CHARACTERIZATION OF HIGH PRESSURE RTM PROCESSES FOR MANUFACTURING OF HIGH PERFORMANCE COMPOSITES

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Abstract
The current paper addresses new variants of the RTM process namely High Pressure-Injection Resin Transfer Molding (HP-IRTM) and High Pressure - Compression Resin Transfer Molding (HP-CRTM) for manufacturing of continuous fiber reinforced composites with high fiber volume content. Both these process utilize High Pressure RTM equipment for precise dosing and mixing of highly reactive epoxy resin and amine hardener with relatively high throughput rates. The objective of the proposed study was to investigate the effects of parameters such as mold gap and resin injection time on the quality of the respective process variants. The influence of these process variables on the quality of the laminates and the mechanical properties is analyzed. Finally the applicability of the HP-RTM process variants for high volume manufacturing of composites is discussed.

1 Introduction
1.1 Classical RTM process
The resin transfer molding (RTM) process is well established for low volume manufacturing applications and has recently gained great interest in the manufacturing of higher volumes. This interest is strongly motivated by the need of light-weighting for the automotive and aerospace industries. 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 equipments. These issues hinder the use of RTM process for high volume manufacturing of the automotive components. In the classical RTM process the three dimensional preform is placed into the cavity and the mold is completely closed. The mold closure leads to compaction of the preform to the final part thickness thereby leading to reduction in the preform permeability. The reactive resin (premixed theromset resin and hardener) is then injected into the cavity under pressure which is typically 1 to 20 bar [1]. The resin flows along the length of the preform and impregnates the fibers. Ideally, once the impregnation process is completed the resin undergoes a curing reaction and after a defined time interval, the part can be demolded. The complete closure of the mold prior to the resin injection leads to reduction in the permeability of the preform, hence resulting in longer resin injection and preform impregnation time. Longer impregnation time in the classical RTM process does not allow use of fast curing resins for high volume manufacturing of the RTM components. An alternative approach to achieve fast resin injection into the mold cavity is to use High Pressure RTM equipment which allows injecting the resin under high throughput...
rate into the cavity. Using such a processing equipment two different variants of the RTM process namely High Pressure-Injection Resin Transfer Molding (HP IRTM) and High Pressure - Compression Resin Transfer Molding (HP CRTM) can be implemented for the manufacturing of continuous fiber reinforced composites.

1.2 HP-IRTM process
The HP-IRTM process differs from the classical RTM process only in terms of the processing equipment for the resin system. In case of classical RTM process low pressure mixing and dosing equipment is used for processing of resins where as in the HP-IRTM process resin mixing and dosing is carried out using a High Pressure RTM equipment. Similar to the classical RTM process, 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. Once the mold is closed, resin and hardener mixture is injected into the cavity under high injection speed. The high throughput rate results into fast filling of the cavity and hence the resin injection time can be reduced significantly. As the resin can be injected into the cavity in shorter time, this process variant allows to use resins with relatively higher reactivity. After resin curing reaction is completed the part can be demolded [2, 3].

![Figure 1. Resin injection sequence in the High Pressure - Injection RTM (HP-IRTM) process](image1)

1.3 HP-CRTM process
The HP-CRTM process is a combination of resin transfer molding (RTM) and compression molding in which similar to HP-IRTM process High Pressure RTM equipment is used for processing of resins.

![Figure 2. Resin injection sequence in the High Pressure - Compression RTM (HP-CRTM) process](image2)

Often the process, if conducted without use of High Pressure RTM equipment, is referred in the literature as Compression RTM process. In this process, the preform is placed into the mold cavity and then the mold is closed partially to obtain a small gap between the mold surface and the fiber preform. The resin is introduced into the gap and flows easily over the
preform and may partially impregnate the preform as well. Once the required amount of resin is injected into the gap and the injection point is closed, the mold closes further and applies high 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 [4].

1.4 Parameters Affecting the HP-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 by the Compression RTM (CRTM) process. Kang et al. has attempted to analyze the CRTM process by studying the experimental and numerical data. Experiments were performed using a vinylester resin and complicated three dimensional preform based on continuous randomly oriented glass fiber mats. The resin was injected into the cavity in 30 s and had a gel time of approx. 9 min. The achieved fiber volume content in the CRTM parts was approximately 17%. The varied process parameters were compression force and compression speed. A close agreement was found between the experimental and numerical data by Kang et al [5]. The effect of mold gap and gap closure speed on the void content and flexural properties of the CRTM laminates was investigated by Ikegawa et al. However the used compression pressure and the cavity pressure were significantly low and varied between 1 to 13 bar. The fiber volume content in the produced laminates was 33% [6]. Chang et al. investigated the effect of injection pressure, mold gap, resin temperature, compression pressure, mold temperature and curing temperature on the CRTM process. Experiments were performed using epoxy resin and bidirectional woven glass fabric and obtained fiber volume content in the laminates was approximately 40%. However the used compression pressure was once again very low and it was varied between only 1 to 2 bar [7]. The reactivity of the chosen resins was, however, significantly low in most of these studies. In case of HP-IRTM process only limited literature is available. Kacaras et al. has investigated the effect of two different epoxy resins and resin injection time on the HP-IRTM and HP-CRTM processes. Mainly the resin injection time was varied between 30 s to 11 s for these investigations. It was found out that the faster injection of resin into the cavity lead to poor impregnation quality of the HP-RTM laminates for one resin system whereas equivalent impregnation quality was obtained for second resin system at all resin injection times [8]. The goal of the current study was to use fast curing resin system to evaluate the effect of below defined process parameters on the HP-RTM laminate properties, as well as to demonstrate high volume production capability of the HP-RTM process variants.

2 Materials
For the characterization of the HP-RTM process variants, an epoxy resin with trade name Resin XB 3585 was chosen. An amine based curing agent with trade name Hardener XB 3458 was used to cure the selected epoxy resin. As reinforcement UD non woven E-glass fabric from SAERTEX GmbH & Co. KG was used. The commercial product number of the chosen glass fabric was S14EU960-01210-01300-487000 and was available with multi-compatible sizing. The used UD non woven glass fabric had a surface area weight of 1218 g/m² with 95% fibers in 0° orientation and 4.5% fibers in 90° orientation as well as additional 0.5% PES based stitching fibers.
3 Processing equipments
A specially designed RTM mold with cavity size 910 mm x 560 mm x 2 mm and an obligatory shear edge was used for the HP-RTM process studies. The mold had an injection point in the middle of the mold from the lower side. Additionally, the mold facilitated two points on either side of the mold for applying vacuum during resin injection or as an exit port for the extra amount of the injected resin. However vacuum was not used during the manufacturing of HP-RTM laminates in the addressed process studies. A hydraulic compression press from the company Dieffenbacher GmbH (press type - DYL630/500) was used to mount the RTM mold as well as to precisely apply defined compression force and to control the mold gap and gap closure speed during the HP-CRTM process study. For the mixing of the chosen epoxy resin and curing agent and for injecting the reactive resin into the mold cavity HP-RTM, equipment from the company KraussMaffei Technologies GmbH was used.

4 Process Parameter Matrix for HP RTM process studies
Table 1 summarizes the process parameters used for studying the HP-RTM process variants. For the investigation of the HP-IRTM process and HP-CRTM process resin injection times of 30 s and 7.5 s were used. For manufacturing of laminates by HP-IRTM process the mold was completely closed using a press force of 4200 kN and resin amount of 690 g (pre-calculated for obtaining complete impregnation of the preform) was injected into the cavity at defined injection times. In case of the HP-CRTM process a mold gap of 1 mm was used for manufacturing the laminates. The resin amount of 690 g was injected into the cavity while the mold was closed at a mold gap of 1 mm and after the resin injection was completed the mold was further closed by applying a press force 3100 kN and 0.2 mm/s gap closure speed to obtain final laminate. The used laminate layup for all process parameter studies was 0/90/0. The epoxy resin was preheated to 80°C to obtain low viscosity whereas the curing agent was maintained at room temperature. After resin injection was completed, the laminates were cured for 240 s and afterwards the laminates were demolded.

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<td>[0/90/0]</td>
<td>80</td>
<td>690</td>
<td>4200</td>
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<td>-</td>
<td>30</td>
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<tr>
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<td>690</td>
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<td>HP-CRTM 1</td>
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<td>HP-CRTM 2</td>
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Table 1. Process parameter matrix used for characterization of HP-RTM process variants

5 Materials characterization
The manufactured laminates were characterized using following norms:
- DIN EN ISO 7822 for characterization of the fiber volume content
- DIN EN ISO 527-5 for characterization of the tensile properties
- DIN EN ISO 14125 for characterization of the flexural properties
- DIN EN ISO 14130 for characterization of the inter-laminar shear strength (ILSS)
6 Results and discussions

6.1 Fiber volume content in the HP RTM Laminates

Figure 3 (a) shows the effect of the HP-RTM process variants on the final part thickness and fiber volume content in the manufactured laminates in correlation to the used resin injection time. As it can be observed, if shorter resin injection time was used for HP-IRTM and HP-CRTM process variants then it resulted in laminates with thickness of 2.4 mm against laminate thickness of 2.5 - 2.6 mm if resin injection time of 30 s was used for the same process variants. The changes in laminate thickness resulted in variation of fiber volume content in the range of 53% to 59%. Figure 3 (b) shows the fiber volume in the laminates normalized to a part thickness of 2.5 mm. The normalization of the fiber volume content results in nearly equivalent fiber volume content in all the laminates indicating that the variation in the experimentally measured fiber volume content is only due to variation in the part thickness.

Figure 3. Part thickness and fiber volume content of HP-RTM laminates (a) Experimentally measured properties from laminates after manufacturing, (b) Normalized properties to 2.5 mm part thickness

6.2 Mechanical properties of the HP-RTM Laminates

One of the goals of the current study was to investigate the effect of the HP-RTM process variants and used respective resin injection time on the quality of the HP-RTM laminates. As during the study the resin injection time was changed from 30 s to 7.5 s (reduction by a factor of 4) the tensile properties of the laminates were measured to find out occurrence of fiber washout / fiber displacement effects. As the occurrence of fiber washout / fiber displacement generally would result in the change of fiber orientation, it may cause variations in the tensile properties. Figure 4 shows the tensile strength and tensile modulus of HP-RTM laminates. The shown properties are normalized to 2.5 mm part thickness. As it can be seen the tensile strength and modulus of all the laminates are almost constant. This indicates that the selection of the HP RTM process variant and reduction of resin injection time from 30 s to 7.5 s did not
affect the fiber orientation of the laminates thereby resulting in no fluctuations in the properties. In order to investigate the effect of process variants and respective resin injection time on the impregnation quality of the laminates flexural properties and inter-laminar shear strength (ILSS) of the laminates were measured.

![Figure 4. Tensile properties of HP-RTM laminates (normalized to part thickness of 2.5 mm)](image)

Figure 5 shows the flexural properties of the HP-RTM laminates. Similar to tensile properties almost identical flexural strength values were measured for the HP-RTM laminates. The flexural modulus of laminates manufactured using HP-CRTM process and 30 s resin injection time exhibited highest modulus value of 37 GPa whereas rest of the HP-RTM laminates exhibited flexural modulus of 33 GPa.

![Figure 5. Flexural properties of HP-RTM laminates (normalized to part thickness of 2.5 mm)](image)
Figure 6. ILSS properties of HP-RTM laminates (normalized to part thickness of 2.5 mm)

Figure 6 shows the ILSS properties of the HP-RTM laminates. The ILSS properties where measured in the lengthwise and crosswise orientation to the mold length i.e. lengthwise and crosswise to the laminate layup of [0/90/0]. Similar to the tensile and flexural properties, the ILSS properties of the HP-RTM laminates were not influenced by the selected process variant and resin injection time. As the ILSS properties were not affected by the process variant as well as resin injection time, nearly equivalent impregnation quality in the laminates is expected. Further process properties such as selection of the process variant and resin injection time on the cavity pressure built-up will be presented at the conference for the addressed study.

7 Summary
In the current study two different HP-RTM process variants, namely High Pressure - Compression RTM (HP-CRTM) and High Pressure - Injection RTM (HP-IRTM) were investigated. The main goal of these investigations was to evaluate the influence of the process variants and selected resin injection time for the respective process variants on the quality of the HP-RTM laminates. The reduction of resin injection time from 30 s to 7.5 s resulted in slight reduction in the laminate thickness manufactured using both the process variants thereby leading to strong deviations in the fiber volume content of the laminates. The mechanical properties of the manufactured laminates were however almost constant and selection of particular process variant and reduction of resin injection time did not show any negative influence on the laminate mechanical properties. Based on the conducted investigations it can be concluded that both the HP-RTM process variants are suitable for reducing the resin injection time significantly and hence both the process variants are capable of utilizing fast curing resins for high volume manufacturing of the RTM components.

8 Outlook
Further investigations are ongoing at Fraunhofer ICT to investigate the effect of the process parameters such as the effect of resin systems, resin viscosities, fiber type, fabric architecture...
on the quality of the laminates manufactured by using the HP-RTM process variants. Simultaneously investigations are ongoing at Fraunhofer ICT to setup an automated and industrial scale HP-RTM process setup to demonstrate the HP-RTM process variants for manufacturing complex shape components.

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