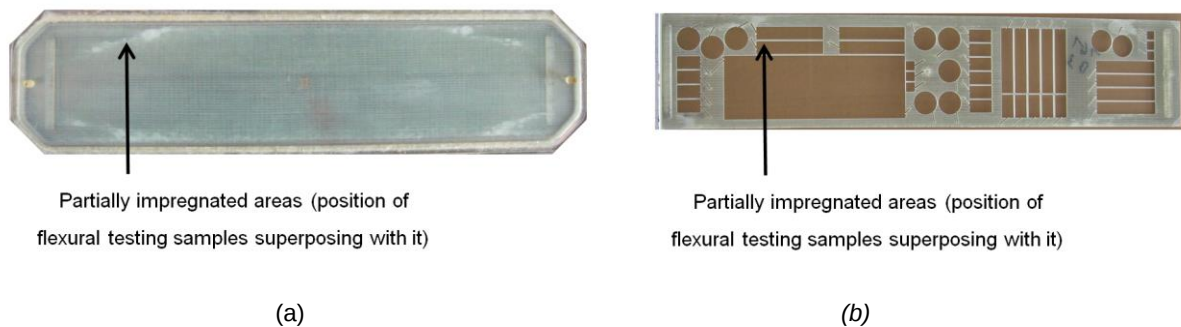
(a)



(b)

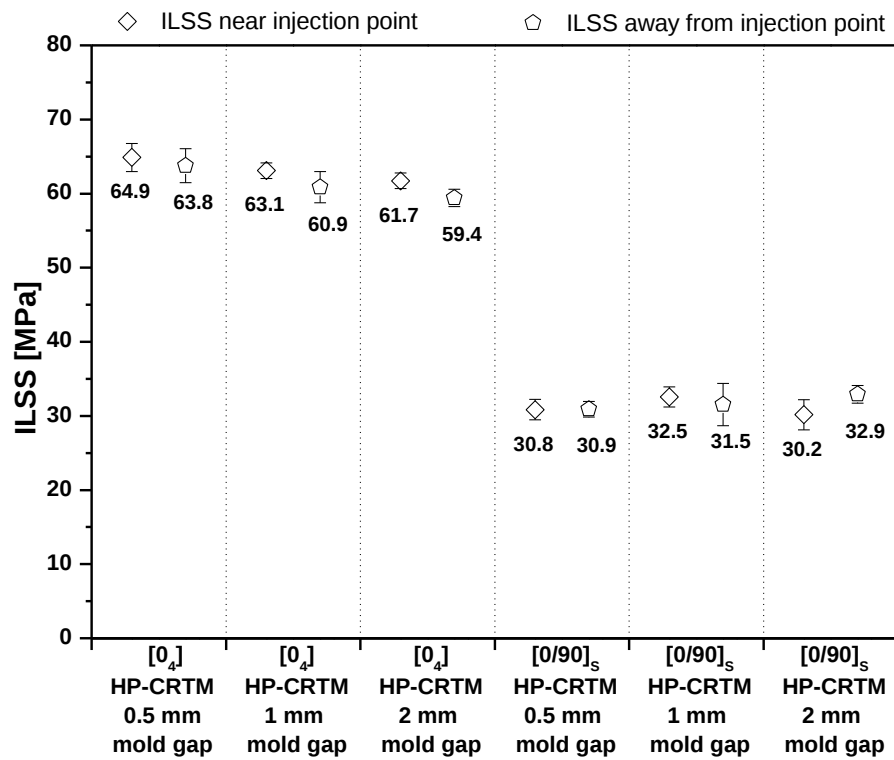
Figure 6.8: Effect of mold gap on flexural properties of the HP-CRTM laminates manufactured using plate mold 1, samples tested lengthwise; (a) Near injection point, (b) Away from injection point

Picture 6.8 shows unidirectional laminate manufactured using a 0.5 mm mold gap and the sample preparation plan used for characterizing all the laminates. As can be seen on the left side of Picture 6.8 such laminates show partially impregnated areas and the position of flexural testing samples was superposing with this partially impregnated areas of the laminates as seen on the right side of Picture 6.8. This must have led to lower flexural strength of 1138 MPa and flexural modulus of 39.8 GPa in the laminates manufactured using a 0.5 mm mold gap in the section away from injection point compared to a flexural strength of 1220 MPa and a flexural modulus of 42.0 GPa measured in the section near injection point for the respective laminates. However, partially impregnated areas did not seem to affect flexural properties of unidirectional laminates significantly, as not all the samples showed this partial impregnation and the standard deviation of flexural properties was within the acceptable range.

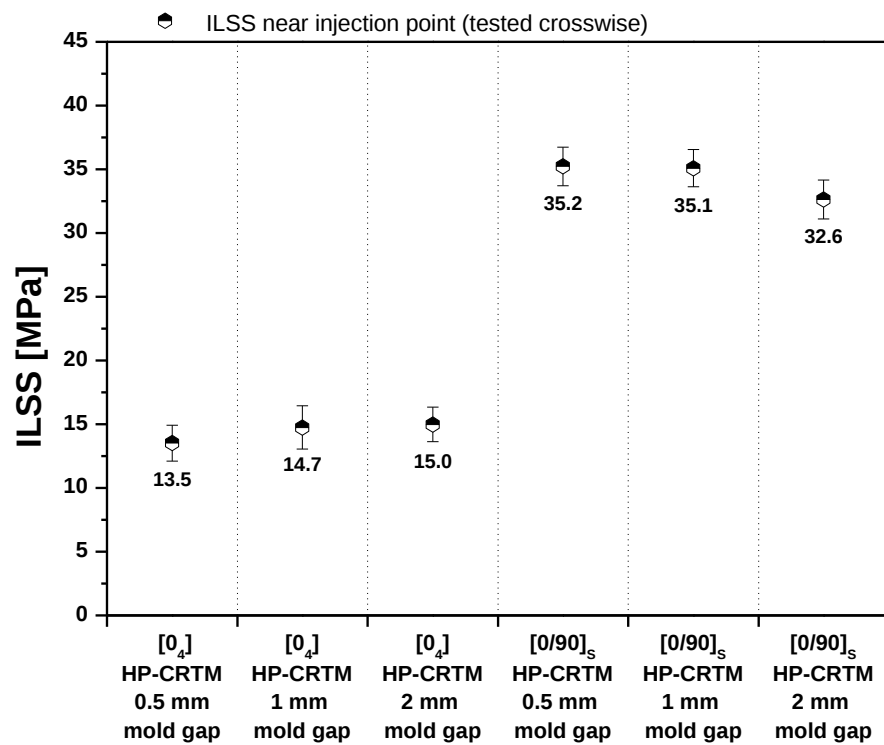


Picture 6.8: Impregnation quality of unidirectional laminates manufactured by the HP-CRTM process using a 0.5 mm mold gap (a) laminate after manufacturing, (b) sample cutting plan of the manufactured laminate

For bidirectional laminates, the average flexural strength values between 1059 - 1116 MPa and the average flexural modulus values between 37.5 - 38.7 GPa were measured in the section near injection point, and the average flexural strength values between 1064 - 1120 MPa and flexural modulus values between 38.2 - 40.0 GPa were measured in the section away from injection point. According to optical observations, bidirectional laminates (Picture 6.7) manufactured using a 0.5 mm mold gap showed the best impregnation quality compared to the laminates manufactured using a 1 mm and 2 mm mold gap which showed relatively high air entrapment in the outer sections of the laminates. However, the presence of the air entrapment did not result in any adverse effects on flexural properties of the laminates manufactured using 1 mm and 2 mm mold gaps. This effect can also be explained on the basis of the sample preparation plan. Flexural testing samples for bidirectional laminates were taken from the areas where no high air entrapment was observed and the areas of high air entrapment for bidirectional laminates were located at the far corners of the laminates.



(a)



(b)

Figure 6.9: Effect of mold gap on ILSS properties of the laminates manufactured by the HP-CRTM process; (a) samples taken from areas near injection point and away from injection point and tested lengthwise, (b) samples tested crosswise

Figure 6.9 (a) shows ILSS properties of the laminates characterized by taking samples from sections near injection point and away from injection point and testing them in lengthwise orientation (samples prepared along the mold length). As observed, ILSS properties of these laminates at the respective laminate layup were almost constant and were not affected by the chosen mold gap. For unidirectional laminates the average ILSS values varied between 59.4 - 64.9 MPa, and for bidirectional laminates the average ILSS values varied between 30.8 - 32.9 MPa in different sections of the laminates.

ILSS properties are generally dependent on the impregnation quality of the laminates, and thus ILSS can be sensitive to the presence of air entrapments or presence of voids in the laminates. The samples were therefore also tested crosswise to the mold length. These samples were taken from the section near injection point. During ILSS testing of crosswise samples, especially for unidirectional laminates, the stress is applied only on the matrix, and the fibers experience very little stress. Hence the presence of air entrapments or any irregularities in the impregnation quality should result in a decrease of ILSS properties of the laminates compared to ILSS properties of a well impregnated laminate. As seen in Figure 6.9 (b), the average ILSS values for the respective laminate layup crosswise to the mold length were also almost identical to each other and were not affected by the chosen mold gap. For unidirectional laminates, ILSS values varied between 13.5 - 15 MPa, and for bidirectional laminates ILSS values varied between 32.6 - 35.2 MPa, indicating that a near-equivalent impregnation quality was achieved in the section where ILSS samples were taken from the manufactured laminates and ILSS was not affected by the chosen mold gap value but ILSS was only affected by laminate layup.

Analysis of void content in laminates

The HP-CRTM laminates manufactured using plate mold 1 showed only slight variations in the mechanical properties at different mold gap values for a particular laminate layup. Hence in the next step, the morphology of the unidirectional and bidirectional laminates was studied. In order to study the morphology of the HP-CRTM laminates scanning electron microscope (SEM) was used and samples taken from sections near injection point and away from injection point were analyzed. The sample surface perpendicular to the mold length and resin flow direction was used for SEM analysis. The summary of SEM analysis in detail is available in annex 9.2. For the characterized samples the areal void content was calculated by measuring the surface area of the pores and surface area of the sample. The area of the pores was calculated using a surface area classification method in which the area of the

pores and the area of the fully impregnated section of the composites were identified as two different surfaces within the sample.

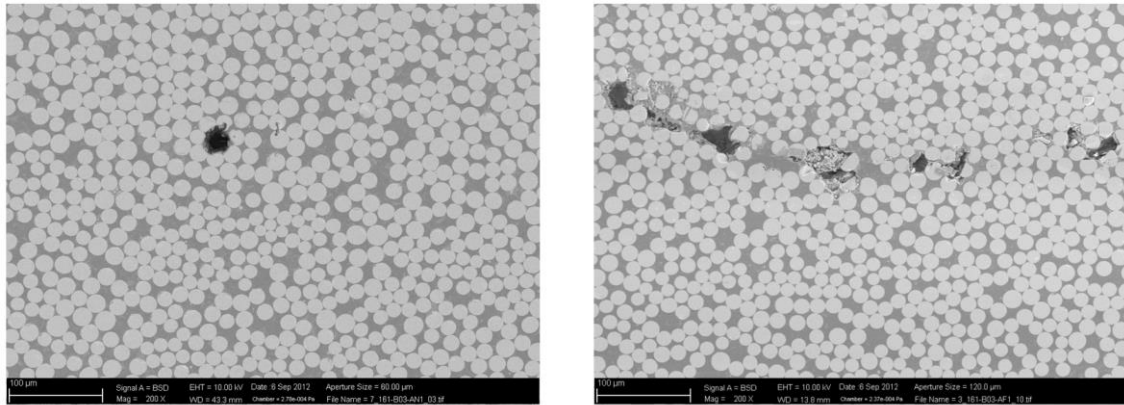
The areal void content in the samples in percentage was calculated using following equation:

$$Void\ content_{areal} = \frac{A_{pores}}{A_{sample}} \cdot 100 \quad \text{Equation 6.8}$$

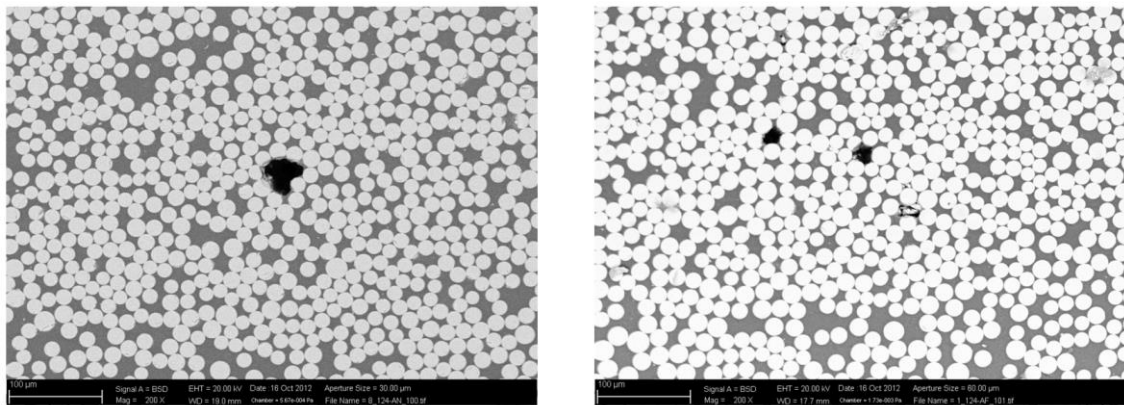
Where, A_{pores} is the sum of the area of all the detected pores in the specific sample area of A_{sample} .

The qualitative results of SEM analysis for unidirectional laminates at a magnification factor of 200 are shown in Picture 6.9. The reproducibility of SEM analysis was tested using unidirectional laminates and their results are given in annex 9.4. For the laminates manufactured using a 0.5 mm mold gap the sample near injection point showed fewer pores (only 4) than the sample away from injection point (48 pores). In the case of laminates manufactured using a 1 mm mold gap nearly the same numbers of pores, 13 pores near injection point and 14 pores away from injection point, were identified. Similar to laminates manufactured using a 0.5 mm mold gap, in laminates manufactured using a 2 mm mold gap the sample near injection point showed fewer pores (only 17) than the sample away from injection point (68 pores). It shall be noted that the number of pores are mentioned to reflect the quantitative results of SEM analysis. For unidirectional laminates these pores were only identified in fiber rovings, and that the free spaces between the rovings (inter-roving spaces) did not show any presence of pores at all using the selected mold gaps.

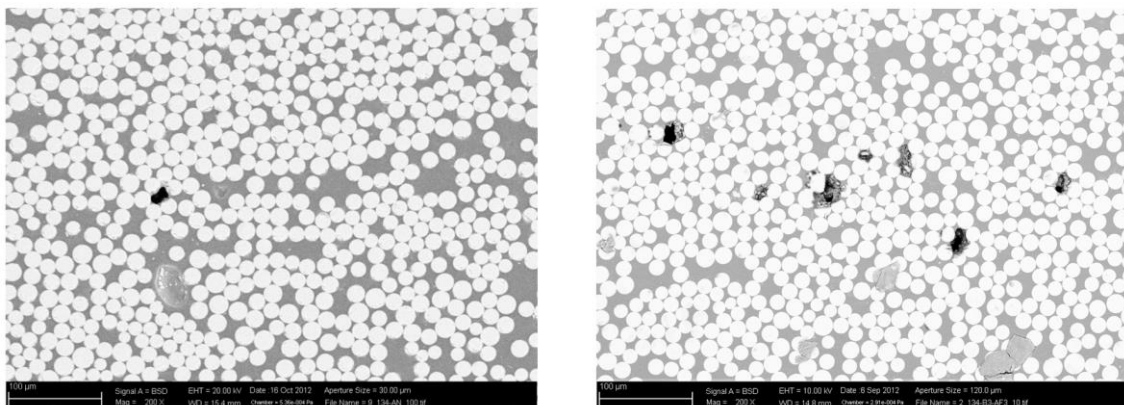
In the case of bidirectional laminates, as can be seen in Picture 6.10, in the laminates manufactured using a mold gap of 0.5 mm and 1 mm, the samples near injection point and away from injection point did not show any pores. It shall be noted that the surface of the bidirectional laminate for SEM analysis included only two unidirectional layers which were orientated in the flow direction, as the remaining two layers, located in the middle of the laminate, were oriented perpendicular to the mold length. For laminates manufactured using a 2 mm mold gap, the sample near injection point showed 19 pores, compared to 45 pores in the sample away from injection point.



0.5 mm mold gap



1 mm mold gap

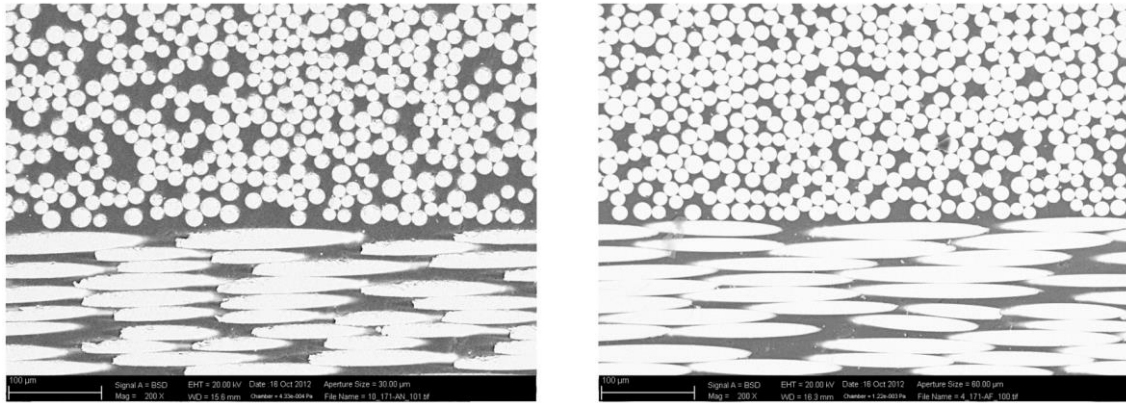


2 mm mold gap

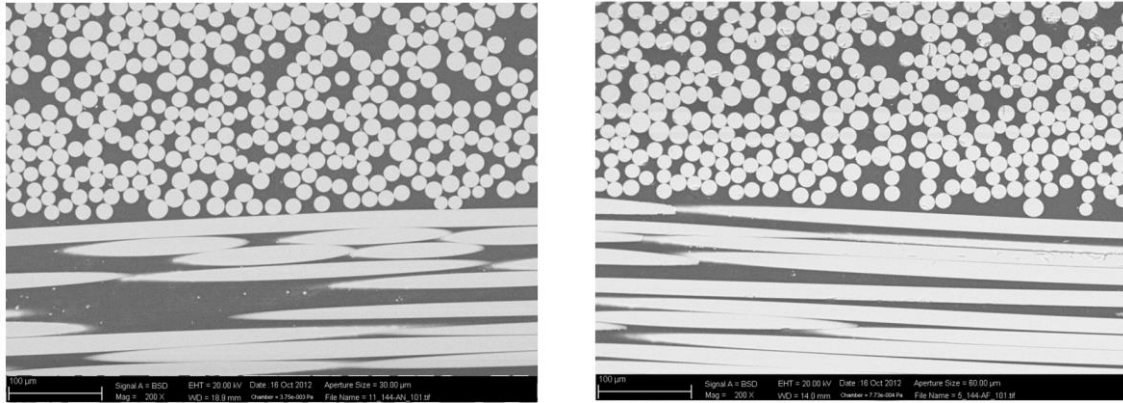
(a)

(b)

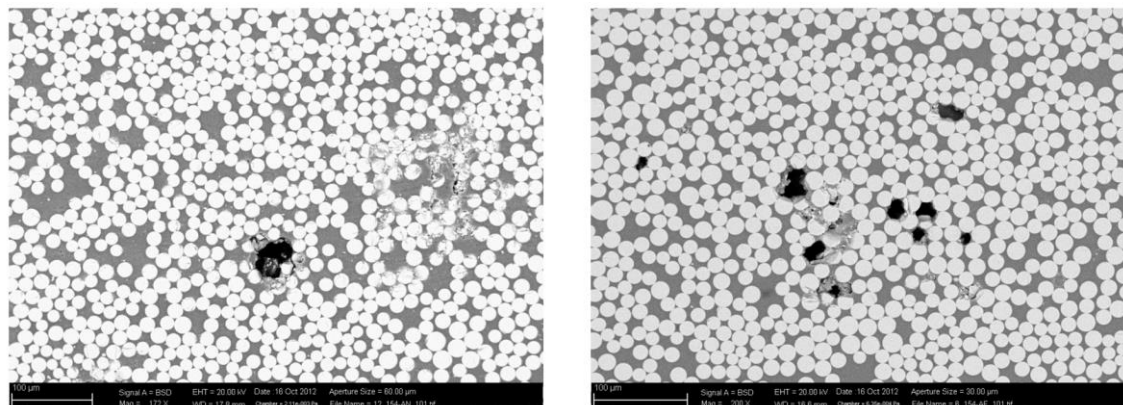
Picture 6.9: SEM micrograph of the unidirectional HP-CRTM laminates manufactured using plate mold 1; (a) near injection point, (b) away from injection point



0.5 mm mold gap



1 mm mold gap



2 mm mold gap

(a)

(b)

Picture 6.10: SEM micrograph of the bidirectional HP-CRTM laminates manufactured using plate mold 1; (a) near injection point, (b) away from injection point

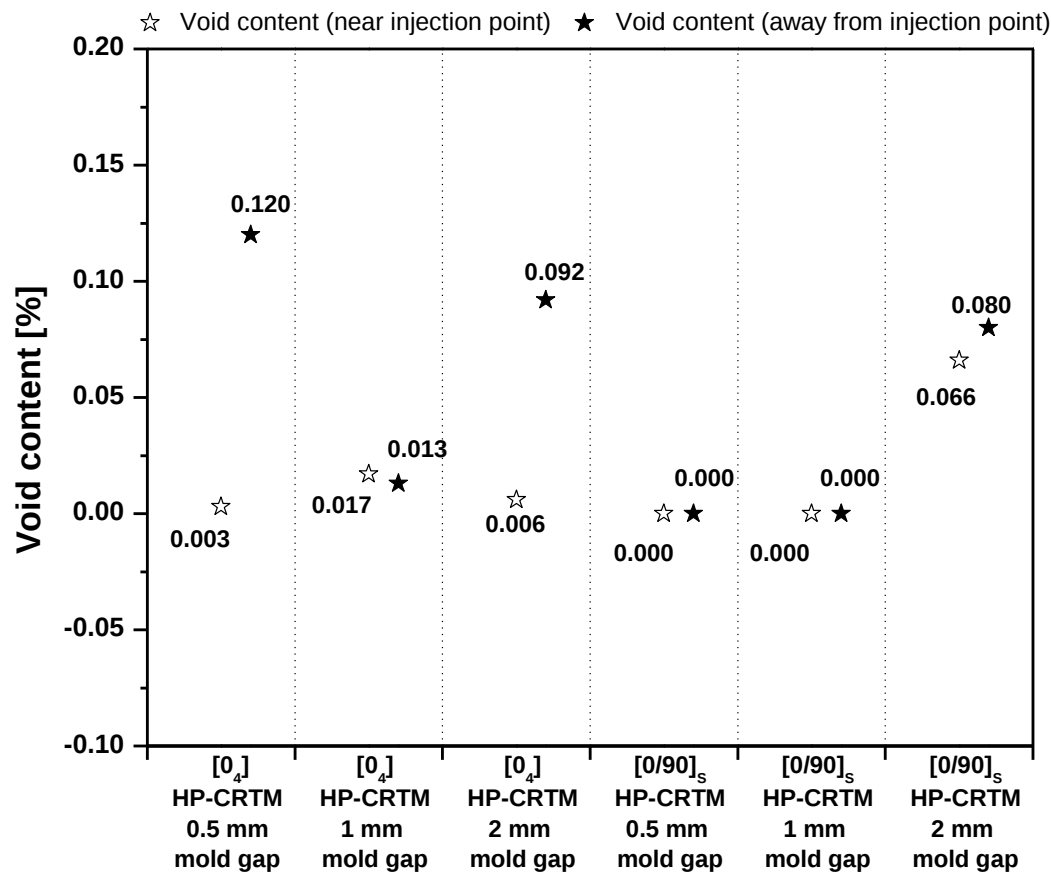


Figure 6.10: Areal void content in the HP-CRTM laminates manufactured using plate mold 1 at different mold gap values

The areal void content in the unidirectional and bidirectional laminates is shown in Figure 6.10 as a function of the selected mold gap value. As can be seen, unidirectional laminates manufactured using a 0.5 mm mold gap show a higher void content (0.12 %) away from injection point than near injection point (0.003 %). For unidirectional laminates manufactured using a 1 mm mold gap near injection point and away from injection point, samples showed almost the same void content of 0.017 % and 0.013 % respectively. Likewise, laminates manufactured using a 2 mm mold gap, similar to laminates manufactured using a 0.5 mm mold gap, showed a higher void content (0.092 %) away from injection point than near injection point (0.006 %). Bidirectional laminates, due to the absence of pores if manufactured using a 0.5 mm and 1 mm mold gap, showed 0 % void content in the analyzed samples (in sections near as well as away from injection point). Using a 2 mm mold gap, a void content of 0.066 % near injection point and 0.08 % away from injection point was measured in bidirectional laminates.

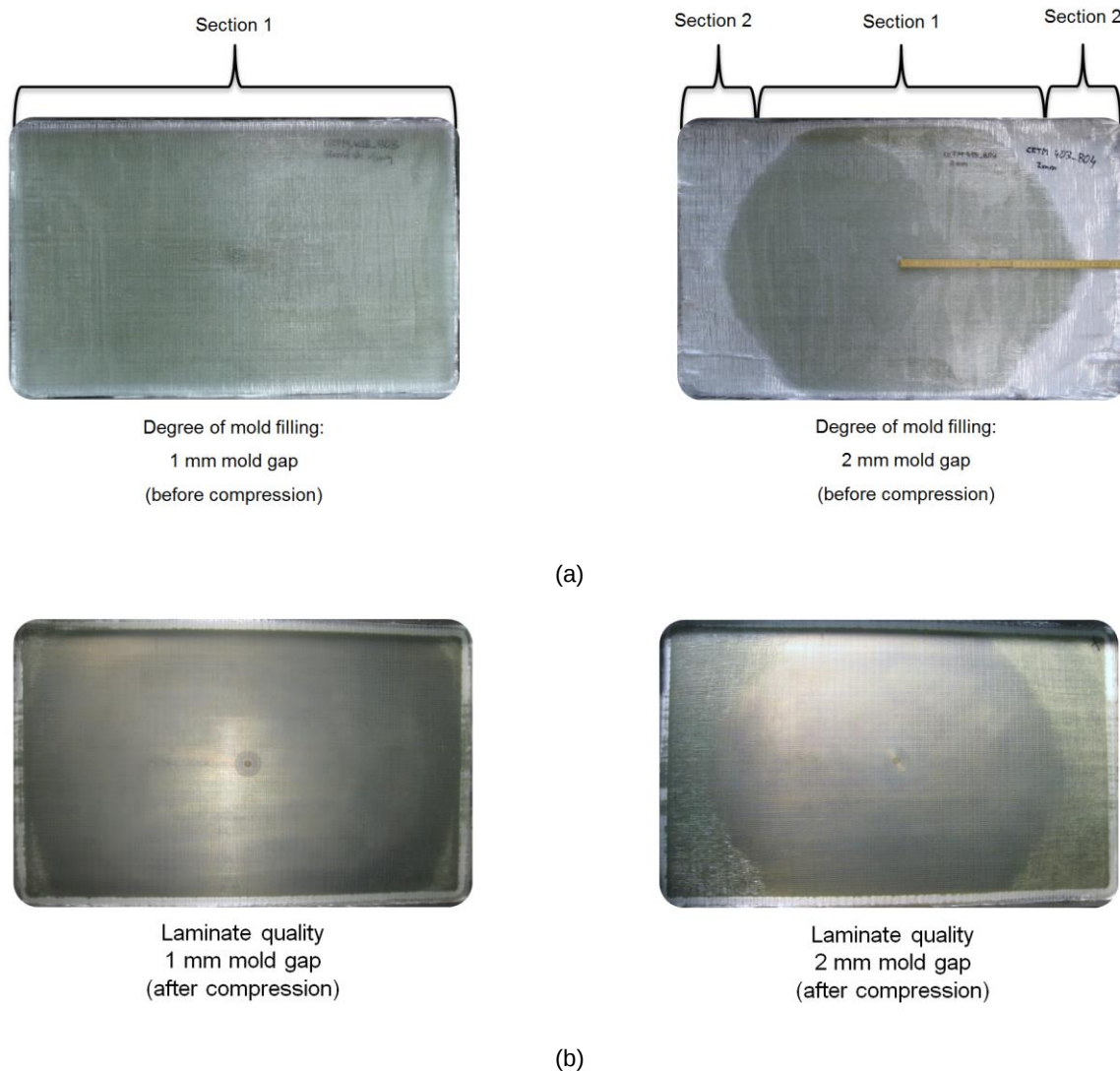
The void content characterization of the laminates indicates, though pores were detected in the unidirectional laminates at all the selected mold gap values and for bidirectional laminates at 2 mm mold gap value, probably, as the void content in all the manufactured laminates was too low (void content lower than 0.2 %) and as these voids, if detected, were always present inside the rovings and not in the free spaces between the rovings, the presence of pores did not affect the mechanical properties such as flexural modulus and ILSS of the laminates. In general these properties are affected by the void content and in literature the influence of void content on such properties was identified and reported for the laminates manufactured using a compression RTM process [70, 71].

6.2.1.3 Laminates manufacturing by HP-CRTM process using plate mold 2

After conducting the mold gap study for the HP-CRTM process using plate mold 1 and evaluating the properties of the laminates, in the next step, new investigations were conducted using the design configuration 1 of plate mold 2. The used laminate layup [0/90/0] for studying the HP-CRTM process by plate mold 2 was slightly different than laminate layup [0/90]_s in the HP-CRTM process study for mold gap evaluation using plate mold 1. The mold gaps of 1 mm and 2 mm were used for the investigations with plate mold 2 which resulted in the effective gaps of 0.2 mm and 1.2 mm respectively. The same resin system as in the mold gap study conducted with plate mold 1 (6.2.1.1) was also used for manufacturing the laminates by using plate mold 2. The required amount of the resin, 670 g, was injected into the partially open mold cavity in 30 s using high-pressure RTM equipment. After the resin was injected into the partially open mold cavity the mold was then closed completely at closure speed of 0.2 mm/s and a compression force of 3100 kN was applied. Further details process parameters applied are given in annex 9.1 [96].

Picture 6.11 shows the results of mold fill study and laminates obtained at the end of the resin injection and before application of compression force (see Picture 6.11 (a)) and laminates obtained after application of compression force and resin curing (see Picture 6.11 (b)) using selected mold gaps. As can be seen in Picture 6.11 (a), with a 1 mm mold gap (effective gap 0.2 mm), the resin covers almost the entire surface of the mold and the fibers were partially impregnated at the end of resin injection and before application of compression force. At a 2 mm mold (effective gap of 1.2 mm), an oval shaped resin flow front progression was observed in the cavity which resembles to approximately 70 % of the entire mold surface. A probable explanation for such an oval shaped resin flow front progress with selection of a 2 mm mold gap can be given on the basis of the mold design and used laminate layup. The gate geometry in plate mold 2 was designed to have cylindrical geometry (also referred as point gate having cylindrical cross section). As the resin was injected into the mold through the point gate, the resin came in direct contact with the

unidirectional outer layer of laminate layup [0/90/0] which was oriented along the length of the mold. Due to the higher permeability of the unidirectional layer in the length direction, the resin flow was more dominant along the length of the mold and less dominant along the mold width. The resin penetrating through the bottom 0° layer to middle 90° orientation layer then experiences a dominant flow along the mold width. As the resin penetrates through the 90° layer to top 0° layer, again the resin flow becomes dominant in the lengthwise direction of the mold if compared to the flow along the mold width. As a result an oval shaped resin flow front progression is observed.



Picture 6.11: HP-CRTM laminates manufacturing at different mold gaps using plate mold 2 - configuration 1; (a) degree of mold filling at the end of resin injection and before compression, (b) Obtained laminates after compression and curing

Similar to the mold fill study for the CRTM process (chapter 6.1.1) in the current study as well two different sections can be identified in terms of impregnation quality of laminates. At 1 mm

and 2 mm mold gaps, the section 1 is partially impregnated after resin injection step and prior to the compression step. Application of compression pressure at 1 mm mold gap therefore leads to compaction of fabrics as well as further impregnation of the fabrics in Z direction in section 1 due to almost no flow of resin along X and Y directions as the mold surface is already covered in X-Y plane. In case of 2 mm mold gap, application of compression pressure leads to compaction of the fabrics as well as further impregnation in the section 1. It also leads to impregnation of dry fabrics in the section 2 due to the resin flow in X and Y directions occurring from additional resin which is squeezed out from section 1. Considering the similarity in the impregnation of laminates as in the feasibility study, the HP-CRTM laminates obtained by plate mold 2 were tested analogue to the feasibility study. Picture 6.11 (b) shows the obtained laminates using 1 mm and 2 mm mold gaps after application of compression force. As can be seen, for both the laminates good quality impregnation was obtained in section 1 which was partially impregnated during resin injection step. It shall however be noted that at a 1 mm mold gap the four corners of the laminates (approx. 100 mm x 50 mm) showed poor impregnation quality if compared to the entire laminate surface. In case of laminate manufactured using a 2 mm mold gap, in section 2 poor quality impregnation was observed. The best impregnation quality was observed in the laminates manufactured using a mold gap of 1 mm (effective gap of 0.2 mm). This observation for [0/90/0] laminate layup is in accordance with results from plate mold 1, where for laminates with layup [0/90]_s the best impregnation was observed at a 0.5 mm mold gap (effective gap 0.2 mm). The similarity in these two laminate layups in which layer/s with 90° orientation were present in the middle of laminates lead to the conclusion that the laminates with good impregnation quality were obtained at an effective gap of 0.2 mm using plate mold 1 as well as plate mold 2.

6.2.1.4 Characterization of HP-CRTM laminates manufactured using plate mold 2

The laminates manufactured using plate mold 2 were characterized as per the sample preparation plan shown in the annex 9.2. Similar to the feasibility study for the CRTM process, samples prepared from the laminates manufactured using plate mold 2 were identified as samples near injection point and samples away from injection point. In case of a 2 mm mold gap, the samples identified as near injection point were located in section 1 which is partially impregnated during resin injection step and further impregnated by application of compression force. The samples identified as away from injection point were located in section 2 which is purely impregnated by the resin flow during the compression step. In case of a 1 mm mold gap, as concluded, only section 1 was identified and section 2 did not exist. Hence in case of the laminates manufactured by using a mold gap of 1 mm the samples identified as near and away from injection point resemble only to the respective

location of the samples taken from the laminates manufactured by using a 2 mm mold gap. The samples were prepared in longitudinal and crosswise orientations. The samples identified as longitudinal samples were prepared along the mold length and parallel to laminate layup and crosswise samples were prepared perpendicular to the mold length and laminate layup.

Fiber volume content in laminates

Figure 6.11 shows the fiber volume content in correlation to the laminate thickness measured in the sections near injection point and away from injection point. As can be seen in the two left side columns of Figure 6.11, the measured thickness of the laminates, independent of selected mold gap value, was slightly higher in the section away from injection point (2.6 mm) than in thickness in the section near injection point (2.5 mm). This variation in the laminate thickness resulted in fiber volume content of 53.9 % near injection point and fiber volume content of 52.3 % away from injection point in laminates manufactured using 1 mm mold gap. For laminates manufactured using a 2 mm mold gap the fiber volume content of 54.1 % was measured near injection point and fiber volume content of 54.6 % was measured away from injection point.

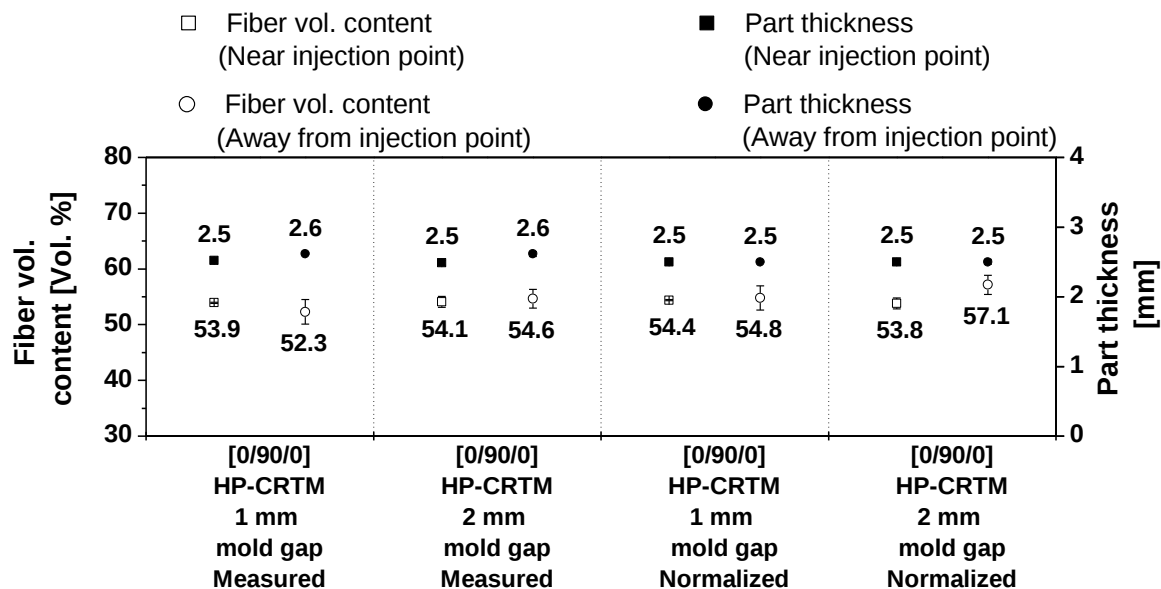


Figure 6.11: Effect of mold gap on fiber volume content and part thickness of the HP-CRTM laminates manufactured using plate mold 2

The two right side columns of in Figure 6.11 shows the fiber volume content in the laminates normalized to a laminate thickness of 2.5 mm. After normalization the laminates

manufactured using a 1 mm mold gap exhibited nearly equivalent fiber volume content of 54.4 % - 54.8 % in both the sections of the laminates. However, the fiber volume content in the section away from injection point was associated with slightly higher standard deviation. In case of a 2 mm mold gap the normalized fiber volume content of 53.8 % was obtained for samples near injection point which was lower than fiber volume content of 57.1 % obtained for samples away from injection point. At a 2 mm mold gap as well higher standard deviation was observed in the fiber volume content in the section away from injection point.

Mechanical properties of laminates

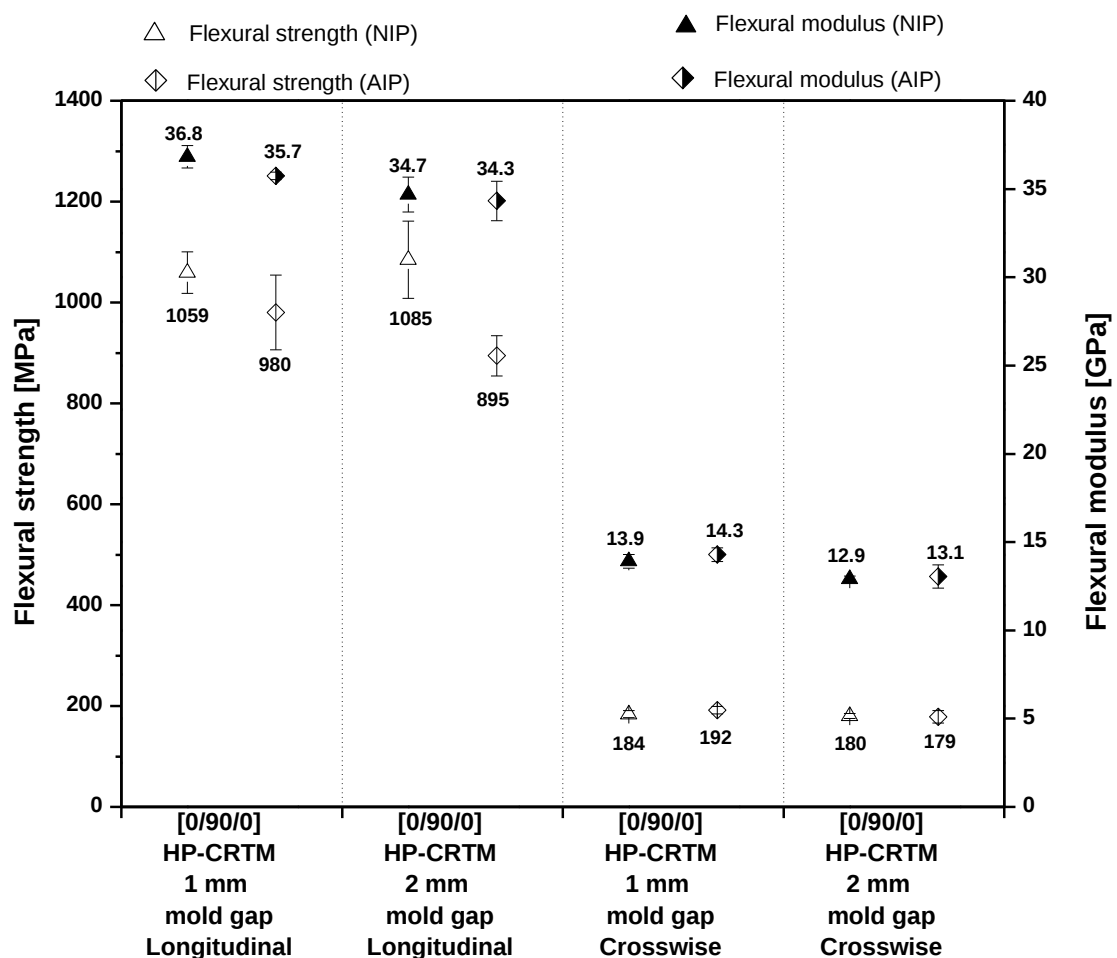


Figure 6.12: Effect of mold gap on flexural properties of the HP-CRTM laminates manufactured using plate mold 2

Figure 6.12 shows flexural properties of the HP-CRTM laminates manufactured using plate mold 2. The properties are normalized to a laminate thickness of 2.5 mm. As can be seen, for laminates manufacturing using a 1 mm mold gap nearly constant flexural modulus of 35.7 - 36.8 GPa and flexural strength of 980 - 1059 MPa was measured lengthwise to

laminate layup [0/90/0] in the sections away and near injection point. In case of a 2 mm mold gap flexural modulus of 34.7 - 34.3 GPa was measured in the sections near and away from injection point. However for laminates manufactured using a 2 mm mold gap lower flexural strength of 895 MPa was measured in the section away from injection point than the measured flexural strength of 1085 MPa near injection point. Flexural modulus and flexural strength crosswise to laminate layup were not influenced by the selected mold gap value and were constant in the sections near and away from injection point. Flexural modulus varied between 12.9 - 14.3 GPa and flexural strength varied between 179 - 192 MPa for the laminates manufactured using 1 mm and 2 mm mold gap values.

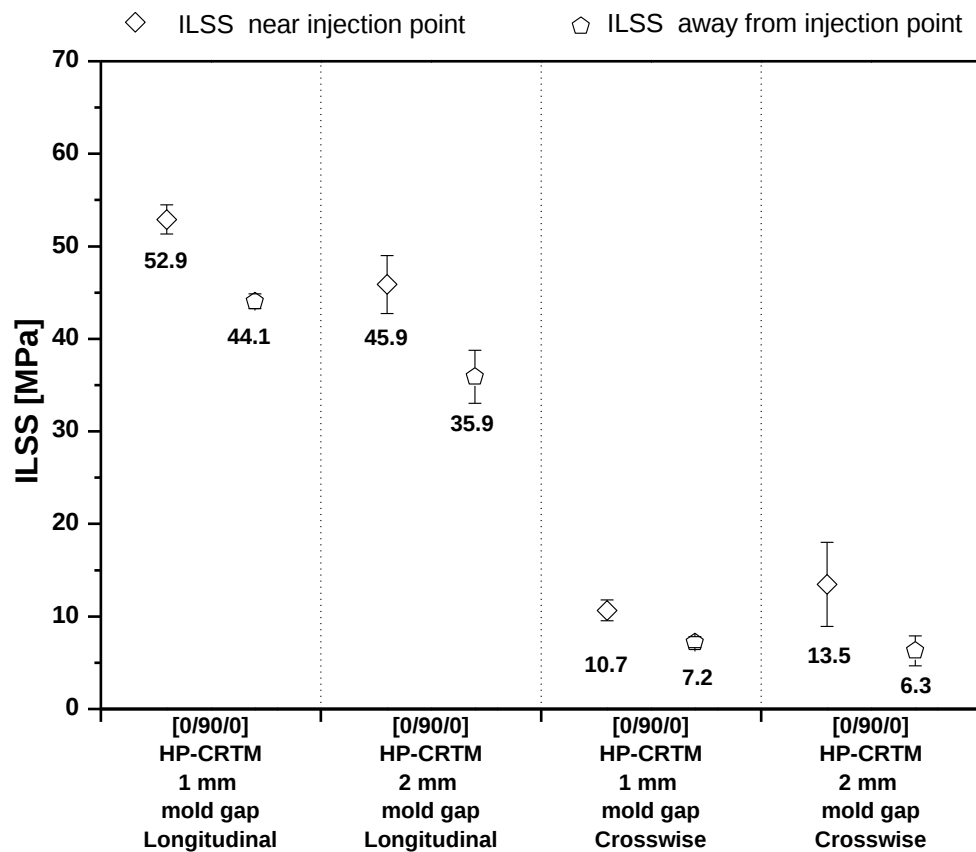


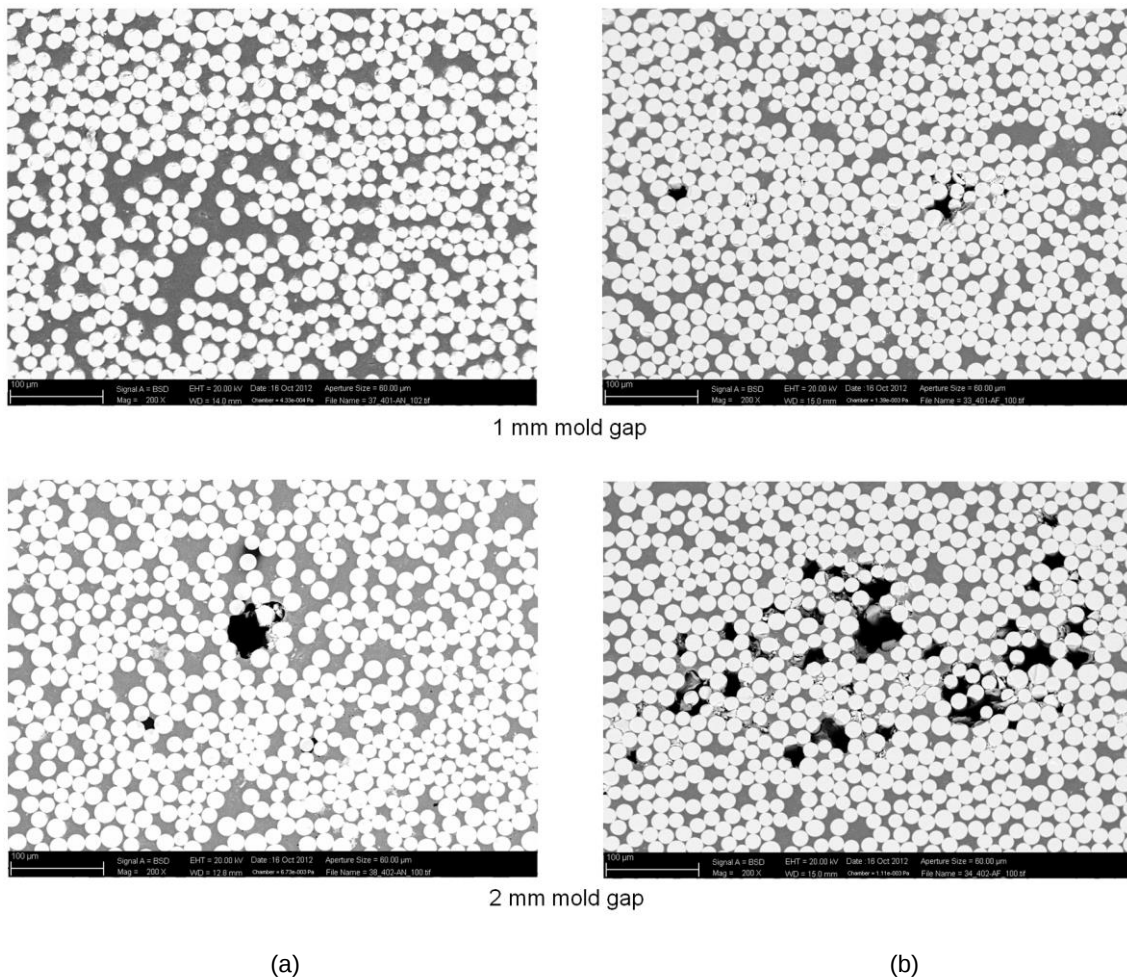
Figure 6.13: Effect of mold gap on ILSS properties of the HP-CRTM laminates manufactured using plate mold 2

ILSS properties of the HP-CRTM laminates are shown in Figure 6.13. ILSS of the laminates is not normalized to a laminate thickness of 2.5 mm. ILSS values in this figure represent actual measured ILSS values at corresponding laminate thickness. As can be seen, a general tendency of drop of ILSS in the section away from injection point as compared to ILSS in the section near injection point was observed for all the laminates. The longitudinal ILSS of the laminates manufactured using a 1 mm mold gap varied between 52.9 MPa near injection point and 44.1 MPa away from injection point which were significantly higher than

ILSS of the laminates manufactured at a 2 mm mold gap which varied between 45.9 MPa to 35.9 MPa in the sections near and away from injection point respectively. The crosswise ILSS of the laminates also showed larger variations between 6.3 MPa to 13.5 MPa at selected mold gap values of 1 mm and 2 mm.

Tensile properties of the HP-CRTM laminates did not show any dependency on the selected mold gap value as generally tensile properties are strongly dominated by the fiber properties and laminate layup. The average longitudinal tensile modulus of 34.21 GPa (standard deviation 0.65 GPa) and longitudinal tensile strength of 689 MPa (standard deviation 63 MPa) were measured in the manufactured laminates using 1 mm and 2 mm mold gap.

Analysis of void content in laminates



Picture 6.12: SEM micrograph of the HP-CRTM laminates manufactured using plate mold 2; (a) near injection point, (b) away from injection point

Picture 6.12 shows the results of SEM analysis for the manufactured laminates using plate mold 2. For laminate manufactured using a 1 mm mold gap the sample near injection point showed presence of lesser pores, only 3 pores, than presence of 35 pores in the sample away from injection point. In case of laminate manufactured at a 2 mm mold gap higher numbers of pores were observed than the laminates manufacture at a 1 mm mold gap. The sample near injection point showed presence of 7 pores only, if compared to presence of 491 pores which were observed in the sample away from injection point in the laminate manufactured using a 2 mm mold gap.

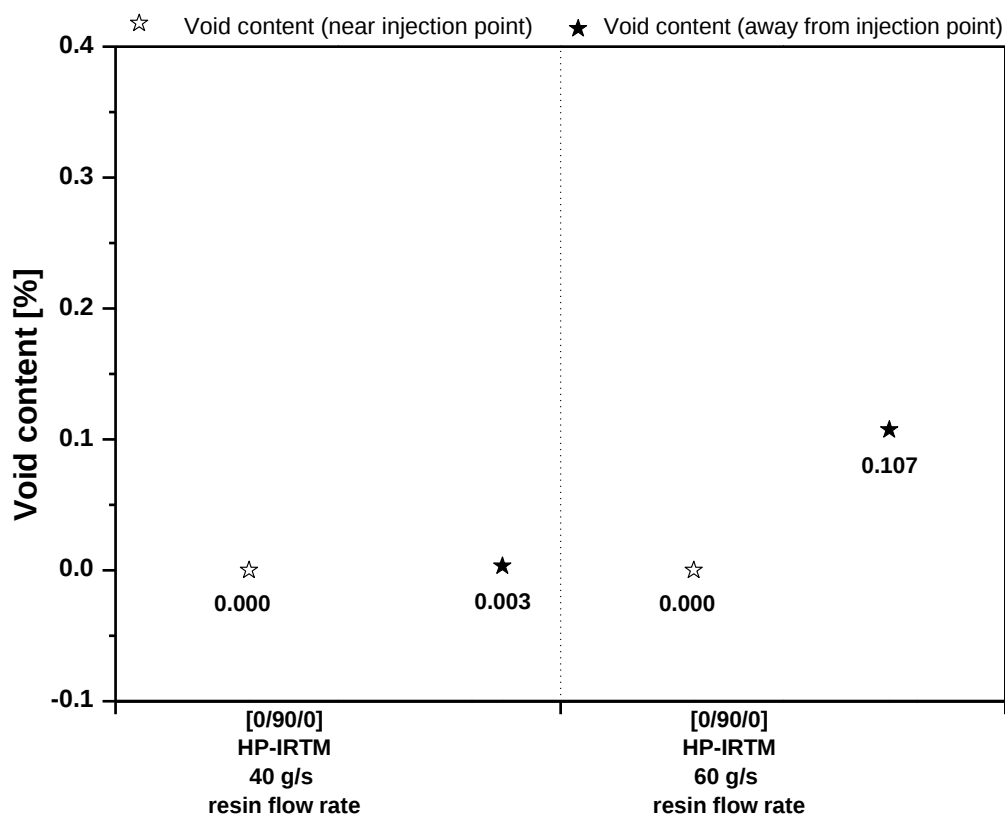


Figure 6.14: Areal void content in the HP-CRTM laminates manufactured using plate mold 2 at different mold gap values

The areal void content in the laminates is shown in Figure 6.14 in dependence of the selected mold gap value. As can be seen at a 1 mm mold gap the sample near injection point shows void content of 0.001 % than the void content of 0.016 % in the sample taken from section away from injection point. At a 2 mm mold gap, if compared to a 1 mm mold gap, the void content in the samples taken near and away from injection point was relatively higher and it measured 0.016 % and 0.304 % respectively. The analysis of the void content can be correlated to the observed mechanical properties. The observed drop of longitudinal flexural strength of the laminates manufactured at a 2 mm mold gap in the section away from

injection point and measured overall lower ILSS of these laminates resulted due to higher void content in these laminates as compared to better impregnation quality of the laminates manufactured at a 1 mm mold gap. In case of a 1 mm mold gap, as can be seen concluded from Figure 6.14 and Picture 6.12, nearly equivalent impregnation quality was observed over the entire laminate surface, however at a 2 mm mold gap only good quality impregnation was observed in the section near injection point and in the section away from injection point poor quality impregnation was observed.

6.2.1.5 Conclusion of mold gap effects on HP-CRTM process

By using a high-pressure RTM equipment it was possible to obtain high resin flow rate for laminates manufacturing. For plate mold 1, using high-pressure RTM equipment the resin injection time of 7.5 s was achieved, if compared to the required resin injection time of 30 s in the feasibility study conducted using a low flow rate classical RTM equipment. The fast flow rate of resin in the mold did not lead to any adverse effects on the fiber orientation. In comparison to the feasibility study the use of preheated resin having low viscosity in the current study led to disappearance of problem of rovings displacement for bidirectional laminates manufactured using plate mold 1.

Based on the conducted investigations using plate mold 1 and plate mold 2 it can be concluded that the impregnation quality of the laminates at a particular laminate layup was strongly affected by the chosen mold gap value. Depending on the selected laminate layup, mold gap value and end laminate thickness different effective gaps and thus different permeability of laminate layups were achieved in both the molds while manufacturing the laminates. Based on the optical observations and REM analysis of the HP-CRTM laminates manufactured using plate mold 1, it was concluded that a selection of a 1 mm mold gap which resulted in 0.7 mm effective gap and selection of a 0.5 mm mold gap which resulted in effective gap of 0.2 mm led to homogeneous impregnation quality respectively in unidirectional and bidirectional laminates ($[0_4]$ and $[0/90]_s$). For unidirectional laminates nearly equivalent void content (void content < 0.02 %) was obtained in the sections near and away from injection point at a 1 mm mold gap, whereas no void content was detected for bidirectional laminates at 1 mm and 0.5 mm mold gap values. For plate mold 1, in general, selection of mold gap only optically showed influences on the impregnation quality of the laminates. However the mechanical properties for both laminate layups were consistent to larger extent at different selected mold gaps of 0.5 mm, 1 mm and 2 mm. For plate mold 2, homogeneous impregnation quality of laminates was achieved at a 1 mm mold gap, effective gap value of 0.2 mm, for the selected laminate layup $[0/90/0]$ and the void content less than 0.02 % was detected in the laminates in the sections near and away from injection point at the this mold gap value. At higher mold gap value, 2 mm, mold gap and effective gap of

1.2 mm, poor impregnation quality was observed in the section away from injection point. The poor impregnation quality led to reduced flexural and ILSS properties of the laminates manufactured using a 2 mm mold gap in the section away from injection point if compared to respective properties in the section near injection point and properties of the laminates manufactured using a 1 mm mold gap. Also the mold fill study and mold gap study led to a conclusion that for the laminates having layup of $[0/90]_s$ and $[0/90/0]$ in which the permeability of the textile layup is higher in both the directions, along the mold length and crosswise to the mold, it is generally favorable to use lower mold gap and thus lower effective gap in the range of 0.2 mm to 0.3 mm to obtain laminates of good impregnation quality. In case of unidirectional laminate layup $[0_4]$ selecting higher mold gap and thus higher effective gap in comparison to bidirectional laminates was concluded to show better impregnation quality in the laminates. Through these HP-CRTM investigations hypothesis 1 was again validated as in feasibility study for CRTM process. A combination of particular laminate layup and effective mold gap led to different end permeability values in mold cavity thus leading to different resin flow velocities and thus resin flow front progression (and resin distribution) during resin injection step. The differences in resin flow resulting due to different selected mold gaps and laminate layups reflected in change of impregnation quality of laminates.

6.2.2 Mold gap closure study for HP-CRTM process

For further investigations of the HP-CRTM process a mold gap closure study was conducted using plate mold 1. The main goal of such mold gap closure study was to systematically evaluate in steps the effect of gap closure on the laminate properties. Laminate layup, thermoset resin type and injection resin amount were similar to the investigation of the effect of mold gap as described in chapter 6.2.1. Further details of the used process parameters are given in annex 9.1 [94].

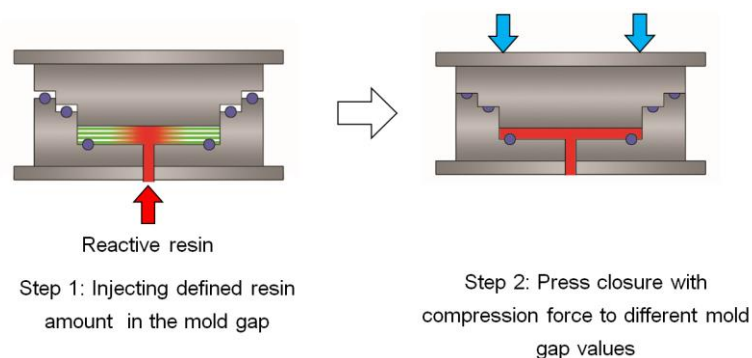
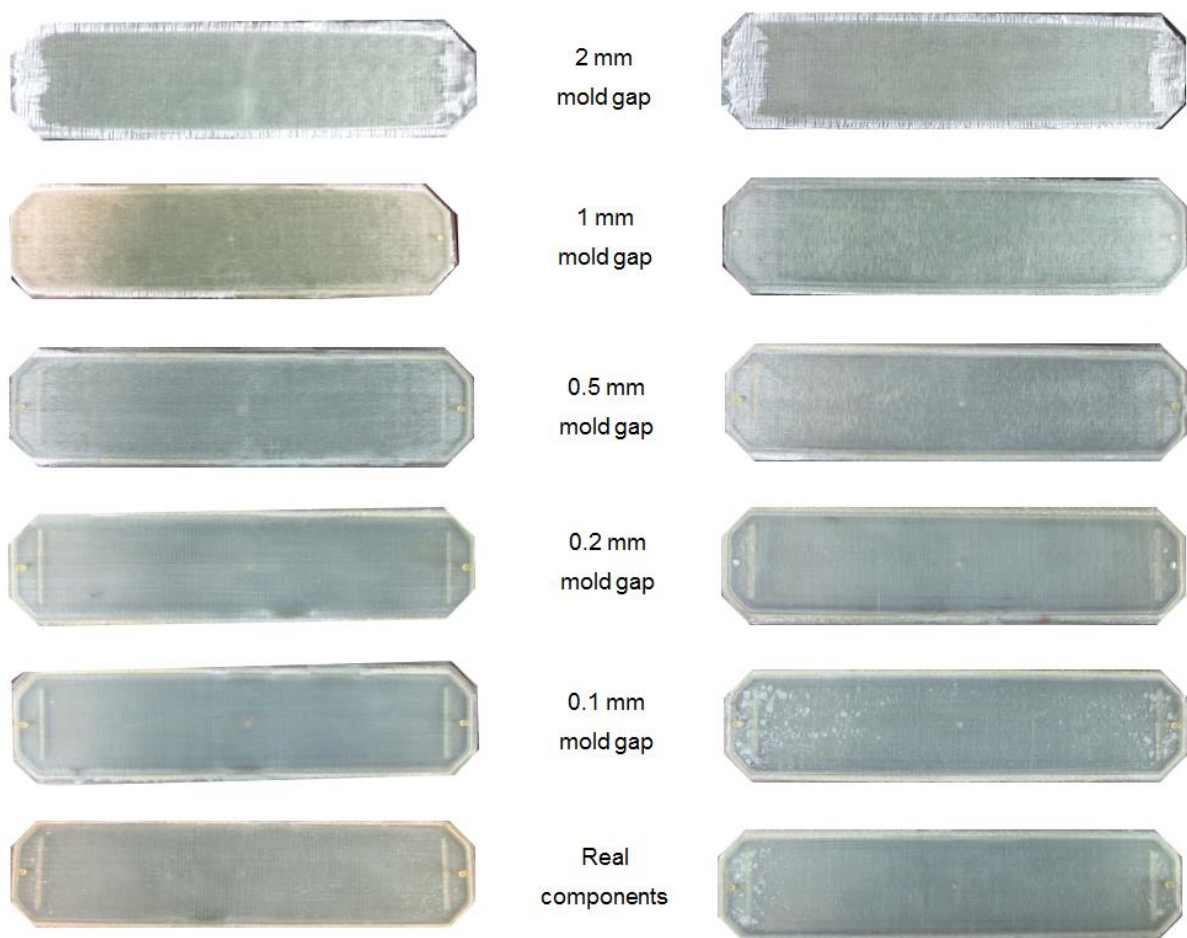


Figure 6.15: Mold closure study for HP-CRTM process; resin injection in partially open mold cavity at defined mold gap of 2 mm and immediate mold closure with compression force to obtain laminates at defined end mold closure values (1 mm, 0.5 mm, 0.2 mm and 0.1 mm)

As shown in Figure 6.15 for each laminate layup the required amount of resin was injected into the partially open mold cavity at a mold gap of 2 mm which led to effective gap of 1.7 mm. After the resin injection was completed, the upper mold half was closed in steps to 1 mm, 0.5 mm, 0.2 mm and 0.1 mm end mold gap values. In order to close the mold in steps to different end mold gap values stainless steel based spacer plates were placed in the mold parting line which restricted the mold from its complete closure though a compression force of 1060 kN was applied by the press during mold closure step. After the mold was closed to a defined end mold gap value the laminates were cured for defined time of 4 min at 100 °C and then the laminates were demolded. Additionally, laminates were manufactured at 2 mm mold gap where the resin was injected into the cavity closed to this mold gap and the laminates were cured without any further mold closure and demolded at this mold gap value.

6.2.2.1 Laminates manufacturing for mold gap closure study



Picture 6.13: Laminates manufactured during the mold gap closure study for the HP-CRTM process;
(a) Unidirectional laminates, (b) bidirectional laminates; real components from study 6.2.1.1 are shown for comparison

Picture 6.13 shows the manufactured laminates at different end mold gap values in the current study and also the real laminates manufactured during the mold gap study (6.2.1.1) where the resin was injected into the mold cavity closed to a gap of 2 mm and then mold gap was completely closed as the resin injection was completed. As can be seen, as the end mold gap value reduced (from top to bottom in the picture) in steps higher compression force was applied on the materials and the resin was forced to flow through the reinforcements thereby leading to longer flow front progression. Also, improvement of laminate impregnation can be observed from top to bottom as the end mold gap value reduced.

6.2.2.2 Characterization of laminates manufactured in mold gap closure study

Similar to earlier studies the fiber volume content, ILSS and flexural properties of the laminates manufactured at different end mold gap values were characterized. The used sample cutting plan, sample geometries and test parameters are given in annex 9.2

Results of mold gap closure study for unidirectional laminates

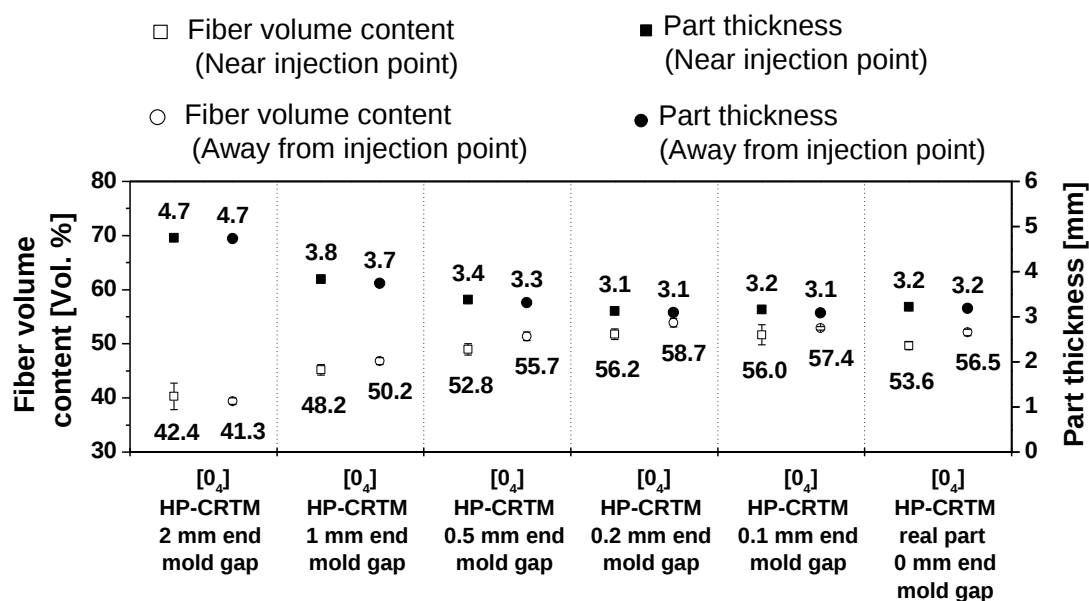


Figure 6.16: Fiber volume content and thickness of unidirectional laminates obtained during mold gap closure study

Figure 6.16 shows the effect of end mold gap values on fiber volume content and thickness of unidirectional laminates manufactured at 2 mm, 1 mm, 0.5 mm, 0.2 mm and 0.1 mm end mold gaps. The thickness and fiber volume content of all the laminates were characterized in the sections near and away from injection point. The thickness and fiber volume content of unidirectional laminates manufactured in the mold gap study in chapter 6.2.1.1 are also included in the shown figure and are indicated as laminates obtained at 0 mm end mold gap. As can be seen, the laminate thickness decreased with reducing end mold gap value which

led to increase in fiber volume content of the laminates. The fiber volume content and thickness of the laminates manufactured using end mold gap values of 0.2 mm, 0.1 mm and 0 mm were almost constant and did not show any significant variations. The cause for constant thickness of the laminates manufactured at 0.2 mm, 0.1 mm and 0 mm end mold gap values can be explained by correlating the measured mold cavity height without any materials, set end mold gap value to be achieved and obtained laminate thickness at these end mold gap values. The mold cavity height of approximately 2.9 mm was measured over the entire mold surface if no materials were placed in the mold cavity. This indicates that though the end mold gap values of 0.2 mm and 0.1 mm were set for obtaining the laminates, ultimately the mold cavity was only closed to a height of 3.1 - 3.2 mm thus resulting in equivalent laminate thickness at 0.2 mm, 0.1 mm and 0 mm end mold gap values. The restriction of the mold cavity closure to the cavity height of 3.1 - 3.2 mm must have resulted due to a combination of used gasket diameter, preform height and preform compressibility. This also indicates that probably, though the spacer plates were used in the mold parting line for achieving the end mold gap values of 0.2 mm and 0.1 mm, same degree of compaction of the laminates was obtained if the end mold gap values were set to 0.2 mm, 0.1 mm and 0 mm.

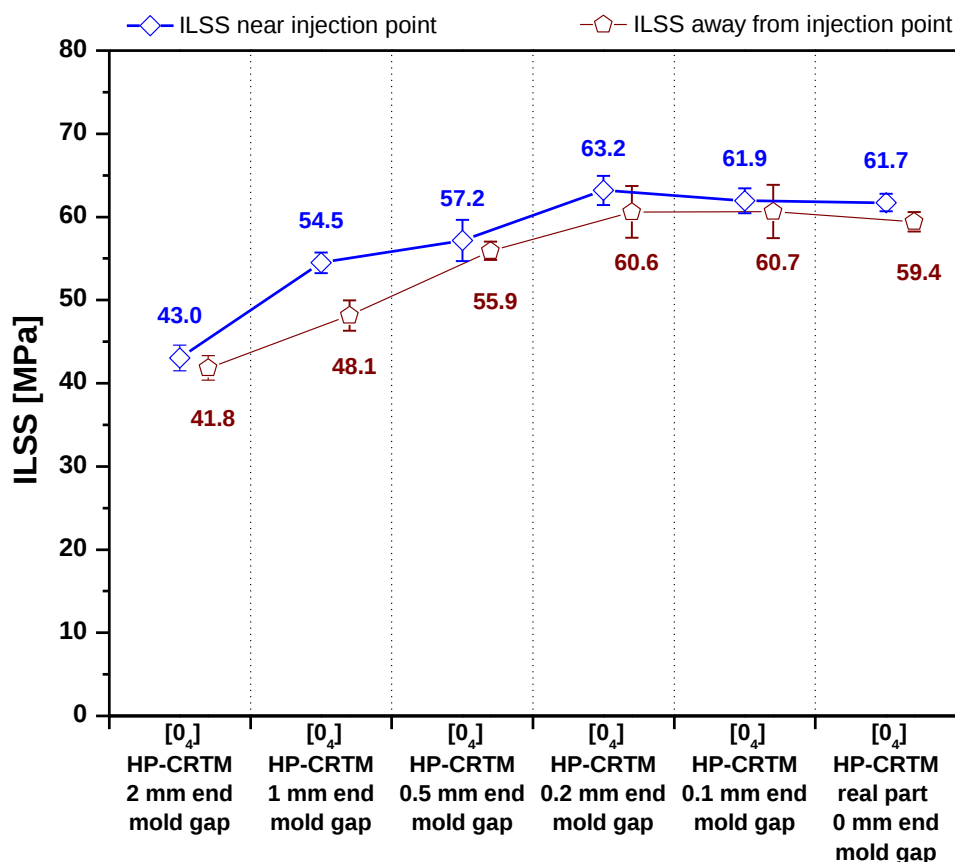


Figure 6.17: ILSS of unidirectional laminates obtained during the mold gap closure study

Figure 6.17 shows ILSS of unidirectional laminates manufactured using different end mold gap values. ILSS is also measured in the sections near and away from injection point. ILSS of the laminates increased with reducing end mold gap values especially for 2 mm, 1 mm and 0.5 mm end mold gaps. Also, as can be seen, at these end mold gap values ILSS of the laminates in the section away from injection point is slightly lower than near injection point at the respective end mold gap values. The increasing values of ILSS with reducing end mold gap values from 2 mm to 0.5 mm can be correlated to the increasing fiber volume content in the laminates at these respective end mold gaps. ILSS of the laminates manufactured using 0.2 mm, 0.1 mm and 0 mm end mold gap values are almost identical to each other and ILSS is constant in the sections near and away from injection point. This effect can be again explained on the basis of the mold cavity height without materials and end laminate thickness of 3.1 - 3.2 mm obtained at 0.2 mm, 0.1 mm and 0 mm end mold gap values. As explained earlier probably the same degree of compaction of the laminates was obtained when the end mold gap values were set to 0 mm, 0.1 mm and 0.2 mm. Hence it led to equivalent fiber volume content and ILSS at these end mold gaps for unidirectional laminates.

Results of mold gap closure study for bidirectional laminates

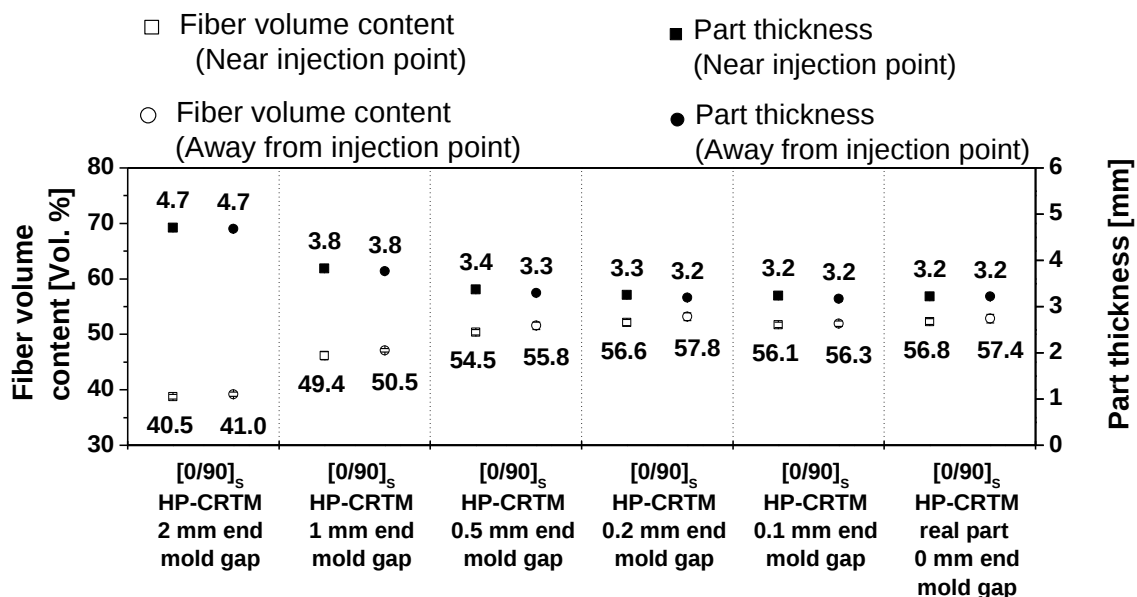


Figure 6.18: Fiber volume content and thickness of bidirectional laminates obtained during the mold gap closure study

Figure 6.18 shows the effect of the end mold gap values on the fiber volume content and thickness of bidirectional laminates manufactured at 2 mm, 1 mm, 0.5 mm, 0.2 mm and 0.1 mm end mold gaps. In this case as well the thickness and fiber volume content of

bidirectional laminates manufactured in the mold gap study in chapter 6.2.1.1 are included in the shown figure and are indicated as laminates obtained at 0 mm end mold gap. Similar to unidirectional laminates also for bidirectional laminates the laminate thickness decreased with reducing end mold gap value which led to increase in fiber volume content of the laminates. Also, the fiber volume content and thickness of bidirectional laminates manufactured using end mold gap values of 0.2 mm, 0.1 mm and 0 mm were almost constant and did not show any significant variations. The cause for constant thickness and equivalent fiber volume content of bidirectional laminates manufactured at 0.2 mm, 0.1 mm and 0 mm end mold gap values can be explained on the same basis as for unidirectional laminates.

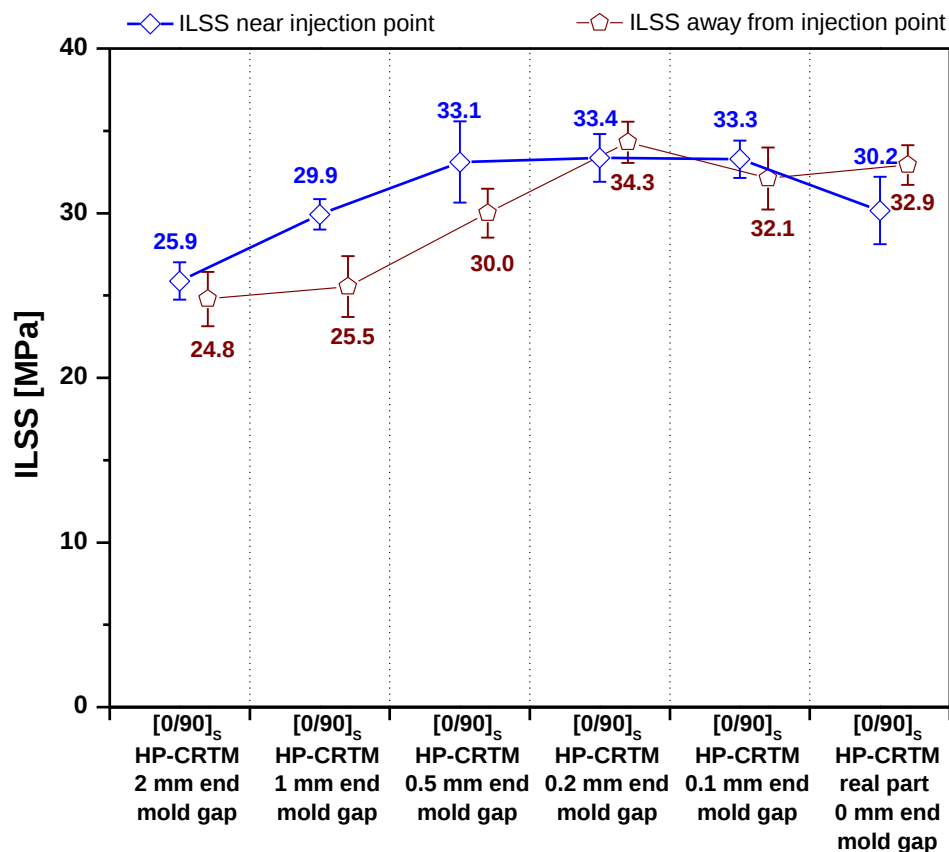


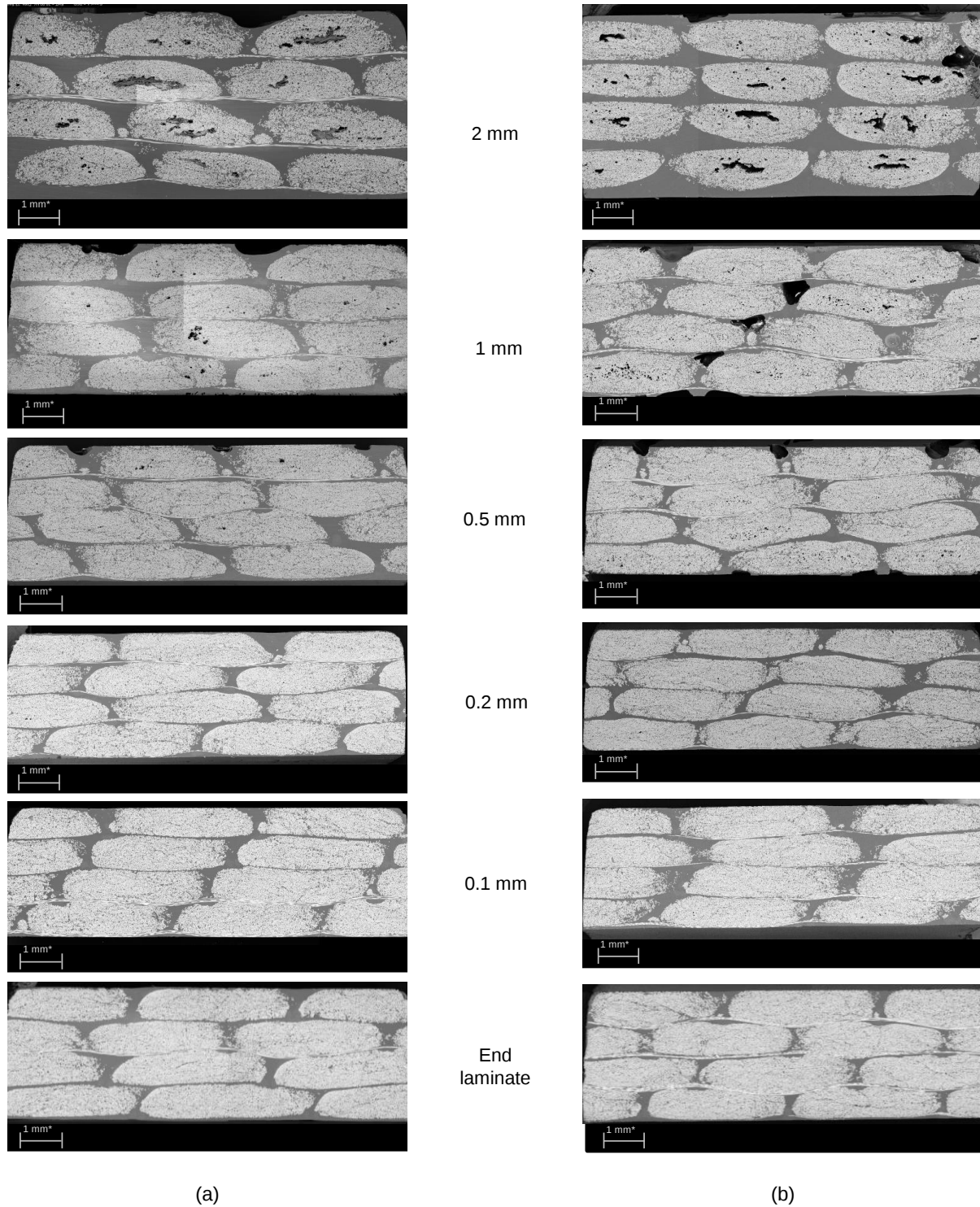
Figure 6.19: ILSS of bidirectional laminates obtained during the mold gap closure study

Figure 6.19 shows ILSS of bidirectional laminates manufactured using different end mold gap values. Similar to unidirectional laminates a general tendency of increase of ILSS of bidirectional laminates with reducing end mold gap values especially for 2 mm, 1 mm and 0.5 mm end mold gaps can be observed which is due to increasing fiber volume content in the laminates at these respective end mold gap values. At these end mold gap values bidirectional laminates as well show lower ILSS in the section away from injection point if compared to ILSS in the section near injection point. As observed in unidirectional laminates,

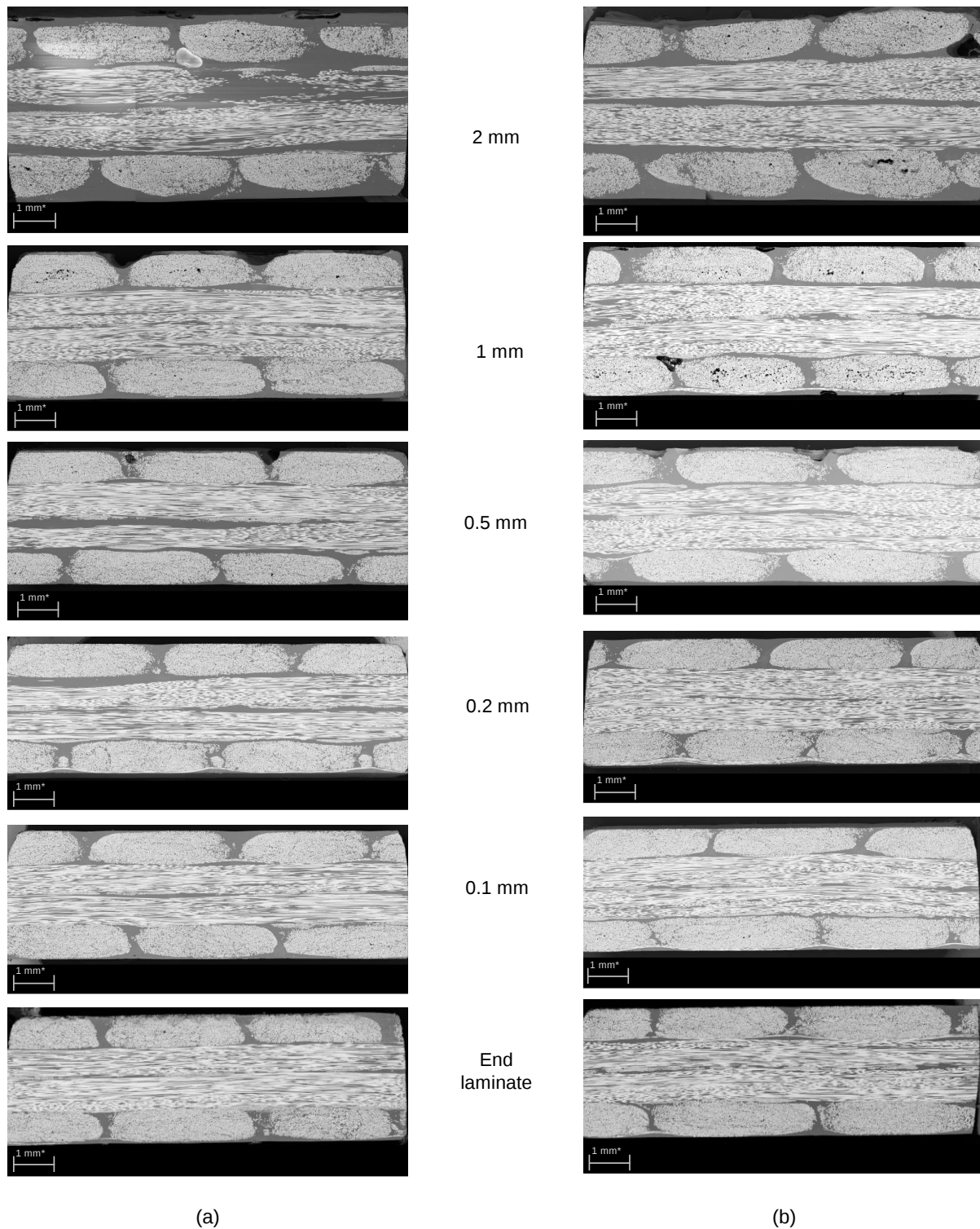
ILSS of bidirectional laminates manufactured using 0.2 mm, 0.1 mm and 0 mm end mold gap values are also almost identical to each other and ILSS is constant in the sections near and away from injection point. The effect of almost identical ILSS of unidirectional laminates at 0.2 mm, 0.1 mm and 0 mm end mold gap values is already explained earlier and the same explanation is also valid for the identical ILSS of bidirectional laminates at these respective end mold gap values.

Flexural properties of the unidirectional and bidirectional laminates in the sections near and away from injection point were also tested. Flexural properties of the laminates showed a similar trend like ILSS properties of respective laminates. Flexural properties of the laminates increased as the mold gap values decreased from 2 mm to 0.5 mm due to increasing fiber volume content. Flexural properties of the laminates were constant and comparable to each other at 0.2 mm, 0.1 mm and 0 mm end mold gap values due to constant fiber volume content and compression of respective laminate layups at these end mold gap values. Also flexural properties in the sections near and away from injection point were constant at respective end mold gap value and respective laminate layup.

Analysis of impregnation quality of laminates manufactured at different end mold gap values



Picture 6.14: SEM micrograph of the unidirectional HP-CRTM laminates manufactured during gap closure study; (a) near injection point, (b) away from injection point



Picture 6.15: SEM micrograph of the bidirectional HP-CRTM laminates manufactured during gap closure study; (a) near injection point, (b) away from injection point

Picture 6.14 and Picture 6.15 show the results of SEM analysis for unidirectional laminates and bidirectional laminates manufactured at different end mold gap values. The samples were taken from the sections near and away from injection point. For both laminate layups,

as seen in the pictures, the impregnation quality of all laminates show a clear improvement as the end mold gap value was reduced from 2 mm to lower values. The impregnation quality in a laminate in the free channels between the rovings is referred hereafter as macroscopic impregnation quality and the impregnation quality inside the roving is referred hereafter as microscopic impregnation quality.

Analysis of impregnation quality at 2 mm end mold gap value

For unidirectional laminates manufactured at 2 mm end mold gap value the resin channels between the rovings in the sample near injection point showed good impregnation quality and hence the sample showed good macroscopic impregnation. The non-impregnated areas can be observed inside the fiber roving indicating that the microscopic impregnation of the laminates was not complete at this end mold gap value. In case of sample taken away from injection point unidirectional laminate shows poor macroscopic and microscopic impregnation as the voids can be observed between the rovings and also inside the fiber roving. A similar effect can be observed also for the bidirectional laminate in which also the samples taken near injection point showed good macroscopic impregnation and poor microscopic impregnation, whereas, a sample taken away from injection point from the same bidirectional laminate shows poor macroscopic and microscopic impregnation quality at 2 mm end mold gap value.

Analysis of impregnation quality at 1 mm end mold gap value

In case of unidirectional laminate the samples near and away from injection point showed improvement of the microscopic impregnation quality as lesser voids were observed inside the fiber rovings if compared to respective samples from the laminate manufactured at 2 mm end mold gap value. Also, in both the samples resin lacking areas/channels were observed on top side of the sample. In case of sample taken away from injection point the macroscopic impregnation quality was further reduced as more voids were observed in the resin channels between the rovings. A probable cause for increased voids between the rovings can be correlated to the compression of materials from 2 mm mold gap to 1 mm end mold gap value. As the mold was closed to a 1 mm mold gap value the materials were slightly compressed and probably the resin was forced to flow inside the rovings pushing the air outside the rovings which was gathered in the channel between the rovings.

In the bidirectional laminate manufactured at a 1 mm end mold gap no significant changes were observed in the macroscopic and microscopic impregnation if compared to the respective samples from laminates manufactured at a 2 mm end mold gap. The sample near

injection point showed good macroscopic impregnation as compared to sample away from injection point. Also, both samples showed presence of voids inside the fiber rovings. Similar to unidirectional laminates bidirectional laminates also showed presence of resin lacking areas/channels on top side of the sample.

Analysis of impregnation quality at 0.5 mm end mold gap value

As the end mold gap value was reduced to 0.5 mm further improvement of the macroscopic and microscopic impregnation was observed in the samples taken near and away from injection point from a unidirectional laminate. The voids were significantly reduced inside the rovings if compared to the sample from laminates manufactured at 2 mm and 1 mm end mold gap values. Also, no voids were observed between the rovings indicating that channels between the rovings were completely filled by resin. This improvement in the macroscopic and microscopic impregnation quality can be again correlated to the compaction of the materials during mold closure step. The resin was injected into the mold cavity at a 2 mm mold gap and then the mold was closed to a 0.5 mm end mold gap value. During the mold closure step the materials were compressed and the resin was forced to impregnate the area inside the rovings. As the cross-section reduced during the mold closure from 2 mm to 0.5 mm mold gap probably the resin amount was enough to fill the channels between the rovings showing good quality macroscopic impregnation. These unidirectional samples as well showed presence of resin lacking areas/channels on top side of the sample. However the size of these channels was significantly lower than the observed channels in the samples from 1 mm end mold gap value.

Similar to unidirectional laminates bidirectional laminates as well showed significant improvement of macroscopic and microscopic impregnation quality at a 0.5 mm end mold gap value. The samples near and away from injection point showed significantly lower voids inside the rovings and complete macroscopic impregnation as channels between the rovings were completely filled with resin. These samples as well showed presence of resin lacking areas/channels on top side of the sample which were smaller than resin channel size in the samples taken from laminates manufactured at a 1 mm end mold gap value.

Analysis of impregnation quality at 0.2 mm, 0.1 mm and 0 mm end mold gap values

As described earlier, the combination of the mold cavity height without any materials, set end mold gap values of 0.2 mm, 0.1 mm and 0 mm and obtained laminate thickness at these end mold gap values resulted in laminates having thickness of 3.1 - 3.2 mm. This indicates that

probably the same degree of compaction of the laminates was obtained when the end mold gap values were set to 0.2 mm, 0.1 mm and 0 mm. This assumption can be well correlated and confirmed with the SEM results obtained for the unidirectional and bidirectional laminates manufactured at 0.2 mm, 0.1 mm and 0 mm end mold gap values.

All the samples taken near and away from injection point from the laminates manufactured at 0.2 mm, 0.1 mm and 0 mm end mold gap values showed nearly same impregnation quality for the respective unidirectional and bidirectional laminate layups. All the samples showed good quality macroscopic impregnation irrespective of the used laminate layup and no presence of resin lacking areas/channels can be observed on top side of the sample indicating that the mold free volume was completely filled with resin and fibers. Also the microscopic impregnation quality of the samples at respective laminate layups is comparable at 0.2 mm, 0.1 mm and 0 mm end mold gap values.

Analysis of void content in laminates

Figure 6.20 shows the areal void content in samples taken near and away from injection point from unidirectional laminates manufactured at different end mold gap values. As can be seen, the void content in the samples reduced as the end mold gap value was reduced from 2 mm to 0.5 mm. The samples taken from laminates manufactured at 0.2 mm, 0.1 mm and 0 mm end mold gap showed almost identical areal void content without any significant deviations as the areal void content values varied between 0 % - 0.092 %. Figure 6.21 shows the areal void content in the samples taken near and away from injection point from bidirectional laminates manufactured at different end mold gap values. In these samples as well, the areal void content in the samples reduced as the end mold gap value was reduced from 2 mm to 0.5 mm.

For bidirectional laminates also the samples taken from laminates manufactured at 0.2 mm, 0.1 mm and 0 mm end mold gap showed almost identical areal void content without any significant deviations as areal void content values varied between 0 % - 0.08 %. The lower void content in bidirectional laminates if compared to unidirectional laminates resulted due to its layup itself as only two unidirectional layers were orientated in flow direction and these two layers were characterized for void content. The remaining two layers, located in middle of the laminate, were oriented perpendicular to the mold length and were not used for void content measurement.

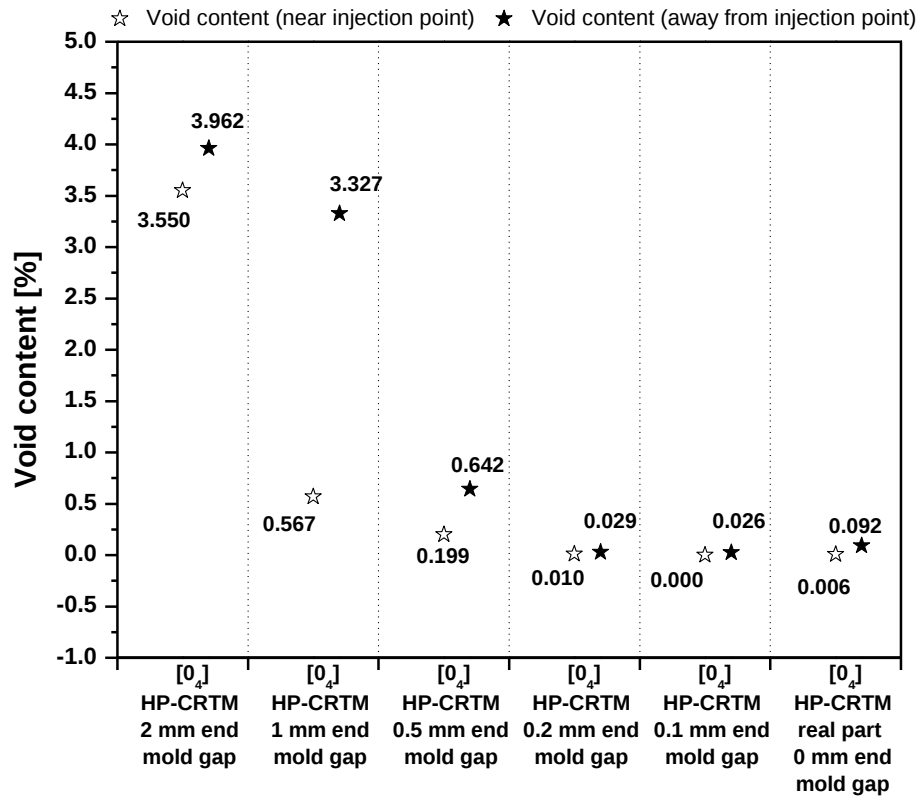


Figure 6.20: Areal void content in the unidirectional HP-CRTM laminates manufactured during gap closure study

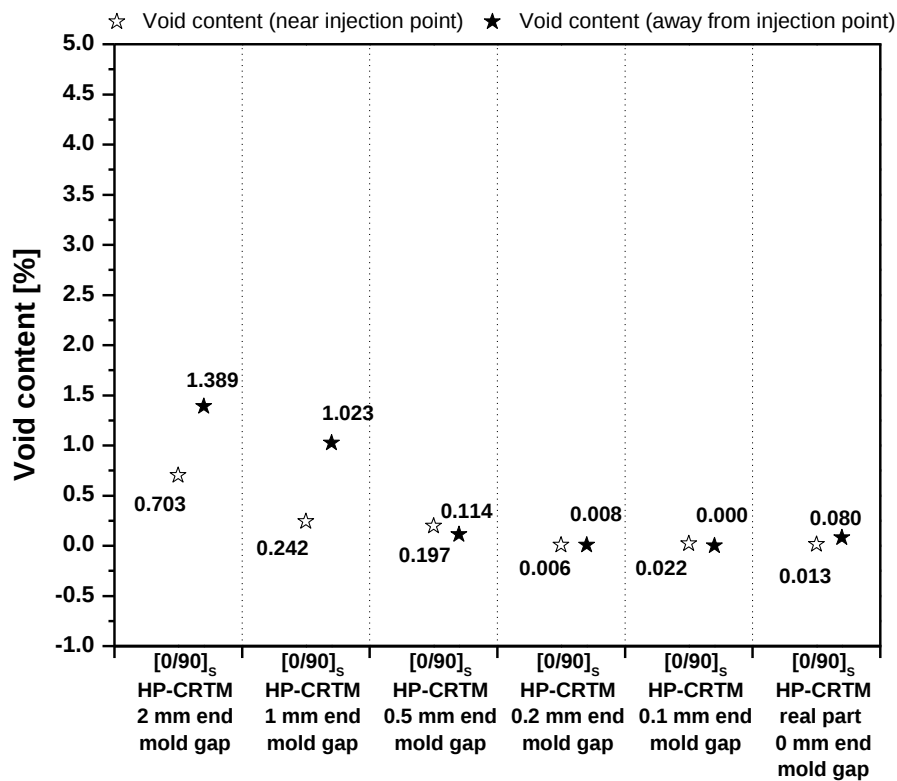


Figure 6.21: Areal void content in the bidirectional HP-CRTM laminates manufactured during gap closure study

6.2.2.3 Conclusion of mold gap closure study for HP-CRTM process

The effect of mold gap closure on the unidirectional and bidirectional laminates manufactured by the HP-CRTM process was investigated in the current study. The resin was injected into the partially closed mold to a mold gap of 2 mm which led to an effective gap of 1.7 mm. After resin injection the mold gap was closed in steps to different end mold gap values of 1 mm, 0.5 mm, 0.2 mm and 0.1 mm and the laminates were manufactured. SEM analysis showed a remarkably high void content in the manufactured laminates at a 2 mm mold gap in the sections near and away from injection point for both selected laminate layups. The samples taken near injection point for both laminate layups showed presence of voids inside the fiber rovings indicating that the microscopic impregnation quality was extremely poor at a 2 mm mold gap. The channels between the rovings did not show any voids and hence the macroscopic impregnation quality of the laminates was very good at a 2 mm mold gap in the section near injection point. This brings to the conclusion that, though the resin was probably injected without any significant cavity pressure built-up into the partially open mold cavity at a mold gap of 2 mm, the resin flow was actually dominant in the free channels between the rovings which led to good quality macroscopic impregnation and poor microscopic impregnation in the laminates. In case of samples taken away from injection point for both laminate layups voids were present inside the roving and in the channels between the rovings indicating that the microscopic and macroscopic impregnation quality was extremely poor at a 2 mm mold gap in the section away from injection point. The samples taken away from injection point showed poor macroscopic impregnation due to predefined amount of resin injected into the cavity which was not yet sufficient to fill the mold free volume completely. The resin amount injected into the cavity was calculated for impregnating the used laminate layups if the mold cavity was completely closed. As the mold was not yet completely closed and was held at a 2 mm mold gap the free volume of the mold was significantly higher than a completely closed mold volume.

As the mold cavity was closed from 2 mm to 1 mm end mold gap value the resin was squeezed into the fiber rovings which led to improvement of the microscopic impregnation in the samples near and away from injection point for both laminate layups leading to the conclusion that the impregnation in z-direction was improved with the mold closure. The samples taken near injection point showed good macroscopic impregnation quality which remained unchanged if compared to samples characterized at a 2 mm mold gap for both laminate layups. This indicates that as the mold was closed from 2 mm to 1 mm mold gap the resin flow took place not only in the z-direction but also in the x-y plane thereby leading to longer flow front progression. The channels between the rovings were entirely filled with resin in the section near injection point even at a 2 mm mold gap and hence with the mold closure the resin was only forced to flow in z-direction and in x-y plane in this section thereby not

affecting the macroscopic impregnation quality. For samples taken away from injection point as the mold cavity was closed from 2 mm to 1 mm the squeezing of the resin into the fiber rovings led to improvement of the microscopic impregnation quality and caused the voids within the laminate to gather into the free channel between the rovings. The voids were gathered in the free channel between the rovings also due to the fact that the resin amount was still not sufficient to fill the free volume of the mold cavity as the mold cavity was closed to obtain end a mold gap of 1 mm. Also the cause of better microscopic impregnation if compared to poor macroscopic impregnation quality in the section away from injection point can be explained on the basis of the capillary forces in the fiber rovings. As the resin amount was not yet sufficient to fill the mold cavity the capillary forces in the fiber rovings may have led to dominant flow inside rovings thereby leaving the channels between the rovings free of resin thereby leading to poor macroscopic impregnation.

The microscopic as well as macroscopic impregnation quality was improved significantly for both laminate layups as the mold was closed to a gap of lower than 0.5 mm for both laminate layups. At the selected mold gaps of 2 mm, 1 mm and 0.5 mm a trend of formation of resin lacking areas/channels was observed on top side of the sample indicating a tendency of movement of voids towards top side surface of the mold. The tendency of formation of resin lacking areas/channels on top side surface of the laminates probably occurred to a combination of resin injection point being from the bottom side of the laminate and density difference of resin and voids. As the resin injection point was located from the bottom side of the laminate, during the resin injection step a dominant resin flow must have occurred initially in the bottom side layers first thereby pushing the voids towards top side surface. Also the resin having higher density must have gathered in the lower side of the laminate and voids having low density were pushed to move towards top side surface of the laminate during the laminate curing.

During the study it was planned to achieve end mold gaps of 0.2 mm and 0.1 mm which ultimately was not possible due to given explanation for constant thickness and fiber volume content of the laminates at 0.2 mm, 0.1 mm and 0 mm end mold gap values. Hence at these end mold gap values of 0.2 mm, 0.1 mm and 0 mm the same degree of laminate compression was probably achieved for both the used laminate layups. The comparable impregnation quality in the investigated SEM samples and ILSS values at these three different end mold gap values for both laminate layups indicate the consistency of the process and reproducibility of the void content. The samples taken near and away from injection point showed very less areal void content which varied between approximately 0 % - 0.1 % in both laminate layups at 0.2 mm, 0.1 mm and 0 mm end mold gap values. The combination of the resin injection point from the bottom side of the mold/laminate layup and

density difference of resin and voids also explains the possible cause for low void content inside the test area of the laminates at the end mold gap values of 0.2 mm, 0.1 mm and 0 mm though no vacuum was used for running the HP-CRTM trials. The air probably gets pushed through the laminate to form the resin lacking areas/channels on the laminate top side surface during the compression step and further the air is probably pushed towards the mold parting line as compression force is applied by the mold closure.

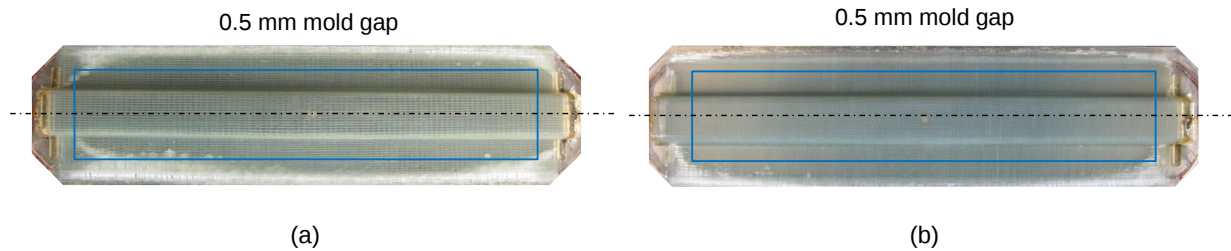
6.2.3 Effect of mold geometry on HP-CRTM process

6.2.3.1 Manufacturing of hat shaped components by using HP-CRTM process

Hat shaped components were manufactured using same laminate layups ($[0_4]$ and $[0/90]_s$) as in the feasibility study and investigation of the HP-CRTM process using different mold gaps. The same epoxy resin system as in the mold gap study (6.2.1) was used for manufacturing hat shaped components in order to be able to compare the properties of flat laminates with the properties of hat shaped components. Similar to the mold gap study the epoxy resin Araldite® LY 564 was preheated to 60 °C and Hardener XB 3458 was maintained at room temperature. Due to the same volume of hat shaped component and flat laminates manufactured from plate mold 1, the same amount of the resin (410 g and 430 g resin for unidirectional and bidirectional laminate layup respectively) was used for manufacturing hat shaped components. The defined amount of resin was injected into the partially open mold cavity in 7.5 s using high-pressure RTM equipment. For manufacturing of hat shaped components a mold gap of 0.5 mm was used. After the resin was injected into the partially open cavity the mold was closed completely at closure speed of 0.1 mm/s to apply a compression force of 1060 kN [94]. The preinvestigations conducted on the hat shaped mold at 2 mm and 1 mm mold gaps showed the resin was flowing out of the mold cavity during the resin injection step. This shows that the gasket of the mold was not compressed enough during resin injection step. It hence indicates that the selected mold gap value for the combination of the component geometry, laminate layup and mold concept was too high. Due to this effect a mold gap of 0.5 mm was used for manufacturing hat shaped components using both laminate layups. The mold was heated to 100 °C and the components were cured in the mold at this temperature for 4 min.

The manufactured hat shaped components using unidirectional and bidirectional laminate layups and a mold gap of 0.5 mm are shown in Picture 6.16. As can be seen in this picture, the outer corner edges of hat shaped components show poor impregnation. However, as mentioned in the mold description, the maximum size of this component is 830 mm x 210 mm of which the actual predefined area for testing is 700 mm x 130 mm. This

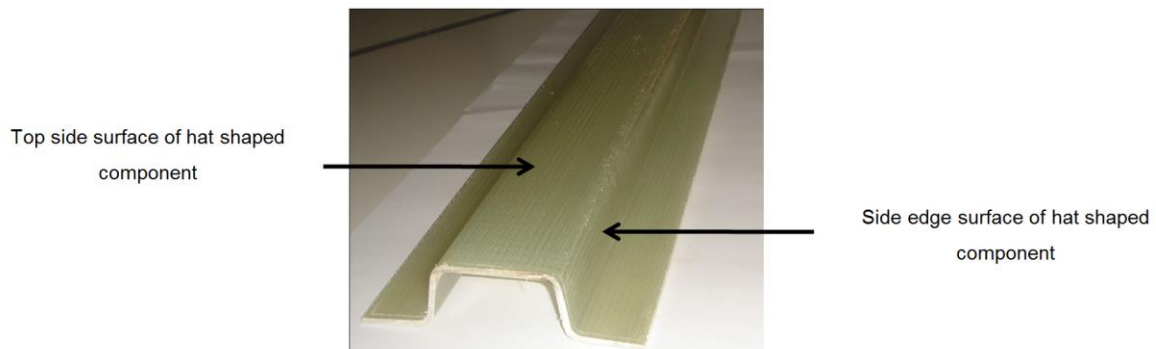
test area is marked in Picture 6.16 by a blue frame and, as can be seen, the components showed fairly good quality impregnation in the defined test area.



Picture 6.16: Impregnation quality of hat shaped components manufactured by the HP-CRTM process;
(a) Laminate layup - $[0_4]$, (b) Laminate layup - $[0/90]_s$

6.2.3.2 Characterization of hat shaped components

The manufactured hat shaped components were characterized by measuring fiber volume content, ILSS and flexural properties. Picture 6.17 shows the surfaces of hat shaped component used for the characterization.



Picture 6.17: Used surfaces for characterization of hat shaped components

As shown, top side and side edge of hat shaped component were used for characterization. In this case as well, samples were taken in the sections near injection point and away from injection point to evaluate the properties of hat shaped component in different sections. The sample preparation plan used is shown in annex 9.2 As hat shaped components were manufactured using a 0.5 mm mold gap, the measured properties shown below are compared with flat laminates manufactured using the same mold gap.

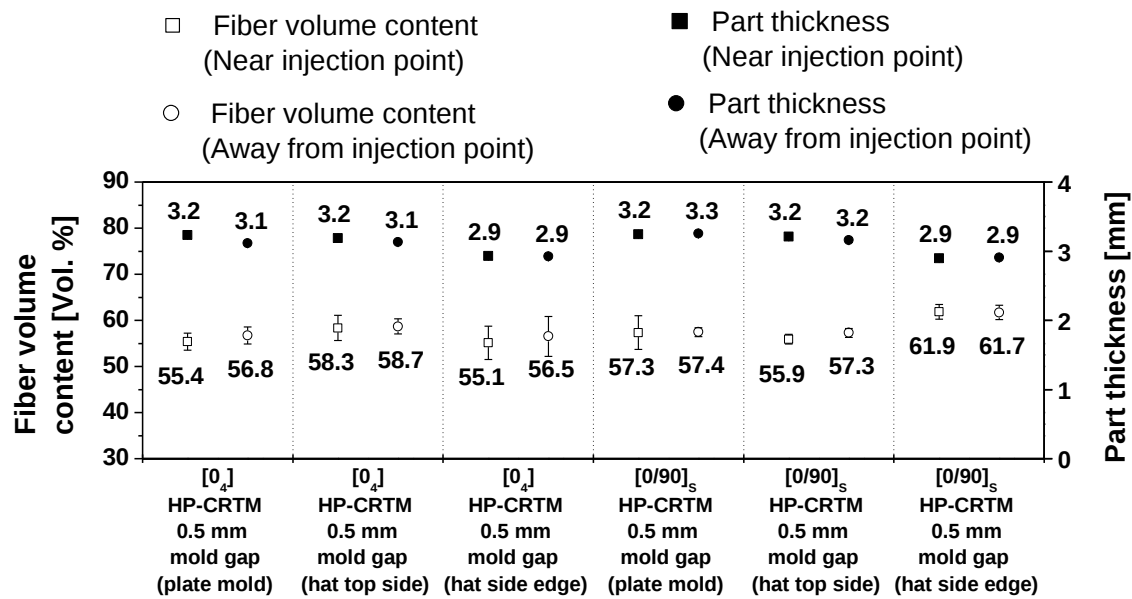


Figure 6.22: Fiber volume content and part thickness of flat laminates and hat shaped components manufactured by the HP-CRTM process

Figure 6.22 shows the fiber volume content in correlation to the laminate thickness measured in the different sections of flat laminates and hat shaped components manufactured by unidirectional and bidirectional laminate layups with a mold gap of 0.5 mm. As can be seen, for unidirectional laminate layup flat laminates and the top side surface of hat shaped components exhibited nearly equivalent thicknesses near injection point (3.2 mm) and away from injection point (3.1 mm). At the equivalent thickness, however, hat shaped components showed slightly higher fiber volume content (58.3 % - 58.7 %) in the top side surface compared to the fiber volume content in flat laminates (55.4 % - 56.8 %). side edge surface of hat shaped components exhibited a constant thickness of 2.9 mm near injection point and away from injection point, which was lower than the top side surface thickness (3.1 - 3.2 mm) of hat shaped components. Though the thickness of sample taken from side edge surface was lower than the thickness of sample taken from top side surface, the measured fiber volume content in sample taken from side edge surface was lower than fiber volume content in sample taken from top side surface of hat shaped component. In general, reduction in the thickness of side edge surface should have resulted in higher fiber volume content compared to the top side surface. However, side edge surface exhibited a fiber volume content of 55.1 % near injection point and 56.5 % away from injection point compared to the fiber volume content of 58.3 % and 58.7 % in the respective sections of the top side surface of hat shaped components. Also, a high standard deviation can be observed in the fiber volume content for side edge surface of hat shaped components.

The observed effect of lower fiber volume content in sample taken from side edge surface of hat shaped components manufactured using unidirectional laminate layups is most likely due to a combination of effects of manual draping of the unidirectional textile layers on the mold core (lower mold half) and application of compression force on side edge surface of hat shaped components, which may have led to roving displacement in side edges of the component.

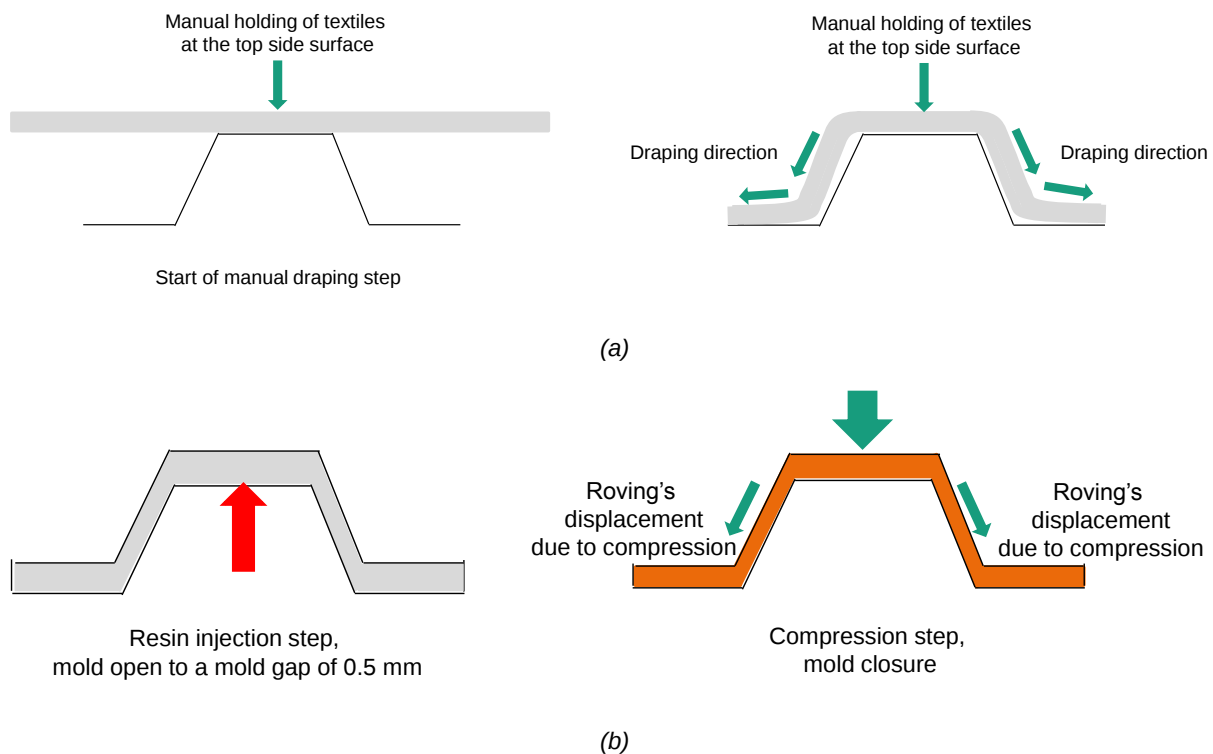
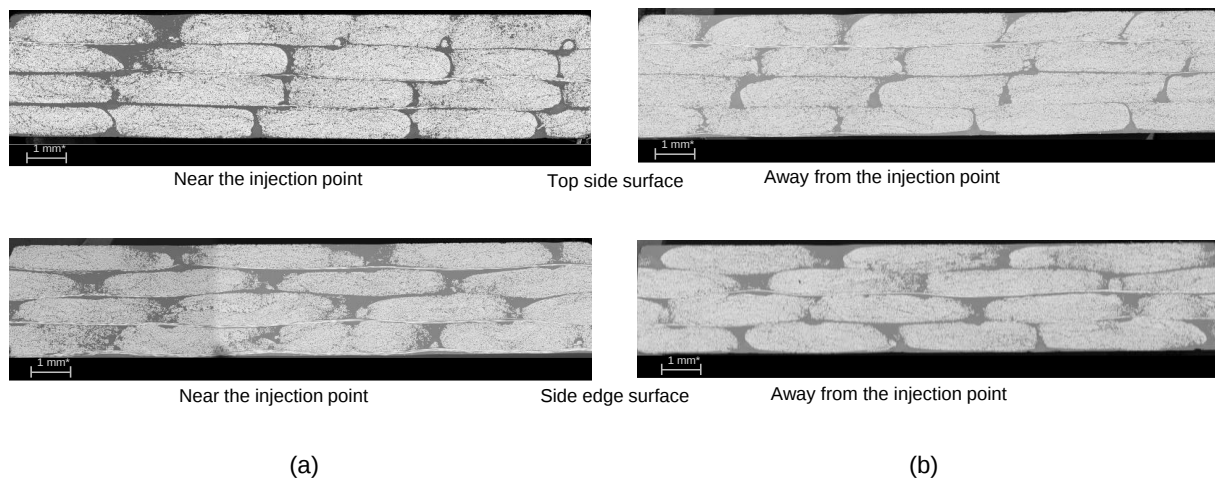


Figure 6.23: Diagram showing manufacturing of a hat shaped component; (a) manual draping of textile reinforcement layers, (b) resin injection step and compression during the manufacture of hat shaped components with a unidirectional laminate layup

Figure 6.23 shows the process steps involved in the manufacturing of hat shaped components. As a first step, four layers of unidirectional reinforcement with a 0° orientation to the mold were manually draped on the mold core. The textiles were centered on the mold core and then draped along the draping direction shown. As the unidirectional rovings of the textiles were well stabilized by using 4 wt % of the glass rovings which were located from the back side of the textile, high draping forces were needed to obtain the shape of the mold core for textile reinforcements. The draping forces were applied along the width of the mold and therefore acted perpendicularly to the orientation of the rovings. This must have caused an increase in the inter-roving distances for the textiles along the draping areas of the component. In the next step, the mold was closed to obtain a mold gap of 0.5 mm and the

required resin was injected into the mold cavity. After the resin injection was completed, the mold gap was closed and compression force was applied. The application of compression force on top side (flat) surface of hat shaped component must have resulted in compaction of the rovings and their stabilization against movement from each other. However, application of compression force on side edge surfaces of the component must have led to roving movement and displacement from each other as the mold gap was closed. Probably for these reasons, lower fiber volume content was measured and high standard deviation of fiber volume content was observed at side edge surface of hat shaped component compared to top side surface.

Picture 6.18 shows the SEM micrograph of samples taken from top side surface and side edge surface of a hat shaped component manufactured using unidirectional laminate layup. As can be seen, increased inter-roving distances were detected in the samples taken near and away from injection point from side edge surface of the component. Relatively lower inter-roving distances were detected in the samples from top side surface of the same component.

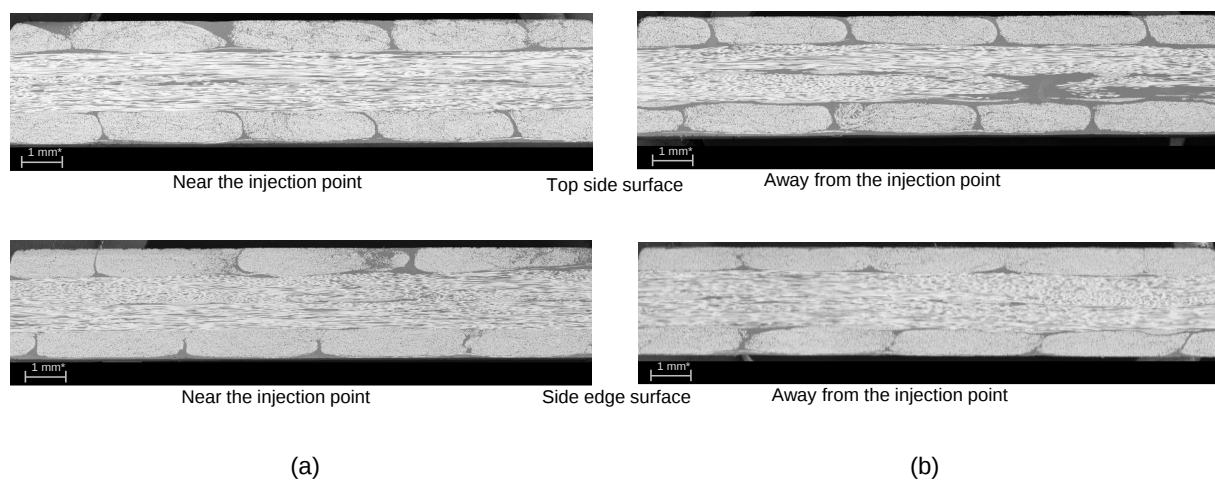


Picture 6.18: SEM micrograph of unidirectional hat shaped components manufactured by the HP-CRTM process; (a) near injection point, (b) away from injection point

In case of bidirectional laminate layup, flat laminates as well as top side surface of hat shaped component showed a nearly equivalent thickness of 3.2 - 3.3 mm near injection point as well as away from injection point, and side edge surface of hat shaped component exhibited a thickness of 2.9 mm in both sections of the component. Hat shaped components showed slight variation in volume content (55.9 % - 57.3 %) in top side surface compared to the nearly constant fiber volume content in flat laminates (57.3 % - 57.4 %). In case of bidirectional laminate layup, reduction in thickness of side edge surface also resulted in

higher fiber volume content than in top side surface. Sample taken from side edge exhibited a fiber volume content of 61.7 % - 61.9 % which is approximately 4-5 % higher than samples taken from top side surface of hat shaped component. The presence of two textile layers in the middle of the laminate layup with a 90° orientation to the mold length must have prevented roving displacement as the rovings within these layers were oriented along the draping forces.

Picture 6.19 shows the SEM micrograph of samples taken from top side surface and side edge surface of a hat shaped component manufactured using bidirectional laminate layup. Unlike the unidirectional layup, in the components manufactured by bidirectional laminate layup reduced inter-rovings distances were detected in the samples taken near and away from injection point. Relatively higher inter-rovings distances were detected in the samples from top side surface of the same component.



Picture 6.19: SEM micrograph of bidirectional hat shaped components manufactured by the HP-CRTM process; (a) near injection point, (b) away from injection point

Figure 6.24 shows ILSS of hat shaped components measured at top side surface and side edge surface. In this figure, ILSS properties of flat laminates are also shown for comparison. For unidirectional laminate layup, independent of component geometry and position of specimen, ILSS values varied between 60.4 - 65.8 MPa. This indicates that ILSS values of hat shaped component were identical in the sections near injection point and away from injection point at top side surface as well as side edge surface. In the case of bidirectional laminates, ILSS of hat shaped component varied between 33.6 - 38.9 MPa, which was slightly higher than ILSS of flat laminates which was approximately 30.8 - 30.9 MPa. Based on ILSS values for hat shaped component it can be concluded that the application of compression force on the flat top side surface and slanting side edge surface of hat shaped

components did not result in any major deviations in ILSS properties. This reconfirms the optically observed near-equivalent impregnation in both these surfaces of hat shaped component.

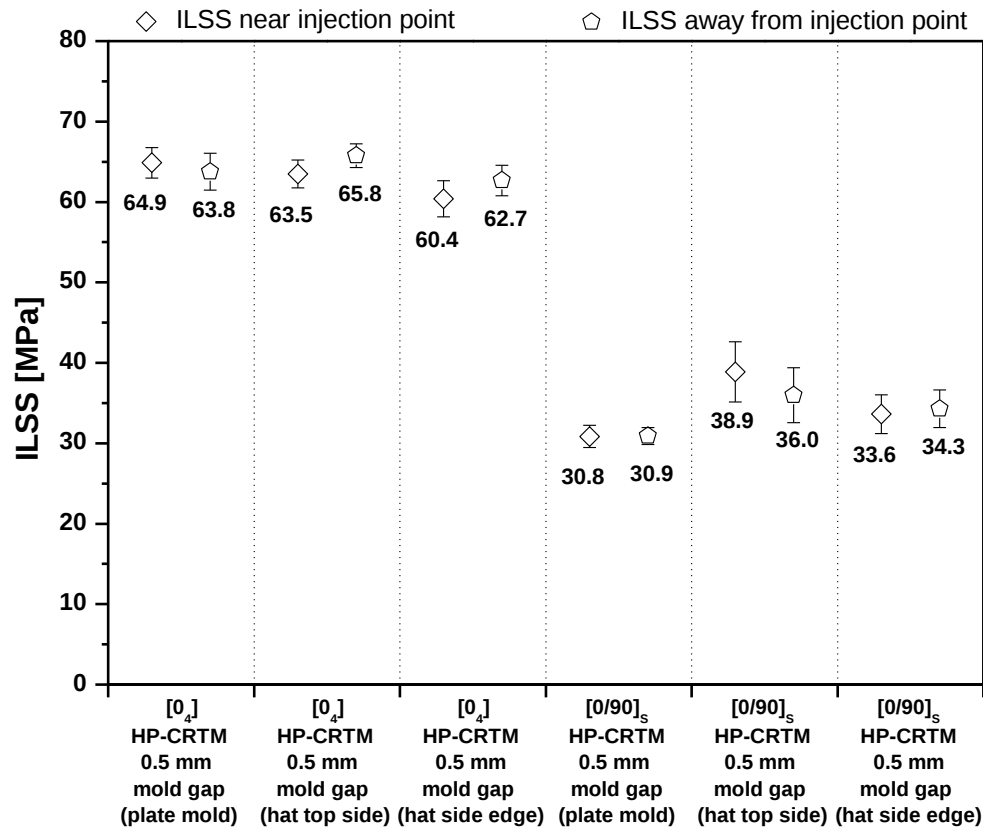


Figure 6.24: ILSS of flat laminates and hat shaped components manufactured by the HP-CRTM process

Figure 6.25 shows flexural properties of hat shaped components and flat laminates manufactured using a unidirectional laminate layup. In the section near injection point (NIP) hat shaped components and flat laminates showed almost identical flexural strength which varied between 1220 - 1123 MPa. In the section away from injection point (AIP) flexural strength of flat laminates measured 1138 MPa and side edge of hat shaped components measured 1173 MPa and these measurements were identical to each other. However, at top side surface of hat shaped component a maximum flexural strength of 1282 MPa was measured away from injection point. Flat laminates near injection point exhibited the highest flexural modulus value of 41.8 GPa compared to flexural modulus of hat shaped components at top side surface and side edge surface, which varied between 36.8 - 37 GPa. In the section away from injection point nearly equivalent flexural modulus in the range of 38.5 - 39.8 GPa was measured for flat laminates as well as at top side surface and side edge surface of hat shaped components. Based on these results, it can be concluded that the roving displacement observed at side edge surface of hat shaped components and slightly

higher fiber volume content in top side surface of hat shaped components did not lead to any derivable influence on flexural properties of these components.

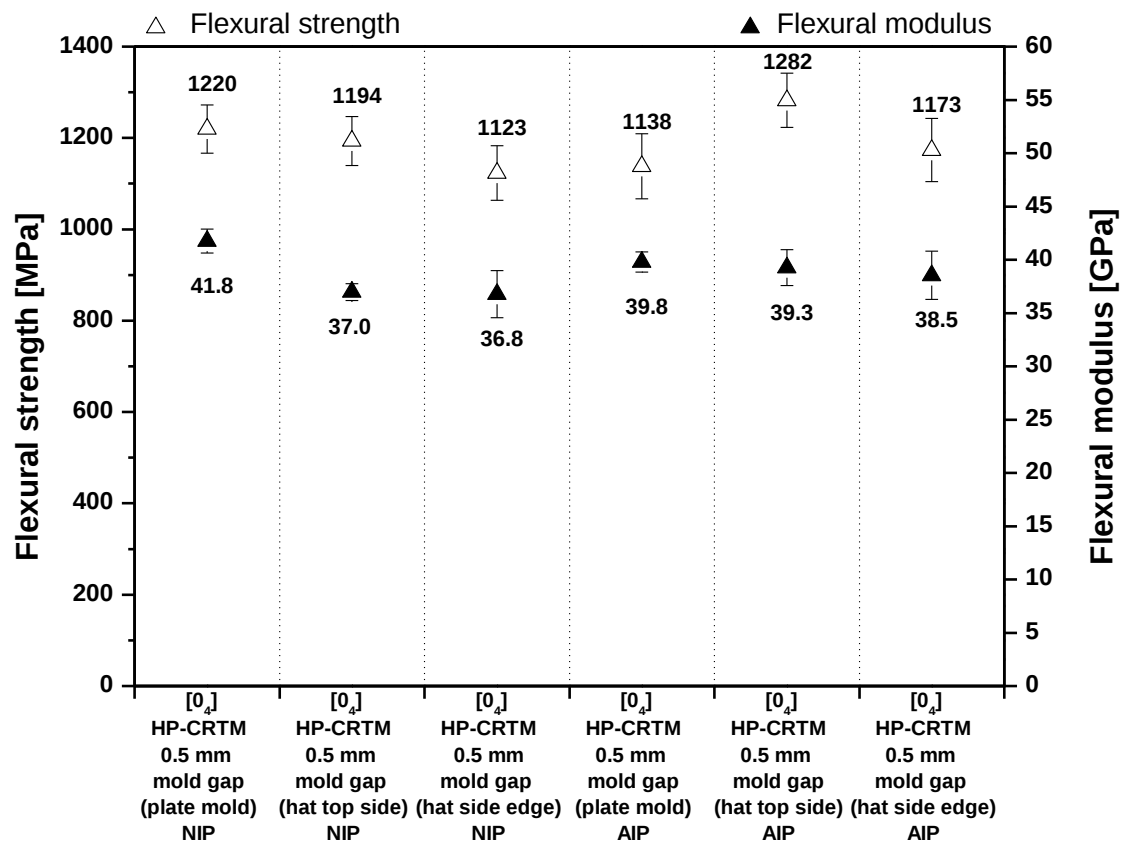


Figure 6.25: Flexural properties of unidirectional flat laminates and hat shaped components manufactured by the HP-CRTM process

In the case of bidirectional laminates, as can be seen in Figure 6.26, a different trend was observed in flexural properties. Flexural strength of flat laminates and hat shaped components at top side surface and side edge surface was almost constant near injection point (NIP) and away from injection point (AIP), and it varied between 1059 - 1107 MPa. In the section near injection point flexural modulus of 38.2 GPa was measured for flat laminates whereas for hat shaped components flexural modulus of 33.6 GPa and 43 GPa was measured at top side surface and side edge surface respectively. Similarly, in the section away from injection point flexural modulus of 39.5 GPa was measured for flat laminates, and for hat shaped components flexural modulus of 35.5 GPa and 43.1 GPa was measured at top side surface and side edge surface respectively. The increase in flexural modulus of bidirectional laminates at side edge surface, compared to top side surface, can be correlated to higher fiber volume content in hat shaped components at side edge surface than in top

side surface. Flexural modulus was nearly constant in the sections near and away from injection point respectively for flat laminates as well as hat shaped components.

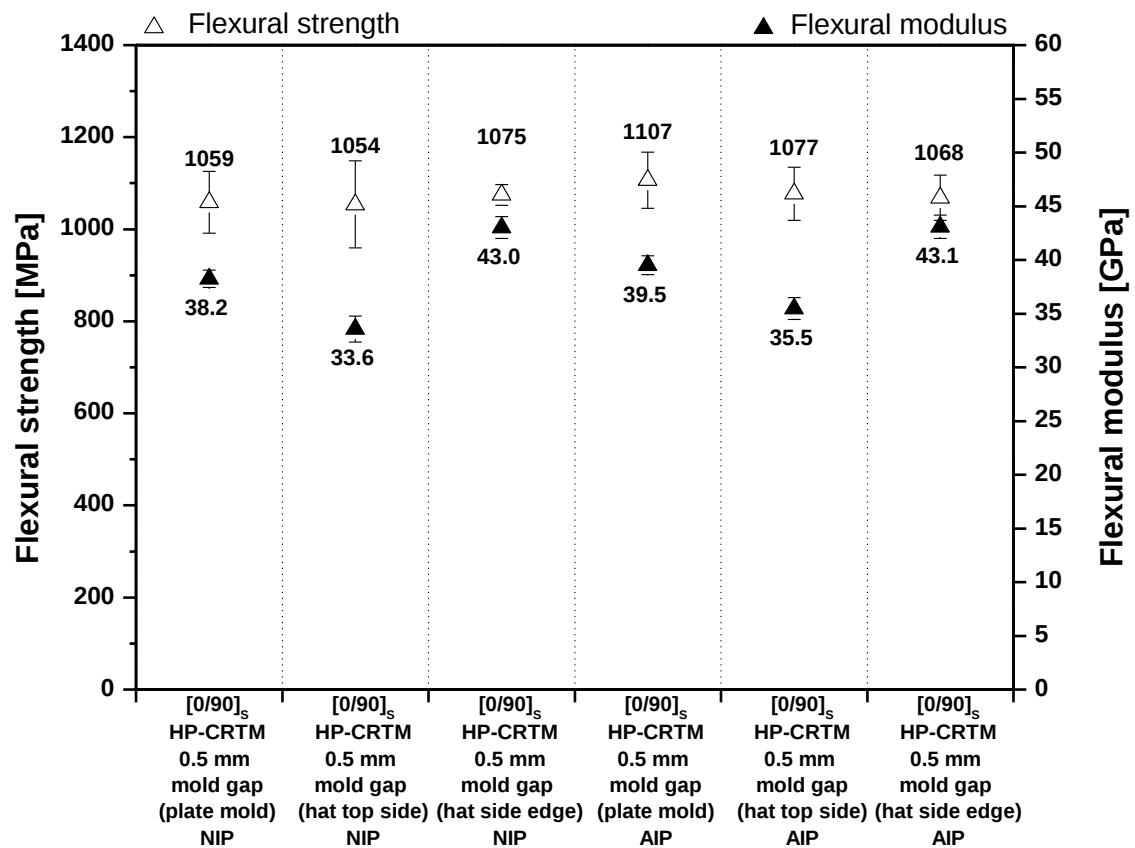


Figure 6.26: Flexural properties of bidirectional flat laminates and hat shaped components manufactured by the HP-CRTM process

6.2.3.3 Conclusion of mold geometry effects for HP-CRTM process

The main goal of manufacturing hat shaped components and their comparison with flat laminates was to study the effect of component geometry on the HP-CRTM process. The molds used for manufacturing flat laminates and hat shaped components were identical to each other in terms of gasket system, injection runner and gate geometry. In the case of unidirectional flat laminates, the best impregnation quality was obtained with a 1 mm mold gap, and for bidirectional flat laminates the best impregnation quality was obtained with a 0.5 mm mold gap. If the component geometry was varied from flat to hat shaped geometry then for both laminate layups it was essential to use the mold gap value of 0.5 mm to avoid the resin flow out of the mold cavity. This indicates that the selection of the mold gap value in the HP-CRTM process can be strongly affected by the component geometry. The hat shaped mold exhibited a higher thickness at top side surface (3.2 mm) and a lower thickness at side edge surface (2.9 mm). For unidirectional laminate layup, significant roving displacement

was observed in samples taken from side edge surface of hat shaped components as a result of a combination of the effects of manual draping of unidirectional textile layers on the mold core (lower mold half) and application of compression force on side edge surface. Such roving displacement was not observed in flat unidirectional laminates. In case of bidirectional laminate layup no significant roving displacement was observed in samples taken from side edge surface of hat shaped component. The occurrence of rovings displacement in hat shaped components with unidirectional laminate layup, and absence of roving displacement in hat shaped components with bidirectional laminate layup, led to changes in the physical and mechanical properties in different areas these components (i.e. measured differences in properties of samples taken from top side and side edge surfaces of the components). This leads to the conclusion that, although the mold gap value was selected to allow the manufacturing of hat shaped components by the HP-CRTM process, variations in the properties of hat shaped components were observed at different sections / areas due to the chosen textile reinforcement and laminate layup.

Through the investigations described above (chapter 6.2), the effect of mold gap and mold gap closure, and hence the effect of stepwise pressure build-up and mold geometry were investigated for the HP-CRTM process. These investigations were essential to understand the main influencing parameters for the HP-CRTM process. As a part of this doctoral thesis both the HP-RTM process variants, HP-CRTM and HP-IRTM processes, were studied for manufacturing glass fiber reinforced composites using plate mold 2. The investigation published by the author showed that it was possible to manufacture glass fiber reinforced laminates by the HP-IRTM process (without using any mold gap/effective gap) using plate mold 2 even under high resin flow rate. This led to a conclusion that the manufacturing of glass fiber reinforced laminates with equivalent physical and mechanical properties was possible by both the HP-RTM process variants, provided that process parameters were adapted and optimized [97-99]. While considering these results for manufacturing glass fiber reinforced composites, as a next step in this doctoral thesis the HP-RTM process variants were characterized to manufacture carbon fiber reinforced plastics (CFRP).

6.3 Characterization of HP-RTM process variants for manufacturing carbon fiber reinforced plastics

For investigating the effect of the HP-RTM process variants on the manufacturing of carbon fiber reinforced plastics plate mold 2 was used in different design configurations. Two different epoxy resin systems, Resin A and Resin B, were used for studying the HP-RTM process variants. The non-crimp fabric having surface area weight of approximately 300 g/m²

and manufactured using Toray carbon fiber type T620S-24K-50C was used as textile reinforcement. Following laminate layups were used for the studies:

1. Layup 1: 5 layers of textile reinforcement based on biaxial fabrics
[0/90 # +45/-45 # 0/90 # -45/+45 # 90/0]
2. Layup 2: 8 layers of textile reinforcement based on biaxial fabrics
[0/90 # +45/-45 # -45/+45 # 90/0]_s
3. Layup 3: 8 layers of textile reinforcement based on uniaxial fabrics [0 # 90 # 0 # 90]_s

Investigations were conducted to evaluate the effect of resin flow rate (resin injection time) on laminate quality. For manufacturing laminates by Resin A and Resin B, 570 g of resin was injected into the cavity for all the experimental series conducted using 5 layers of textile reinforcement. This resin amount was slightly higher than the theoretically calculated resin amount required for impregnating laminates of size 910 mm x 560 mm x 1.9 mm size with approximately 49 % fiber volume content. For manufacturing laminates using 8 layers of textile reinforcement 670 g of resin was injected into the cavity in 30 s for both the HP-RTM process variants and this resin amount was slightly higher than the theoretically calculated resin amount required for impregnating laminates of size 910 mm x 560 mm x 2.4 mm size with approximately 55 % fiber volume content. All the laminates were cured in mold at a mold temperature of 120 °C for 300 s after resin injection. Further details of process parameters are given in annex 9.1 [100-105].

6.3.1 Selection of mold gap and its effect on cavity pressure profile of HP-RTM process variants

The selection of the mold gap for the resin injection step is an important factor which determines permeability of the textile reinforcement during the resin flow through the mold cavity. In case of HP-IRTM process, if compared to HP-CRTM process, no mold gap is used and thus the permeability of the reinforcement is expected to be lower in the HP-IRTM process than in the HP-CRTM process. In the studies conducted for glass fibers using plate mold 1 and plate mold 2 (design configuration 1), using an effective mold gap of 0.2 mm high quality impregnation was obtained for bidirectional laminate layup in chapter 6.2. For the study of the HP-CRTM process variant for manufacturing carbon fiber reinforced composites thus an effective gap of 0.3 mm was selected. The HP-CRTM and HP-IRTM process variants have been studied in literature indicating the relevance of a 0.3 mm mold gap on the cavity pressure profile during the resin injection step. The same mold as in thesis work, plate mold 2 with design configuration 2 having film gate geometry, was used for studying the HP-RTM process variants [106].

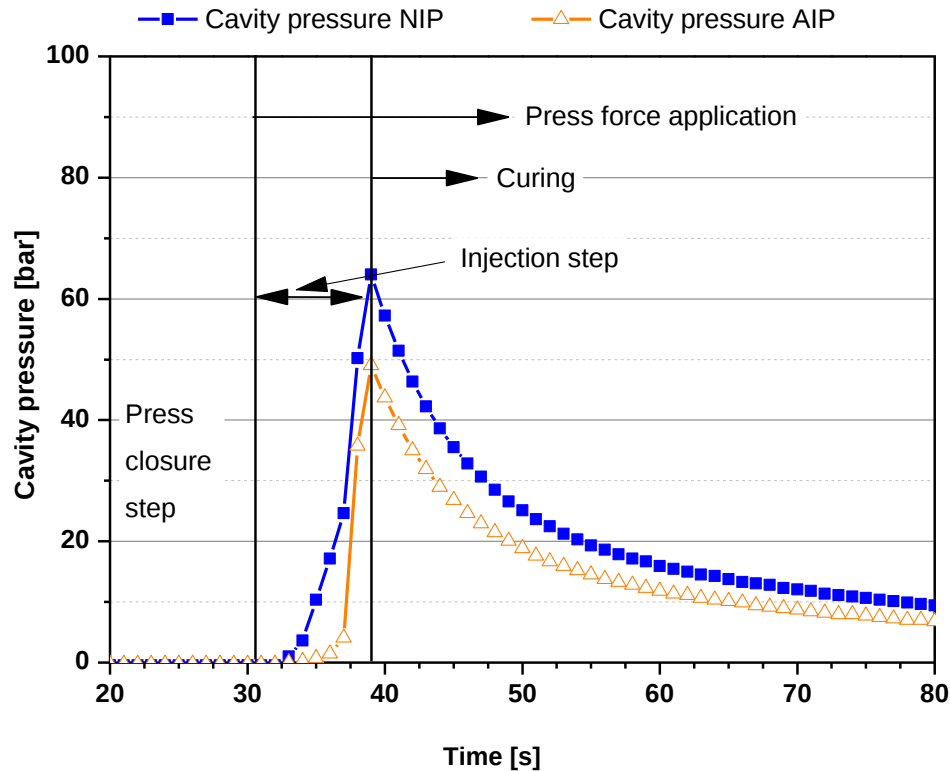


Figure 6.27: HP-IRTM process analysis for laminate manufacturing using 5 layers of reinforcement [106]

Figure 6.27 shows the summary of the process data from the injection equipment for a laminate manufactured using a HP-IRTM process and resin flow rate of 80 g/s. This figure is divided into three important sections, namely press closure, resin injection and curing. The press closure step is purely a function of the selected press closure profile and it may vary in accordance with the used profile. In the HP-IRTM process the required press force is built on the mold at the end of press closure step. The typical process characteristic of HP-IRTM and HP-CRTM can be seen in the resin injection step. The resin injection step started after 31 s from the start of the process cycle. During this process step the high-pressure mixing head of the injection equipment opened and the reactive resin mixture was injected into the cavity at a defined flow rate in 8.1 s time. During the resin injection step the cavity pressure increased continuously indicating that the impregnation of fibers was carried out in the HP-IRTM process during resin injection step. As the pressure sensors were placed at a distance of 180 mm and 410 mm from the injection point, an increase in the cavity pressure was realized with a time delay of 2 s near injection point (NIP), and 6 s away from the injection (AIP) point after the resin injection step was started. The maximum measured cavity pressure in the mold was 62 bar near injection point and 49 bar away from injection point. Once the required amount of resin was injected into the cavity the mixing head was closed which was followed by the resin curing step and then the laminate was demolded. During the curing step a

continuous drop in the cavity pressure was observed which is attributed to a combination of resin shrinkage and minor resin flow out of cavity through the gaskets. As during the curing step a drop in cavity pressure was observed the HP-IRTM process is analyzed only for 80 s from the process start.

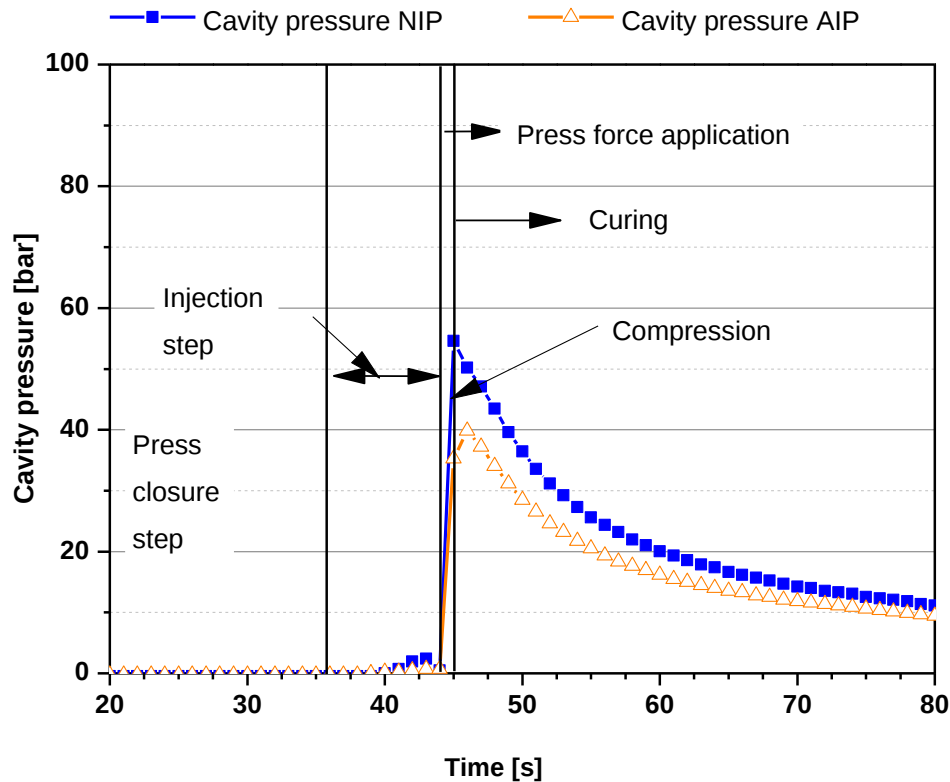


Figure 6.28: HP-CRTM process analysis for laminate manufacturing using 5 layers of reinforcement at 0.3 mm mold gap [106]

Figure 6.28 shows the summary of the process data for a laminate manufactured using the HP-CRTM process and resin flow rate of 80 g/s. If compared to the HP-IRTM process, an additional compression step can be observed in the process data summary. In the press closure step the press was closed to a defined mold gap of 0.3 mm and in this case the press force is not yet applied on the mold. The resin injection step started after 35 s from the start of the process cycle step. During the resin injection step in this experiment resin injection was carried out as well at defined resin flow rate in 8.1 s in a partially open mold cavity. After resin injection the mixing head was closed and then the compression step was carried out. The press closed the partially open mold completely, applying defined compression force of 3100 kN. During the compression step, which lasted for 2-3 s, there was an immediate increase in the cavity pressure which indicates that the resin was squeezed in the fibers thereby leading to their impregnation by the resin. The observed maximum cavity pressure near and away from injection point was approximately 54 bar and

39 bar respectively. After the compression step, the curing step was carried out and then the laminate was demolded. Unlike the HP-IRTM process, almost no increase of cavity pressure near or away from injection point was observed during the whole resin injection step. This indicates that there was no compaction of the textile layers as the press closed to a mold gap of 0.3 mm (effective gap 0.3 mm) and enough permeability of the textiles must have been retained homogeneously over the entire laminate area during the resin injection step. As no cavity pressure was built up during the resin injection step, it appears that likely the macroscopic resin flow was dominant during the resin injection step and that only a limited microscopic impregnation of the laminates was obtained during this step. As the cavity pressure rose significantly during the compression step, majorly the microscopic impregnation of the roving filaments must have taken place during the compression step. These investigations conducted using glass fibers in this thesis and based on the literature and effective gap of 0.3 mm was selected for the HP-CRTM process for manufacturing carbon fiber reinforced laminates. For manufacturing laminates using 5 layers of reinforcement and 8 layers of reinforcement mold gaps of 0.3 mm and 1 mm were selected respectively for HP-CRTM process which led to an effective mold gap of approximately 0.3 mm.

6.3.2 Effect of resin viscosity and process variant on laminate manufacturing

During laminate manufacturing by selected process variants the real-time process parameters from the HP-RTM injection equipment were exported and analyzed to evaluate the effect of selected process parameters on the stability of the injection process and laminate quality. The actual injection pressure of the used HP-RTM equipment in this thesis work was not measured as no sensor was placed directly between the mixing head and the resin injection point of the mold. The mixing head pressure sensors of resin and hardener as they were just located before the mixing area of the head only indicate the trend of injection pressure variation during the HP-RTM process on materials pressure variations during constant flow rate dosing of these materials. In order to study the effect of resin viscosity and process variant on the laminate manufacturing laminate layup 1 based on 5 layers of biaxial reinforcement [0/90 # +45/-45 # 0/90 # -45/+45 # 90/0] was used along with the design configuration 1 of plate mold 2 having point injection gate geometry.

HP-IRTM process analysis

Figure 6.29 (a) shows the summary of process data for a laminate manufactured using the HP-IRTM process, Resin A and 30 s resin injection time. In the press closure step the mold was closed as the upper half of the press was moved down from its maximum

opening height, and a defined press force of 4200 kN was applied on the mold, which contained 5 layers of textile reinforcement with defined laminate layup. In this step the high-pressure mixing head remained closed and the equipment re-circulated the resin and hardener through the high-pressure mixing head under a defined pressure which was 117 bar for resin and 124 bar for hardener.

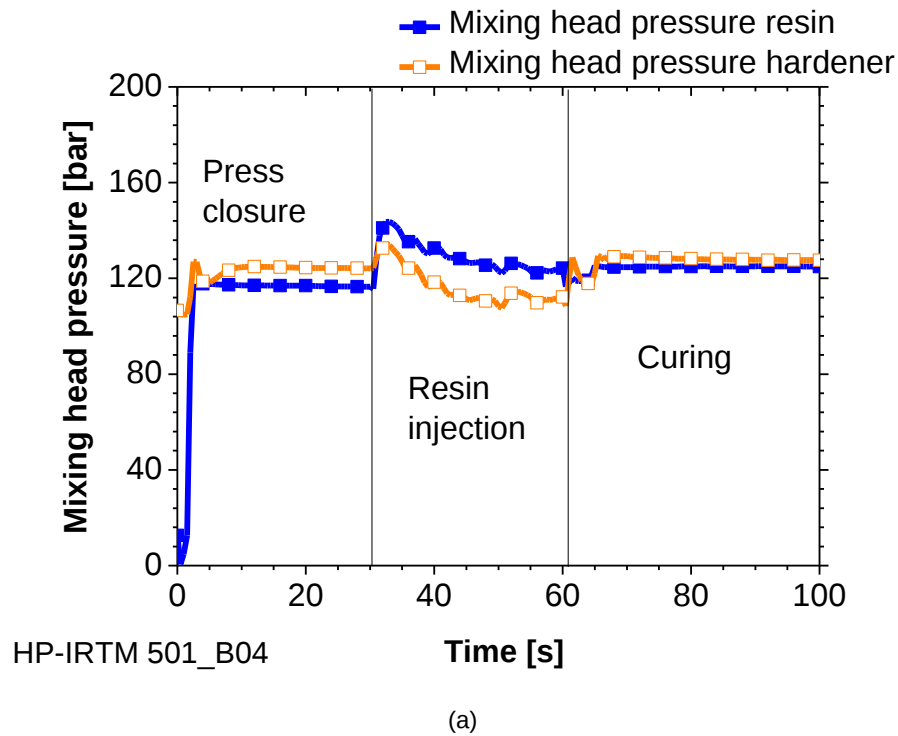


Figure 6.29: HP-IRTM process analysis for laminate manufacturing using 5 layers of reinforcement, Resin A, 30 s resin injection time and point gate; (a) process data, (b) laminate quality near injection point

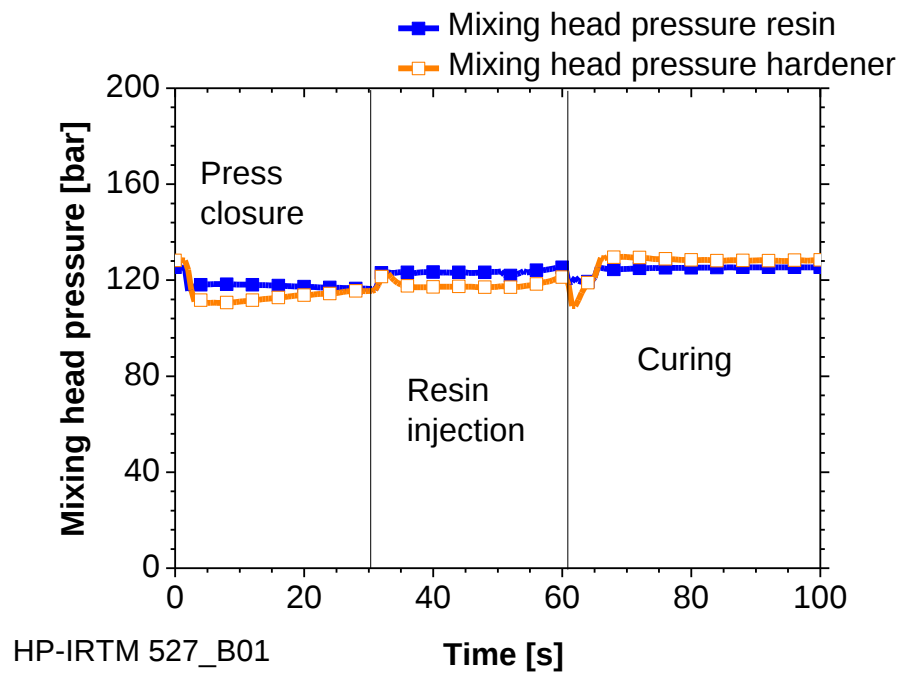
During the resin injection step, the high-pressure mixing head opened, resulting in resin and hardener mixing under high pressure, and the reactive resin mixture was injected into the cavity at a defined flow rate which was approximately 19 g/s in this experiment. As can be observed in Figure 6.29 (a), as the resin injection step started the resin and hardener pressure increased suddenly to 142 bar from an initial value of 117 bar, and to 132 bar from an initial value of 124 bar respectively, and during the whole resin injection step the resin and hardener pressure fluctuated. Once the required amount of resin was injected into the cavity the mixing head was closed again and the resin and hardener were shifted back to re-circulation mode. After the resin injection step was completed, the curing step followed during which the laminate was cured for 300 s and then the laminate was demolded. Figure 6.29 (b) shows a picture of the area near injection point (laminate photographed from cavity side in which the injection point is located) of the laminate manufactured by the HP-IRTM process using Resin A and 30 s resin injection time. A strong displacement of the rovings can be observed directly at the injection point. This roving displacement can be correlated with a sudden increase of resin and hardener pressure at the start of the resin injection step, and with subsequent fluctuation in the resin and hardener pressure throughout the whole injection step.

As mentioned earlier the mold, when closed completely, exhibited a cavity height from 1.55 mm to 1.75 mm near and away from injection point respectively. Hence a complete closure of the mold with a press force of 4200 kN led to strong compaction of the textile layers. This compaction of the textile layers must have reduced the permeability of the textile layers drastically and as a result at the start of the resin injection step itself resin and hardener pressure increased immediately. The later drop in resin and hardener pressure and pressure variations can be explained by the displacement of rovings in the textile layers, leading to continuous changes in the local permeability of the textiles. The variations in pressure can also be correlated with differential macroscopic and microscopic resin flow through textiles. As the resin flows through free spaces between rovings (macroscopic flow through regions with high permeability) the resistance to resin flow decreases, leading to pressure drop, and as the resin flows through a roving and between fiber filaments (microscopic flow through regions with low permeability) the resistance to resin flow increases, leading to pressure increase.

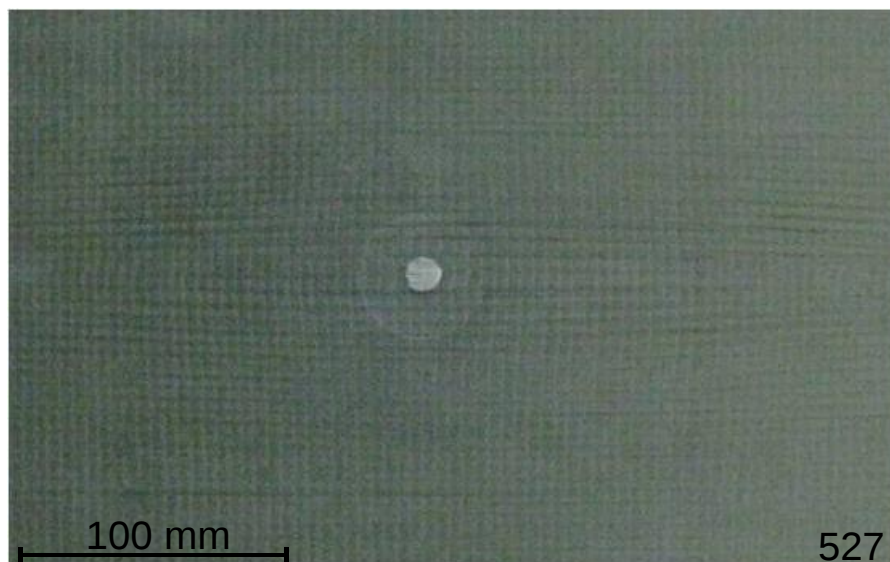
Figure 6.30 shows the summary of the comparative experiment for a laminate manufactured using the HP-IRTM process, Resin B and 30 s resin injection time. For Resin B the resin and hardener pressure increased slightly to 122 bar from an initial value of 117 bar, and to 121 bar from an initial value of 114 bar respectively during the whole resin injection step and

resin and hardener pressure did not show any fluctuations which however were observed for the Resin A as seen in Figure 6.29 (a).

Figure 6.30 (b) shows picture of the area near injection point of the laminate manufactured using Resin B. If compared to the laminate manufactured by Resin A, only slight displacement of the rovings was observed directly at the injection point in the cured laminates after demolding.



(a)



(b)

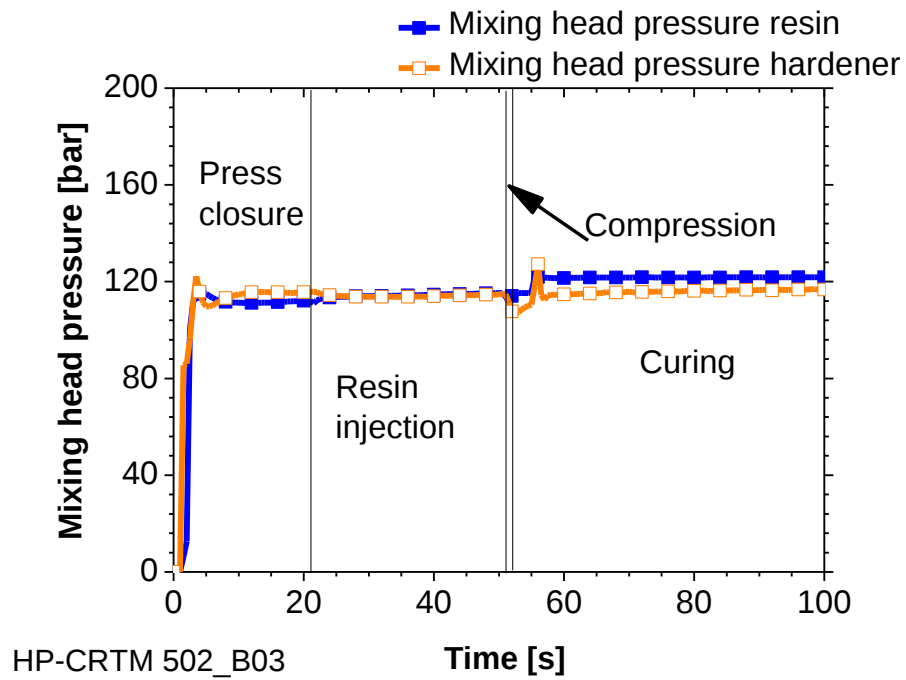
Figure 6.30: HP-IRTM process analysis for laminate manufacturing with 5 layers of reinforcement, Resin B, 30 s resin injection time and point gate; (a) process data, (b) laminate quality near injection point

The observed effect of only slight rovings displacement for the laminate manufactured using Resin B if compared to higher rovings displacement in the laminate manufactured using Resin A can be explained on the basis of the impregnation viscosity of the used resin systems. The impregnation viscosity of Resin B was only 20 mPa.s if compared to the relatively higher impregnation viscosity of Resin A which was 40 mPa.s during the conducted experiments. Hence Resin B could flow through the compacted textiles having low permeability near injection point relatively without causing any rovings displacement if compared to Resin A. Also, low viscosity of the resin did not cause any fluctuations on the resin and hardener pressure in the mixing head. The experiments conducted using Resin A and Resin B indicates that the resin viscosity plays an important role in the resin flow through the compacted carbon fiber based textiles in the HP-IRTM process and if the resin viscosity is high then it may lead to a problem of the rovings displacement.

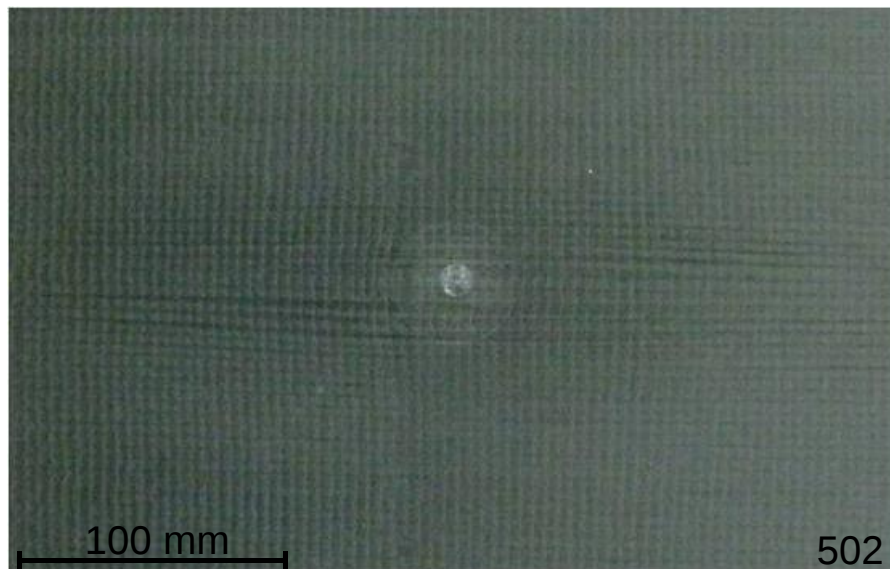
HP-CRTM process analysis

Figure 6.31 (a) shows the summary of the process data for a laminate manufactured using the HP-CRTM process, Resin A and 30 s resin injection time. If compared to the HP IRTM process, an additional compression step can be observed in the process data summary. In the press closure step the press is closed to a defined mold gap of 0.3 mm. During the resin injection step, the high-pressure mixing head opened, resulting in resin and hardener mixing under high pressure and resin injection at defined resin flow rate in 30 s in a partially open mold cavity. Unlike the HP-IRTM process conducted using Resin A, as the resin injection step started, the resin and hardener pressure did not increase during this HP-CRTM experiment and resin and hardener pressure during the injection step remained constant at 115 bar.

Figure 6.31 (b) shows picture of the area near injection point of the laminate manufactured by the HP CRTM process using Resin A and 30 s resin injection time. In comparison to the laminates manufactured by the HP IRTM process with 30 s resin injection time, very little roving displacement was observed in the area near injection point in the laminate manufactured by the HP CRTM process. This indicates that there was no significant compaction of the textile layers as the press closed to a mold gap of 0.3 mm, and enough permeability of the textiles must have been retained homogeneously over the entire laminate area during the resin injection step.

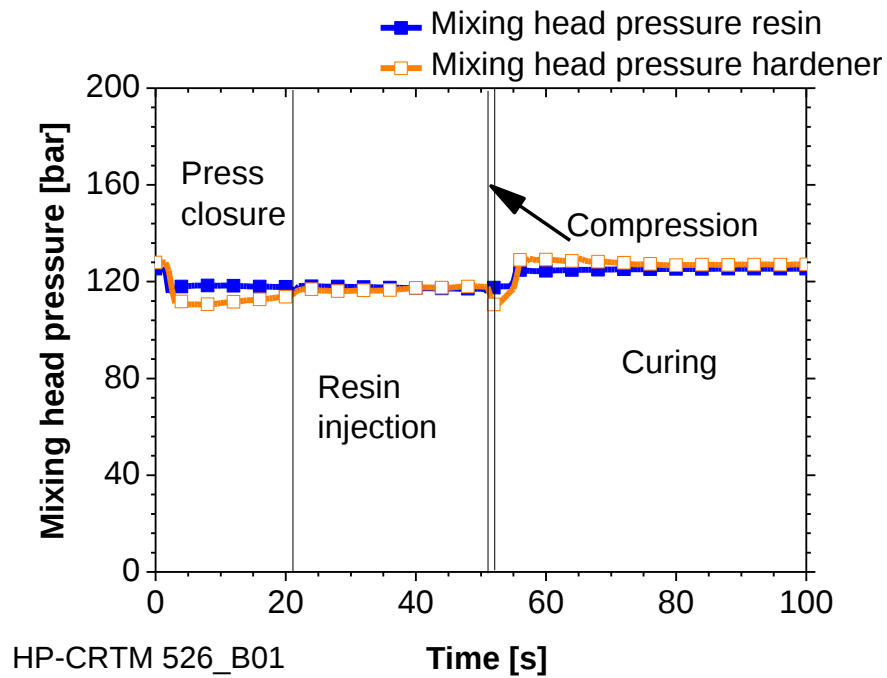


(a)

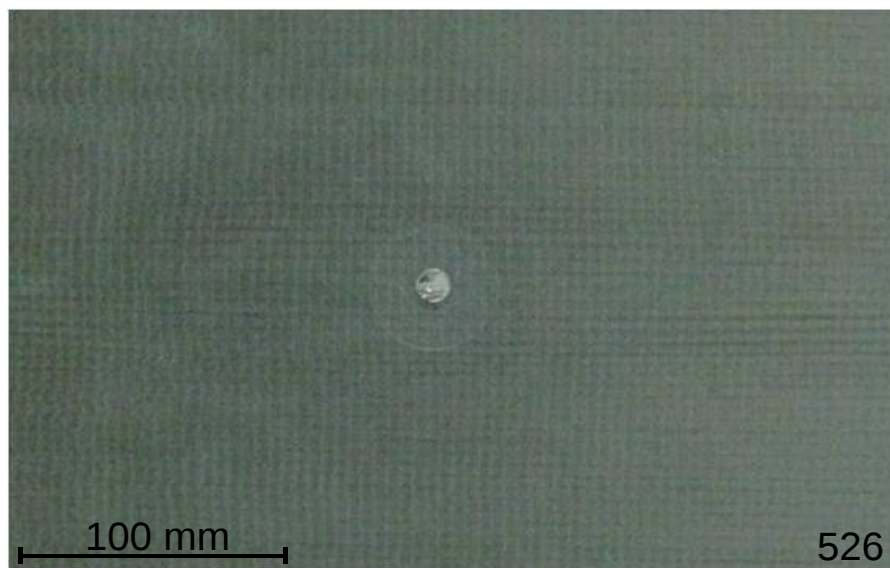


(b)

Figure 6.31: HP-CRTM process analysis for laminate manufacturing using 5 layers of reinforcement, Resin A, 30 s resin injection time and point gate; (a) process data, (b) laminate quality near injection point



(a)



(b)

Figure 6.32: HP-CRTM process analysis for laminate manufacturing using 5 layers of reinforcement, Resin B, 30 s resin injection time and point gate; (a) process data, (b) laminate quality near injection point

Figure 6.32 (a) shows the summary of the comparative experiment for a laminate manufactured using the HP-CRTM process, Resin B and 30 s resin injection time. During resin injection step, in this HP-CRTM experiment also the resin and hardener pressure did not increase and pressure values remained constant till end of resin injection step. Almost no roving displacement was observed in the area near injection point in the laminate

manufactured using Resin B as seen in Figure 6.32 (b). This observed effect of no rovings displacement in the laminate manufactured using the HP-CRTM and Resin B if compared to the observed slight rovings displacement in the comparative laminate manufactured using Resin A is correlated to the impregnation viscosity of the used resin systems. The Resin B having low impregnation viscosity, if compared to the relatively higher impregnation viscosity of the Resin A, showed a significant improvement in the laminate quality near injection point especially in terms of rovings displacement in the HP-CRTM process. A similar effect of resin viscosity on the rovings displacement was observed for other resin systems if the resin viscosity was too high during the resin injection step [107].

6.3.3 Effect of carbon fiber volume content on HP-RTM process variants

The studies conducted using carbon fibers in chapter 6.3.2 indicated that it was possible to obtain laminates by using HP-IRTM and HP-CRTM process variants using Resin A and design configuration 1 of plate mold 2. In this study using 5 layers of reinforcement (each layer 300 g/m²) at 1.7 mm laminate thickness approximately 49 % fiber volume content was measured in the laminates experimentally. In order to understand the relevance of HP-RTM process variants for manufacturing laminates of high fiber volume content, 8 layers of reinforcement were used as in laminate layup 2 for the next studies to achieve fiber volume content of approximately 56 % at 2.4 mm laminate thickness.

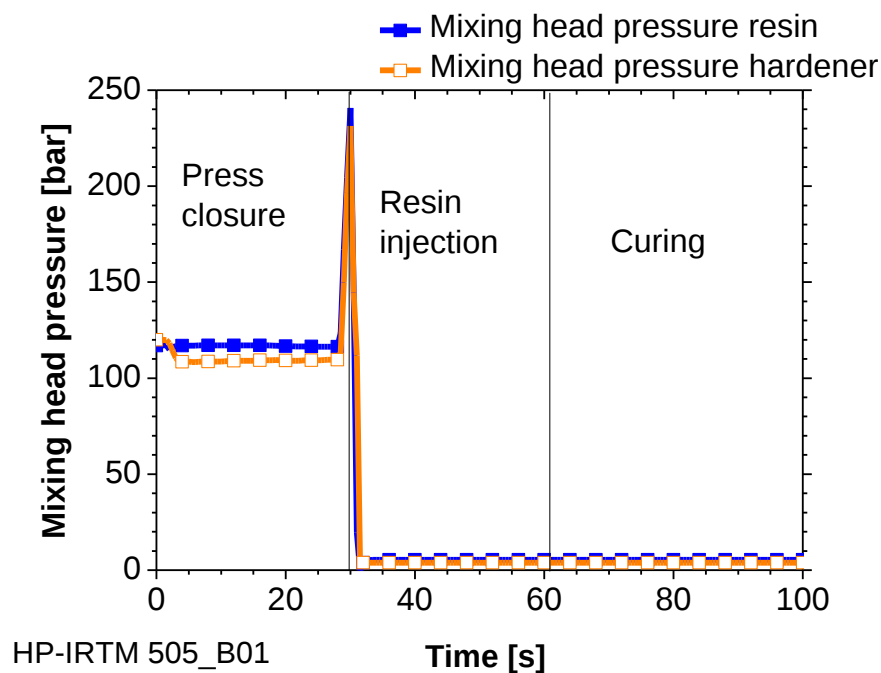


Figure 6.33: Process data from HP-IRTM process analysis for laminate manufacturing using 8 layers of reinforcement, Resin A, 30 s resin injection time and point gate

Figure 6.33 shows the summary of the process data for a laminate manufactured using the HP-IRTM process, Resin A, 30 s resin injection time and 8 layers of textile reinforcement. After the press closure was completed the resin injection step started, and as can be seen, within 2 s the pressure on the resin and hardener in the mixing head reached a value of more than 230 bar. Due to pressure increase in the mixing head to more than 230 bar the equipment stopped due to safety shutdown of the equipment. This indicates that as the mold was closed by the press the 8 layers of textile reinforcement were compacted to very high extent, than the compaction of 5 layers of reinforcement in earlier experiments in chapter 6.3.2, thereby leading to strong reduction of the permeability of the textiles.

The high degree of compaction of 8 layers if compared to 5 layers can be confirmed on the basis of dependency of permeability on the fiber volume content. The correlation between permeability of the textile reinforcements and fiber volume content can be explained on the basis of following equation:

$$K = C_1 d^2 B^3 / (1 - B)^2 \quad \text{Equation 6.9}$$

Where, C_1 is a constant, d is the fiber diameter, B is the porosity ($1 - V_f$) and V_f is the fiber volume content of the component. As 5 layers and 8 layers of reinforcement were based on the same fabric product, the effect of fiber diameter effect on the permeability for these two laminate layups can be treated as a constant thus leading to following permeability relationship.

$$K_{5 \text{ layers}} = C_0 (1 - 0.49)^3 / (1 - [1 - 0.49])^2$$

$$K_{8 \text{ layers}} = C_0 (1 - 0.56)^3 / (1 - [1 - 0.56])^2$$

Where, $K_{5 \text{ layers}}$ and $K_{8 \text{ layers}}$ represent the permeability of 5 layers and 8 layers of reinforcement at 49 % and 56 % fiber volume content respectively and C_0 is the new constant which include effect of diameter on permeability along with constant C .

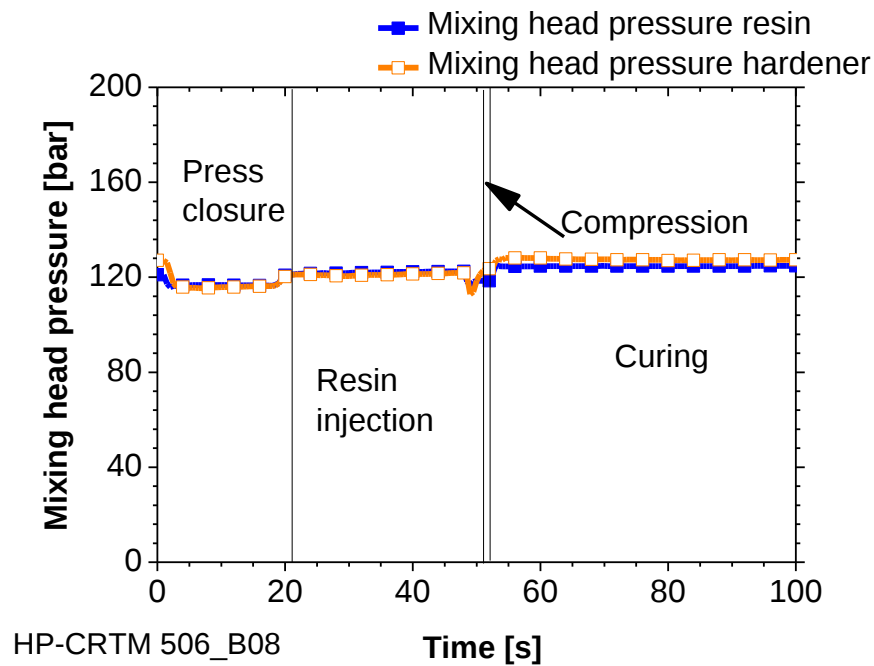
The above equations result in:

$$K_{5 \text{ layers}} = 0.55 C_0 m^2$$

$$K_{8 \text{ layers}} = 0.27 C_0 m^2$$

As can be seen, the permeability of 8 layers of reinforcement at 56 % fiber volume content drops approximately by a factor of 2 if compared to 5 layers of textile reinforcement at 49 % fiber volume content. The reduced permeability of the textiles led to too high pressure built-up in the mixing head and hence it was not possible to manufacture a laminate using 8 layers of reinforcement by the HP-IRTM process. The mathematical correlation between permeability of 5 layers and 8 layers of reinforcements and HP-IRTM experiments conducted

using these materials validated hypothesis 3 according to which for a selection of specific mold geometry, and resin viscosity (η), resin flow rate (Q) and mold filling time (T) being defined as constants, the differences in permeability (K) shall result in different pressure gradients (dp/dx). In conducted experiments pressure gradient was not directly measured, but pressure profile on materials was recorded to reflect influence of permeability on resin injection process.



(a)



(b)

Figure 6.34: HP-CRTM process analysis for laminate manufacturing using 8 layers of reinforcement, Resin A, 30 s resin injection time and point gate; (a) process data, (b) laminate quality near injection point

Figure 6.34 (a) shows the summary of the process data for a laminate manufactured using the HP-CRTM process, Resin A, 30 s resin injection time and 8 layers of textile reinforcement. During the press closure step the press was closed to leave a mold gap of 1 mm between upper and lower mold halves. This led to an effective gap of 0.3 mm in the mold during resin injection step.

Unlike the HP-IRTM process, completion of the resin injection step was achieved during this HP-CRTM experiment without any significant pressure increase in the mixing and no fluctuations were observed on the materials pressure. Figure 6.34 (b) shows picture of the area near injection point of the manufactured laminate. As can be seen, very little roving displacement was observed in the area near injection point. The possibility of manufacturing the laminate using 8 layers of reinforcement by HP-CRTM process, if compared to non-successful HP-IRTM process, can be explained on the basis of fiber volume content / porosity of the fibers in the partially open mold cavity at the defined effective mold gap of 1 mm. Due to the mold gap of 1 mm, the mold cavity height increased from 1.7 mm to 2.7 mm. The fiber volume content of the 8 layers of reinforcement in partially open mold cavity at 2.7 mm cavity height can be calculated as follows:

$$V_{f \text{ at } 0.3 \text{ mm gap}} = \left[\frac{V_{\text{fibers in cavity}}}{V_{\text{cavity}}} \right] \cdot 100 \quad \text{Equation 6.10}$$

Where, $V_{\text{fibers in cavity}}$ is the total fiber volume in the free cavity volume (V_{cavity}).

$V_{\text{fibers in cavity}}$ can be calculated as follows:

$$V_{\text{fibers in cavity}} = \left[\frac{n \cdot A_{\text{mold}} \cdot \rho_{\text{fabrics}}}{\rho_{\text{fibers}}} \right] \quad \text{Equation 6.11}$$

Where: n is the number of textile layers, A_{mold} is the surface area of the mold, ρ_{fabrics} is the areal density of the fabric and ρ_{fibers} is the density of fiber material. While considering the area of the mold as 0.91 m x 0.56 m (length x width), cavity height of 2.7 mm, 8 number of layers having 0.3 kg/m² areal density and 1800 kg/m³ as carbon fiber density leads to:

$$V_{\text{fibers in cavity}} = 679 \text{ cm}^3, V_{\text{cavity}} = 1376 \text{ cm}^3, \text{ and } V_{f \text{ at } 0.3 \text{ mm gap}} = 49\%$$

The theoretical fiber volume content of 49 % and thus resulting porosity of 51 % at 0.3 mm effective gap thus leads to the nearly equivalent permeability ($0.55C_0 \text{ m}^2$) for the 8 layers of reinforcement in the HP-CRTM process as for the 5 layers of reinforcement in the HP-IRTM process thus making it feasible to manufacture the laminate in the HP-CRTM process. This again validated the hypothesis 2 according to which the resin flow rate and mold filling time are affected by permeability of textile reinforcements in mold cavity.

6.3.4 Effect of an alternative gate geometry on HP-RTM process variants

The previous investigations carried out for manufacturing of the carbon fiber reinforced composites (chapter 6.3.2 and chapter 6.3.3) by the HP-RTM process variants were conducted using point gate geometry for the resin injection. The investigations showed that it was not possible to manufacture laminates with 8 layers of reinforcement (fiber volume content approx. 55 %) by the HP-IRTM process. If Resin A was used then often fiber rovings disorientation was observed near the resin injection area in the laminates manufactured using 5 layers of reinforcement. Hence in the next step of investigations plate mold 2 was modified from configuration 1 to configuration 2 in which the point injection gate was modified to a film injection gate. For a constant injection pressure system (variable resin flow rate) the relationship of mold filling time and injection gate strategy is given by following equation 3.3. According to this equation the mold filling time T is directly proportional to the constant C whose value is determined by the gating choice. For plate mold 2 the injection gate diameter was 12 mm for a characteristic mold length of 910 mm. So the value of constant C for the design configuration 1 of plate mold 2 is determined according to equation 3.4 as follows:

$$C_{plate\ mold\ 2\ configuration\ 1} = 0.25 \left[\left(\frac{12}{910} \right)^2 - 1 + 2 \ln \left(1 / \frac{12}{910} \right) \right] = 2.05$$

Whereas, for the design configuration 2 of plate mold 2 the constant C is as follows:

$$C_{plate\ mold\ 2\ configuration\ 2} = \frac{1}{4} = 0.25$$

As the conducted experiments in chapter 6.3.2 and chapter 6.3.3 were with the constant flow rate, the mold filling time was actually predefined by the flow rate value and required resin injection amount. For manufacturing laminates with 8 layers of reinforcement 670 g of resin was required. At defined resin injection time of 30 s led to 22.3 g/s of resin flow rate. The correlation of required resin injection pressure for design configuration 1 and design configuration 2 of plate mold 2 can be given as:

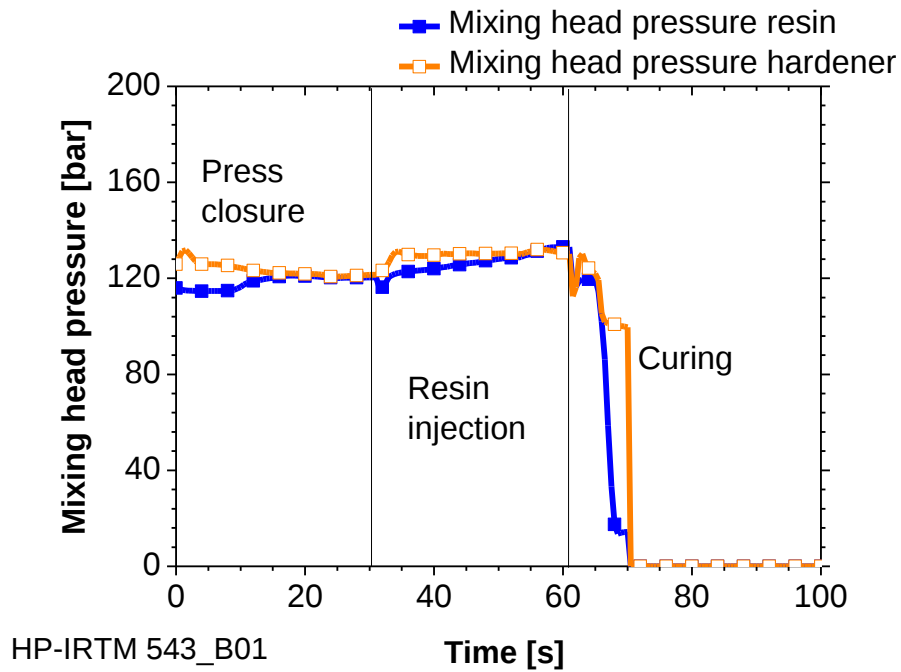
$$T_{plate\ mold\ 2\ configuration\ 1} = \frac{2\eta l^2(1-V_f)}{P_{configuration\ 1}K}$$

$$T_{plate\ mold\ 2\ configuration\ 2} = 0.25 \frac{\eta l^2(1-V_f)}{P_{configuration\ 2}K}$$

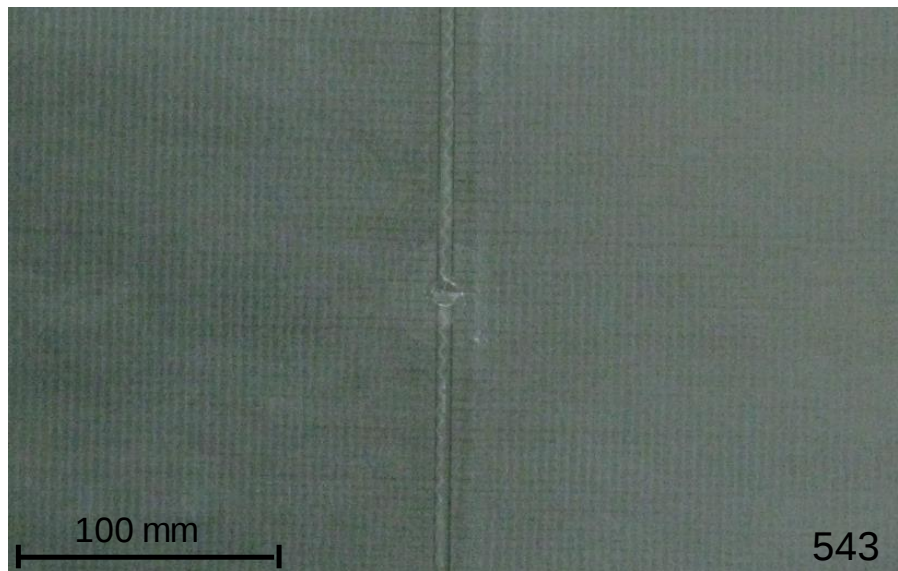
As, $T_{plate\ mold\ 2\ configuration\ 1} = T_{plate\ mold\ 2\ configuration\ 2}$, and values of l , V_f , and K can be treated constant it leads to:

$$P_{configuration\ 2} = \frac{1}{8} P_{configuration\ 1}$$

This indicates that using a film gate it shall be possible to reduce the required injection pressure in the HP-IRTM process by a factor of 8 if compared to the point gate geometry.



(a)



(b)

Figure 6.35: HP-IRTM process analysis for laminate manufacturing using 8 layers of reinforcement, Resin A, 30 s resin injection time and film gate; (a) process data, (b) laminate quality near injection point

Figure 6.35 (a) shows the summary of the process data for a laminate manufactured using the HP-IRTM process, Resin A, 30 s resin injection time, 8 layers of reinforcement and

modified film gate. Unlike the comparative laminate manufactured using the point gate (see Figure 6.33), by using the film gate it was possible to achieve the whole resin injection step and the required amount of resin was injected into the mold cavity in 30 s without any safety shutdown of the equipment. In this experiment the resin and hardener pressure did not increase after starting the resin injection step and during the whole resin injection step the resin and hardener pressure did not show any fluctuations. The achievement of whole resin injection step and non-fluctuating pressure profile of the resin and hardener can be explained as above on the basis of the film gate geometry which shall require 8 times lower pressure if compared to the point gate. Figure 6.35 (b) shows picture of the area near injection point of the laminates manufactured by Resin A, 8 layers of textile reinforcement, 30 s resin injection time and film gate geometry using HP-IRTM. As can be seen using a film gate laminate was obtained by the HP-RTM process variant without any rovings displacement. In an extended study using film gate confirmed that using 5 layers and 8 layers of reinforcement laminates without any rovings displacement were obtained using both the HP-RTM process variants [101-103].

The mathematical correlation between injection pressure by using point gate and film gate and validation of laminate manufacturing feasibility using 8 layers of reinforcements only by film gate (non-successful laminate manufacturing if point gate was used) validated hypothesis 4 according to which for a selection of specific mold geometry, and resin viscosity (η), permeability (K), resin flow rate (Q) and mold filling time (T) being kept constant, the differences in cross sectional area for resin flow (A_{flow}) resulted due to selection of different gate geometries shall influence different pressure gradients (dp/dx). Again, in conducted experiments pressure gradient was not directly measured, but pressure profile on materials was recorded to reflect influence of gate geometry on resin injection process.

6.3.5 Characterizing effect of gate geometry on laminate properties

Figure 6.36 shows ILSS properties of the laminates manufactured by HP-RTM process variants, Resin A and 5 layers (layup 1) of reinforcement. As can be seen, different laminates manufactured using point gate and film gate geometries along with resin injection time variation were used for characterizing ILSS. ILSS properties of the laminates were measured directly at the injection point (IP), near injection point (NIP) and away from injection point (AIP). It can be observed for the HP-IRTM process variant, ILSS properties at the injection point tend to drop with reduced resin injection time (30 s resin injection time compared to 15 s resin injection time) if a point gate geometry was used. Also ILSS values at the injection

point showed relatively high standard deviation as compared to ILSS values in other regions (NIP and AIP) of the laminate.

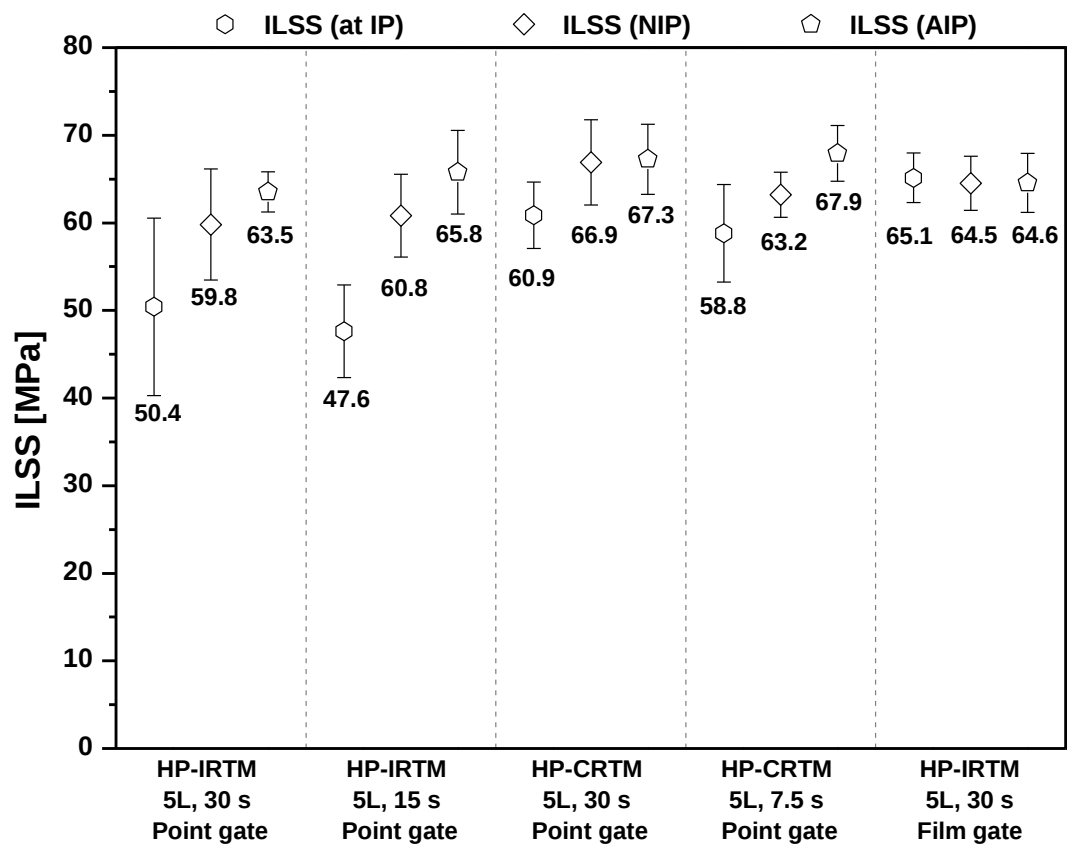
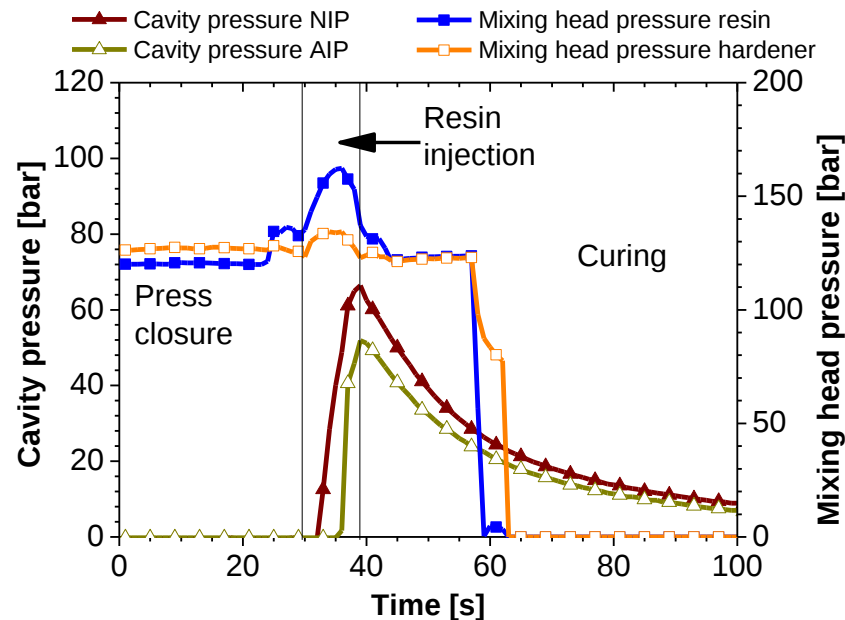


Figure 6.36: ILSS of laminates manufactured by HP-RTM process variants, Resin A, 5 layers of reinforcement and different gate geometries

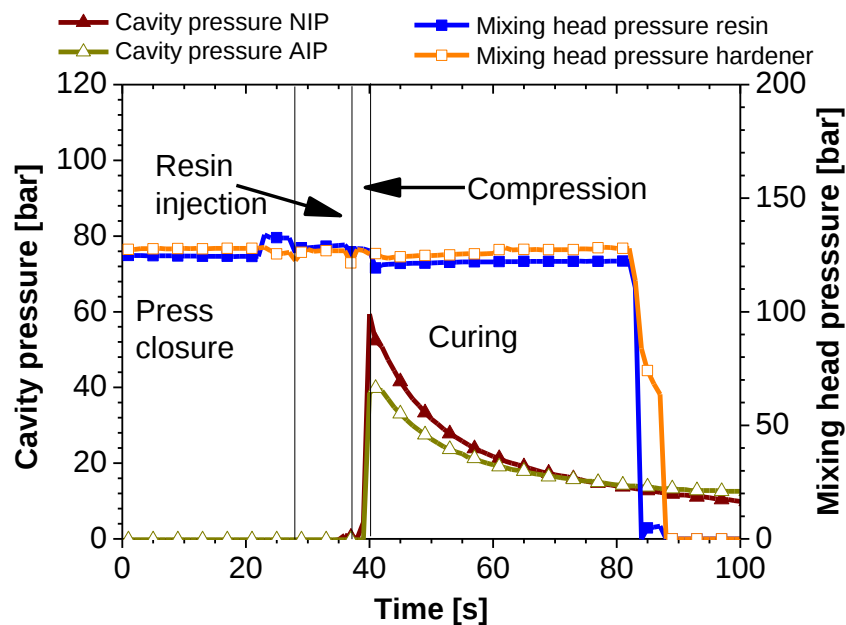
The drop in ILSS values and high standard deviation for HP-IRTM laminates can be attributed to the higher roving displacement observed in the laminates when the point gate was used. The roving displacement resulted in local variations of resin content (and hence fiber volume content) in the test specimen, thereby leading to a larger deviation in ILSS properties. In comparison to the HP-IRTM process, the HP-CRTM process at 30 s resin injection time and point injection gate geometry leads to nearly equivalent ILSS properties in all the sections of a laminate. Also, the average ILSS values of the HP-CRTM laminates are higher than HP-IRTM laminates at 30 s resin injection time. However with 7.5 s resin injection time, ILSS properties at the injection point show again a high standard deviation, attributed to increased roving displacement. In case of HP-IRTM process, if the point gate geometry was changed to film gate geometry, constant ILSS properties were observed in all the three sections of the laminates. This indicates that homogeneous quality was observed in the laminates manufactured using film gate geometry.

These investigations carried out in chapter 6.3.4 and chapter 6.3.5 thus indicates that special attention shall be given for the resin injection gate geometry selection during the mold design for implementing the HP-RTM process variants for manufacturing CFRP.

6.3.6 Correlating HP-RTM process variants for high-volume manufacturing



(a)



(b)

Figure 6.37: HP-RTM process analysis for manufacturing laminate using 8 layers of reinforcement, film gate and 80 g/s resin flow rate; (a) process data for HP-IRTM process, (b) process data for HP-CRTM process

As concluded above by using a film gate it was possible to obtain laminates of equivalent quality by HP-IRTM and HP-CRTM process variants. However, the selection of one of these two process variants especially for high-volume manufacturing of automotive components is not yet clear. Hence additional experiments were conducted to understand the effect of these process parameters for high-volume manufacturing. The laminates were manufactured using both the process variants at using design configuration 2 of plate mold 2, 80 g/s resin flow rate and laminate layup 2 based on 8 layers of reinforcement. The higher resin flow rate was specially considered as for industrial scale manufacturing of automotive components it's essential to achieve as low injection time as possible to allow use of highly reactive resins to reduce the overall cycle time of the process.

As can be seen in Figure 6.37 using both the HP-RTM process variants the resin was injected into the mold cavity in approximately 9 s. The film gate was located in the middle of the mold which led to resin flow length of approximately 460 mm on either side of the film gate during the resin injection step. As can be seen, during the resin injection step in the HP-IRTM process the pressure on the resin and hardener in the mixing head increased continuously. The pressure changed from an initial value of 130 bar to maximum value of 160 bar for the resin and from an initial value of 126 bar to maximum value of 135 bar in case of the hardener. In case of HP-CRTM process during the resin injection step the pressure on the resin and hardener in the mixing head did not increase significantly and the resin and hardener pressure remained at approximately 130 bar throughout the whole resin injection step. As explained earlier, it shall be noted that this drastic increase in the pressure value of resin from 130 bar to 160 bar in case of HP-IRTM process resulted due resin flow through compressed textiles having low permeability. Whereas due to uncompressed textiles in the HP-CRTM process the permeability of the textiles was not reduced and the resin could flow without any pressure built-up in the mold cavity.

Figure 6.38 shows the effect of resin flow rate on resin pressure change in the HP-IRTM process. These investigations were conducted using design configuration 3 of plate mold 2 and layup 3 based on 8 layers of reinforcement. As can be seen for this new design configuration of plate mold 2 and new layup 3 at 80 g/s resin flow rate the change of resin pressure from an initial value of 125 bar to maximum value of 158 bar was observed. This pressure change is nearly same as in case of HP-IRTM process experiment conducted using design configuration 2 of plate mold 2 and layup 2 at 80 g/s as seen in Figure 6.37 (a) indicating that the pressure change was not affected by laminate layup as in both the experiments constant fiber volume content was targeted to be achieved through use of same fiber areal density and cavity height. As can be seen, the difference between resin pressure before starting injection step and maximum resin pressure during resin injection step

increases as resin flow rate increases from 20 g/s to 100 g/s. It appears that maximum pressure value during the process is the function of selected resin flow rate and maximum pressure value further also can be affected by selected resin viscosity as studied earlier.

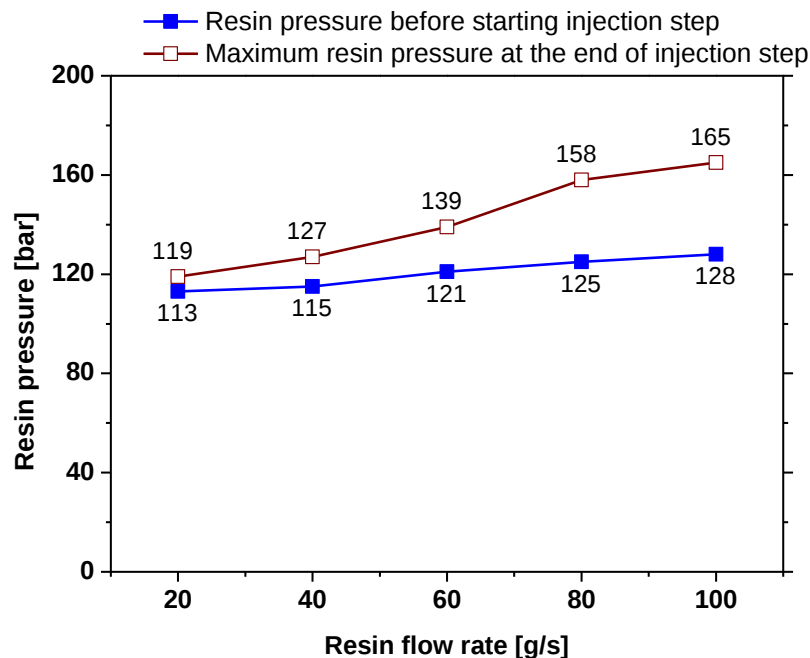


Figure 6.38: Correlation of resin pressure change in HP-IRTM process at different resin flow rates

It shall be noted that in the conducted experiments (Figure 6.37 and Figure 6.38) the resin flow length was only approximately 450 - 460 mm on either side of the film gate. In case of real automotive component manufacturing, e.g. manufacturing of an automotive roof panel or floor structure, the resin shall flow even longer distances in the cavity. In such cases, in order to still implement fast curing resin systems for high-volume manufacturing it is highly essential to inject the resin in short time at even higher flow rate than 100 g/s into mold cavity before reaching the gel point of the resin. In such cases the resin flow through the compacted textiles may still lead to increase of materials pressure in the mixing head thereby not allowing the use of maximum allowed flow rate of the equipment for the quick resin injection into the mold cavity. Also, new generation of resin systems are under development which allow resin curing in 1 to 2 min cure cycle time leading to a need for impregnation of textile reinforcements under high resin throughput rates [107-109]. Further parameters such as used binder concentration in the textile preform and achieved compaction of the preform in a preforming process may lead to further reduction in the permeability of the textiles. If longer flow length shall be achieved in the mold cavity and the textile reinforcement does not have enough permeability then using a small mold gap may help to implement the HP-RTM

process for manufacturing of high performance CFRP based components having larger dimensions. The selection of the process variant for an industrial scale manufacturing will be further determined by the complexity of the component, preform permeability and desired fiber volume content, flow length in the mold cavity and chosen injection gate geometry.

7 Summary and Outlook

Summary

Due to the increasing demand for development of light-weight materials for structural automotive component manufacturing, high-performance composites based on use of glass fibers and especially carbon fibers have gained lots of interest in research and development for new materials and process technologies. This increasing demand provided motivation for investigating the new variants of Resin Transfer Molding (RTM) process for their capability for high-volume manufacturing of fiber reinforced composites.

In order to achieve short resin cure cycle time ($t \leq 5$ min) a variety of fast cure resin systems were characterized within the scope of this thesis. For manufacturing of the structural automotive components it is desirable to achieve high fiber volume content between 50-60 % in the components. During the experimental investigations the manufacturing of glass and carbon fiber reinforced plastics (CFRP) was studied using different resin injection equipment, RTM process variants and mold technologies. In the classical RTM process during the infiltration step as the mold is closed and textile reinforcements are compacted. The compaction of the reinforcement reduces the permeability of the textile and thus it may lead to a problem of longer resin injection time or incomplete resin injection into the mold cavity. The conducted feasibility study showed that using the CRTM process quick resin injection in the mold cavity can be obtained due to higher permeability of the non-compacted textiles and also quick impregnation of the laminates due shorter time required for the compression step. The investigation of the CRTM process however was conducted using a low pressure RTM equipment which led to a resin injection time of 30 - 33 s in the CRTM process for the resin amount of 260 g. It hence indicated the need for robust newly developed process equipment. Thus in later investigations, robust equipment technology based on use of high precision and fast acting compression press, and high-pressure RTM equipment being capable of quick and safe processing of fast cure resin systems were used to study selected RTM process variants for their implementation for industrial scale manufacturing.

The detailed investigations of the HP-CRTM process showed that by using a high-pressure RTM equipment significant reduction of the resin injection time was possible. The impregnation quality of the laminates for a particular laminate layup was strongly affected by chosen mold gap value and resulting effective gap value. The effective mold gap was determined based on selected laminate layup, mold gap value and end laminate thickness. The investigation of mold gap closure for the HP-CRTM process showed that at different end mold gap values differential macroscopic and microscopic impregnation quality was obtained in the laminates which was further affected by the selected laminate layup. As the HP-CRTM process was further investigated using a hat shaped component geometry,

the relevance of gasket systems for avoiding the resin flow out of the mold cavity became more important and it was necessary to select low mold gap values to avoid the resin flow out of the mold cavity during resin injection step. The combination of effects of manual draping on the mold core (lower mold half) and application of compression force on side edge surface of hat shaped components led to rovings displacement in side edges of hat shaped component for a unidirectional laminate layup whereas this effect was not observed for bidirectional laminates. This indicated that while implementing HP-CRTM process for manufacturing complex shaped components is essential to adapt the mold gap, gasket concept and preform quality as per the requirements of the component and mold design concept.

For manufacturing CFRP by the HP-RTM process the use of HP-CRTM process showed additional advantages over HP-IRTM process in terms of obtaining less rovings displacement, achieving shorter resin injection time and high fiber volume content, and manufacturing laminates of homogeneous quality if the point gate geometry was used. During the investigations it was essential to rework on the gate concept to achieve manufacturing of laminates of high fiber volume content up to 55 % by the HP-IRTM process. The study of HP-RTM process variants for manufacturing CFRP showed that the gate concept and the impregnation viscosity of the resin system plays a significant role in optimizing the HP-RTM process for high-volume manufacturing where shorter resin injection time shall be achieved.

Outlook

As mentioned earlier, the selection of the HP-RTM process variant for an industrial scale manufacturing will be further determined by the complexity of the component, preform permeability and desired fiber volume content, flow length in the mold cavity and chosen injection gate geometry. In order to understand the effect of the component geometry on the selection of the HP-RTM process variants different geometries as shown in Figure 7.1 are recommended for further studies.

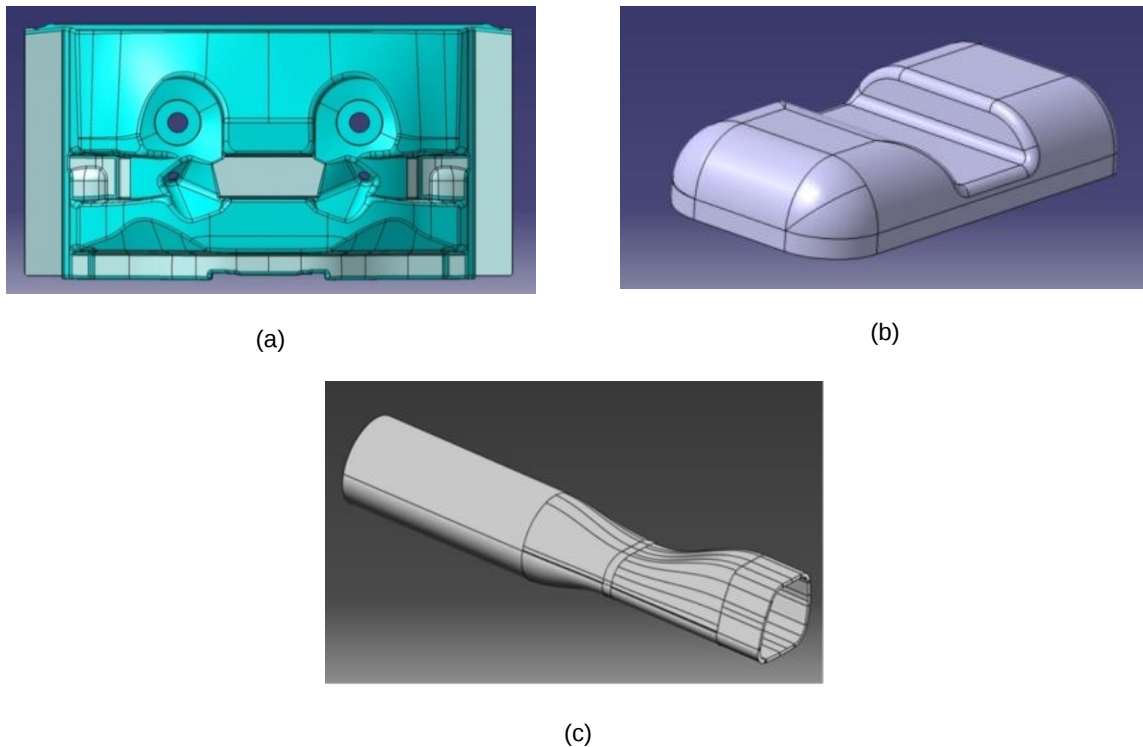


Figure 7.1: Technology demonstrators for investigating high-volume manufacturing capability of HP-RTM process variants; (a) Firewall of an automotive, (b) Complex convex-concave form, (c) hollow generic component of different cross sections [110]

The investigations carried out in this thesis form the basis for mold design, and selection of injection strategy, and optimization of process parameters such as mold gap and resin injection time for manufacturing such components with shorter cycle time. For high-volume manufacturing it is also essential to evaluate and develop automated preform manufacturing process and desired process automation. The correlation of different process steps involved in entire HP-RTM process chain (refer Figure 3.3) shall be investigated and optimized to demonstrate the process for high-volume manufacturing of structural automotive components. Further the relevance of the HP-RTM process variants for manufacturing external automotive panels of class-A surface quality and manufacturing of functions

integrated components (hollow components, sandwich materials etc.) need to be investigated in future for opening new application fields for carbon fiber reinforced composites.

8 References

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9 Annex

9.1 Detailed process parameters

Table 9.1: Detailed process parameters from chapters 6.1 and 6.2

Process and series	Laminate layup	Resin amount	Mold gap	Gap closure speed	Cavity height w/o materials	Preform thickness	Laminate thickness	Effective gap	Injection time
		[g]	[mm]	[mm/s]	mm	[mm]	[mm]	[mm]	[s]
Process parameters from feasibility study for the CRTM process using plate mold 1; Resin and hardener processed at room temperature; Allowed mixing head pressure max 6 bar; Press force 1060 kN									
RTM	[0 ₄]	-	0	-	2.9	4.1	3.0	-0.1	400
CRTM 1	[0 ₄]	260	1	0.2	2.9	4.1	3.0	0.9	30-33
CRTM 2	[0 ₄]	260	2	0.2	2.9	4.1	3.0	1.9	30-33
RTM	[0/90] _s	-	0	-	2.9	4.1	3.0	-0.1	400
CRTM 3	[0/90] _s	260	1	0.2	2.9	4.1	3.0	0.9	30-33
CRTM 4	[0/90] _s	260	2	0.2	2.9	4.1	3.0	1.9	30-33
Process parameters from detailed investigation of the HP-CRTM process for manufacturing glass fiber reinforced composites mold gap study using plate mold 1; Resin processed at 60°C and hardener processed at room temperature; Allowed mixing head pressure 70 bar; Press force 1060 kN									
HP-CRTM 11	[0 ₄]	410	0.5	0.1	2.9	4.1	3.2	0.2	7.5
HP-CRTM 12	[0 ₄]	410	1.0	0.2	2.9	4.1	3.2	0.7	7.5
HP-CRTM 13	[0 ₄]	410	2.0	0.2	2.9	4.1	3.2	1.7	7.5
HP-CRTM 14	[0/90] _s	420	0.5	0.1	2.9	4.1	3.2	0.2	7.5
HP-CRTM 15	[0/90] _s	420	1.0	0.2	2.9	4.1	3.2	0.7	7.5
HP-CRTM 16	[0/90] _s	420	2.0	0.2	2.9	4.1	3.2	1.7	7.5
Process parameters from detailed investigation of the HP-CRTM process for manufacturing glass fiber reinforced composites, mold gap study using plate mold 2; Resin processed at 60°C and hardener processed at room temperature; Allowed mixing head pressure 70 bar; Press force 3100 kN									
HP-CRTM 17	[0/90/0]	670	1.0	0.2	1.65	3.0	2.5	0.2	30
HP-CRTM 18	[0/90/0]	670	2.0	0.2	1.65	3.0	2.5	1.2	30
Process parameters from mold gap closure study for the HP-CRTM process; Resin processed at 60°C and hardener processed at room temperature; Allowed mixing head pressure 70 bar; Press force 1060 kN									
HP-CRTM 31	[0 ₄]	410	2.0	0.2	2.9	4.1	4.7	1.7	7.5
HP-CRTM 32	[0 ₄]	410	1.0	0.2	2.9	4.1	3.8	1.7	7.5
HP-CRTM 33	[0 ₄]	410	0.5	0.2	2.9	4.1	3.4	1.7	7.5
HP-CRTM 34	[0 ₄]	420	0.2	0.2	2.9	4.1	3.1	1.7	7.5
HP-CRTM 35	[0 ₄]	420	0.1	0.2	2.9	4.1	3.2	1.7	7.5
HP-CRTM 41	[0/90] _s	420	2.0	0.2	2.9	4.1	4.7	1.7	7.5
HP-CRTM 42	[0/90] _s	420	1.0	0.2	2.9	4.1	3.8	1.7	7.5
HP-CRTM 43	[0/90] _s	420	0.5	0.2	2.9	4.1	3.4	1.7	7.5
HP-CRTM 44	[0/90] _s	420	0.2	0.2	2.9	4.1	3.2	1.7	7.5
HP-CRTM 45	[0/90] _s	420	0.1	0.2	2.9	4.1	3.2	1.7	7.5
Process parameters from detailed investigation of the HP-CRTM process for manufacturing glass fiber reinforced composites, effect of mold geometry (hat-shaped component); Resin processed at 60°C and hardener processed at room temperature; Allowed mixing head pressure 100 bar; Press force 1060 kN									
HP-CRTM 51	[0 ₄]	410	0.5	0.1	2.9	4.1	3.2	0.2	7.5
HP-CRTM 52	[0/90] _s	420	0.5	0.1	2.9	4.1	3.2	0.2	7.5

Note: Effective gap = (cavity height without materials + mold gap) – (end possible laminate thickness achieved in the process series)

Note: Laminate thickness considered as 3.2 mm for calculating the effective gap and the initial mold gap of 2 mm was used for all the manufactured laminates in the mold gap closure study to calculate the effective gap

Table 9.2: Detailed process parameters from chapter 6.3

Process and series	Laminate layup	Resin amount	Mold gap	Gap closure speed	Cavity height w/o materials	Preform thickness	Laminate thickness	Effective gap	Injection time
		[g]	[mm]	[mm/s]	mm	[mm]	[mm]	[mm]	[s]
Process parameters from characterization of the HP-RTM process variants for manufacturing carbon fiber reinforced composites by using Resin A and point gate; Resin processed at 80°C and hardener processed at room temperature; Allowed mixing head pressure 120 bar; Press force 4200 kN (HP-IRTM process) and 3100 kN (HP-CRTM process)									
HP-IRTM 501	5 L	570	0	0.1	1.65	1.7	1.7	0.0	30
HP-IRTM 505	8 L	670	0	0.2	1.65	2.6	2.4	-0.8	30
HP-CRTM 502	5 L	570	0.3	0.1	1.65	1.7	1.7	0.3	30
HP-CRTM 506	8 L	670	1.0	0.2	1.65	2.6	2.4	0.3	30
Process parameters from characterization of the HP-RTM process variants for manufacturing carbon fiber reinforced composites by using Resin A and film gate; Resin processed at 80°C and hardener processed at room temperature; Allowed mixing head pressure 120 bar; Press force 4200 kN (HP-IRTM process) and 3100 kN (HP-CRTM process)									
HP-IRTM 543	8 L	670	0	0.2	1.65	2.6	2.4	-0.8	30
Process parameters from characterization of the HP-RTM process variants for manufacturing carbon fiber reinforced composites by using Resin B and point gate; Resin processed at 80°C and hardener processed at room temperature; Allowed mixing head pressure 120 bar; Press force 4200 kN (HP-IRTM process) and 3100 kN (HP-CRTM process)									
HP-IRTM 527	5 L	570	0	0.1	1.65	1.7	1.7	0.0	30
HP-CRTM 526	5 L	570	0.3	0.1	1.65	1.7	1.7	0.3	30

9.2 Sample preparation plan and test specifications

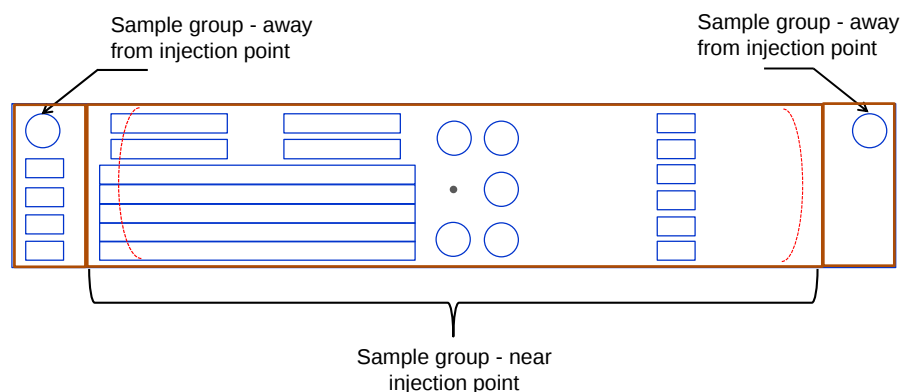


Figure 9.1: Sample preparation plan for characterization of CRTM laminates in the process feasibility study; dotted red line indicates the resin flow front progression after resin injection and before compression at 2 mm mold gap; (chapter 6.1); detailed sample dimensions are given in table 9.3

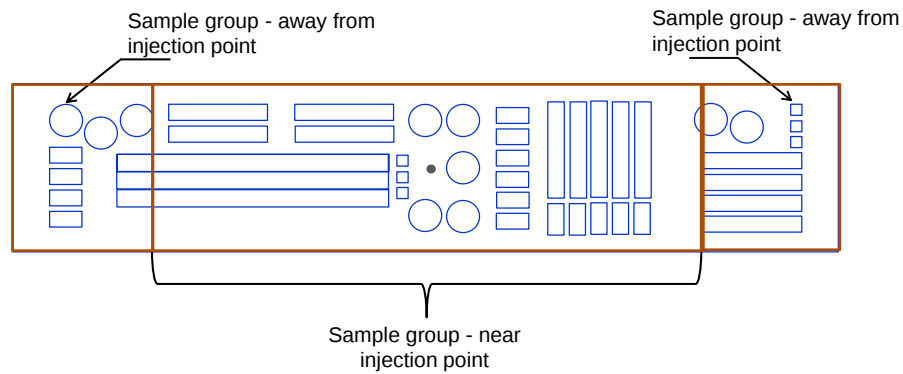


Figure 9.2: Sample preparation plan for characterization of HP-CRTM laminates manufactured using different mold gaps and plate mold 1; (chapter 6.2.1.1 and chapter 6.2.1.2); detailed sample dimensions are given in table 9.3

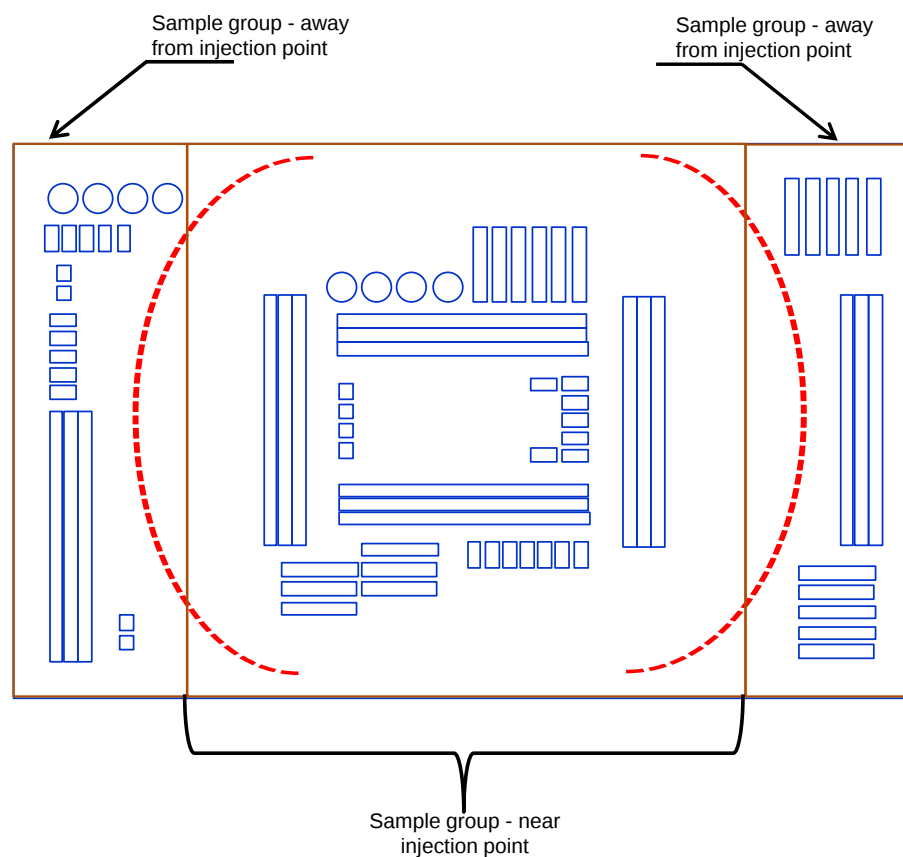
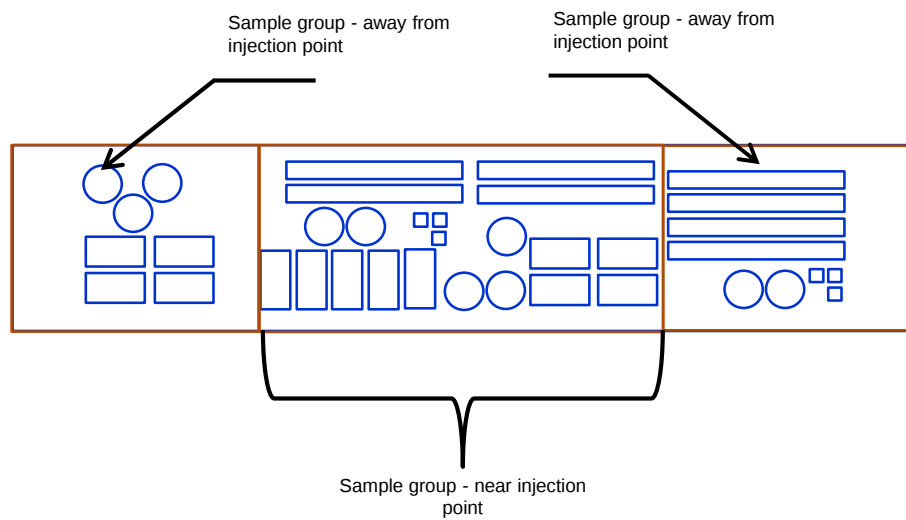
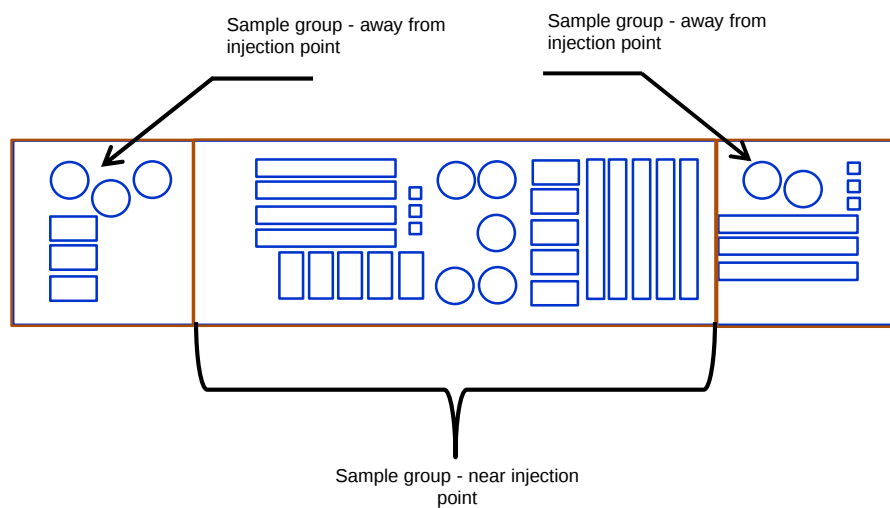


Figure 9.3: Sample preparation plan for characterization of HP-CRTM laminates manufactured using different mold gaps and plate mold 2; (chapter 6.2.1.3 and chapter 6.2.1.4); detailed sample dimensions are given in table 9.3



(a)



(b)

Figure 9.4: Sample preparation plan for characterization of the laminates manufactured by HP-CRTM process in the mold gap closure study; sample plan for (a) laminates with approx. 5 mm thickness, (b) laminates with approx. 4 mm and 3.5 mm thickness; detailed sample dimensions are given in table 9.3

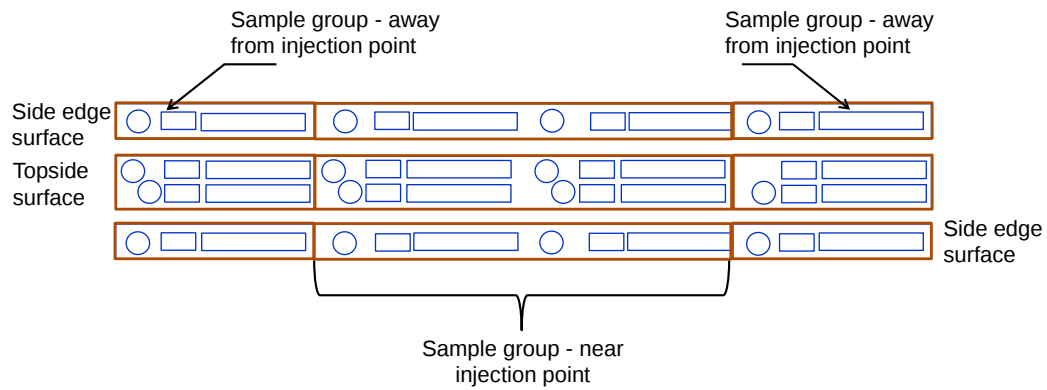


Figure 9.5: Sample preparation plan for characterization of cap-shaped components manufactured by the HP-CRTM process; detailed sample dimensions are given in table 9.3

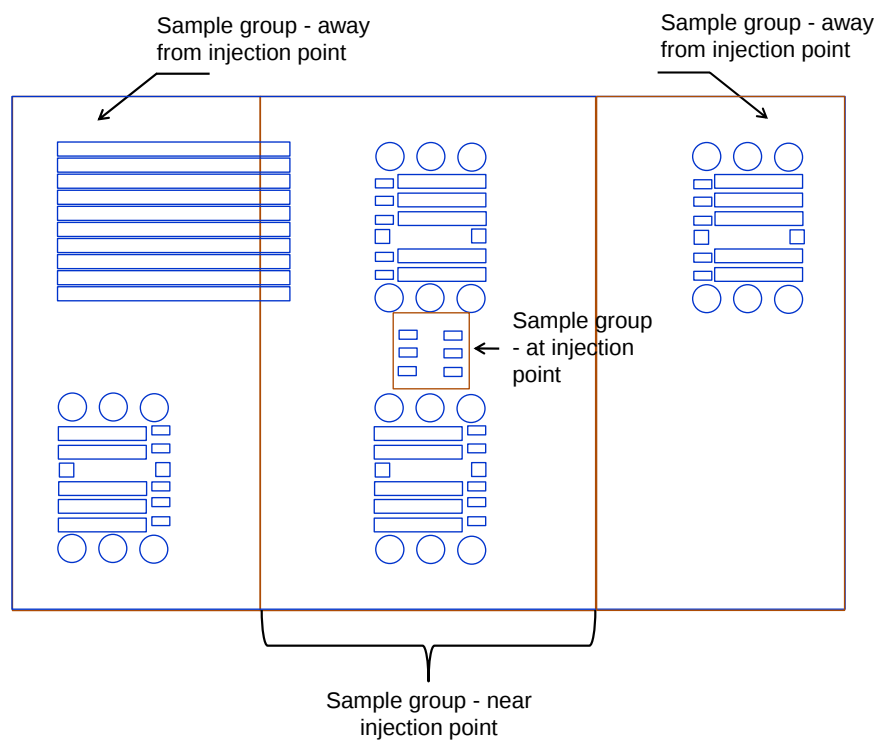


Figure 9.6: Sample preparation plan for characterization of laminates manufactured by HP-RTM process variants using carbon fiber reinforcements; detailed sample dimensions are given in table 9.4

Table 9.3: Test parameters from characterization of CRTM laminates and HP-CRTM laminates manufactured using glass fiber reinforcements

Test method / Norm	Sample thicknesse	Sample geometry		Measurement length / Support length	Test parameters
		Rectangular samples			
	[mm]	length (mm)	width [mm]	L (mm)	
Feasibility study - plate mold 1					
Tensile testing / DIN EN ISO 527	3	250	15	100	2 mm/min
Flexural testing / DIN EN ISO 14125	3	90	15	60	2 mm/min
Inter laminar shear strength (ILSS) / DIN EN ISO 14130	3	30	15	15	1 mm/min
Fiber vol. content measurement	3	Circular samples - diameter 30 mm		-	600°C for 90 min
Effect of mold gap on laminates manufactured by HP-CRTM process - plate mold 1					
Tensile testing / DIN EN ISO 527	3.2	250	15	100	2 mm/min
Flexural testing / DIN EN ISO 14125	3.2	90	15	64	2 mm/min
Inter laminar shear strength (ILSS) / DIN EN ISO 14130	3.2	30	15	15	1 mm/min
Fiber vol. content measurement	3.2	Circular samples - diameter 30 mm		-	600°C for 90 min
Morphology test sample	3.2	10	10	-	-
Effect of mold gap on laminates manufactured by HP-CRTM process - plate mold 2					
Tensile testing / DIN EN ISO 527	2.5	250	15	100	2 mm/min
Flexural testing / DIN EN ISO 14125	2.5	75	15	52	2 mm/min
Inter laminar shear strength (ILSS) / DIN EN ISO 14130	2.5	25.5	12.75	13	1 mm/min
Fiber vol. content measurement	2.5	Circular samples - diameter 30 mm		-	600°C for 90 min
Morphology test sample	2.5	15	15	-	-
Mold gap closure study - plate mold 1					
Flexural testing / DIN EN ISO 14125	4.8	150	15	100	2 mm/min
	3.8	120	15	76	2 mm/min
	3.4	105	15	64	2 mm/min
	3.0 - 3.2	90	15	64	2 mm/min
Inter laminar shear strength (ILSS) / DIN EN ISO 14130	5.0	50	25	25	1 mm/min
	4.0	40	20	20	1 mm/min
	3.5	35	17.5	18	1 mm/min
	3.0 - 3.2	30	15	15	1 mm/min
Fiber vol. content measurement	3.0 - 5.0	Circular samples - diameter 30 mm		-	600°C for 90 min
Morphology test sample	3.0 - 4.8	10	10	-	-

Table 9.4: Test parameters from characterization of HP-RTM laminates manufactured using carbon fiber reinforcements

Test method / Norm	Sample thicknesse	Sample geometry		Measurement length / Support length	Test parameters
		Rectangular samples			
	[mm]	length (mm)	width [mm]	L (mm)	
Laminates manufactured using 5 layers of textile reinforcement and plate mold 2					
Tensile testing / DIN EN ISO 527	1.9	250	15	100	2 mm/min
Flexural testing / DIN EN ISO 14125	1.9	95	15	76	2 mm/min
Inter laminar shear strength (ILSS) / DIN EN ISO 14130	1.9	19	9.5	9.5	1 mm/min
Fiber vol. content measurement	1.9	Circular samples - diameter 30 mm		-	600°C for 90 min
Laminates manufactured using 8 layers of textile reinforcement and plate mold 2					
Tensile testing / DIN EN ISO 527	2.4	250	15	100	2 mm/min
Flexural testing / DIN EN ISO 14125	2.4	120	15	96	2 mm/min
Inter laminar shear strength (ILSS) / DIN EN ISO 14130	2.4	24	12	12	1 mm/min
Fiber vol. content measurement	2.4	Circular samples - diameter 30 mm		-	600°C for 90 min
Morphology test samples for all types of laminates	1.9 - 2.4	15	15	-	-

9.3 Test parameters for studying the curing reaction of resins

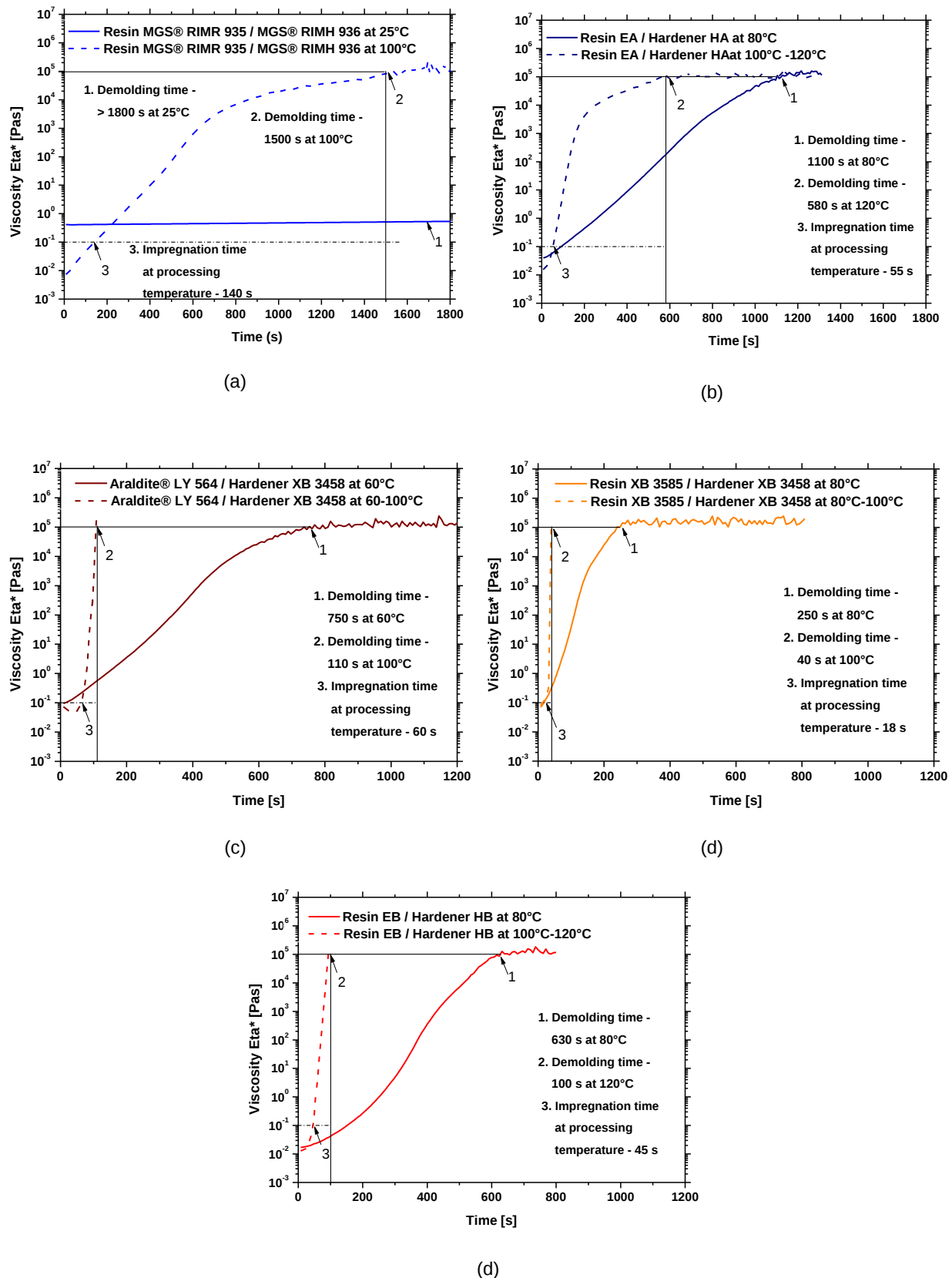


Figure 9.7 Processing properties of different epoxy resins

(a) EPIKOTE™ Resin MGS® RIMR 935 / EPIKURE™ MGS® RIMH 936: Injected at RT into the mold; cured at 100 °C mold temperature

(b) Resin A: Injected at 80°C into the mold; cured at 120 °C mold temperature

(c) Araldite® LY 564 / Hardener XB 3458: Injected at 60 °C into the mold; cured at 100 °C mold temperature

(d) Resin XB 3585 / Hardener XB 3458: Injected at 80 °C into the mold; cured at 100 °C mold temperature

(e) Resin B: Injected at 80 °C into the mold; cured at 120 °C mold temperature

Chemo-rheology test parameters

Plate-plate geometry with 40 mm diameter; Sample thickness 1 mm;

Sample amount approximately 2.5 g (Epoxy resin and hardener)

Dynamic time sweep test

Frequency: 80 rad/s

Strain: 80 %; Strain adjustment: 30 %

Maximum allowed torque: 0.002 Nm; Minimum allowed torque: 2.5 E-06 Nm

Dynamic temperature ramp test

Start temperature: defined as per resin

Max allowed temperature: defined as per resin

Heating rate: 80 °C/min (however maximum heating rate of the machine was around 50 °C/min -60 °C/min)

Frequency: 80 rad/s

Strain: 80 %; Strain adjustment: 30 %

Maximum allowed torque: 0.002 Nm; Minimum allowed torque: 2.5 E-06 Nm

9.4 Reproducibility of void content analysis

Table 9.5: Void content measurement in unidirectional glass fiber reinforced samples manufactured in chapter 6.2.1.1

Laminate layup	Mold gap	Sample position	Sample cross section area mm ²	Number of pores / voids	Sum area of pores mm ²	Void content %	Average void content
Unidirectional [0 ₄]	0.5 mm	Near injection point (NIP)	30.52	1	0.001	0.003	0.011
			31.52	30	0.004	0.014	
			35.47	31	0.005	0.015	
		Away from injection point (AIP)	31.40	45	0.022	0.070	0.027
			32.12	16	0.003	0.009	
			30.23	5	0.000	0.001	
	1 mm	Near injection point (NIP)	31.165	8	0.004	0.013	0.009
			31.56	6	0.002	0.007	
			31.50	6	0.002	0.006	
		Away from injection point (AIP)	31.949	15	0.006	0.019	0.006
			31.442	19	0.002	0.000	
			31.691	2	0.000	0.001	
	2 mm	Near injection point (NIP)	32.176	4	0.002	0.006	0.020
			30.84	3	0.015	0.050	
			31.65	5	0.002	0.005	
		Away from injection point (AIP)	32.423	52	0.026	0.080	0.134
			31.73	61	0.054	0.171	
			31.74	144	0.048	0.150	

Personal Information:

Name	Raman Chaudhari
Date of birth	02.07.1982, Bhusawal, India
Nationality	Indian
Marital status	Married

Professional Designation:

Since 04/2011	Team Leader - High Performance Composites at Fraunhofer Institute for Chemical Technology (ICT)
Since 09/2010	Research employee at Institute of Vehicle System Technology (FAST), Karlsruhe Institute of Technology (KIT); collaterally agreed for maintaining working place at Fraunhofer ICT
Since 04/2009	Part time research employee at Fraunhofer ICT
09/2007 - 08/2010	Research employee at Institute of Materials Science and Engineering (<i>iwk I</i>), Karlsruhe Institute of Technology (KIT); collaterally agreed for maintaining working place at Fraunhofer ICT
05/2007 - 08/2007	Research employee at Fraunhofer ICT
11/2006 - 04/2007	Post graduate assistant at Fraunhofer ICT

Educational Qualifications:

04/2004-09/2006	Master of Science (M.Sc.) Master of Applied Polymer Science from Martin-Luther-University, Halle-Wittenberg, Germany
07/1999-06/2003	Bachelor of Engineering (B.E.) Bachelor of Polymer Engineering from Pune University, India

The current developments in the Resin Transfer Molding (RTM) process for the automotive industry are strongly driven by the need for automotive light-weight design and high volume manufacturing capacity. In order to adapt the RTM process for industrial scale manufacturing of the automotive components it is essential to implement newly developed fast curing resin systems. If such resin systems shall be implemented in the RTM process, then it raises the necessity to achieve shorter resin injection time in this process. For serving this purpose, modern equipment technology such as precise compression press and high pressure RTM equipment were used for all the scientific and technological investigations in this particular thesis.

The scope of investigations included studying different high pressure RTM (HP RTM) process variants to understand their effects on the manufacturing of high performance composites. The important parameters such as mold gap created during the resin injection step, laminate layup, resin viscosity, resin injection rates and mold geometry were investigated through several process studies. Through the achieved results a basis has been formed for the industrialization of the HP RTM process chain.

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Herausgeber:
Fraunhofer-Institut für Chemische Technologie ICT
Joseph-von-Fraunhofer-Straße 7
76327 Pfinztal (Berghausen)
Telefon +49 721 4640-0
Telefax +49 721 4640-111
info@ict.fraunhofer.de
www.ict.fraunhofer.de

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