Tooling Concepts for Quality Improvement of Composite Panels

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by

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Summary

The scope of this thesis is to develop new tooling concepts and production techniques to improve the quality of composite skin panels. The research focuses on closed mould manufacturing techniques using solid-solid and solid-flexible tool sets. Subsequently the project introduces prepreg/resin infusion concept as an additional way to reduce manufacturing defects like voids and porosities in the composite panels. The closed mould tool setup was optimized with repeatable tests for both tooling sets. The composite laminates produced had no visible defects like fiber flow-out and wrinkles on the surface. The panels produced with both tooling sets were mechanically tested for inter laminar shear strength and the cross-sections were analyzed under optical microscope to determine the void content. The prepreg/resin infusion technique was used with the developed mould setup as an attempt to eliminate voids in the produced laminates. However, the attempt was not successful due to different processing temperatures of liquid resin and prepreg material used in the project. Therefore, it is recommended to use both prepreg and liquid resin with similar chemical and processing conditions. Furthermore, it is advised more research has to be conducted for improving infusion concepts with the closed mould tool setup used in this project.

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Introduction

The composite industry has made significant advancement over the years in terms of new materials and processing techniques. The major beneficiaries of these advancements are the automotive and aerospace industries. Manufacturing of structural components made of fiber composites can produce cost savings as a result of reduced weight, lower maintenance effort, thereby reducing overall manufacturing costs. Due to a high strength to weight ratio, high specific stiffness and non-corrosive behaviour, the carbon fiber lend itself for applications in airframe structures. Stiffened composite panels are manufactured using moulding processes like co-curing, co-bonding and secondary bonding [10,21,27]. In the co-curing technique the prepreg skin and stiffener are cured simultaneously as shown in Figure 1.1 [14]. This offers advantages in terms of cost reduction due to less material usage and a simpler production technique [10].

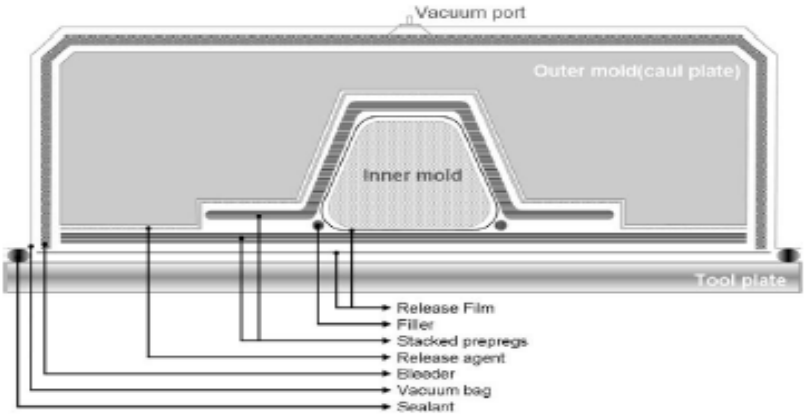


Figure 1.1: Schematic representation of co-curing process

Co-curing production techniques often cause defects in composite panels like porosity, variation in thickness and fiber waviness. This leads to rework to remove the defects, thereby increasing the overall manufacturing costs. Another area for the origin of defects in composite panels is the tooling used during manufacturing. Some of the defects related to tooling as investigated by researchers are in and out-of-plane undulations, wrinkles on the skin etc which can be caused by thermal mismatch between the tool and the composite material or misalignment of a tool or material during the manufacturing process. To reduce or eliminate these defects in composite panels, new manufacturing and tooling concepts for Carbon Fiber Reinforced Polymer (CFRP) are being proliferated. A possible solution is to improve the tooling by using tool materials with comparable thermal coefficients to the composite materials. Another solution is to combine the existing manufacturing techniques like dry fiber/prepreg/resin infusion or prepreg/resin infusion in a single process as is investigated by Kaps and Wiedemann [25]. Veronique Carlier *et al.* [5] also describes a similar approach by injecting pressurized resin in a closed mould with pre-impregnated fiber pre-form to reduce/eliminate any gas bubbles originating during curing of the resin.

In this research two different tooling concepts: solid-solid and solid-flexible in a closed mould system will be investigated. Along with the tooling, the use of pressurized resin with prepreg as a manufacturing technique to improve the quality of the composite panel will also be researched. Therefore, the project puts forward the main research question as: *How does the use of prepreg/resin infusion process with different tooling concepts contribute to the elimination of manufacturing defects and making the fabrication process more economical?*

This question can be answered by investigating the following focal points:

- Investigating the effect of different process parameters on the quality of composite laminate.
- Studying the effect of using different tooling concepts on the quality of composite laminate.
- Investigating Prepreg/Resin Infusion process.

For the first sub-question the prepreg material is tested under different process conditions to find out the optimum scenario that can be utilized for testing in this project. The material is tested for two different layup sequences: 0-90 and unidirectional, two cure cycles and two manufacturing process: autoclave curing and out-of-autoclave (hot press) curing. From this analysis one case will be selected which can be used in the second phase for further improvement.

To answer the second sub-question an experimental setup (closed mould) is prepared with two tooling concepts: solid-solid and solid-flexible configuration. To optimize the setup repeated tests are conducted with both tooling configurations. The specimens manufactured from these setups are inspected visually for any surface defects and cross-sections are observed under optical microscope.

Investigation of prepreg/resin infusion forms the third phase of the project. Rheological tests are performed to understand the compatibility of the prepreg resin and liquid epoxy resin. This is followed by the actual infusion trial with both the tooling concepts.

To start off the project, a literature study was done to give an overview of the different manufacturing techniques and tooling concepts that are adopted by industries and researchers over the years. This would form a firm background for finding a new concept for manufacturing. The literature study is summarized in Chapter 2. Before starting the actual tests, preliminary tests are conducted on the material used in the project. The results are analyzed and described in Chapter 3. Chapter 4 gives the evolution of the closed mould tool setup with product quality . Chapter 5 describes the tests conducted with the new tool setup. The laminates are inspected for the laminate quality, the changes made to the setup to improve the defects and the inspection tests done on the laminates. Chapter 6 describes the third phase of the project explaining the prepreg/resin infusion test. The project is concluded in chapter 7 followed by recommendations for future work.

2

Literature Study

An extensive literature study was performed for preparation of this project. This section provides a brief review of the actual study done. The objective is to get a basic understanding of the thermoset manufacturing concepts on which this thesis project is based and it forms a background for implementing integrated technology. It is necessary to know how the processes work individually and how the industry uses them for fabricating Carbon Fiber Reinforced Polymer (CFRP) components. The research is divided in two sections, standard manufacturing techniques and tooling concepts.

2.1. Standard Manufacturing Processes for CFRP Panels

2.1.1. Hand Lay-up

The most basic manufacturing technique for a composite part is hand layup. This method typically consists of laying up cut fibers on the mould and then impregnating the layers with resin by using a brush or a spatula. The laminate stack is vacuum bagged to remove any entrapped air and is cured in the oven. As the process is labor intensive, this technique is used to manufacture prototypes or small series production. A disadvantage of using this process is the porosity in the material, since the process depends on the expertise of the technician[4].

2.1.2. Pultrusion

Similar to the extrusion of aluminum to produce long profiles with constant cross section, there is a Fiber Reinforced Plastic (FRP) manufacturing process called pultrusion. CFRP profiles, such as T-stringers, or hat-stiffeners used as aircraft parts or sub-assemblies can be fabricated using this process. The fibers are shaped prior to the impregnation with resin. In contrast to the extrusion process, where the material is pushed through the tool under high pressure and temperature, pultrusion is characterized by pulling the impregnated fibers through a heated cavity to produce the desired geometry[23], as shown in 2.1

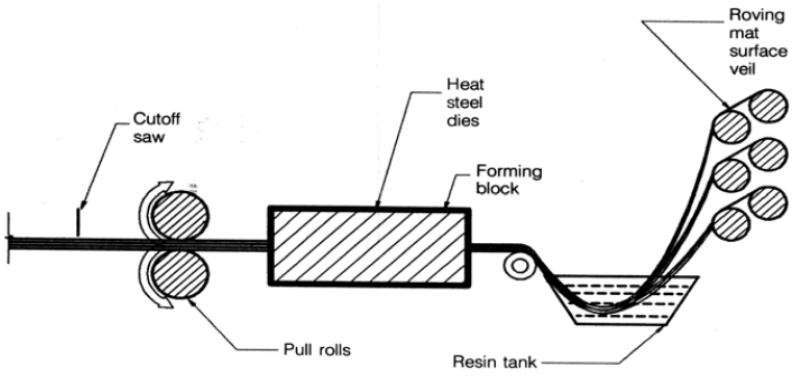


Figure 2.1: Basic pultrusion process

Pultrusion is a very efficient and economical means of producing long length, constant cross section parts. The cured section emerging from the die is grasped, pulling the pre-cured profile through, at a constant rate. After leaving the die the part is trimmed to specified length and often post cured. A disadvantage of with this technique is the inability to fabricate parts with varying cross-section, which limits its application.

2.1.3. Prepreg Based Processes

Most CFRP aircraft parts nowadays are based on prepregs. Prepregs are matrix materials that are already impregnated with a sufficient amount of partially cured resin (B-stage prepreg). The prepreg material is typically stored at cold temperatures around –18 °C. The sealed material is brought to room temperature to avoid condensation and absorption of moisture and unpacked for cutting to desired shape. The material is draped in the mould manually, vacuum bagged and cured at high temperature and pressure in the autoclave.

For large sized parts like wing skins, manually draping the prepreg becomes time consuming and labour intensive. In this case automated tape laying (ATL) technology is used. The process uses impregnated unidirectional tape which is carried by the laying head of the machine[2]. Although this process is preferred for straight profiles or mildly curved profiles like skin panels, but with modern ATL machines it can be used for complex geometries like U-shaped spars.

The biggest advantage of prepreg material is the ease of handling and manufacturing is less time consuming. Whereas using dry fibers requires close monitoring of void content and is time consuming because of resin infusion in the later stages.

2.2. Infusion Technologies

A more common manufacturing method for high performance CFRP parts is resin-infusion, which involves impregnating the dry fibers with resin after positioning and draping the fibers in the mould. The resin is forced in the fibers because of vacuum inside the closed part and due to pressure on the resin system. Resin infusion technologies are known since the 1960s, when the RTM process was introduced. These technologies are adapted to manufacture diverse structural components. It is widely used to fabricate wind mill blades, with a length of up to 35 m and beyond.

2.2.1. Resin Transfer Moulding (RTM)

Resin Transfer Moulding (RTM) is a fairly simple process. The process typically consists of matched closed mould often made of metal. The dry preform is placed inside the mould cavity. The mixed resin is injected in the mould at moderate pressure (6 bar) through injection ports (figure 2.2). To assure proper impregnation, the viscosity of the resin should be low, which is mostly achieved by warming the resin. The positioning of gating and venting strongly affects the quality of the finished part. RTM parts do not necessarily require autoclave curing but they do require a post cure cycle for high temperature applications. One drawback in this

process is that a large closing force is required to clamp the moulds during the entire process, to counteract the injection pressure.

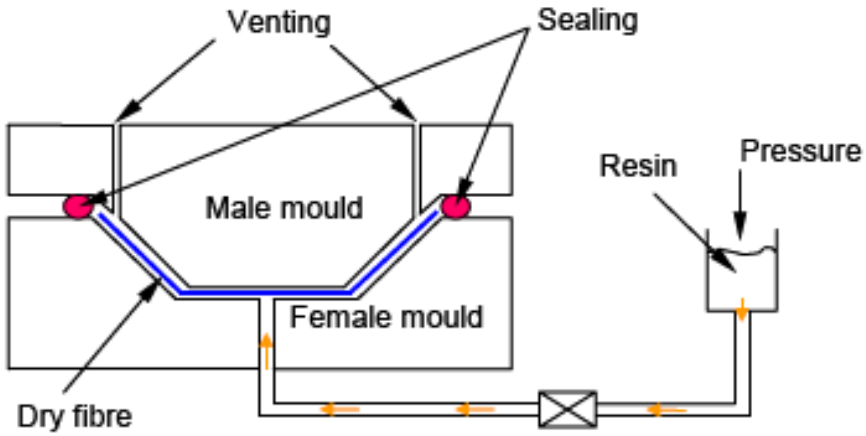


Figure 2.2: Working principle of Resin Transfer Moulding process

RTM is a very versatile process as it can be used to produce simple as well as complex geometries with good surface quality and finish. The dry preform used for the process is a relatively cheaper material than prepreg and it can be stored at room temperature. The void content in RTM parts can be achieved as low as 0-2 percent range, but the process requires very close monitoring throughout.

There are several variants of this process like Vacuum Assisted Resin Transfer Molding (VARTM) which uses vacuum to draw in the resin to impregnate the fibers. Hassan Mahfuz, *et al.*[21] utilised VARTM process to inject composite skin-stiffener assembly in one single step. "Light RTM" (LRTM) is another variant of RTM that uses two part mould. The LRTM mould typically consists of a rigid half-female cavity made from high temperature low shrink thermoset resin and semi-flexible upper half made from thermoset vinyl ester resin[26]. As compared to traditional RTM, LRTM offers advantage in terms of minimum tooling structure, low tooling cost and increased production rates over open mold process.

Another variant of RTM process is the Modified Vacuum Infusion Process (MVI) which is patented by Airbus. The process uses a semi-closed mold, where a vacuum bag on the non-tooling side covers the fiber layers. The basic technique in this process is to use two discrete vacuum bags, an inner bag next to the laminate which removes the entrapped air in the dry preform and an outer bag which is separated by a breather fabric. The outer bag provides compaction to the entire preform. Therefore, this technique significantly reduces the void content and increases the fiber volume ratio, thereby improving the overall mechanical strength of the laminate.

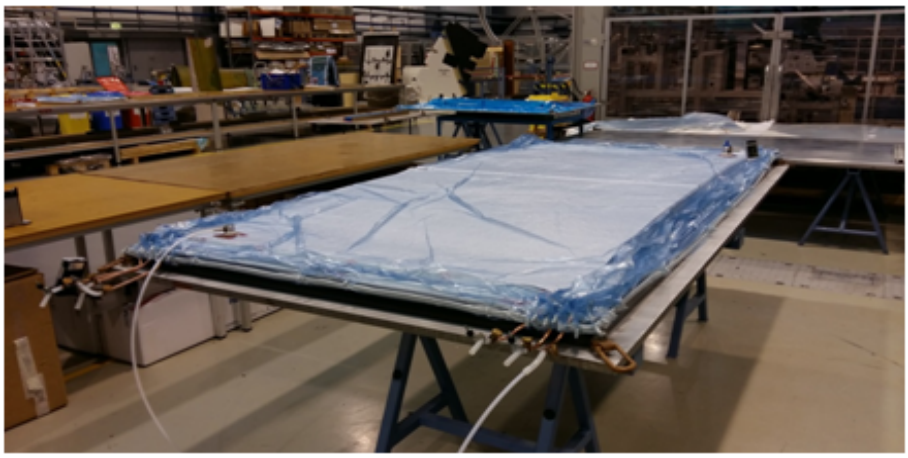


Figure 2.3: Modified Vacuum Infusion Process

The Resin Film Infusion (RFI) is a low cost manufacturing process as compared to prepreg or other processes. In this process, the resin is applied to the tool as thermoset resin films, which are located underneath or between the fiber layers. The preform assembly is vacuum bagged and cured in autoclave. Under high temperature and pressure the resin film liquefies and impregnates the fiber in thickness direction. This method allows to feed exactly the needed amount of resin for the composite part. Additionally, it avoids any resin lines during the autoclave cycle. The biggest advantage of using this process is very short distance of resin infusion which is in the thickness direction.

2.3. Co-curing Processes

Stiffened composite panels are being widely used in aircraft construction as primary structures. A key feature in this is the skin-stiffener assembly. Commonly used techniques for the assembly process are co-curing, co-bonding and secondary bonding. Out of these processes co-curing is a preferred choice. The main advantage of using co-curing process; reduced stress concentration because of elimination of holes, reduced assembly time and less material usage which results is cost saving[8]. Another important factor is the quality check of co-cured panels. The stiffener and skin are simultaneously cured as one panel, so the quality check is done for the entire panel as opposed to co-bonding and secondary bonding where a separate quality check is required for the bondline and for the entire panel. Some examples of aircraft components that are made using co-curing techniques are vertical fins, tail cones, horizontal stabilizers, pressure bulkheads, etc[10].

Liquid Composite Moulding Techniques(LCM), like Vacuum Assisted Resin Infusion(VARI), Resin Transfer Moulding(RTM) and Resin Film Infusion(RFI) have the advantage of being applied on complex geometries because of ease of shaping fiber architecture and they are fast processes. On the other hand, the laminates produced from these processes result in low fiber volume fraction, limiting their use in primary loaded structures[7]. Co-LCM is a new co-curing process that combines the prepreg and LCM processes combining the advantages of both the techniques, thereby giving a potential solution for improved quality and reduced manufacturing cost[19].

Northrop Grumman company reported the first use of the co-RTM process for integral composite structures. Co-RTM uses dry or binderized preform in the tool cavity. The process includes prepreg skin panel within the tool, injecting resin into the preform and curing the entire preform assembly in an autoclave or oven[12]. The process provides a significant overall cost reduction as compared to typical fabrication processes. Manufacturing of F-15 tail using co-RTM process had a 17 % overall cost reduction. Further more superior dimensional control and a light weight stiffened composite panel are achieved through this process[18].

Ma X Q,*et al.*[19] developed co-VARI process which combined VARI and prepreg process to manufacture T-shaped stiffened skin structure. Their investigation revealed increase in fiber volume fraction without any void defects. Another strategy of using integrated co-curing process, is co-RFI process which combines resin film infusion and prepreg process to fabricate skin-stiffener assembly[20]. The resin film is placed between the T or I-shaped dry fiber reinforcement and prepreg skin layers as shown in figure 2.4[23]. Xuqiang Ma *et al.*[20] investigated mechanical performance of co-RFI stiffened skin at the interface region with no obvious defects and good pull-off properties.

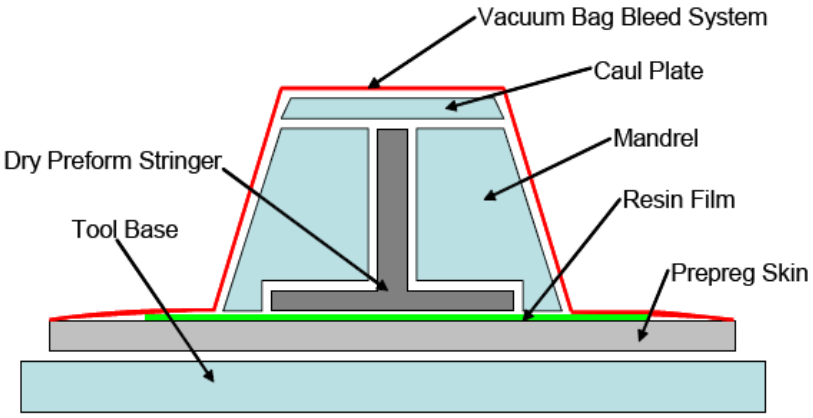


Figure 2.4: Schematic representation of RFI stringer preform on prepreg skin

2.4. Tooling Concepts

Apart from manufacturing techniques, tooling is an important parameter that determines the final quality of the laminate. Tools can be made from a variety of materials, but the material selection certainly depends on a lot of production factors. For low production rate parts that are cured at low temperatures and pressure, materials like fiberglass, high density foam, clay or wood/plaster tools can be used. For high production rate parts which require high dimensional accuracy with excellent surface finish, metal tools are used that can withstand high temperatures as well as production cycles. For high performance composite parts tools are made from carbon fiber, graphite or metals like aluminium, steel and invar.

Hard tooling materials generally include aluminum, steel and invar. Metal tools are robust, damage tolerant and deliver excellent surface finish. One key issue with tooling is the coefficient of thermal expansion(CTE) mismatch. Commonly used metal tools like steel and aluminum though are easily affordable show CTE mismatch when used for manufacturing composite parts. Another problem investigated by researchers is fiber waviness which is caused by tool slippage during manufacturing. This can be prevented by securing the tool with bolts or weights around the tool to restrict the movement[28].

Soft tooling material includes tools made from flexible rubber material and sometimes inflatable tools. These are easily constructed as compared to metal tools, but are more susceptible to wear with use. Composite tools have better CTE performance as compared to metal tools. The CTE of soft tool matches the CTE of composite panel. During cure cycle the thermal expansion of tool and part are very similar, therefore the dimensional accuracy of the composite panel is maintained[22].

G.-H. Kim *et al.*[14] manufactured co-cured hat stiffened panel using an inflatable tool made of silicon. The tool is expanded as a result of air pressure, which helps in consolidating the composite part. Their investigation revealed no noticeable wrinkles on the panel surface. However, the manufacturing cost and time involved in using inflatable tool was higher than for other metal or composite tools.

Elastomeric Tooling Technology (ETT) uses the advantage of hard tooling which provides dimensional accuracy to the laminate and soft tooling providing even pressure distribution. Because of this combined property elastomers are being widely used in the fabrication of isogrid panels[15,16] and Advanced Grid Stiffened Structures (AGSs)[11,13]. A key feature for ETT is the high coefficient of thermal expansion and good thermal stability for soft tools which are made from silicon rubber. On application of heat the elastomeric tool expands, compacting the composite laminate[22,24,29].

Flexible rubber tooling or elastomeric tooling is in use for many years for providing uniform pressure on composites in autoclave curing. Composite laminates are cured in an autoclave or an oven in order to provide even pressure for part consolidation. But often, inspite of using autoclave the problem of corner voids and laminate bridging occurs as shown in Figure: 2.5[22]. This problem can be controlled by applying additional external pressure using elastomeric tooling from the vacuum bag side of the laminate which prevents the slipping of fibers during consolidation.

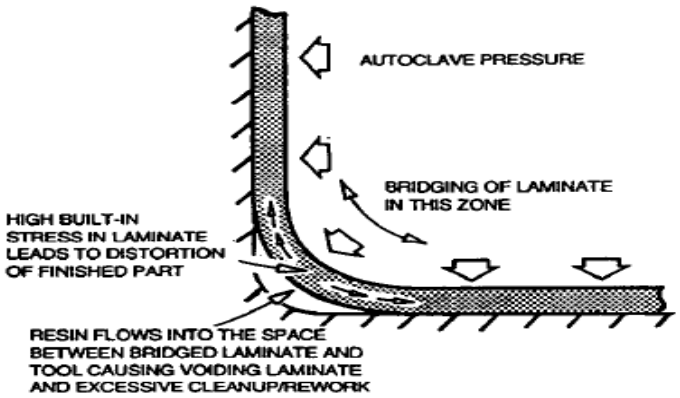


Figure 2.5: Corner voids in composite laminate

Reinforced elastomeric tooling use polyacrylic rubber material used as airpads[22]. Airpads provide uniform pressure distribution during curing cycle in autoclave. The surface finish on the vacuum bag side of the part is comparable to metal tool finish.

The design of isogrid and AGS structures is different from that of T-shaped or I-shaped stiffeners. The panel has stiffeners and ribs in the form of a grid structure. Two low cost tooling methods have been proposed, hybrid tooling method and expansion block method[13]. Hybrid tooling uses a base tool (made of tooling epoxy) which provides stiffness to the part and an expansion tool(silicone rubber) which provides compaction to the composite panel. In the expansion block method, the base tool which is made of stiffer material like aluminium and expansion blocks made of high CTE material is used to provide compaction[11].

The research on these tooling methods can provide a good understanding of how these techniques can be implemented for T-shaped or I-shaped stiffener combined with co-LCM process.

2.5. Concluding Remarks

Stiffened composite panels are designed for a variety of load carrying conditions in aircraft construction. The skin-stiffener assembly is usually manufactured using moulding processes like co-curing, co-bonding and secondary bonding. Co-curing is generally a preferred choice out of the three processes, as it cures the the stiffener and skin in a single cycle and does not require extra steps. But manufacturing of stiffened panels with prepreg can become laborious and time consuming. Therefore, a new co-curing technique called co-LCM was introduced. This includes co-RTM, co-VARI or co-RFI process. Many researchers[7,18,18,19,25] have described co-LCM processes as promising low cost alternatives to solely using prepreg or individual LCM processes. The benefits include better mechanical performance, increase in fiber volume fraction and overall improved laminate quality.

Tooling is another parameter that considerably affects the quality of composite panel. Researchers have studied the effect of using different tooling schemes[27]. Both hard and soft tooling have their advantages and disadvantages. The use of flexible or soft tooling which includes inflatable tools and elastomers has been researched. It is observed that flexible tooling provides better CTE performance and increase in fiber volume fraction[22,24,27–29]. Not enough information is available when both co-LCM and flexible tooling are used together. Using both concepts will help in reducing defects thereby improving the quality of laminate produced.

If composite panels are manufactured using co-LCM with different tooling schemes, it can help answer the research question presented in this literature report. The following focal points can be validated-:

- The use of prepreg and resin infusion in single process
- It will help in establishing a comparative analysis of effects on the laminate, by using different tooling schemes

- It will help understand the effects of different process parameters such as type of resin, temperature and pressure conditions during manufacturing on composite laminate.

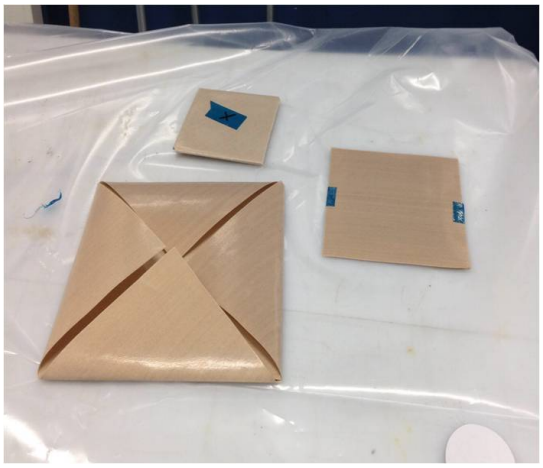
3

Preliminary Test Phase

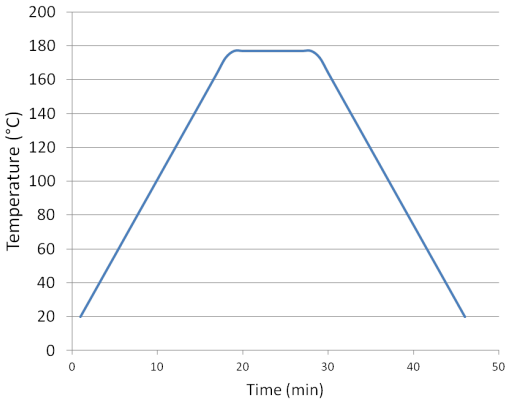
3.1. Material test

3.1.1. Joos press cure cycle

In this preliminary test phase the available prepreg material was tested under different processing conditions to do a comparative analysis of consolidation quality and void content with process cycle in the Joos Press and autoclave. The material used is UD carbon prepreg provided by TenCate Advanced Composites USA Inc. Two samples were prepared, crossply (0-90) and unidirectional each with 10 layers each. Each stack was enclosed in teflon sheet as shown in Figure 3.1a to avoid any fiber slippage during consolidation in the Joos press. These stacks of prepreg material were placed between stainless steel press-protector plates which were thoroughly coated with Marbocote release agent. The plates were positioned in the Joos press and consolidated. The cure cycle consists of increasing the temperature from 20 °C to 177 °C at a heat up rate of 9 °C per minute with 6 bar pressure. Since, the actual cure cycle information for UD carbon prepreg was not available at the initial stages, the 9°C per minute cycle was taken from a similar thermoset material.



(a) Prepreg stack with teflon covering



(b) Cure cycle for first preliminary test

Figure 3.1: Preliminary test setup and cure-cycle

A second test was done with the same prepreg material with the same layup sequence and press arrangement but different cure cycle. The cure cycle used for this test was given by the material supplier (received after a month from the first test) which consists of increasing the temperature from 20 °C to 71 °C at 1°C per minute, hold time for 60 minutes, second ramp up to 100°C at 1°C per minute, then hold for 120 minutes and finally cool down to room temperature at 1°C per minute. The temperature cycle is shown in the Figure 3.2.

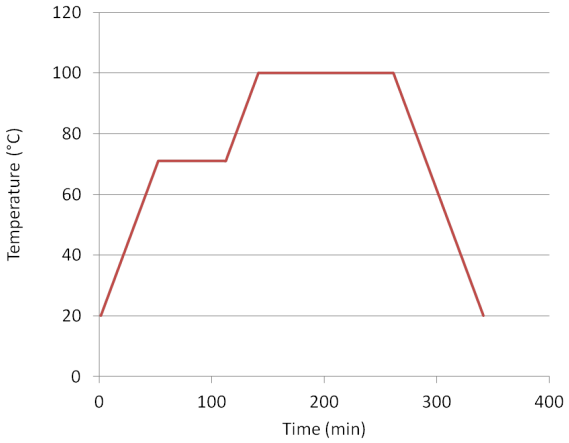


Figure 3.2: Cure cycle for second preliminary test

3.1.2. Autoclave process

For the autoclave setup, the prepreg stacks were placed between aluminum caul plates which were coated with release agent. A large aluminum base plate was prepared with release agent and the stacks were vacuum bagged as shown in the Figure 3.3. The cure cycle used to consolidate the material in this process was the same as provided by the material supplier as shown in the Figure 3.2.

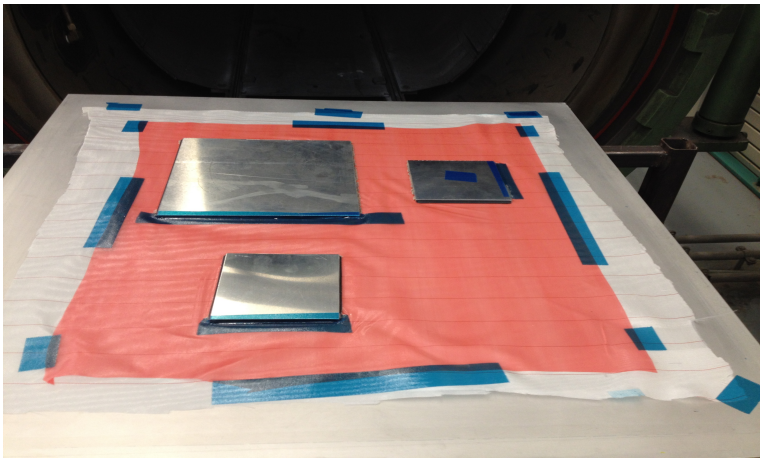


Figure 3.3: Autoclave setup

3.2. Inspection

3.2.1. Thickness Measurement

After consolidation, the laminates were inspected for any visible flaws on the surface. For measuring the thickness of the laminate, the surface was divided in small sections of 10×20 mm. Each of these sections was measured individually for its thickness using a vernier calliper or micrometer. The thickness of all these sections was averaged to get the mean (average) thickness of the laminate.

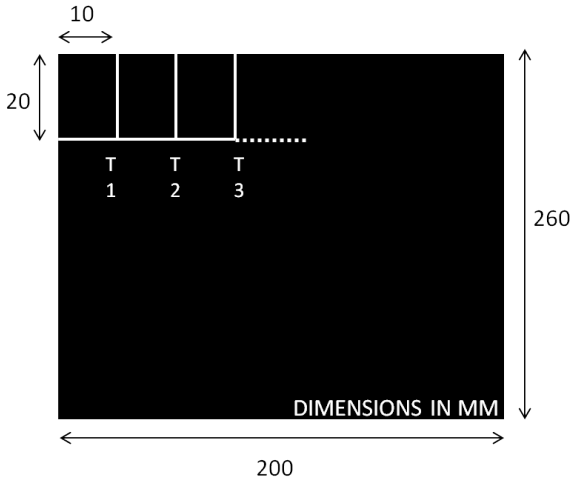


Figure 3.4: Schematic representation of thickness measurement

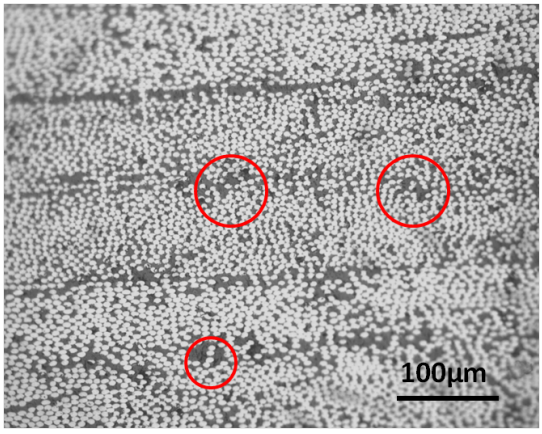
3.2.2. Microscopy

The samples from the laminates were cut with a diamond blade and cleaned in an ultrasonic ethanol bath to remove any contamination from the cutting process. The samples were embedded in Technovit 4071 resin to analyze the cross-section. The embedded samples were sanded with 180, 320, 1000 and 2400 grit sanding paper and polished with plates coated with 6, 3 and 1 μ m diamond paste. The cross-sections were analyzed using Leica optical microscope at $\times$20 magnification. The image analysis software Lyca Qwin was used for analyzing the cross-sections of the specimens. The software analyzes individual pixels that make the image cross-section. The built in analysis tools in the software calculates total number of voids which are seen as black pixels in the image, fibers as white and matrix as grey area. The total area of the captured image with voids, fibers and matrix is taken as 100% and the constituent % is calculated with the help of software tools by selecting the voids or fibers or matrix area in the image.

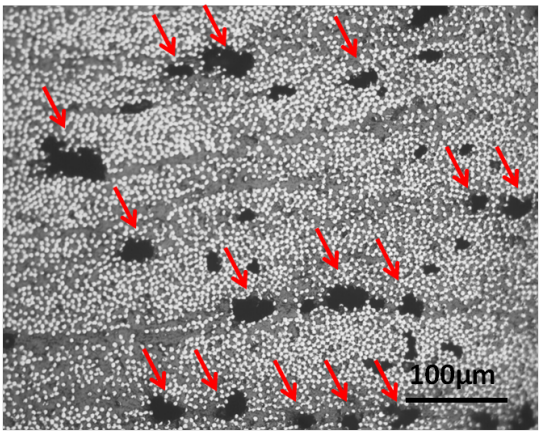
3.3. Preliminary Test Analysis

In this phase three tests were performed, two in the Joos press with different temperature cycles and one in the autoclave. The purpose of doing these tests was to do a comparative study on the void content variation in the laminates.

The first test with 9 °C per minute ramp up rate gives a void content of 3.91% for the 0-90 sample and 5.97% for the UD sample. The cross-section images with void scatter are shown in Figure 3.6. On comparing both the cross sectional images the UD sample shows voids scattered over much larger area (Figure 3.5b). Whereas in 0-90 sample the voids are visible in a limited region (Figure 3.5a).



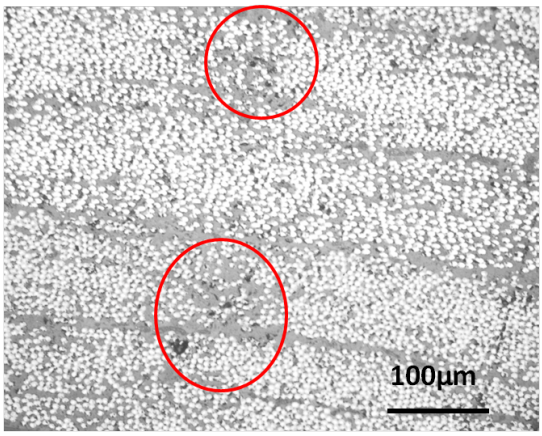
(a) Void scatter in 0-90 sample



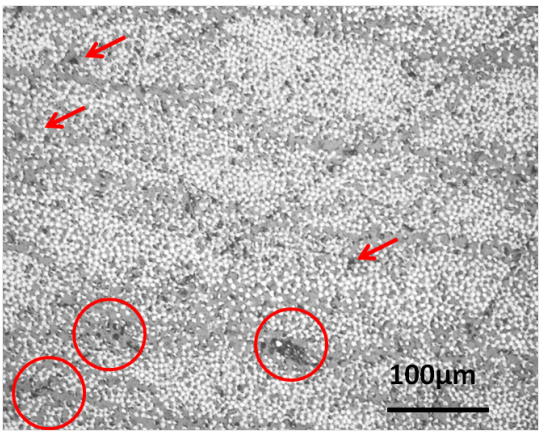
(b) Void scatter in UD sample

Figure 3.5: Cross-sectional analysis for 9 °C per minute ramp up rate with X20 magnification

The second test with 1 °C per minute ramp up rate gives a void content of 3.62% in 0-90 sample and 4.28% in UD sample. The cross sectional images can be seen in Figure 3.6. The void scatter in the UD sample is spread over a wider region but it is less dominant as compared to the UD sample in the first test (Figure 3.6b). In the 0-90 sample the scatter is again limited to a smaller region if compared to the UD sample (Figure 3.6a). This gives an indication that cure cycle and hold time affect the consolidation of the laminate. The void content for 0-90 panels was comparable but in the second test the UD sample shows improvement as the void scatter decreased by ± 30% .

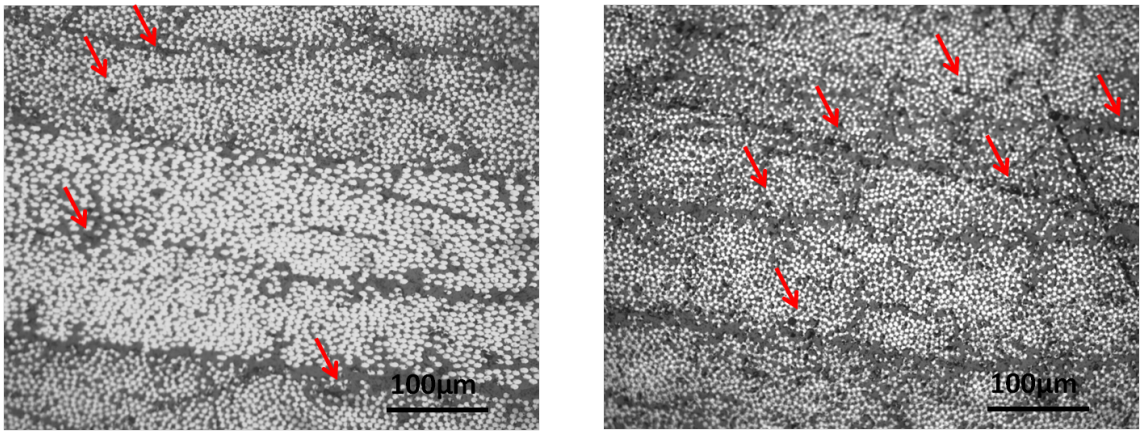


(a) Void scatter in 0-90 sample



(b) Void scatter in UD sample

Figure 3.6: Cross-sectional analysis for 1 °C per minute ramp up rate samples with X20 magnification



(a) Void scatter in 0-90 sample

(b) Void scatter in UD sample

Figure 3.7: Cross-sectional analysis for autoclave sample consolidated with 1 °C per minute ramp up rate X20 magnification

The autoclave test was done to compare the results with the Joos press method. The void content for 0-90 sample was 2.82% and for UD it was 3.53%. The void scatter is shown in Figure 3.7 In all the above cross-sections it was observed that UD layup sequence shows a higher void content % than 0-90 layup sequence. This is due to fact that when the UD layers are stacked, the fibers overlap each other and so does any kind of porosity between the fibers. However, in a 0-90 layup sequence the UD fabric is stacked perpendicular to the preceding layer which reduces the porosity between the layers. This results in a lower void % in 0-90 layup sequence. The layup sequence is shown in Figure 3.8.

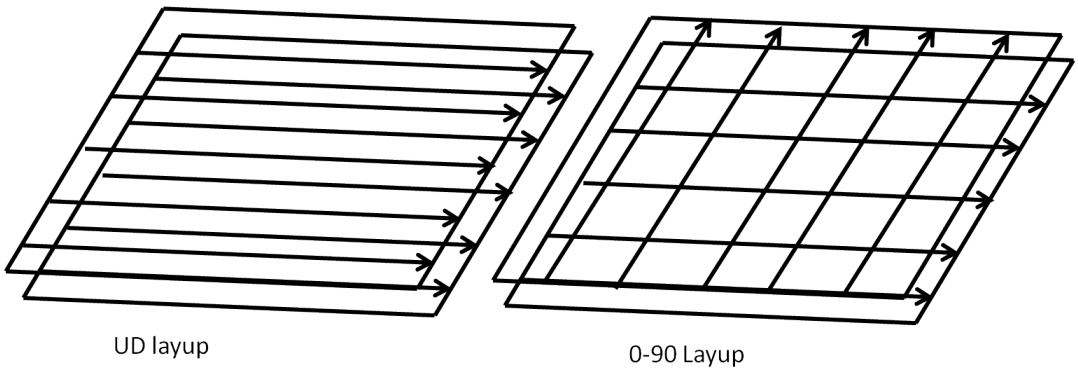


Figure 3.8: Schematic representation of layup sequence UD and 0-90

The thickness measurement for the autoclave samples was very consistent with overall thickness of laminate 1.45 mm with a variation of ± 0.01 mm. Whereas the variation in the Joos press laminate was measured as 1.41 mm with variation of ± 0.04 mm. The difference in variation can be attributed to the presence of teflon sheet which had a rough texture. Also, because of the homogeneous pressure and vacuum conditions inside the autoclave which makes a difference in the laminate quality and void content.

3.4. Concluding Remarks

After conducting the preliminary tests on the prepreg material under different process conditions it was decided to proceed with further testing on UD layup as it offers more improvement opportunity in terms of void content percentage. The cure cycle used in Phase 1, 2 and 3 of the project will be as given by the material supplier also shown in Figure 3.2. The results from preliminary phase are consolidated in the table below.

Table 3.1: Results for press cycle with 9°C/min ramp up rate

Layup sequence	**Thickness measurement**	**Void%**
0-90	1.41 mm	3.91
UD	1.41 mm	5.97

Table 3.2: Results for press cycle with 1°C/min ramp up rate

Layup sequence	**Thickness measurement**	**Void%**
0-90	1.41 mm	3.62
UD	1.41 mm	4.28

Table 3.3: Results for Autoclave test with 1°C/min ramp up rate

Layup sequence	**Thickness measurement**	**Void%**
0-90	1.45 mm	2.82
UD	1.45 mm	3.53

4

Tool Development

The requirement of the experimental setup for this project was to have a closed mould system so that resin infusion can be carried out in the same setup in the later phase of the project. The intention was to have three broad components a base, mid assembly and lid which will facilitate in having a closed setup. Two of the components, the base and mid cylinder were already available from an old equipment around which the rest of the components of the setup were constructed to complete the setup. These existing components contributed to sizing the complete mould. Initially, it was decided to optimize the setup for small scale samples so that its easier to identify and improve any tooling related issues. These improvements can then be implemented for a scaled up version.

In this chapter, the evolution and description of the experimental setup is provided. While conducting the tests, a number of technical issues such as fiber flow-out, excessive pressure buildup, silicon creep etc arose which led to modifications in the tool setup. Each of these problems are explained along with its influence on the changes made in the setup. It was necessary to optimize the tooling setup so as to make the process repeatable and reproducible. A brief summary of the old and the new setup is compared at the end of the chapter.

4.1. Setup Assembly

In a broader sense the setup is divided in three portions: laminate bed, mid assembly and top assembly. A detailed view of the tool setup is shown in Figure 4.1 and the description of the individual parts is given in Table 4.1.

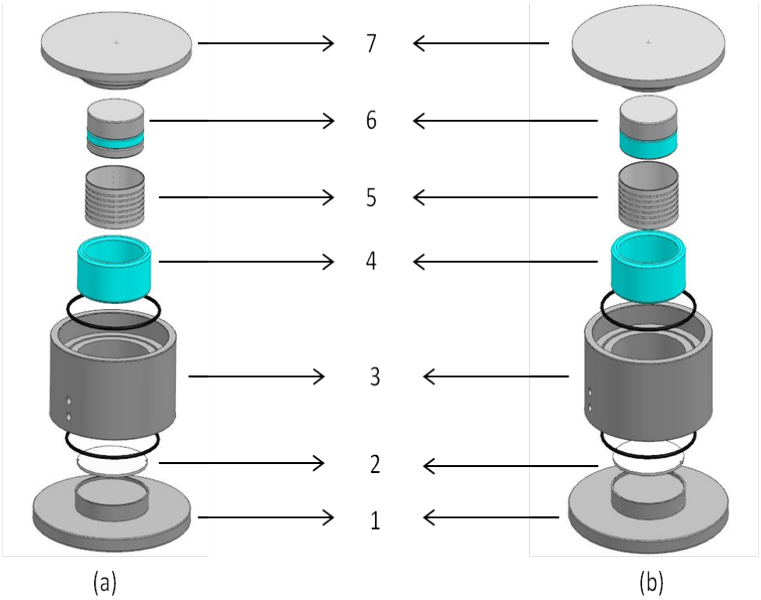


Figure 4.1: Exploded view of tool setup. (a) setup with solid-solid tool (6). (b) setup with solid-flexible tool (6)

Table 4.1: Sequential setup assembly

S.No	Part Description	Material	Quantity
1	Laminate bed	Aluminum	1
2	Laminate bed base	Polyoxymethylene (POM)	1
3	Outer cylinder wall	Aluminum	1
4	Inner silicon ring	Silastic M	1
5	Inner metal ring	Aluminum	1
6	solid-solid/solid-flexible tool	Aluminum and Silicon	1
7	Top lid	Aluminum	1

4.1.1. Laminate Bed

Laminate bed forms the lower part of the setup assembly as shown in Figure 4.2. As the name suggests, the purpose of this part is to place/position the carbon prepreg layers on this bed. On the bottom face of the "bed", a thermocouple is attached to record the temperature during processing. The laminate bed has two grooves on either side for inlet and outlet infusion channels. In the initial test trials the laminate bed base was made of material Polyoxymethylene (POM) which has similar properties as teflon and it is easier to demould the laminate after consolidation.

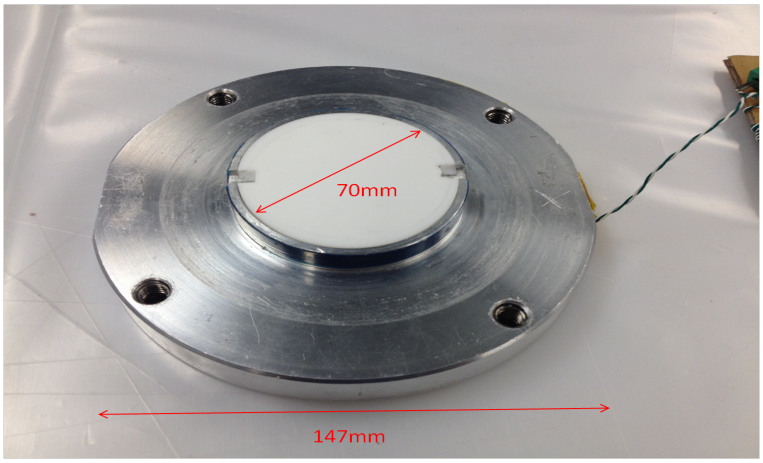


Figure 4.2: Laminate bed with Polyoxymethylene (POM) as base material

4.1.2. Mid Assembly

The mid assembly is positioned on top of the laminate bed and consists of the following parts (compare Table 4.1) :

3 Outer Cylinder Wall

4 Inner Silicon Ring and [5] Metal Ring

6 Solid-Solid/Solid-Flexible tool

[3] Outer Cylinder Wall

The outer cylinder wall consists of inlet and outlet port for infusion. On the upper and lower side of this wall are grooves for O-rings which help in vacuum sealing the entire assembly (Figure 4.3). The inner silicon and metal ring as well as solid-solid/solid-flexible tool in enclosed within this cylinder wall.

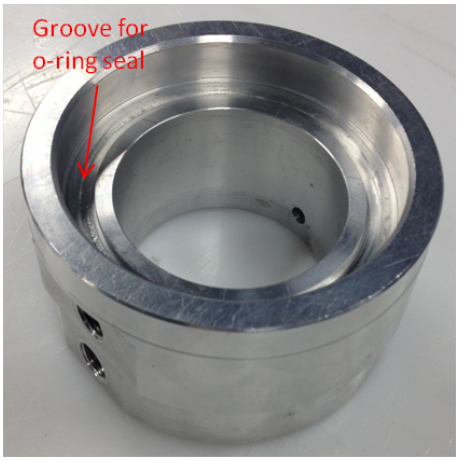


Figure 4.3: Outer cylinder wall

[4] Inner Silicon Ring and [5] Metal Ring

The function of the inner metal ring is to position the prepreg layers. For the first couple of tests this ring was made of aluminum (Figure 4.4). But with repeated tests at higher closing force of around 15 kN, there was a permanent deformation in the aluminum ring. This affected the laminate quality with fiber flow-out around the edges. This ring was replaced by a stainless steel ring to avoid any deformation owing to its stronger material properties .

The silicon rings used in the setup were casted with Silastic M material in separate moulds and cured at room temperature for 24 hours. The description of the material is provided in Appendix A. The inner metal ring is encased within the silicon ring as shown in Figure 4.5. It protects the cylinder walls from any resin spill during the infusion process. As the setup is heated during the processing, the silicon ring expands in vertical and horizontal direction closing any air gaps.

Figure 4.4: Inner metal ring

Figure 4.5: Inner metal ring encased within silicon ring

[6] Solid-Solid/Solid-Flexible Tool

This is the key element of the setup which provides the consolidation pressure to the prepreg layers. The tool assemblies can be seen in the Figure 4.6 and Figure 4.7. In solid-solid tool the pressure is transferred from solid top lid to the solid contacting surface through the inner silicon ring as shown in Figure 4.6. The inner silicon ring expands on application of heat during the process exerting pressure vertically and horizontally closing any gaps. The vertical pressure from silicon ring acts on the bottom solid surface which in turn provides pressure to the prepreg layers.

In case of solid-flexible tool shown in Figure 4.7, the surface in contact with the prepreg layers is the flexible silicon surface. The pressure from the top solid lid is transfered to the silicon block. The silicon block expands both in vertical and horizontal direction providing pressure to the prepreg layers.

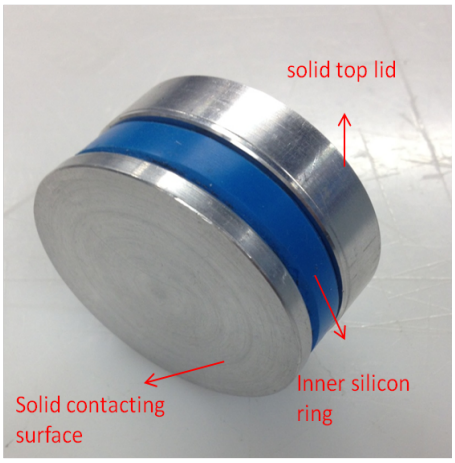


Figure 4.6: Solid-solid tool

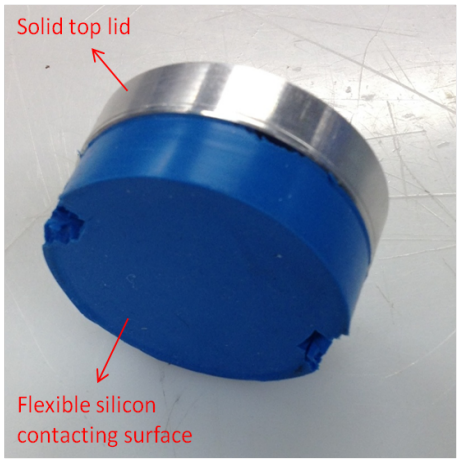


Figure 4.7: Solid-flexible tool

The consolidation pressure desired for the setup is 6 bar (P).The diameter of both the solid-solid tool, solid-flexible tool and the laminate is 54 mm (D). The corresponding closing force required for the setup was

mathematically calculated with the following equations (4.1a) (4.1b).

$$Pressure(P) = Force/Area \tag{4.1a}$$

$$Area = \frac{1}{4}\pi D^2 \tag{4.1b}$$

The calculated closing force for the mould setup was 1500N.

4.1.3. Top Assembly

The top assembly consists of an aluminum lid that closes the complete setup. With the help of an o-ring between the top lid and outer cylinder wall the setup is vacuum sealed. The complete view of the tool setup is shown in Figure 4.8.

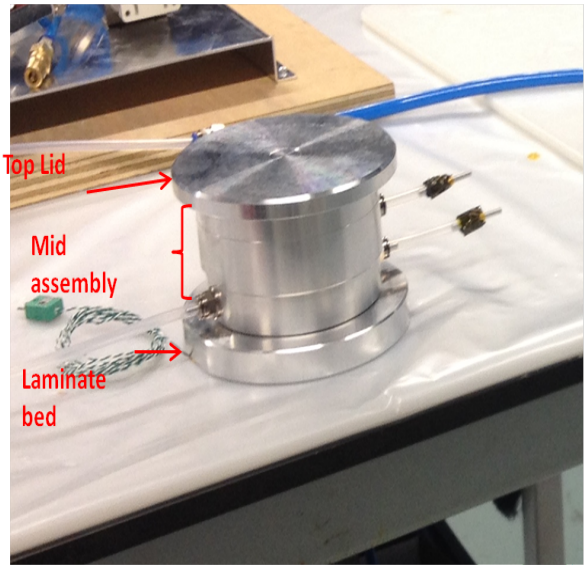


Figure 4.8: Setup Assembly

4.2. Phase 1

In this phase, the tests were conducted using the tool setup described in previous section. A brief overview of the tests is shown in Table 4.2

Table 4.2: Panel Overview

Phase 1:Panels with fibre flowout			
Test Number	**Tool configuration**	**Description**	**Change in setup**
4.1	solid-solid	fiber flowout	filler disc
4.2	solid-solid	no flowout	
4.3	solid-flexible	fiber flowout	bolt assembly
4.4	solid-flexible	surface waviness	cutouts in silicon block
4.5	solid-flexible	silicon creep	silicon block with lower ring
4.6	solid-flexible	silicon creep	embedded flexible tooling

4.2.1. Solid-Solid Tooling Test Procedure 4.1

The first test was conducted with solid-solid tooling configuration Figure 4.9. The layup sequence for the plies was $[0]_{10}$. The setup was placed between the Joos press plates. The setup was kept under vacuum for 15 minutes before starting the process cycle

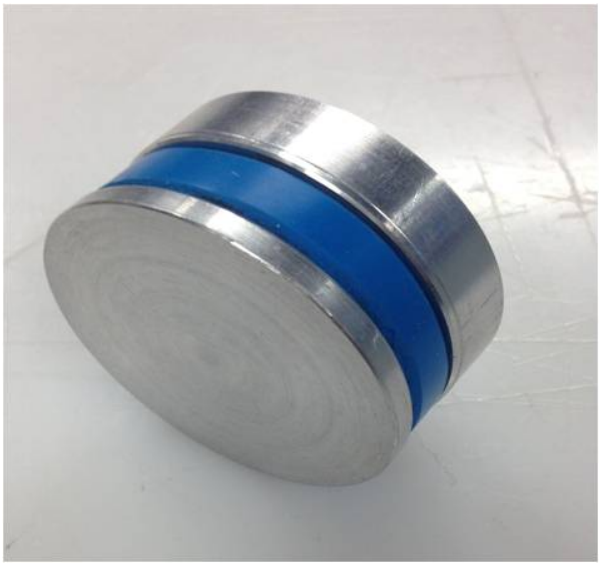


Figure 4.9: Solid-solid tooling

The process was carried out with the temperature cycle provided in Figure 3.2. The setup is demoulded after completion of the process.

Inspection

The test sample produced was inspected visually for any surface defects. Also, the thickness was measured throughout the surface. The consolidated laminate shows significant fiber flow-out around the edges. The top view of the sample is shown in the Figure 4.10.

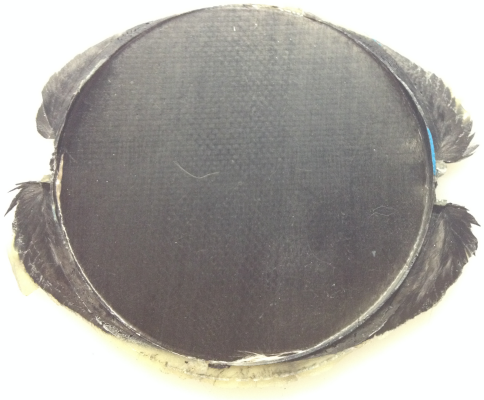


Figure 4.10: Panel 4.1: Fiber flow-out around the edges

Thickness Measurement

From visual and surface inspection it was clear that the edges with fiber flow-out had significant thinning in the cross-sectional thickness as compared to the center of the laminate. For precise measurement, thickness was measured with a vernier caliper. The average thickness of the laminate measured 1.15 mm. The thickness ranged between 1.02-1.05 mm near the edges of the laminate and near the center 1.1- 1.15 mm. This can be seen in the Figure 4.11. The difference in thickness measurement is due to excessive squeeze out of resin and fibers near the edges as compared to the center of the laminate.

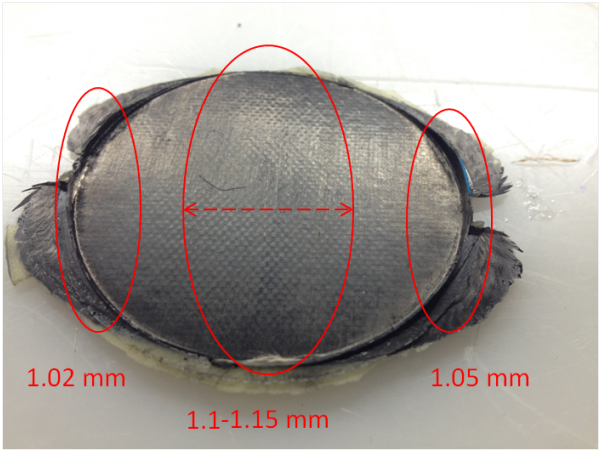
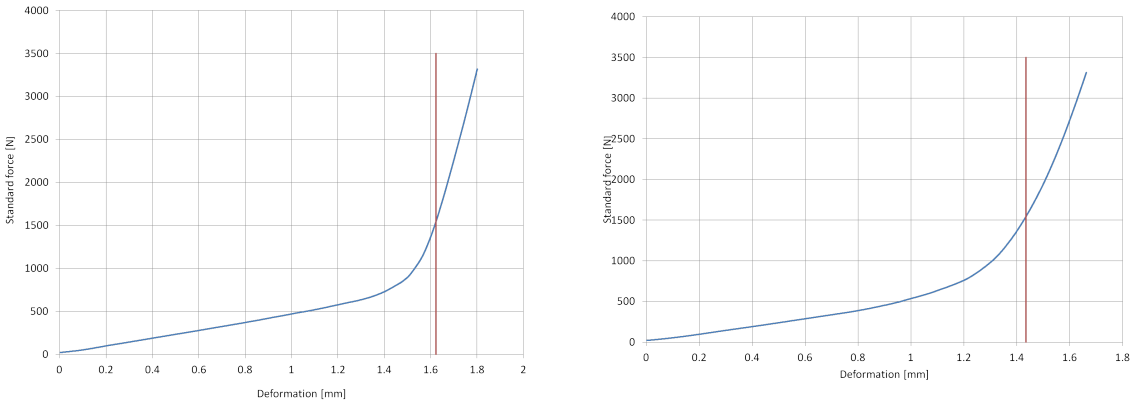


Figure 4.11: Panel 4.1: Thickness variation in the sample

Test Analysis

The fiber flow-out was caused due to insufficient pressure on top of solid-solid tool (Figure 4.9) when the mould was closed. Upon inspection it was found out that the load from the press plates was not getting transmitted to the tool, owing to a design error which meant that the consolidation pressure of 6 bar was not getting created within the setup. There was a clearance of ~ 3.2 mm in between the top lid and solid-solid tool top surface which did not allow the transmission of the closing force to the laminate. To establish the correct procedure of transmitting 1500N closing force onto the laminate, through the solid-solid tool; the tool was subjected to a compression test. The graphs obtained from the test are shown in Figure 4.12



(a) Compression test for solid-solid tool

(b) Compression test for solid-flexible tool

Figure 4.12: Compression test for calculating closing force

From the compression test plots, it was concluded that to transmit a force of 1500N and thereby creating a consolidation pressure of ~ 6 bar, the solid-solid tool has to show a displacement of 1.62 mm under the force of 1500N (4.12a) and the solid-flexible tool has to show a displacement of 1.45 mm under the same force of 1500N (4.12b). The clearance of 3.2 mm and deformation of 1.62 mm in the solid-solid tool, led to sizing of a filler disc with thickness 4.82 mm. Similarly, the clearance of 3.2 mm and deformation of 1.45 mm in the solid-flexible tool, led to sizing the filler disc of thickness 4.65 mm (Figure 4.13). Introducing a filler disc eliminated the issues of clearance, improper load transfer and also the inner metal and silicon ring shift. The filler disc was placed on top of solid-solid tool surface.

Figure 4.13: Aluminum filler disc

Subsequent tests were conducted with the filler disc in the setup. The laminates produced in the test 4.2 revealed negligible flow-out near the edges (Figure 4.14). The overall thickness measured was 1.14 mm with a variation of ± 0.01-0.02 mm which is due to the indentation of the teflon sheet on the top surface of the laminate layers.

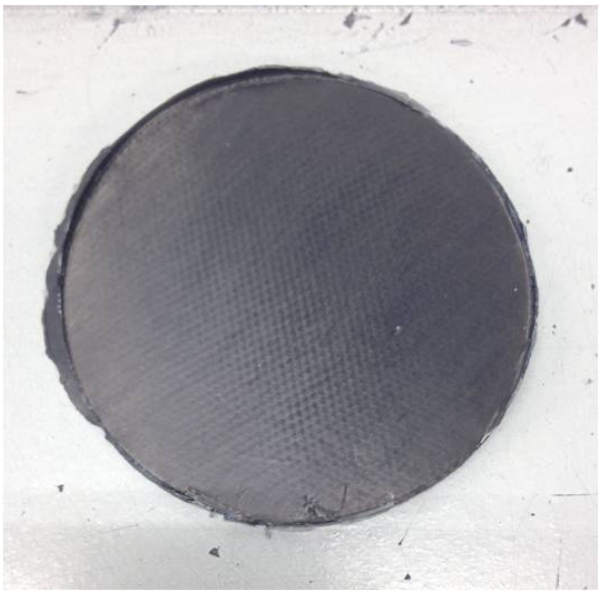
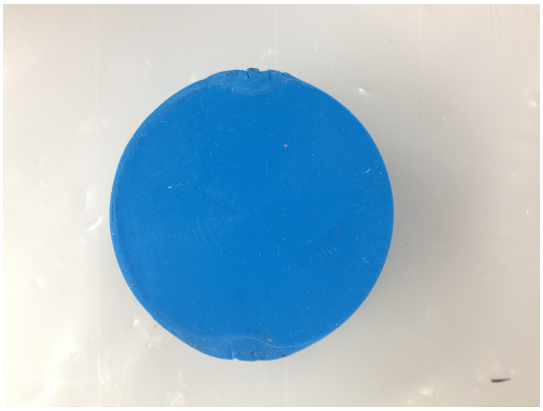


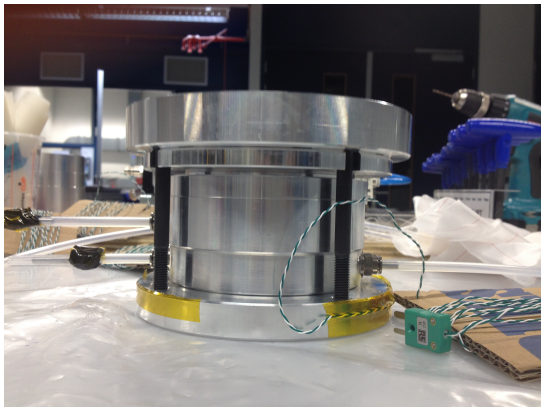
Figure 4.14: Panel 4.2 : Negligible fiber flow-out

4.2.2. Solid-Flexible Tooling Test Procedure 4.3

In the following tests the consolidation pressure provided to the laminate was with the help of solid-flexible tool configuration. The aim is to know the difference in the consolidation quality with respect to solid-solid tool tested in the previous section. The pressure buildup is due to the expansion of flexible silicon rubber tool during the heat up of the process. The surface of the rubber tool can be seen in the Figure 4.15a.



(a) Solid-flexible bottom surface



(b) Setup with bolts

Figure 4.15: Setup for trial 4.3

The mould setup used was the same as described in previous section with the filler disc on top of the solid-flexible tool for pressure. Another addition made to the set up was the bolt fixture as shown in Figure 4.15b. After assembling all the parts of the tool, the mould was closed with bolts on top. This was done to prevent any kind of tool misalignment that might happen while transferring the entire setup from the work table to the Joos press. The layup sequence of the layers and the process cycle was the same as for the tests conducted before. While assembling the setup, a misalignment was caused in the laminate position because the thermocouple was attached to the bottom of the mould at the end. Instead it should have been attached to the mould in the beginning to avoid any movement of the inner tooling.

Visual Inspection

After the process cycle was completed in the press, the setup was demoulded. There was excessive resin leakage around the laminate bed which made the demoulding difficult and damaged the lower o-ring as shown in Figure 4.16. The laminate showed flow-out near one edge and a very rough surface finish as compared to the solid-solid tooling samples. This can be seen in the Figure 4.17. Another observation was the blockage of the inlet and outlet tubing due to silicon creep in the tube cavity.

Figure 4.16: Resin leakage during processing in the lower o-ring.

Figure 4.17: Fiber flowout

Thickness Measurement

The measurement was done using a vernier caliper. Since the surface had rough patches and waviness the average thickness of the laminate measured 1.07 mm. Starting from the flow-out edge to the center of the laminate, thickness varied: 0.85, 0.95, 1.11 and 1.09 mm. This is also represented the Figure 4.18.

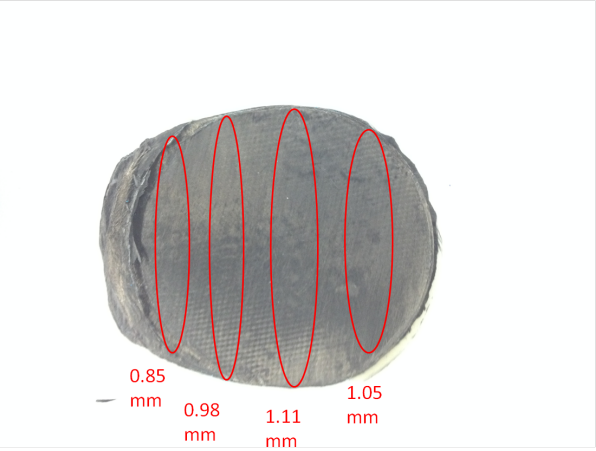


Figure 4.18: Thickness measurement

Test Analysis

- As described earlier, the thermocouple was attached at the bottom after placing prepreg layers inside the mould which might have caused the layers to shift from the position causing fiber flow-out.
- The silicon creep in the tubing suggests that if same configuration is used for further testing, silicon will continue to creep in any empty cavity during processing. This can hamper the infusion trial, blocking the resin inlet tube. Therefore, small cut outs were made on the silicon surface for test procedure 4.4.

4.2.3. Test Procedure 4.4

This test was conducted to accommodate the suggestions prescribed in the previous test. To compensate for the silicon creep, small cavities were created at the opposite edges of the silicon block as shown in Figure 4.19, to give clearance between the inlet/outlet tube and the silicon surface. All other test conditions were kept similar including the layup and temperature cycle in the press.

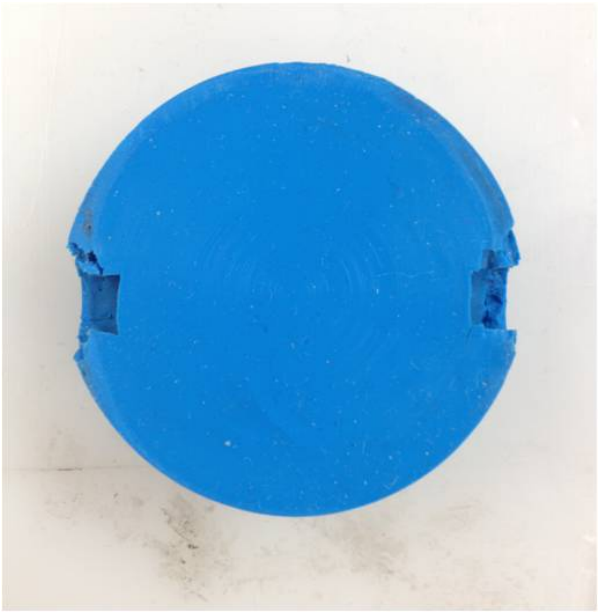


Figure 4.19: Silicon block with cut outs near inlet and outlet

Visual Inspection

On demoulding the setup, the first observation was the resin leakage at the lower o-ring. As compared to

the last test, the o-ring had no damage but slight resin deposition. There was no fiber flow-out near the edges of the laminate. However, the surface had a prominent wrinkle in the center of the laminate (Figure 4.20).

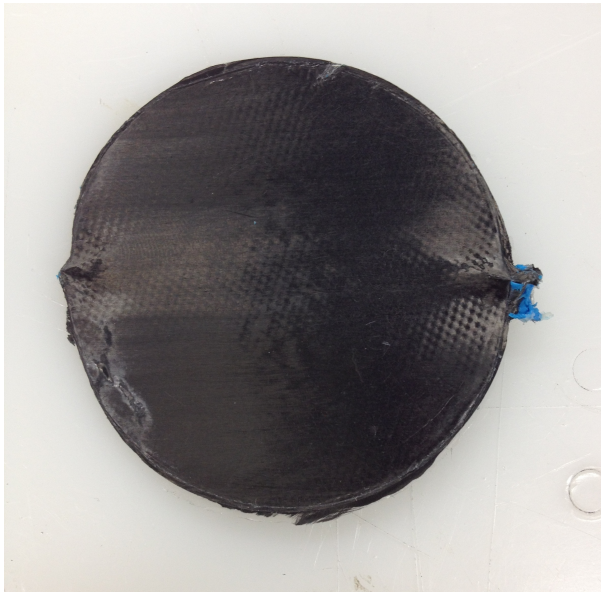


Figure 4.20: Test panel 4.4: Top View

Thickness Measurement

As the panel had rough surface and wrinkles, the measurement was taken at different sections of the laminate surface. The thickness varied in the range : 1.08-1.37mm. The maximum thickness being in the mid section. This is also represented in the Figure 4.21.

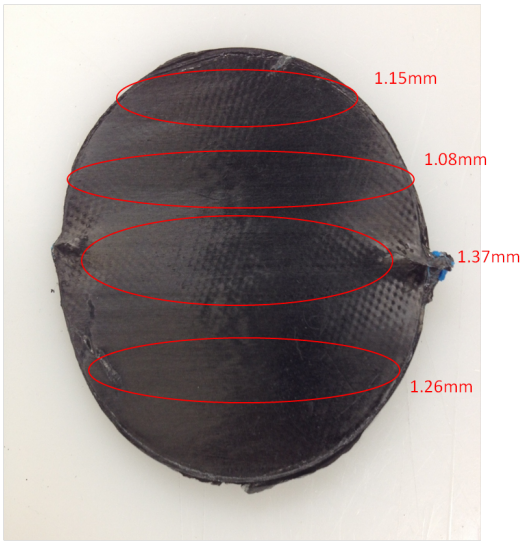


Figure 4.21: Thickness variation for test sample 5.4

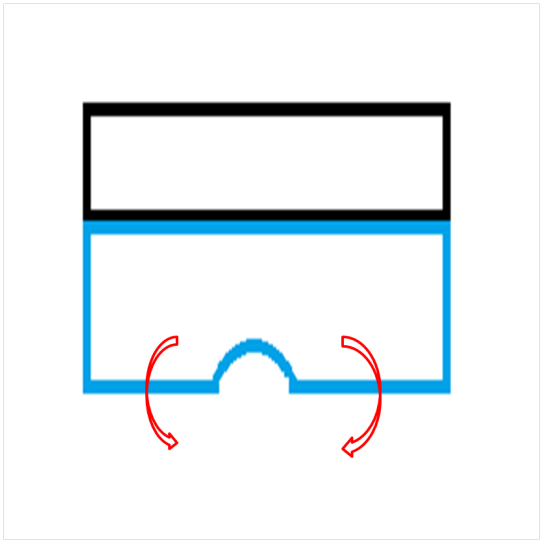


Figure 4.22: Schematic representation of deformation in silicon block

The cavities created in the silicon block resulted in inconsistent pressure buildup. The silicon block deformed along the axis of the cutout resulting in less pressure in the mid section. This led to the laminate take the shape created by the axial deformation of silicon block. This resulted in the formation of wrinkle in the middle of the laminate with maximum thickness of 1.37 mm. The bending in the silicon block is also represented in Figure 4.22.

Test Analysis

Even though the flow-out was reduced in this test, surface waviness and silicon creep in the tubing pose

a major challenge for further testing. Retests were suggested by making changes to the solid-flexible tool to avoid the persisting problems of creep in the infusion tube.

4.2.4. Test Procedure 4.5

The test was repeated by casting a new silicon block and inserting a metal ring boundary around the lower edge to prevent silicon creep. All testing conditions were kept similar. The new solid-flexible configuration is shown in the Figure 4.23 .

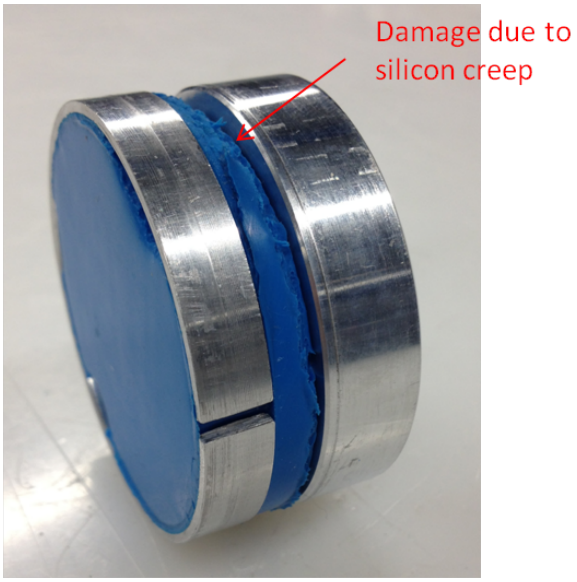


Figure 4.23: Aluminum ring at lower edge

Test Analysis

The test sample manufactured after this retest had minimum fiber flow-out around the edges (Figure 4.24). However, the silicon block got damaged in the midsection because of creep as can be seen in the Figure 4.23 above.

Figure 4.24: Test Panel 4.5: no fiber flow-out

To optimize solid-flexible tool configuration few trials were conducted by cutting the aluminum ring to

allow easy expansion of silicon rubber during the process (Figure 4.23). Even with this change the silicon creep was not contained. At this point, certain changes were made to the tool configuration as explained below.

A cavity of 2 mm depth was milled into the bottom surface of the solid-solid tool which would in turn accommodate a silicon sheet of thickness 2 mm. The silicon sheet was embedded in such a way that a flush surface is obtained with the bottom surface of the solid-solid tool as shown in Figure 4.25. The silicon sheet had the same hardness as the silicon rubber used in the previous configuration. This concept had no creep and did not result in blockage of the inlet and outlet infusion channels during the process.

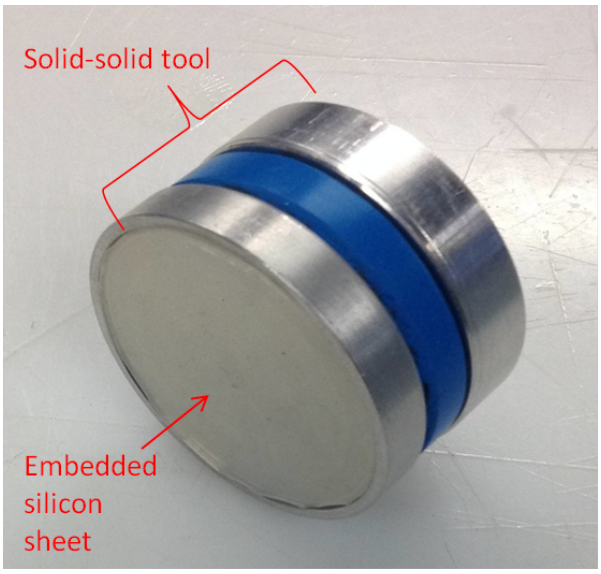


Figure 4.25: Solid-flexible tool with embedded silicon sheet

Another problem identified at this stage was the deformability of the POM bed base. In the first few tests, the laminate had significant fiber flow-out around the edges. This was partly due to the expansion ability of the POM bed which allowed the fibers to creep between the edges of laminate bed and inner metal ring of the mid assembly (Figure 4.27). The laminate bed material was changed to aluminum to improve the surface quality, to mitigate the flow-out problem and also to overcome the issue of deformability of the laminate bed base (refer Figure 4.26).

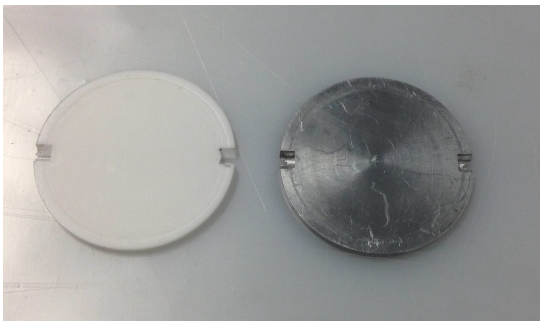


Figure 4.26: Changed aluminum bed base

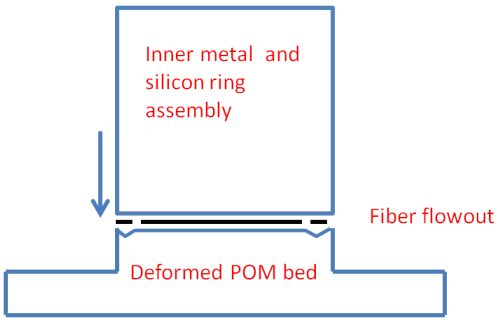
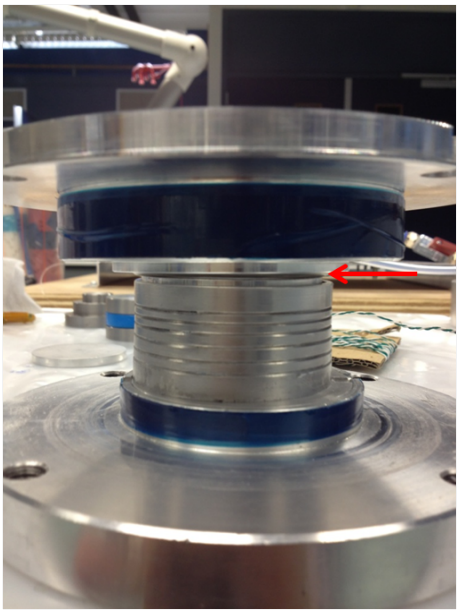


Figure 4.27: Schematic representation of fiber flow-out due to POM bed base

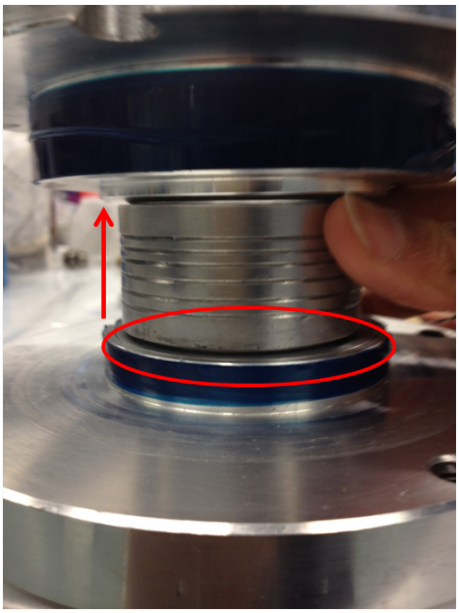
4.2.5. Filler Disc Anomaly

As described in the previous tests aluminum filler disc was used between the top lid and solid-solid/flexible tool to provide the consolidation pressure. However, the tests carried out with that setup still resulted in fiber flow-out around the edges. This was due to the fact that the inner aluminum ring had tendency to move in vertical direction because the pressure from the top lid was active directly on the filler material instead of the inner aluminum ring during the process. This allowed the fibers to escape under the metal ring. The

misalignment is simulated in the figure below.



(a) Pressure on filler disc



(b) Tool movement during process creating gap in the lower edge

Figure 4.28: Simulation of misalignment in the setup

This misalignment was corrected by removing the filler disc from the assembly and changing the inner metal ring with a new stainless steel ring with increased height (Figure 4.29). To fit the lid in the setup the design was modified by machining a 3 mm groove as shown in the Figure 4.30. In this arrangement the pressure from the top lid is transferred directly to the inner metal ring restricting any movement in the ring thereby controlling the flow-out of the fibers. The solid-solid and solid-flexible tool configurations were also changed to stainless steel material to avoid any thermal mismatch in the setup. The height of solid-solid and solid-flexible tool was increased to 30 mm which was previously 28 mm so as to compensate the removal of filler disc. An overview of change in tool parts is also shown in Table 4.3.

Figure 4.29: Stainless steel inner metal ring

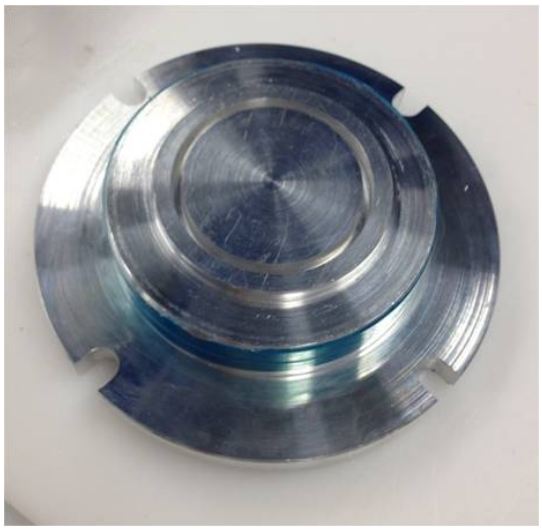


Figure 4.30: Integrated groove design

Because of the changes made in the design of the setup it was necessary to conduct another compression test to know the pressure buildup inside the setup. As described before, the required force of 6 bar pressure is 1500N (1.5kN). Therefore it was important to do the compression test with 1500 N as starting force. The test was carried out in a compression bench. The temperature was increased from 20 °C to 71 °C and was

recorded with thermocouple monitor. Figure 4.31 shows the variation of force build up with time. As can be seen at 65 °C, the force was 3977 N which corresponds to 17.4 Bar pressure. This test confirms that pressure buildup during the heat up due to expansion of silicon ring in solid-solid tool inside the setup was too high as compared to 1500 N which is the requirement throughout the process.

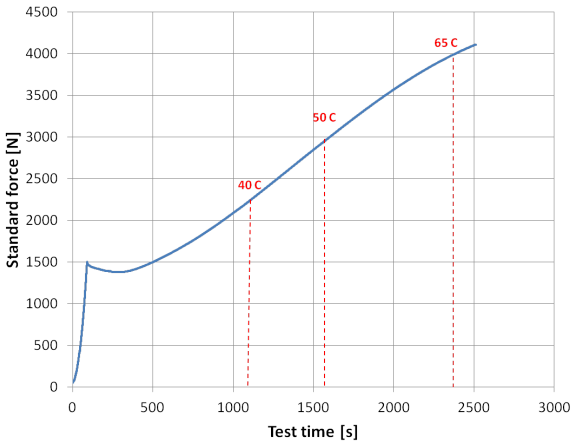


Figure 4.31: Compression test at increased temperature

Another challenge was the default starting force of 5kN for the Joos Press setup in DASML which is again too high for the requirement of this test (1.5kN). Therefore, to control the force given to the setup it was decided to use external spring on top of the tool and stands around the setup as shown in the Figure 4.32.

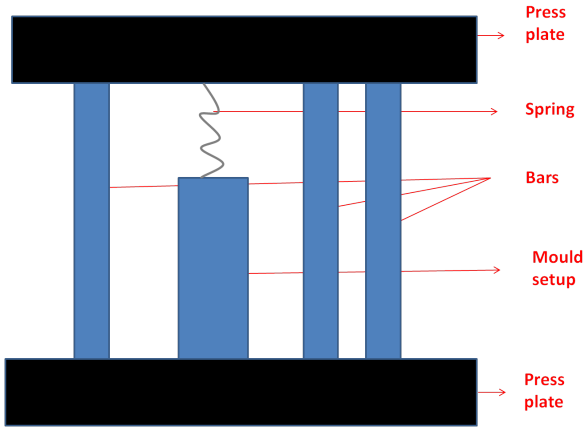


Figure 4.32: Schematic representation of spring arrangement in the press

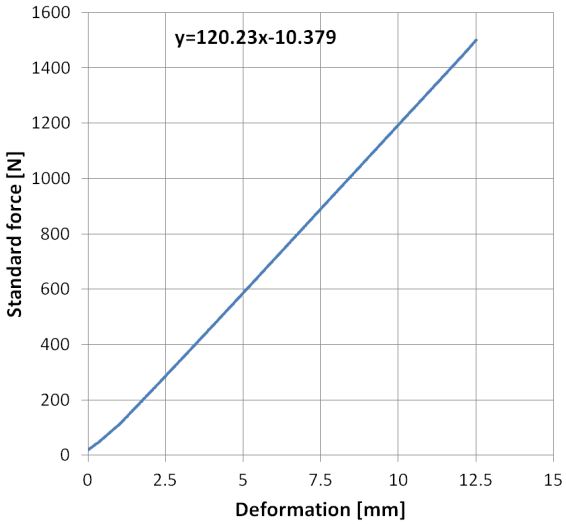


Figure 4.33: Compression test for spring setup

The total height of the tool setup with the spring attachment was measured as 322.8 mm. The spring setup was tested in the compression bench to get a maximum displacement in the spring that can give 6 bar pressure on the laminate. The spring displacement measured for the required pressure was 12.5 mm (Figure 4.33). This displacement in the spring is controlled by three stainless steel bars surrounding the setup. That means when the press plates are closed with a force of 10 KN, this load is taken by the stands which are 310.3 mm high which in turn compresses the spring to 12.5 mm, resulting in 6 bar pressure on the laminate.

4.3. Concluding Remarks

By the end of phase-1, the tool setup was optimized by containing the issue of any fiber flow-out in the laminate which was caused by tooling anomalies. The solid-flexible tool configuration was changed by embedding a silicon sheet in the tool to avoid any silicon creep in the infusion tubes. Since the pressure build up during the process cycle inside the tool was found out to be not consistent, spring setup was added to the tool to control the force transmitted to the system which in turn provided 6 bar pressure to the prepreg layers throughout the process.

4.4. Overview of Change in Tool Components

Table 4.3 provides an overview of the parts that were used in the experimental setup in Phase 1 and the changes made to them for the new setup which is described in Phase 2.

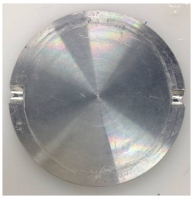
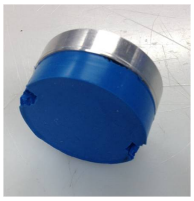
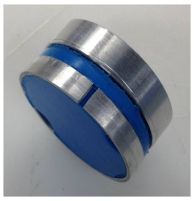
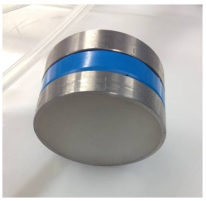
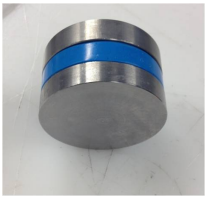
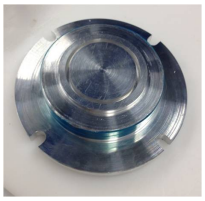
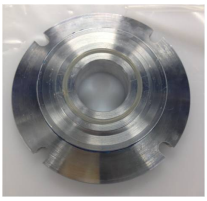
Part description and reason for change	**Original setup**	**Change in setup**
1.Laminate Bed (POM to aluminum) Fiber flow-out due to deformation in POM bed		
2. Inner Metal Ring (Aluminum to stainless steel) deformation in aluminum inner ring		
3. Solid-Flexible Tool due to silicon creep in the infusion tube, tool modified with aluminum ring at lower edge		
4. Solid-Flexible Tool due to damage in silicon block, configuration changed to embedded silicon sheet		
5. Solid-Solid Tool (Aluminum to stainless steel) changed to stronger material to avoid thermal mismatch		
6. Top Lid (integrated groove design to hollow center) to accommodate spring setup		

Table 4.3: Overview of old Vs new setup

5

New Tool Setup

5.1. Phase 2: Panels with New Tool Setup

In the second phase of testing, some major design changes were implemented in the experimental setup. The new working setup is shown in the Figure 5.1 with the parts described in the Table 5.1.

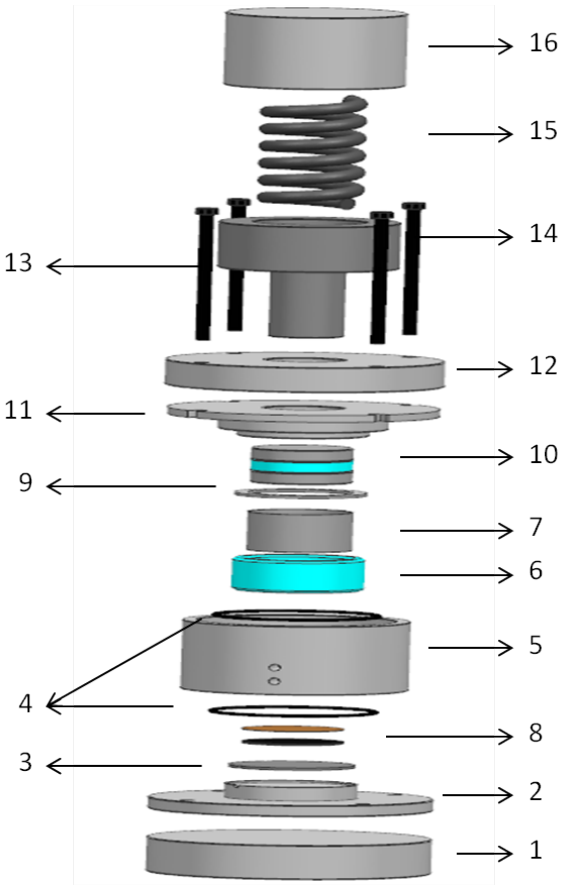


Figure 5.1: Exploded view of tool setup

Table 5.1: Part description for new setup

S.No	Part Descrption	Material	Quantity
1.	Base plate	Aluminum	1
2.	Laminate bed	Aluminum	1
3.	Laminate bed base	Aluminum	1
4.	O-ring seal	Viton	2
5.	Outer cylinder wall	Aluminum	1
6.	Inner silicon ring	Silastic M	1
7.	Inner metal ring	stainless steel	1
8.	Layup + teflon sheet	Carbon prepreg UD	10 layers
9.	Hollow ring	Aluminum	1
10.	solid-solid/solid-flexible tool	Stainless steel and silicon	1
11.	First lid	Aluminum	1
12.	Bolt lid	Aluminum	1
13.	Bolts	Stainless steel	4
14.	Piston	Stainless steel	1
15.	Spring	Stainless steel	1
16.	Spring Cap	Aluminum	1

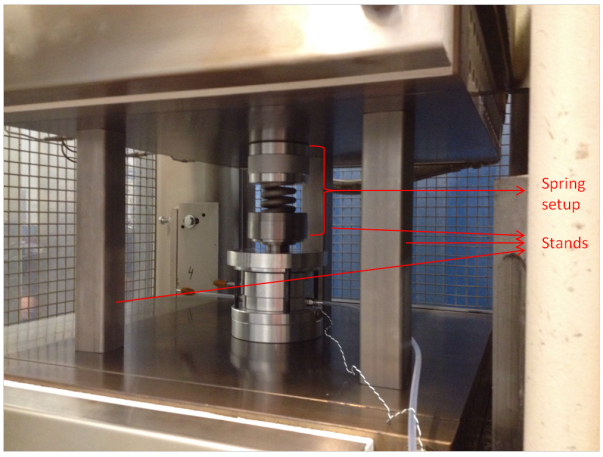


Figure 5.2: New experimental setup with external spring and stands

To make sure the setup is repeatable and the consolidated laminate quality is consistent, each tool configuration (solid-solid and solid-flexible) is tested with 5 repeatable trials with the same process conditions. The complete setup in the hot press is shown in Figure 5.2. After finishing the cure cycle in the press, the setup is demoulded and cleaned with acetone to remove any traces of cured resin. The consolidated laminate is visually inspected for any surface flaws, thickness measurement is done on several points of the laminate, strength of the laminate is tested with three point bending analysis and cross-sections are analyzed under optical microscope.

5.1.1. Visual Inspection

After completing each trial, the laminates were visually inspected for flaws like wrinkles on the surface and fiber slippage. With both solid-solid and solid-flexible configuration the surface of the laminate had no wrinkles, nor fiber flow-out was observed for all 10 trials conducted.

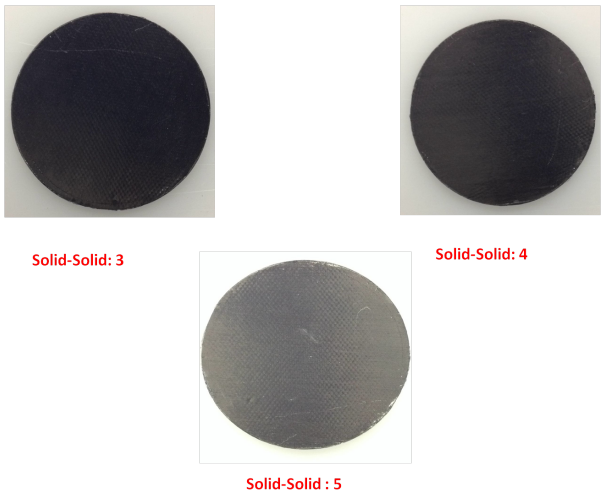


Figure 5.3: Surface finish: solid-solid configuration test panels

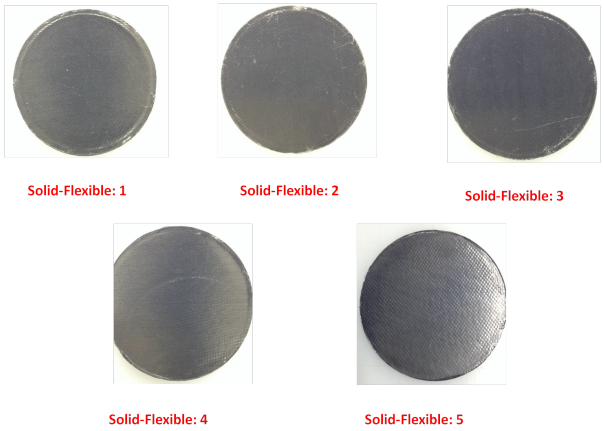


Figure 5.4: Surface finish: solid-flexible configuration test panels

5.1.2. Thickness Measurement

For precise measurement of laminate thickness a micrometer was used with accuracy of 0.01 mm. Each laminate was divided in several sections of 10×20 mm area. This was done to cut small samples for 3 point bending and microscopic analysis which is described in later sections of this chapter. To get a better understanding if there is any variation in the consolidated thickness, thickness was measured at 12 points all over the laminate.

The test laminates produced with both solid-solid and solid-flexible tool configuration were 54 mm diameter. However, the measuring diameter for solid-flexible test laminate was taken 49 mm. This was due to the fact that silicon sheet was embedded inside the aluminum cavity with a 2 mm edge around. So the effective measuring diameter in this case was taken which was consolidated under the silicon sheet surface. Figure 5.6 and 5.5 show the thickness measurement markings on the laminate surface. The arrows marked on the panels represent the direction of the fiber which help in analyzing the cross-section under microscope.

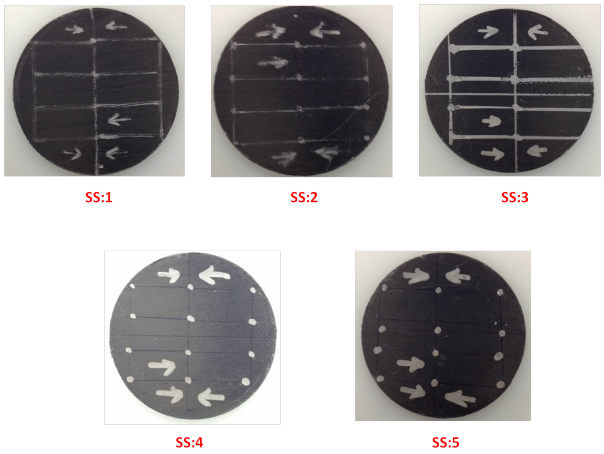


Figure 5.5: Thickness Measurement: solid-solid tool consolidated test laminates

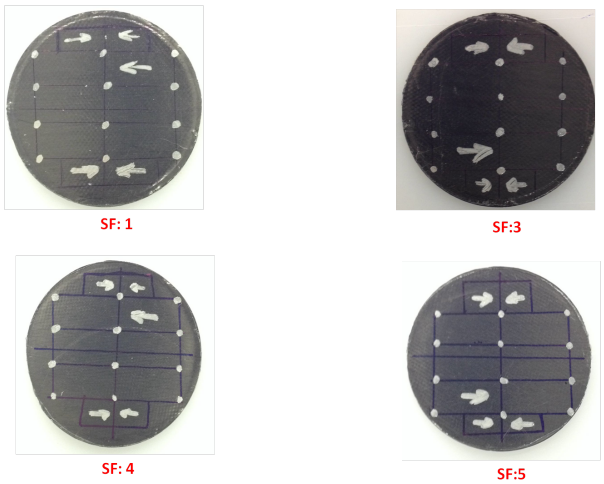


Figure 5.6: Thickness Measurement: solid-flexible consolidated test laminates

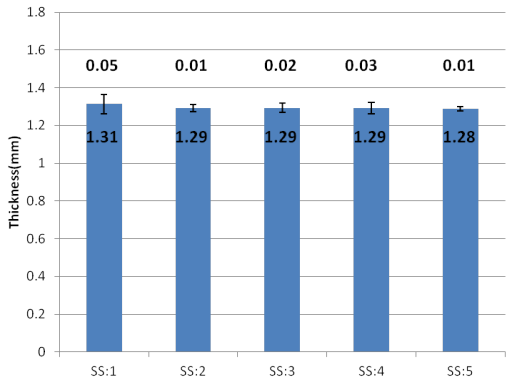


Figure 5.7: Thickness variation for solid-solid tool laminates

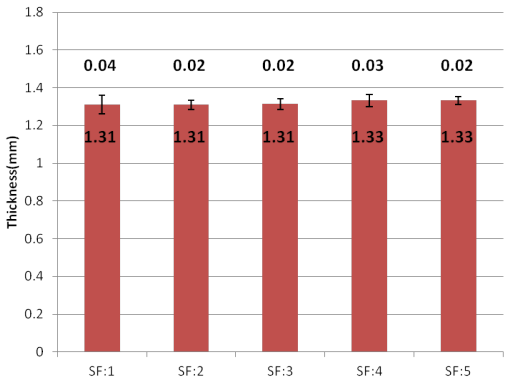


Figure 5.8: Thickness variation for solid-flexible tool laminates

Figure 5.7 and 5.8 show the variation of thickness for each laminate produced with solid-solid tool and solid-flexible tool respectively. In solid-solid tool group (Figure 5.7) the average thickness measured for all five laminates :1.28-1.31 mm (as represented inside the bars on the graph). The deviation of thickness from the averaged measurement within each sample is in the range 0.01-0.05 mm (represented above the bars in the graph) which is due to the impression left by the teflon sheet on the top surface of the laminate. In solid-flexible group the variation in thickness for each laminate is 0.02-0.04 mm and the average thickness in all five laminates measured in the range of 1.31-1.33 mm. This variation is partly due to the teflon sheet impression

and partly due to non uniform pressure distribution by the flexible silicon sheet surface in this setup. The overall comparison of thickness between solid-solid group and solid-flexible group is represented in Figure 5.9. Thickness for solid-solid group was averaged as 1.29 mm and for solid-flexible group as 1.32 mm. The comparison is based on average thickness as the variation is in the range of 0.01-0.03 mm. These results are compared with the preliminary test results (described in CH-3) for UD prepreg with 9°C and 1°C ramp cycle consolidated using Joos press and 1°C ramp cycle consolidated using autoclave which were done without any tooling setup.

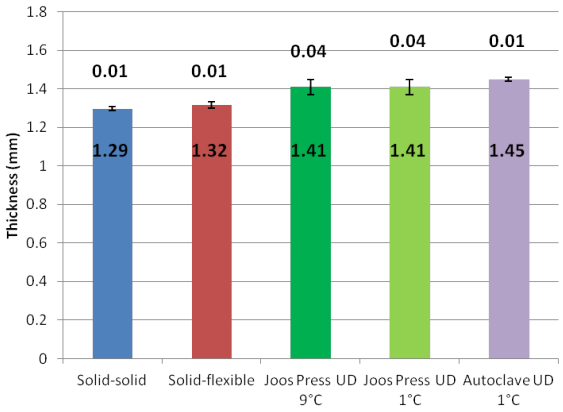


Figure 5.9: Comparison thickness variation solid-solid, solid-flexible panels and preliminary test results

5.1.3. Inter-laminar shear Test (3 point bending analysis)

After successfully manufacturing the composite laminates with solid-solid and solid-flexible tool without any fiber flow-out, the panels were tested mechanically under short beam strength analysis. Five test samples with dimensions 20 mm length ×10 mm width were taken from each laminate and tested with three point bending load setup as shown in Figure 5.10.

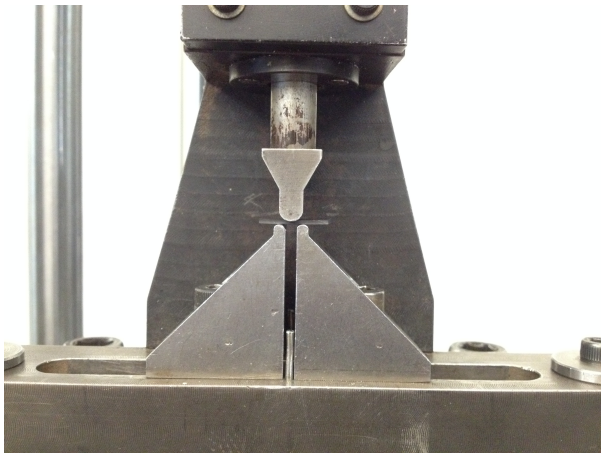


Figure 5.10: Three Point bending setup

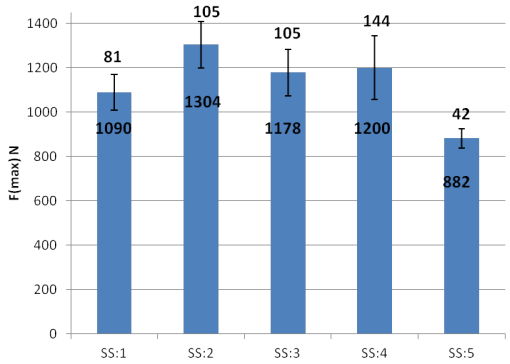


Figure 5.11: Strength Variation for solid-solid tool samples

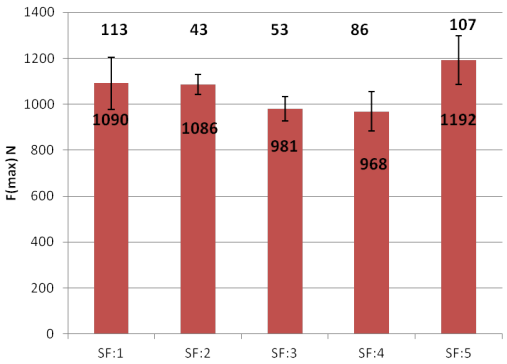


Figure 5.12: Strength variation for solid-flexible tool samples

Figure 5.11 and 5.12 show the variation of maximum failure strength in solid-solid samples and solid-flexible samples respectively. In solid-solid group the overall strength measured from SS:1-SS:5 was in the range 882-1304 N. The deviation in measurement from SS:1-SS:5 was in the range 42-144 N. Similarly, in solid-flexible group the overall strength was in the range 981-1192 N and the deviation in measurement: 43-113 N. One major reason for the variation of strength within both the tool groups was due to variation in cutting precise length and width of the samples with the diamond blade. Figure 5.13 compares overall strength variation in both groups. The average strength in solid-solid group was 1131 N and for solid-flexible group was 1064 N.

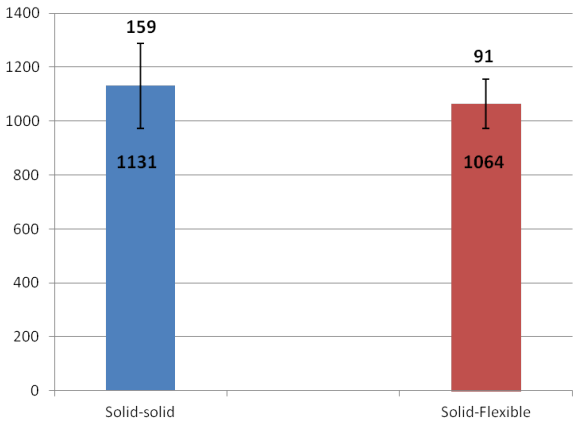


Figure 5.13: Comparison strength variation solid-solid and solid-flexible tool

5.1.4. Optical Microscopy

The samples for optical microscopy were prepared in similar manner as described in section 3.2.2. Samples were cut with diamond blade and cleaned in ethanol bath. The samples were embedded in the embedding resin followed by grinding and polishing to analyze the cross-sectional area under the optical microscope.

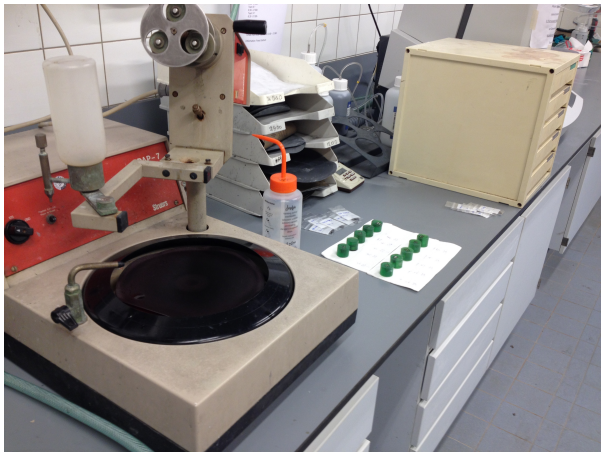


Figure 5.14: Sample Preparation

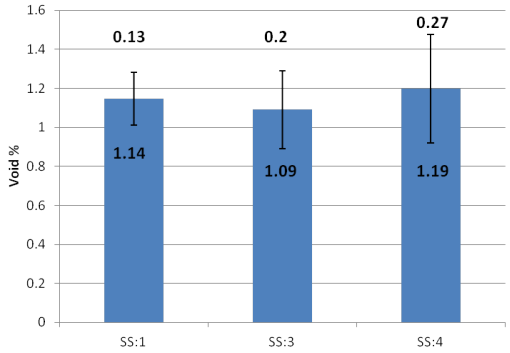


Figure 5.15: Void % of solid-solid tool samples

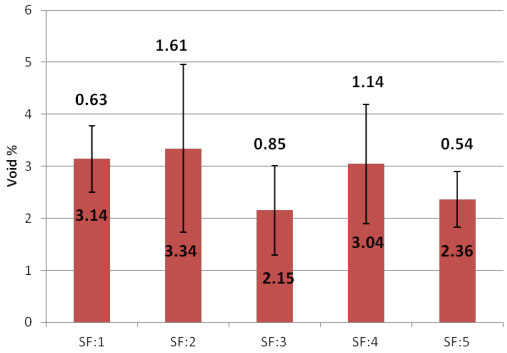


Figure 5.16: Void % of solid-flexible tool samples

Figure 5.15 and 5.16 show the variation of void % in solid-solid laminates and solid-flexible laminates respectively. Two of the samples from solid-solid group were not measurable under microscope due to damages made in the preparation process while embedding the samples in the resin. On comparing the void% for samples in both the tool groups, following trend was observed:

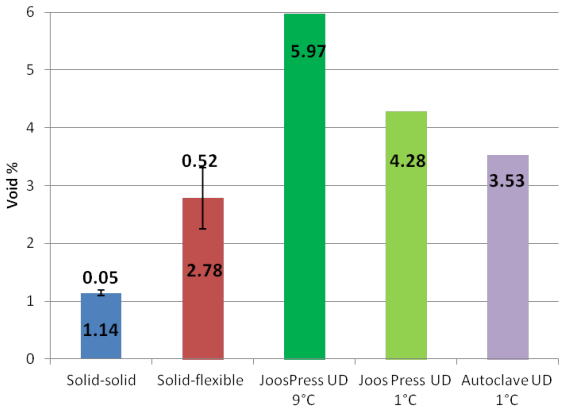
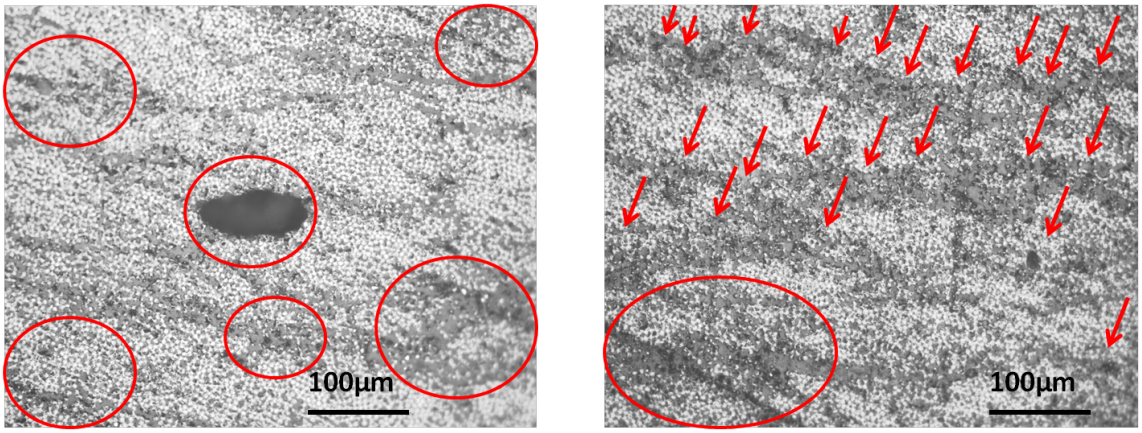


Figure 5.17: Comparison solid-solid and solid-flexible tool groups

Again this variation can be attributed to the pressure distribution by a solid surface and a flexible surface. As sample 2 and 5 from solid-solid group were not measurable under microscope, for effective comparative analysis equivalent samples from both groups were chosen. The comparative analysis is shown in Figure 5.17. From this data it can be concluded that solid-solid samples show lower average void content of 1.14% than solid-flexible samples with 2.78% void content. This is due to uniform pressure distribution delivered by the

solid-solid tool as compared to solid-flexible tool used in the current setup. Figure 5.17 also compares the void % with the preliminary tests results described in Ch-3. The void % in both solid-solid and solid-flexible group is much improved when compared with the preliminary-trial conducted on same UD sample without any tooling setup.

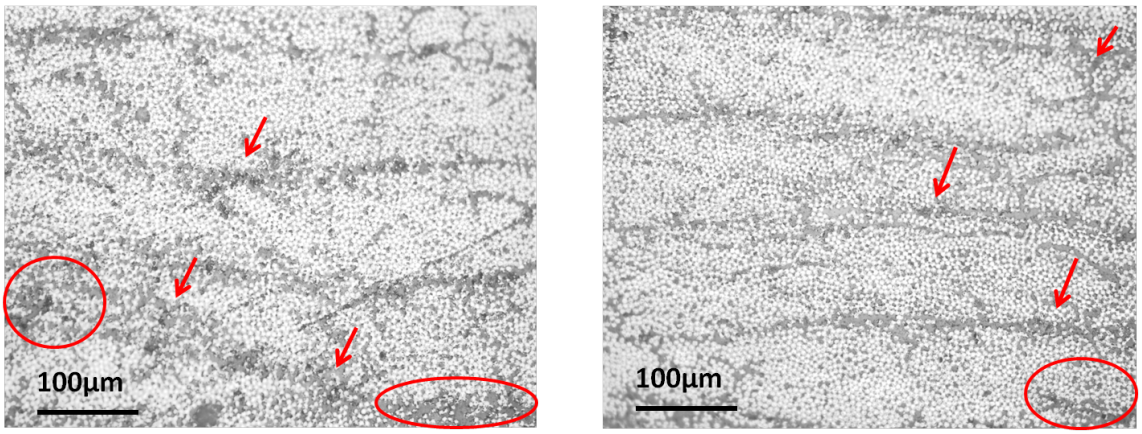
The cross-sectional images as captured by the microscopic camera of the samples are given below. Figure 5.18 show the cross-sectional analysis of sample 1 and sample 2 for solid-flexible tool. The cross-section shows voids scattered over the entire image area for both the samples. Whereas, in solid-solid tool samples (Figure 5.19) void scatter is minimal over the image area. This analysis gives an indication that the pressure distribution from solid-solid tool surface in the current setup is uniform throughout resulting in low void formation as compared to solid-flexible tool surface which has more void formation.



(a) Void scatter solid-flexible sample-1

(b) Void scatter solid-flexible sample-2

Figure 5.18: Cross-sectional overview for solid-flexible tool laminates with X20 magnification



(a) Void scatter solid-solid tool sample-1

(b) Void scatter solid-solid tool sample-3

Figure 5.19: Cross-sectional analysis for solid-solid tool laminates with X20 magnification

5.2. Discussion and Conclusion

In phase 3 tests were conducted with new tool setup as shown in Figure 5.1. The panels produced with this setup showed no fiber flow-out throughout the 10 tests conducted with both solid-solid and solid-flexible tool. The panels were inspected for thickness variation, short beam strength analysis and cross-sectional analysis. On comparing solid-solid and solid-flexible group the following data was observed.

Table 5.2: Phase 2 Results

Test	**Solid-Solid**	**Solid-Flexible**
Thickness	1.29 mm	1.32 mm
Shear strength	1131 N	1064 N
Void %	1.14	2.78

The above results are based on average values from both solid-solid and solid-flexible tool configurations. From table 5.2 it was observed that void% (V) is proportional to thickness (T) of the sample and inversely proportional to shear strength (S) of the sample.

Both the tooling configurations were subjected to similar temperature, pressure and closing force. The only difference being in the contacting surface to the prepreg layers. Under temperature and pressure the flexible silicon surface distributes pressure differently than solid surface. This results in a small difference of thickness in solid-flexible panels than solid-solid panels. The relative decrease in thickness in solid-solid panels with respect to solid-flexible panels was 2.27%. The void % was observed to decrease by 58.99% which resulted in the increase of shear strength, in this case by 6.29%. Therefore, it can be concluded that panels produced with solid-flexible tool group had more thickness than solid-solid tool group, resulting in higher void content and lower shear strength values.

6

Phase 3- Infusion Trial

This chapter describes the last phase of the project which involves infusing resin in the prepreg layers. This technique was basically proposed to reduce the void content in the laminate. The acceptable void percentage in aerospace application as described in the literature is <2%[3]. As discussed in the previous chapter, the consolidated prepreg laminates show maximum void content in the range 1.19% to 3.34% in solid-solid and solid-flexible tool samples respectively. Therefore, in order to reduce void percentage even further during manufacturing this hybrid technique was proposed.

Void formation during the process can be primarily due to the air entrapped in the layup, moisture and volatile dissolved in the resin[9,17]. The idea behind this test was to infuse the resin at high pressure in the prepreg layers so that the resin can fill any voids that might form during the curing of the prepreg layers[5]. For this procedure one key requirement was the nature of resin in the prepreg and the liquid resin. Having the same type of resin system eliminates any compatibility issues and improves laminate quality[5].

The prepreg material used throughout this project was provided by Tencate Advanced Composites USA which is no longer in production. Therefore, to get the similar liquid resin was not possible. After consulting Tencate about the type of resin that can be used with this prepreg material, it was decided to use a low viscosity two part epoxy resin system.

Epikote resin 04908 was a readily available two part epoxy resin system in the DASML used for infusion purposes. However, this resin system had a different chemical nature than the resin of the prepreg. Using different resin systems might not give the required chemical compatibility and result in weak mechanical properties of the laminate produced. Therefore, both carbon prepreg and liquid epoxy resin were characterized physically to get a better understanding of the nature of the resin in each system.

6.1. Differential Scanning Calorimetry (DSC) Test

The Epikote resin 04908 (liquid resin) is generally used for infusion purposes and curing at room temperature. The complete material description is provided in Appendix A. The idea was to infuse the liquid resin at the same temperature when the prepreg resin becomes viscous which is at 71 °C (Figure 3.2) so that both resin systems can become miscible and cure at the same temperature. Therefore, it was important to conduct DSC test to know how the increased temperature affects the curing of the liquid resin. The Differential Scanning Calorimetry experiment determines the heat flow associated with phase transition as a function of temperature. Both prepreg and resin samples were tested for comparative analysis.

The DSC test for prepreg was performed with the same temperature profile as in phase 2. The heat generation versus temperature graph obtained is given in the 6.1.

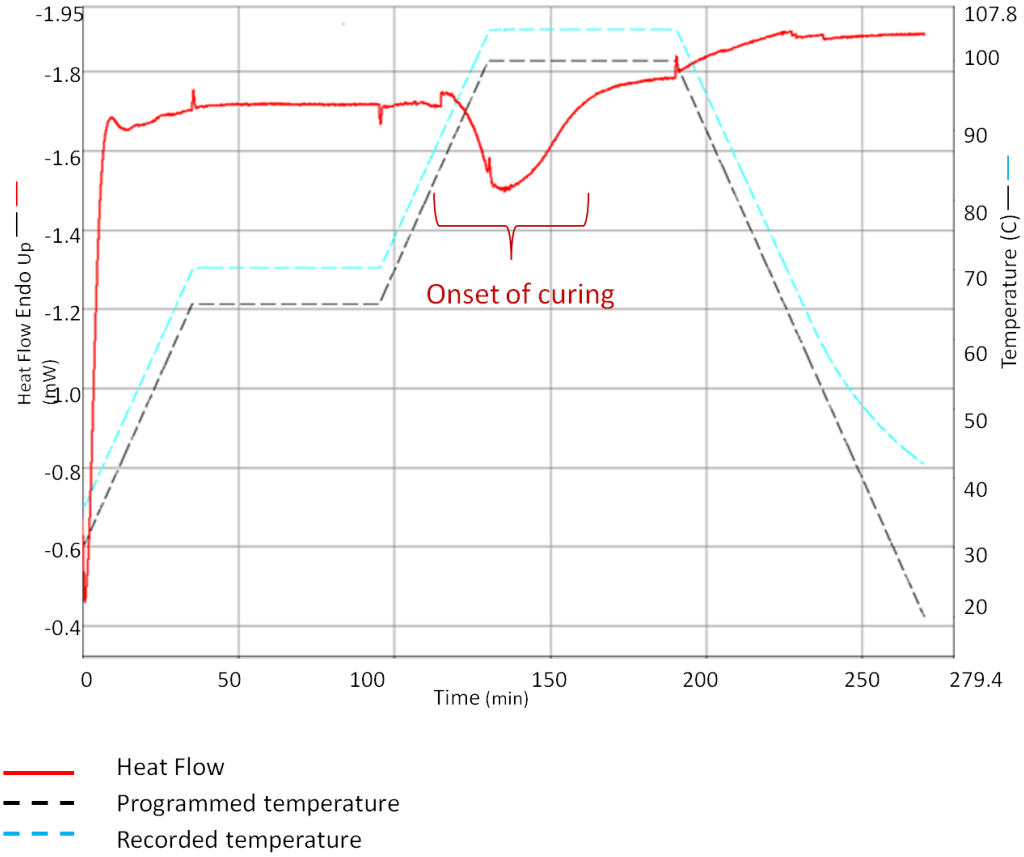


Figure 6.1: Heat flow curve for carbon prepreg sample

Since the resin used has room temperature application, it was decided to conduct the test lower than 71 °C to find out the optimum temperature for infusion. Therefore, the DSC test for Epikote resin was performed from 55 °C to 100 °C with a rate of 1 °C/min and hold time of 120 mins at 100 °C. The test was conducted with same ramp up temperature conditions as for the prepreg material to simulate the actual testing conditions. The heat flow curve is represented in Figure 6.2.

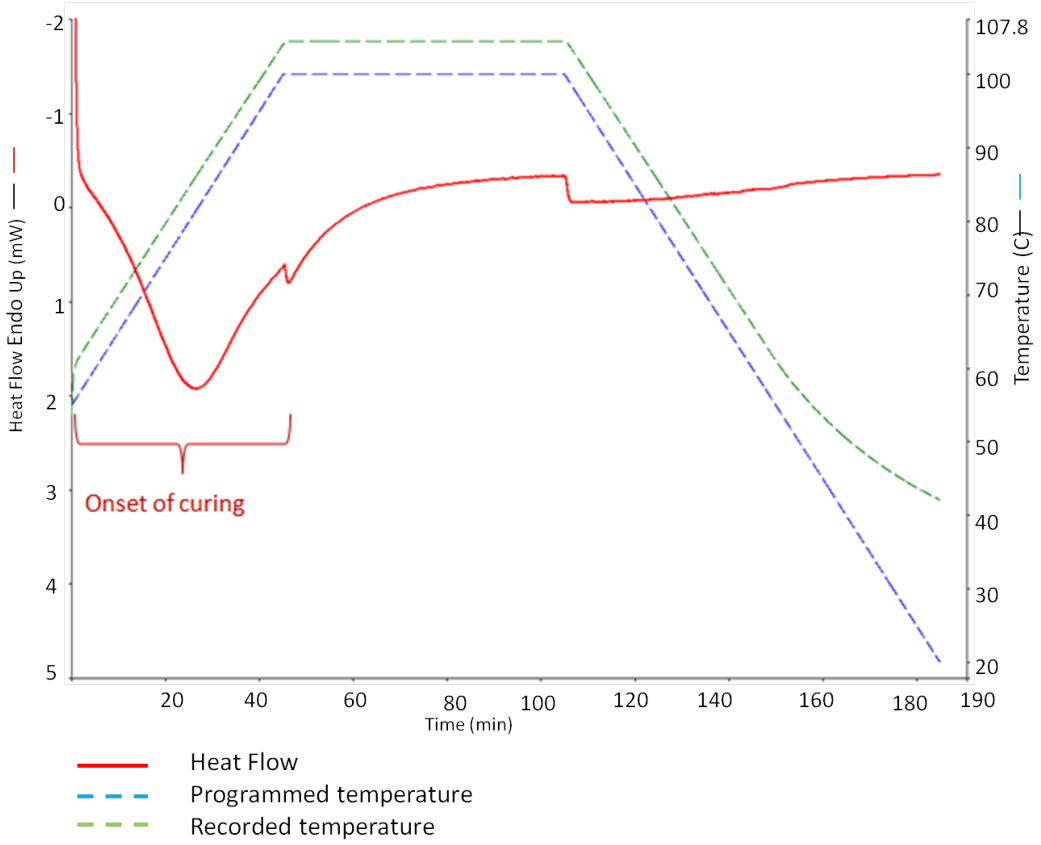


Figure 6.2: Heat flow curve for Epikote resin sample

6.1.1. DSC Test Analysis

The above graphs represent the heat flow of the prepreg and resin sample recorded with respect to time and temperature. It can be observed in Figure 6.1 the onset of curing starts from 90 °C which is seen as a V-shape in the curve. Whereas for liquid resin the curing starts from 65 °C. This indicates that liquid resin cures at a lower temperature than the prepreg. The test also confirms that the resin infusion at 71 °C might result in immediate curing of the resin because of high temperature. Therefore, it was decided to lower the temperature of resin infusion from 71 to 60°C for the actual infusion trial to avoid curing of resin as soon as the resin is infused and also keeping the infusion temperature close to the viscous temperature range of the prepreg resin.

6.2. Fourier Transform Infrared Spectroscopy (FTIR)

The principle of FTIR analysis relies on the fact that most molecules absorb light in the infrared region of the electromagnetic spectrum of light. The absorption results in change in vibration and rotation of the molecules. The vibrational frequency of the molecules determines the absorption of the molecule bonds [1,6]. For the present case, the liquid resin and carbon prepreg are analysed in FTIR analysis. The absorption pattern will reveal the molecules present in both systems .

The epoxy resin used in this phase was Epikote Resin 04908 and Epikure curing agent 04908 which is a two part resin system. The complete details and properties are given in the Appendix A. The test sample: 5 mm prepreg samples and 5 mg epoxy resin were prepared in a small aluminum pan and placed in the Infrared testing machine. The absorption pattern of prepreg and epoxy resin is given in the Figure 6.3 below.

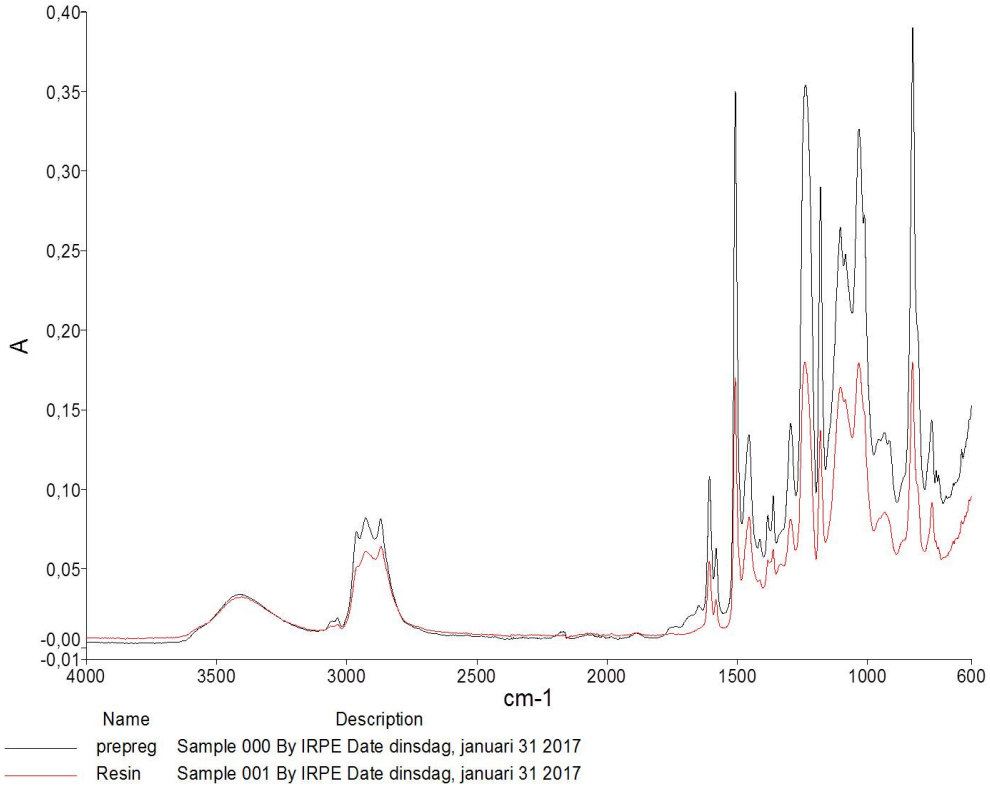


Figure 6.3: FTIR Analysis for prepreg and epoxy resin

The figure shows infrared spectrums of prepreg in black and Epikote epoxy resin in red. The overlapping spectrum of prepreg and resin gives the indication of groups thats are present or absent in both systems. Each group of molecules gives rise to a particular absorption band in the spectrum due to vibration of the molecules. The major absorption bands in the observed spectrum : 3000-3500 cm^{-1} hydroxyl group or amino group vibration, 3000-2500 cm^{-1} aromatic group vibration and 2000-600 cm^{-1} corresponds to Acetylenic groups[6].

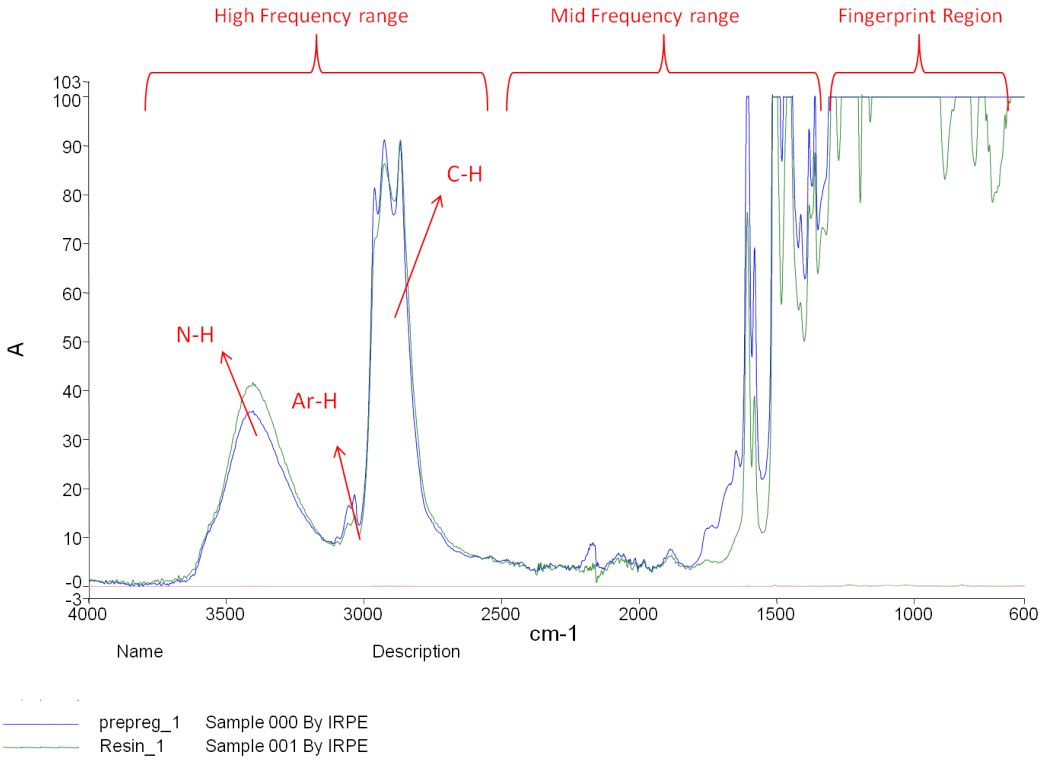


Figure 6.4: Zoomed in view of the absorption peaks

A zoomed in view of the spectrum is shown in the Figure 6.4 with prepreg spectrum in blue and Epikote resin spectrum in green. The spectrum is analyzed in three broad ranges 3500-2500 cm^{-1} which is high frequency range, 2500-1600 cm^{-1} mid frequency range and 1500-600 cm^{-1} low frequency range as major peak were observed here.

6.2.1. High Frequency Range

Both prepreg and epoxy resin show similar absorption patterns in the high frequency range at 3400cm^{-1}. This corresponds to the vibration of -OH atoms representing presence of water molecules. Also presence of primary amine -NH group can be suspected which also falls in the 3400 cm^{-1} frequency range.

Absorption band at 3000 cm^{-1} frequency corresponds to the presence of Aromatic ring vibration for both prepreg and resin.

The frequency encountered below 3000 cm^{-1} and above 2500 cm^{-1} generally corresponds to skeletal vibrations. In this particular case, the absorption between 2970-2930cm^{-1} indicates the -CH vibration of methyl and methylene group [6].

6.2.2. Mid Frequency Range and Fingerprint Region

In the mid frequency range and fingerprint region the peaks have same absorption frequencies but stronger intensity for carbon prepreg as it can absorb more incident light.

6.2.3. FTIR Test Analysis

Overall the two spectrums represent very similar absorption peaks with some difference in absorption intensities for carbon prepreg and liquid resin.

6.3. Infusion Trial

From this point onwards the the mould setup used in Phase-2 will be referred as System A and the infusion chamber as System B as shown in Figure 6.5 for the convenience of the reader.

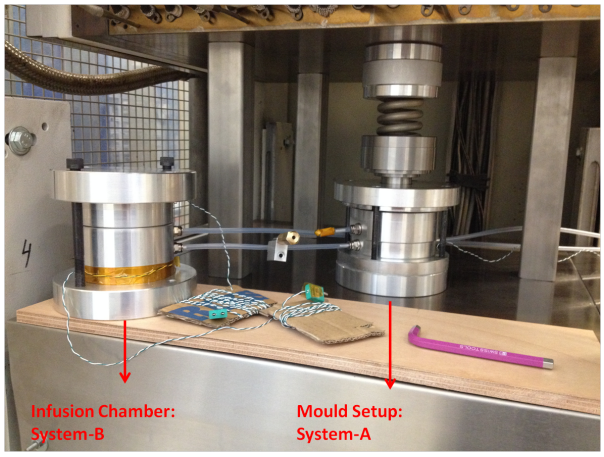


Figure 6.5: Infusion trial setup in Joos Press

6.3.1. Setup and Process

Based on the DSC test conducted on both prepreg and liquid resin it was decided to infuse the resin at 60 °C. The cure profile used for this test is shown in the Figure 6.7. For carrying out the infusion process the same experimental setup (System A) was used as in Phase-2 with the addition of the infusion chamber (System B). System B has two ports: port [2] for pressurizing the resin inside the chamber and port [3] for resin outlet which connects to the resin inlet of System A as shown in Figure 6.6.

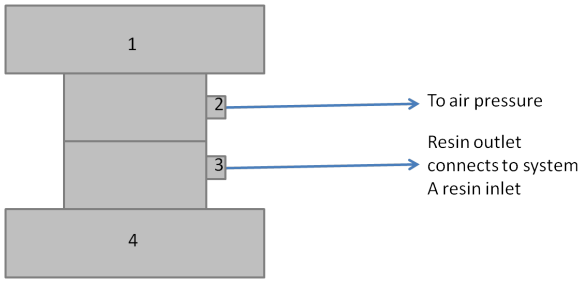


Figure 6.6: Schematic representation of infusion chamber (SystemB)

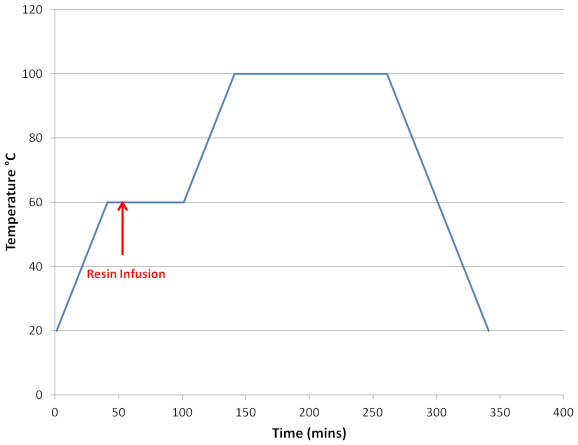
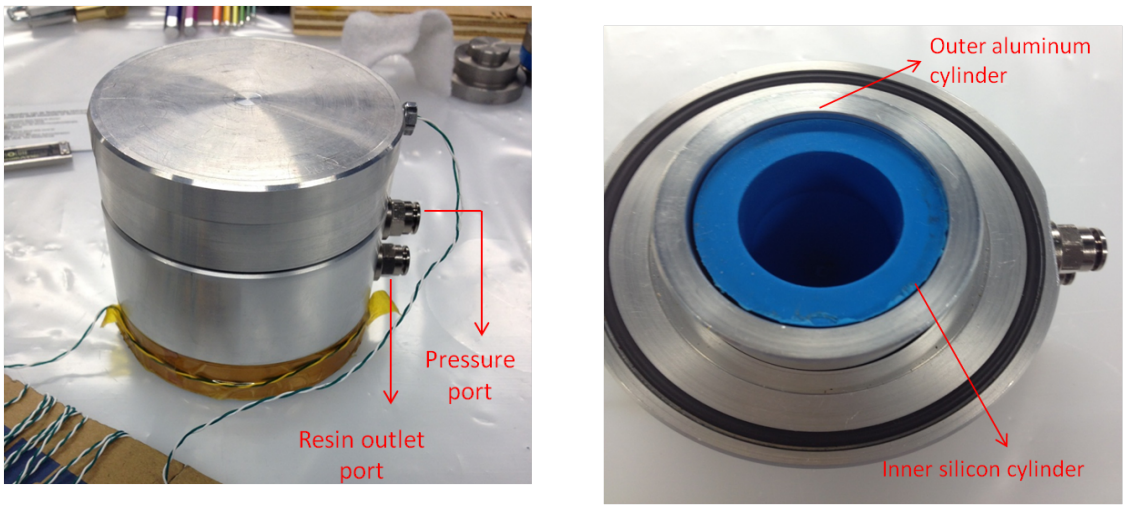


Figure 6.7: Cure cycle used for infusion trial

The inside of the chamber has two cylinders. Outer cylinder made of aluminum and inner with silicon. The inner cylinder contains the resin and prevents any resin spillage to the outer cylinder during the process, see Figure 6.8a and 6.8b.This arrangement is encapsulated between the top lid [1] and base plate [4] (Figure 6.6). The complete setup for infusion test is shown in the Figure 6.5



(a) Infusion chamber with upperpressure port and resin outlet port

(b) Inner chamber

Figure 6.8: Infusion Chamber

As can be seen from Figure 6.5, system A was positioned inside the press plates and system B was placed on a wooden board outside the press. Since the resin would be infused at room temperature the wooden board serves as an insulator protecting system B from the adjacent hot plates of the press. The infusion chamber (System B) and the laminate setup (System A) were connected with infusion lines. The infusion line connecting system A and system B was clamped during the first temperature ramp up of the cure cycle to get a complete vacuum 5 mbar in system A and to avoid resin leakage in the system. The mixed resin was brought in the infusion chamber (System B) and was secured with bolts. The chamber was slowly pressurized by connecting the pressure port to compressed air supply. When the temperature in system A reaches 60 °C the system is allowed to stabilize for 10 minutes before the clamp was opened for the resin to flow. The infusion chamber was disconnected after the infusion of resin and the laminate was consolidated with the programmed temperature cycle.

6.4. Issues during trial

- **Infusion line clamping**
  As described in previous section the infusion line was closed tightly with a clamp to make sure that system A was under vacuum and had no infiltration of resin from system B. Since the infusion tube was tightly secured with the clamp, on releasing it the infusion tube at that point was severely deformed due to the clamping force and this prevented the resin to have a smooth and easy flow into system A (Figure 6.9).

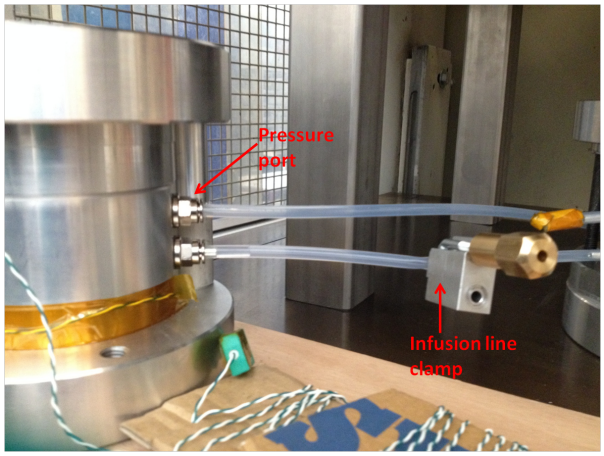


Figure 6.9: Infusion line clamping

- **Resin curing**

  On demoulding system A cured resin traces were found around the inner cylinder wall and silicon ring for both trials conducted with solid-solid and solid-flexible tool as shown in Figure 6.10. The laminate itself showed cured resin around the inlet edge. This suggests that even though the resin was infused in system A, the resin cured as soon as it touched the heated aluminum cylinder wall. The resin was infused when the system A reached 60 °C. As system A has a closed environment, the pressure and temperature conditions inside speeds up the curing of liquid resin.

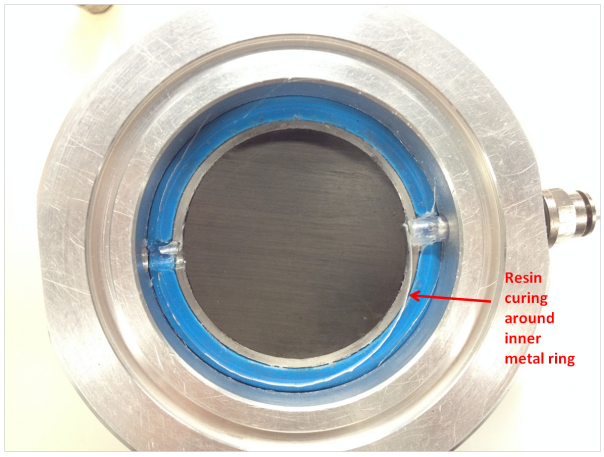


Figure 6.10: Demoulding after process completion

6.5. Results

The trial was conducted with both solid-solid and solid-flexible tooling. The laminates produced were inspected visually and under optical microscope for cross-sectional analysis.

- **Thickness Measurement**

  The laminates showed no fiber flow-out and surface variation except for the resin curing around the edges of the laminates. The average thickness for laminate produced with solid-solid and solid-flexible tooling measured 1.37 mm and 1.35 mm respectively. The thickness measurement data is represented in Figure 6.12

Figure 6.11: Surface quality solid-solid tool panel

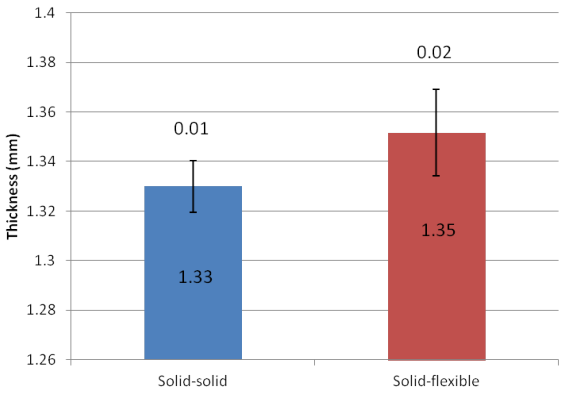


Figure 6.12: Thickness variation in solid-solid and solid-flexible infusion panels

- **Optical Microscopy**

  Three samples were prepared from each laminate for cross sectional analysis under microscope. Figure 6.14 show the void scatter (circled in red) in the cross section for solid-flexible (Figure 6.14a) and solid-solid laminate (Figure 6.14b). The void percentage measured in solid-flexible sample: 3.29 % and for solid-solid sample : 1.59 %. The variation in measurement is shown in Figure 6.13.

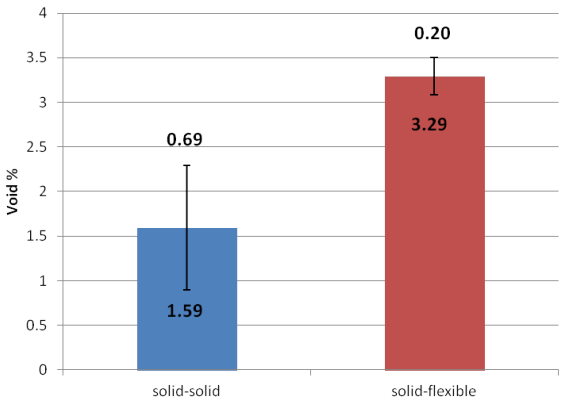
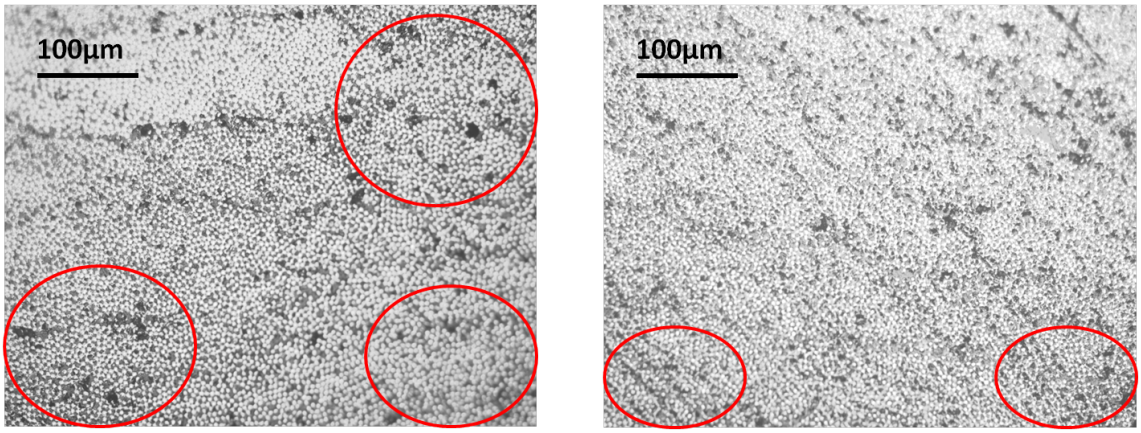


Figure 6.13: Variation in void % for solid-solid and solid-flexible infusion samples



(a) Void scatter for solid-flexible sample

(b) Void scatter for solid-solid sample

Figure 6.14: Cross sectional analysis for infusion trial samples X20 magnification

The void scatter in case of solid-solid sample is much less as compared to samples produced in Phase 2. But it cannot be constructively concluded the low void content was due to infusion of resin as the resin cured much earlier than prepreg layers. This is also described in section 6.4. Similarly in case of solid-flexible sample the void content was measured around 3.29%. This was due to the air entrapped in the infusion tubes which escaped the tube first before the resin flow, while opening the clamp during the first infusion trial.

6.6. Concluding Remarks

The purpose of conducting the infusion trial was to get an understanding how this technique can be used with the current closed mould tool setup. Right from the conceptual stage of the project both system A and system B were designed with the basic understanding gained from the literature study. However, with the progress of the project, changes were made in the setup not only to optimize the setup but also to accommodate different testing limitations. Therefore, some issues were encountered while conducting this trial. Both system A and system B were built in-house keeping in consideration the requirement of the project and the available testing conditions.

However, not all conditions were satisfied which resulted in a "Successful Failure" of Phase-3. As can be seen in the FTIR analysis of prepreg and epoxy resin, the chemical nature of prepreg resin and liquid resin was very similar which was a success in this case. However, the different curing temperatures of prepreg resin and epoxy resin led to failure of actual infusion trial. The desired intension of resin infusion to create a pressurized boundary and reducing any voids or porosity was not achieved owing to issues during the trial.

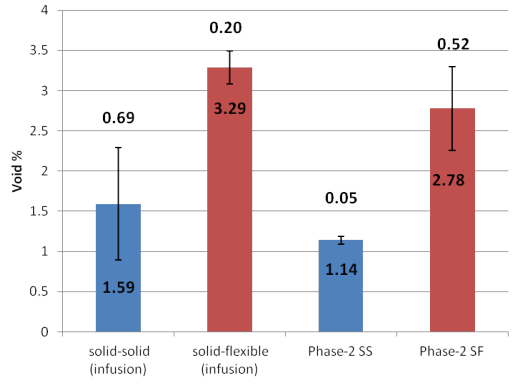


Figure 6.15: Comparison of Void % with phase-2 results

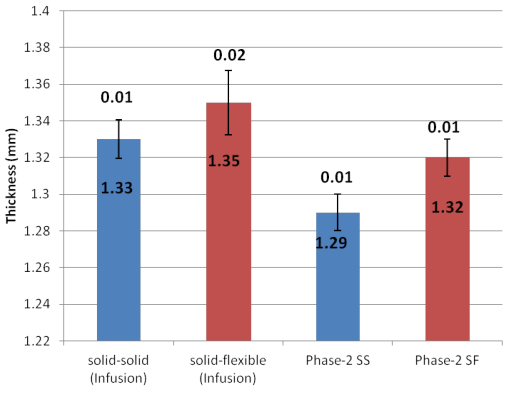


Figure 6.16: Comparison of thickness measurement with phase-2 results

Comparing the thickness of both solid-solid and solid-flexible samples, there is 0.02 mm difference in thickness measurement obtained through infusion process. The void% is higher in solid-flexible sample than

solid-solid sample. However, the void % in both samples as compared to the Phase-2 samples was not improved and found to be elevated. The results are shown in figure 6.15 and figure 6.16.

Conducting more tests at lower infusion temperature does not serve the purpose of the concept as both resin systems have different curing temperatures and cannot combine chemically. Therefore, for improving the proposed concept of prepreg- resin infusion, same cure temperature conditions are required for both liquid resin and prepreg so that both can cure at the same temperature reducing the void content in the panel.

7

Conclusions and Recommendations

7.1. Conclusion

This chapter describes the conclusions that can be inferred after conducting this research. The key research question that were formulated in this thesis:

How does use of prepreg/resin infusion process with different tooling schemes contribute to the elimination of manufacturing defects and improving the quality of the laminates ?

To answer this question the following focal points were researched.

1. **Investigating effect of different process parameters on the quality of composite laminate.**

   The material used in the process was TenCate carbon prepreg. Pretrials were conducted on the material with two different layups (0-90 and unidirectional layup), different cure cycles and two different curing processes as described in chapter 3. The produced samples were inspected visually and under optical microscope.

   In terms of layup sequence UD layup showed the optimum case scenario for improvement with 5.9%, 4.2% and 3.5% void content for 9 °C/min ramp up cycle, 1 °C/min temperature cycle and autoclave curing respectively. Therefore, UD layup was considered for further analysis.

2. **Effect of using different tooling schemes on quality of composite laminate.**

   To investigate tooling schemes, the setup was designed and tested which is described in chapter 4. Multiple trials were conducted to optimize the experimental setup. For both solid-solid and solid-flexible tool configurations, 10 trials were conducted to show the repeatability of the setup as well as to improve the quality of the laminate produced.

   The surface quality of samples produced with both tool configurations is comparable with slight variation in the thickness measurement. In terms of void content, solid-solid configuration shows better results than solid-flexible because of homogeneous pressure distribution in the former case with current tooling setup.

3. **Investigating prepreg/resing infusion process with different tooling schemes.**

   For the last subquestion which also forms the basis for the main research question, DSC tests were conducted on both prepreg and epoxy resin to optimize the curing profile for the infusion trial. Infrared test was conducted to test the compatibility of both prepreg and liquid resin system this is also described in chapter-6. From process point of view, the infusion test conducted was a "successful-failure" . This is due to the fact that both chemical and processing compatibility is required for this concept to be a complete success. Even though the chemical nature of both the carbon prepreg and epoxy resin were found out to be very similar, the curing temperatures of both systems were not the same. This led to the failure of the trial as the desired attempt to improve the quality of the laminate by reducing the void content was not achieved.

7.2. Recommendations

This section describes the recommendations that can be suggested to improve the current experimental setup for further research and accuracy of results obtained.

1. **Increased panel size**

   The test panels manufactured with the setup measured 54 mm diameter. In order to increase the sample size to large scale one of the parameters is increasing the tool dimensions proportional to the size of product required. This requires further testing in the solid-solid and solid-flexible tool configuration to find out the equivalent pressure buildup and the corresponding closing force required in a similar closed mould setup.

2. **Number of plies**

   Another parameter that can be considered in further research is the number of plies used for consolidation. In this thesis all trials were conducted with 10 plies and the closing force was calculated based on the number of plies. The experimental setup can be modified to accommodate the increase in number of plies.

3. **Prepreg/resin infusion concept**

   Another recommendation is based on the prepreg/resin infusion concept used in the project. Further testing is required by using prepreg and liquid resin with same chemical and processing conditions. Improvements can be made in the infusion concept. Having multiple infusion lines is a possible option to speed up and accurately infuse the resin. In this case either the resin can be infused from the bottom directly in contact with the laminate or from the top. However this option is not valid for the current project as the setup was kept directly on the press plates which has no inbuilt infusion lines.

   In Phase 3 of this project, the resin was infused when system A reached 60 °C as explained in section 6.3.1. Another concept for prepreg/resin infusion would be by spreading the resin on top of the prepreg layers before closing the mould. In this strategy the setup can be used in the hot press without the use of vacuum pump and infusion chamber. When the resin in the prepreg starts getting viscous (close to 70°C), the liquid resin from above the layers will start filling any porosity in the prepreg and can combine with the prepreg resin. The cross-section of the consolidated laminate can be studied under microscope to measure the void%.

   Increasing the part size also influences resin infusion concept. Along with changing the tool dimensions as discussed before, the infusion system would also require a redesign to accommodate the amount of resin required for infusion. The infusion channels need to be positioned along the circumference of the part so that entire area is infused in an effective manner as shown in figure 7.1.

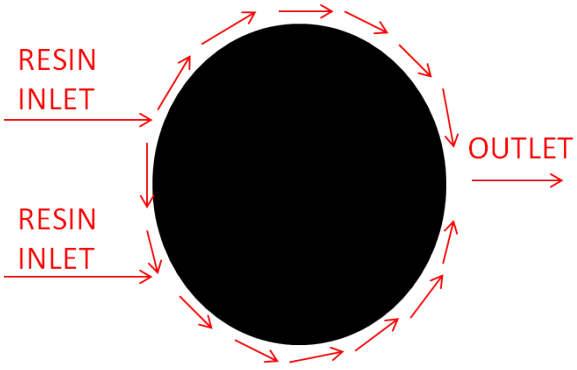


Figure 7.1: Schematic representation of infusion concept

4. **Mechanical testing**

   Due to limited size of the test panels produced in the project, the mechanical testing was limited to inter laminar shear strength of the panels. Increasing the size of test panels gives the option of conducting further testing like peel test to determine the mechanical strength of panels.

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A

Material description

A.1. Carbon Prepreg

The carbon prepreg material used throughout the project is manufactured by TenCate Advanced Composites. The material description as given by the company is provided below.

TENCATE

TENCATE ADVANCED COMPOSITES
Aerospace-Armor
18410 Butter Field Blvd.
Morgan Hill, CA 95037

Objective: To develop an OOA process for low temperature resin system.
LR #: 11500
Material Description: TR50S/FL5-131E
Material Lot No.: 082211-1T2
Prepare by: Henry Villareal
Lay-up: LU-1.5
Cure profile:
-1°F/min to 160°F hold for 1 hr. then 1°F/min t0 212°F for 2 hrs.
- Half of the panel was PC@250° for 1 hr.
Summary:
We used 2 ways of debulking:
Set A.4 ply debulk with TX1040 and non woven breather every 5 – 10 mins
Set B.4ply debulk with FEP every 5- 10 mins.
All laminates were cured in an oven with thermocouple and vacuum sensor attached. After cure we C-scan the laminates.

Mechanical Properties

Mechanical Properties	212°F cure Set A	212°F cure Set B	250°F PC Set A	250°F PC Set B
CS(ksi)	138.04	136.97	149.13	158.56
CM(msi)	15.06	15.47	14.67	15.32
SBS(ksi)	12.37	12.72	12.82	12.64
TG(°C)	110.08	118.71	126.73	125.24

Figure A.1: Carbon prepreg datasheet

A.2. Epoxy Resin

The material description of epoxy resin used in phase 3 ,CH-6 for resin infusion is provided below.

HEXION™
Specialty Chemicals

Technical Information
Division Epoxy/Phenolic Resins
Epoxy Resins

EPIKOTE Resin 04908
EPIKURE Curing Agent 04908

Features

- Certified by German Lyod
- Low viscosity
- Extended pot life
- Low exothermic heat

Application

Low viscous resin system designed for infusion applications with excellent wetting and adhesion characteristics on fibreglass, carbon- or aramid-fibres, particularly in boats and yacht-building and production of rotor blades. This system make it possible to manufacture construction elements of a superior quality, with outstanding surface characteristics and good resistance to thermal deformation and weathering.

Table A.1: Product physical properties (at the time of manufacturing)

Property	Unit	EPIKOTE Resin 04908	EPIKURE Curing Agent 04908
Viscosity at 25°C	mPa.s	500 ± 250	10 ± 5
Epoxy equivalent weight	g/equiv.	165 ± 3	
Amine equvivalent weight	g/equiv.		50
Density at 20°C	gcm^3	1.15±0.02	0.93±0.02
refractive index at 25°C		1.540±0.003	1.468±0.003
Mixing viscosity at 25°C	mPa.s	130±10	130±10
Pot life at 25°C	minutes	300±50	300±50
Tg(TMA)	°C	82	82

Processing Details

Mixing ratio
EPIKOTE™ Resin 04908 100 parts by weight
EPIKURE™ Curing Agent 04908 30 parts by weight

Mixing tolerance
The maximum allowable mixing tolerance is ±2 pbw, but it is particularly important to observe the recommend mixing ratio as exactly as possible. Adding more or less hardener will not effect a faster or slower reaction - but an incomplete curing which cannot correct in any way. Resin and hardener must be mixed very thoroughly. Mix until no clouding is visible in the mixing container. Pay special attention to the walls and the bottom o f t h e mixing container. Processing temperature A good processing temperature is in the range between 25 °C and 35 °C. Higher processing temperatures are possible but will shorten the pot life. A rise in temperature of 10 °C reduces the pot life by approx. 50%. Different temperatures during processing have no significant effect on the strength of the hardened product. ; Do not mix large quantities at elevated processing temperatures. The mixture will heat up fast because of the dissipating reaction heat (exothermic reaction). This can result in temperatures of more than 200 °C in the mixing container. Exemplify curing cycle: 4 - 6 h at 80 °C

Table A.2: Properties of the cured, non-reinforced resin system: (curing: 4 h at 70 ° C + 6 h at 80 °C)

Property	Unit	Value
Density	gcm^3	1.15
Tensile strength	MPa	74
Tensile strain	%	9.4
Modulus in tensile	MPa	2900
Flexural strength	MPa	112
Modulus in flexure	MPa	3100
Water absorption after 24 h 23° C	pbw	0.180
Water absorption after 168 h 23° C	pbw	0.432

Table A.3: Properties of the cured, reinforced resin system (curing: 4 h at 70°C + 6 h at 80°C)

Property	Unit	Value
Tensile strength	MPa	447
Flexural strength	MPa	588
Modulus in flexure	MPa	23400
ILSS	MPa	40
Tg(DMTA)	° C	89

The values are measured on laminates made with glass fabric 181/lnterglas 91745.

Shelf life

The resin and hardener can be stored at 20 - 25 ° C for at least 12 months in their carefully sealed original containers. It is rarely possible that the resin or the hardener crystallize at temperatures below 15 ° C. The crystallisation is visible as a clouding or solidification of the content of the container. Before processing, the crystallisation must be removed by warming up. Slow warming up to 50 - 60 ° C in a water bath or oven and

stirring or shaking will clarify the contents in the container without any loss of quality. Use only completely clarify products. Before warming up, open containers slightly to permit equalization of pressure. Caution during warm up! Do not warm up over open flame!

Precautions

For information about safe handling of EPIKOTE epoxy resins and EPIKURE Curing Agents, please note the corresponding Safety Data Sheet.

A.3. Silastic M

The silicon inner rings used in the experimental setup were casted by following the guidelines given in the material description below.

DOW CORNING

Silastic® M RTV Silicone Rubber

Product Information

Silicone rubber for prototyping, architectural and furniture component applications

FEATURES

- High inhibition resistance
- Formulated to work with both rigid and foam polyurethanes
- Good cut-growth resistance
- High durometer hardness
- Low shrink
- Cures at room temperature within 16 hours or heat curable

COMPOSITION

- Two-part silicone rubber supplied as a pourable fluid; cures to a firm, flexible rubber

USES

Silastic® M RTV Silicone Rubber is designed especially for use with urethane foams and other casting plastics.

TYPICAL PROPERTIES

These values are not intended for use in preparing specifications.

Table A.4: Properties of the cured, reinforced resin system (curing: 4 h at 70°C + 6 h at 80°C)

Test	Unit	Result
As Supplied		
Appearance, Base		White
Curing Agent		Regal Blue
Base to Curing Agent Mixing Ratio, by weight		10:1
Base Viscosity at 77°F (25°C)	poise	1300
As Catalyzed		
Appearance		Regal Blue
Viscosity 1 at 77°F (25°C)	poise	900
Cure Time 2 at 77°F (25°C)	hours	16
As Cured3		
Durometer Hardness, Shore A	points	59
Tensile Strength	psi	650
Elongation, Die C	percent	250
Tear Strength, Die B	ppi	90
Specfic Gravity at 77°F (25°C)		1.29
Linear Shrink		Nil

DESCRIPTION

Silastic M RTV Silicone Rubber is a two-part, flexible mold making material designed especially for use with urethane foams and other casting plastics. This high-strength, tear resistant product cures at room or elevated temperatures by an addition reaction. The special features of Silastic M RTV Silicone Rubber help provide a long mold life, highly detailed reproductions and simplified handling. Silastic M RTV Silicone Rubber base is white, and its curing agent is regal blue to aid inspection for uniform blending. An easy-to-mix ratio of 10:1 base to curing agent helps ensure accurate measuring and blending by hand or machine. The material cures in unlimited thickness, regardless of part configuration or degree of confinement.

HOW TO USE

Pattern Preparation

Certain contaminants used in mold making operations can prevent Silastic M RTV Silicone Rubber from curing. Patterns to be molded should be thoroughly cleaned to remove grease, oil and other surface contaminants. Care should also be taken to ensure that corners, crevices and draws are free of dirt or particles of foreign matter. A light “blow over” with compressed air is advised when the pattern has convoluted draws or undercuts. Then, the original model or pattern should be placed in a light frame of cardboard, foil, wood or other material. There should be approximately 3/8-inch clearance on all sides and over the top of the pattern. The patterns should be attached securely to the bottom of the frame so it does not float. A pattern release agent should then be wiped or sprayed on the pattern. Spreading a light coat of release agent on the sides and underside of the top of the frame will facilitate release. A good pattern release agent can be made by combining 5 percent petroleum jelly and 95 percent solvent. Combine the materials and let stand overnight – then shake by hand to provide a good mix.

Addition of Curing Agent

Automatic mixing equipment handles Silastic M RTV Silicone Rubber efficiently. The product is deaired before shipment when packaged in drums. Silastic M RTV Silicone Rubber curing agent should be mixed into the base material just before use (with either manual or mechanical stirring) in the amounts of 10 parts base to one part curing agent by weight. For the best curing results, use metal cans, clean glassware or unwaxed paper containers for mixing the base and curing agent. Inclusion of air during mixing may cause avoids in the finished mold. Entrapped air may be removed by applying a vacuum of 28 to 29 inches of mercury. Under such a vacuum, the material will expand to three to four times its original volume. As the froth collapses, the mixture will recede to its original volume. The vacuum should be held one or two minutes longer before releasing. Pressure casting may be substituted with equal success.

Working Time
Silastic M RTV Silicone Rubber remains a flowable, pourable material for 1-1/2 hours after the curing agent is added.

Curing
The cure of Silastic M RTV Silicone Rubber occurs by a reaction between the base polymer and the curing agent. Polymerization requires 24 hours after the addition of the curing agent at room temperature. This material will not revert or depolymerize, even under conditions of elevated temperature and confinement. Vulcanization can be accelerated by heating the catalyzed material. However, this will increase the shrinkage from nil to 0.3 percent. A part 1/4-inch thick will set up within 30 minutes if the temperature is maintained at 150°F (65°C). The rate at which thicker sections will set up depends on the size and shape of the piece. Vulcanization will not be accelerated at the center of the piece until the entire mass has reached the elevated temperature. Average setup times at various temperatures for 1/4-inch moldings are as follows:

Temperature	**Demold Time**
77°F (25°C)	16 hours
125°F (52°C)	60 minutes
150°F (65°C)	30 minutes
200°F (93°C)	15 minutes
250°F (121°C)	7 minutes
300°F (149°C)	5 minutes

Inhibition of Cure
Silastic M RTV Silicone Rubber is formulated to have greater resistance to inhibition. However, localized inhibition of cure may be encountered at the interface when Silastic M RTV Silicone Rubber comes in contact with certain contaminants during the curing process. Among materials found to cause inhibition are sulfur containing and organometallic salt-containing compounds (such as organic rubbers), and condensation-cure RTV silicones. Surfaces previously in contact with any of the above materials may also cause inhibition. If in doubt, test for compatibility by brushing a small amount of catalyzed Silastic M RTV Silicone Rubber over a localized area of the service to be reproduced. Inhibition has occurred if the rubber is gummy or uncured after the curing period has elapsed.

USE LIMITATIONS
These products are neither tested nor represented as suitable for medical or pharmaceutical uses.

STORAGE AND SHELF LIFE
Silastic M RTV Silicone Rubber base and curing agent should be stored in closed containers at or below room temperature. The materials have a shelf life of 12 months from date of manufacture. Refer to product packaging for “Use By” date.

PACKAGING
Silastic M RTV Silicone Rubber base is supplied with Silastic M RTV Silicone Rubber curing agent in matched-lot 1.1-, 9.9-, 49.5- and 495-lb (0.5-, 4.4-, 22- and 224-kg) kits. All weights, net.

SAFE HANDLING INFORMATION
PRODUCT SAFETY INFORMATION REQUIRED FOR SAFE USE IS NOT INCLUDED. BEFORE HANDLING, READ PRODUCT AND MATERIAL SAFETY DATA SHEETS AND CONTAINER LABELS FOR SAFE USE, PHYSICAL AND HEALTH HAZARD INFORMATION. THE MATERIAL SAFETY DATA SHEET IS AVAILABLE FROM YOUR DOW CORNING REPRESENTATIVE, OR DISTRIBUTOR, OR BY WRITING TO DOW CORNING CUSTOMER SERVICE, OR BY CALLING (517) 496-6000.

WARRANTY INFORMATION – PLEASE READ CAREFULLY
The information contained herein is offered in good faith and is believed to be accurate. However, because conditions and methods of use of our products are beyond our control, this information should not be used in substitution for customer’s tests to ensure that Dow Corning’s products are safe, effective, and fully satis-

factory for the intended end use. Dow Corning's sole warranty is that the product will meet the Dow Corning sales specifications in effect at the time of shipment. Your exclusive remedy for breach of such warranty is limited to refund of purchase price or replacement of any product shown to be other than as warranted. Dow Corning specifically disclaims any other express or implied warranty of fitness for a particular purpose or merchantability. Unless Dow Corning provides you with a specific, duly signed endorsement of fitness for use, Dow Corning disclaims liability for any incidental or consequential damages. Suggestions of use shall not be taken as inducements to infringe any patent.

B

Test Plan

PRE TRIAL SERIES					
TEST DATE	**TASK DESCRIPTION**	**INSPECTION**	**CURE CYCLE**	**COMMENT**	
30th September,2016	Carbon prepreg samples : 2 crossply samples, 1 UD laminate (10 layers). Without closed mould Press plates	ILSS Optical Microscopy	Straight ramp-up (10 min dwell) 177°C 9°C/min 6bar	ILS thin samples few good results	
				OM 0-90 voids: 3.92% UD voids 5.9%	
24th Feburary, 2017	Consolidation test (JoosPress) 1 crossply 1 UD sample without closed mould Press plates	ILSS Optial Microscopy Thickness Measurment	Pr cycle: 120°C		
28th October,2016	Leak test RTM mould RTM system check in Joos Press	Visual Inspection laminate quality	120°C Vacuum pressure 5mbar Inside pressure 7 bar	•Perfect vacuum 5 mbar no leakage •Tested pressure low for laminate consolidation •Additional hard ring required between the mould cap and chamber	
29th November, 2016	Consolidation test in Autoclave	ILSS Optial Microscopy Thickness Measurment	Pr cycle: 120°C		
2nd December, 2016	Vacuum check in oven at 100°C Solid- Flexible configuration	Leak check	Heat up rate in oven till 100°C	top valve open in intervals of 10°C no leakage at b\w 60-70°C laminate flowout near edges Pressure difference created during closing in the inner cylinder	New top lid design with cavity follow up test SP:2.5
13th December, 2016	Hardness measurement silicon rubber	SHORE-A DUROMETER		•silastic M: 65 shore •glass bead rubber: 80 shore •slilicone sheet : 65 shore	
14th December, 2016	Compression Test inner cylinder (To test pressure build up inside closed mould)	check movement of inner cylinder at 1.5kN		inner silicon ring takes up entire load for expansion, preventing load transfere to the laminate	
16th December, 2016	Compression Test inner cylinder (To test pressure build up inside closed mould)	displacement and change in force at room temperature, at 70°C and 100°C	ramp up temperature in compression oven	•At 40°C: 2166.7N •at 50°C: 2947.6N= 12.9 Bar •At 60° C : 3977.9N=17.4 Bar	new design: shaft/piston with o-rings for controlled
11th December, 2017	vacuum test for infusion system				
25,26th January, 2017	TGA: prepreg prepreg+resin resin	Prepreg +resin : Infrared analysis	same as press cycle	prepreg resin prepreg+resin	Follow up : DSC Test
21st Feburary, 2017	DSC Test		same as press cycle	prepreg resin prepreg+resin	

PHASE 1

	TEST DATE	TASK DESCRIPTION	INSPECTION	CURE CYCLE	COMMENT	
CONSOLIDATION TEST WITH CLOSED MOULD						
TRIAL 1	31st October,2016	Solid-Solid Tooling Specimen id: 1.1	ILSS Optical Microscopy Thickness Measurement	120°C at 1°C/min Dwell: 60 mins	•Laminate flowout. •Extra ring required for pressure. •Inconsistant Thickness	
	2nd November, 2016	Solid-Solid Tooling Specimen id: 1.2		Ramp up 71°C at 1°C/min Dwell: 60min Ramp up 100°C/min Dwell: 120 min	•Laminate flowout. •Damage to inner mould ring •Thickness: 1.5mm	
	4th November, 2016	Solid-Solid Tooling Specimen id: 1.3	ILSS Optical Microscopy Thickness Measurement	Ramp up 71°C at 1°C/min Dwell: 60min Ramp up 100°C/min Dwell: 120 min Post Cure: 120°C for 1hour	•no flowout •Thickness :1.13mm	
	16th November,2016	Solid-Solid Tooling Specimen ID:1.4	ILSS Optical Microscopy Thickness Measurement	Ramp up 71°C at 1°C/min Dwell: 60min Ramp up 100°C/min Dwell: 120 min	•Metal ring with ridge •hard ring: 4.88mm •Perfect deformation on teflon plate •no laminate flow out •no resin around lower O-ring •Thickness: 1.14mm	
TRIAL 2	10th November, 2016	Solid-Flexible Tooling Specimen id: 2.1	ILSS Optical Microscopy Thickness Measurement	Ramp up 71°C at 1°C/min Dwell: 60min Ramp up 100°C/min Dwell: 120 min	•laminate flow out •thickness : 1.14mm •thickness at flowout region: 0.8mm •Pressure build up too high •High deformation in Bottom silicone ring •Replace 4.88mm hard ring with 4.70mm	
	11th November, 2016	Solid-Flexible Tooling Specimen id: 2.2	ILSS Optical Microscopy Thickness Measurement	Ramp up 71°C at 1°C/min Dwell: 60min Ramp up 100°C/min Dwell: 120 min	•laminate flowout •resin around lower o-ring •Use metal ring with 0.2mm ridge •Thickness: 0.96,	
	15th November,2016	Solid-Flexible Tooling Specimen id: 2.3	ILSS Optical Microscopy Thickness	Ramp up 71°C at 1°C/min Dwell: 60min Ramp up 100°C/min	•Metal ring with ridge •hard ring: 4.70mm •Surface waviness •no laminate flow out	
	25th November, 2016	Solid-Flexible Tooling Specimen id: 2.4	ILSS Optical Microscopy Thickness Measurement	Ramp up 71°C at 1°C/min Dwell: 60min Ramp up 100°C/min Dwell: 120 min	•Silicon block with metal ring •Thickness: 1.18mm •Deformation in lower teflon ring •Retest with Aluminium base	
	5th December,2016	Solid-Flexible Tooling Specimen id: 2.5	ILSS Optical Microscopy Thickness Measurement	Ramp up 71°C at 1°C/min Dwell: 60min Ramp up 100°C/min Dwell: 120 min	Lower aluminium ring cut open for easy silicon expansion. •no laminate flowout •damage to silicon block •silicon creep in the cylinder •surface waviness on the laminate, non homogenous pressure distribution	
	8th December, 2016	Solid-Flexible Tooling Specimen ID: 2.6	ILSS Optical Microscopy Thickness Measurement	Ramp up 71°C at 1°C/min Dwell: 60min Ramp up 100°C/min Dwell: 120 min	•tape around laminate bed to stop tool movement •countersunk edges slilicon block •plastic tubing closed du to silicon creep •no laminate flowout, thickness variation,surface waviness	•New stainless steel inner ring cylinder •silicon hollow tube as filler gauge(no teflon rings)
	12th December,2016	Solid-Flexible Tooling Specimen ID: 2.7	ILSS Optical Microscopy Thickness Measurement	Temperature ramp up to 120°C no dwell period in between	no laminate flowout	
	22nd December,2016	Solid-Flexible Tooling Specimen Id: 2.8 (silicone rubber sheet)	visual inspection laminate quality	cure to 100°C 10kN force	Joos press default starting force 5kN •External spring with stands around the mould to control the force	FOLLOW UP TEST : SS-1 (sheet 1)

PHASE 2						
	TEST DATE	**TASK DESCRIPTION**	**INSPECTION**	**CURE CYCLE**	**COMMENT**	
TRIAL 1	3rd January, 2017	SOLID-SOLID TOOLING 1	ILSS, OPTICAL MICROSCOPY, THICKNESS MEASUREMENT	Pr CYCLE: 100°C	•No laminate flowout	
	4th January, 2017	SOLID-SOLID TOOLING 2	ILSS, OPTICAL MICROSCOPY, THICKNESS MEASUREMENT	Pr CYCLE: 100°C	•No laminate flowout •Laminate bed misalignment (not flat throughout)	
	5th January, 2017	SOLID-SOLID TOOLING 3	ILSS, OPTICAL MICROSCOPY, THICKNESS	Pr CYCLE: 100°C	•No laminate flowout	
	6th January, 2017	SOLID-SOLID TOOLING 4	ILSS, OPTICAL MICROSCOPY, THICKNESS	Pr CYCLE: 100°C	•No laminate flowout	
	6th January, 2017	SOLID-SOLID TOOLING 5	ILSS, OPTICAL MICROSCOPY, THICKNESS	Pr CYCLE: 100°C	•No laminate flowout	
SOLID-FLEXIBLE TOOLING CONSOLIDATION TEST						
TRIAL 2	9th January, 2017	SOLID-FLEXIBLE TOOLING 1	ILSS, OPTICAL MICROSCOPY, THICKNESS	Pr CYCLE: 100°C	•no laminate flowout	
	10th January, 2017	SOLID-FLEXIBLE TOOLING 2	ILSS, OPTICAL MICROSCOPY, THICKNESS MEASUREMENT	Pr CYCLE: 100°C	•no laminate flowout	
	11th January, 2017	SOLID-FLEXIBLE TOOLING 3	ILSS, OPTICAL MICROSCOPY, THICKNESS MEASUREMENT	Pr CYCLE: 100°C	•no laminate flowout	inner silicone ring expansion :more relaxation time needed before next cycle. New ring used for this cycle
	12th January, 2017	SOLID-FLEXIBLE TOOLING 4	ILSS, OPTICAL MICROSCOPY, THICKNESS	Pr CYCLE: 100°C	•no laminate flowout	
	13th January,2017	SOLID-FLEXIBLE TOOLING 5	ILSS, OPTICAL MICROSCOPY, THICKNESS	Pr CYCLE: 100°C	•no laminate flowout	
INFUSION TEST						
TRIAL 3	13th Feburary, 2017	SOLID-SOLID tooling		Pr CYCLE: 100°C Post Cure: 120°C	•no resin leakage in the upper chamber during infusion overclaming infusion	
TRIAL 3	17th Feburary, 2017	SOLID-FLEXIBLE tooling		Pr CYCLE: 100°C Post Cure: 120°C	•no resin leakage in the upper chamber during infusion resin curing cylinder wall	