COMPARATIVE CHARACTERIZATION OF OUT-OF-AUTOCLAVE MATERIALS MADE BY AUTOMATED FIBER PLACEMENT AND HAND-LAY-UP PROCESSES

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ABSTRACT

Comparative characterization of Out-of-Autoclave Materials made by Automated Fiber Placement and Hand-Lay-Up Processes

Kulbir Singh Madhok

Automated Fiber Placement (AFP) process along with Out-of-Autoclave (OOA) prepreg technology are important to reducing costs involved in making primary aero-structures ranging from a simple flat parts to intricate three-dimensional parts. This combination promises fast, reliable and cost-effective manufacturing. Avoiding the autoclave during the curing process not only reduces the overhead costs but also opens up opportunities for the use of light weight tooling. However, lack of experience in using the AFP machine with the OOA prepreg and the susceptibility of having voids, resin-rich areas in a part manufactured by the OOA prepreg are the major roadblocks, preventing the aerospace industry from implementing this combination of AFP and OOA prepreg technologies.

This dissertation aims to characterize the mechanical parameters of two different OOA prepreg materials using the AFP process. Using the same OOA prepreg material, the mechanical properties and the quality of the laminates manufactured by the AFP process will be compared with the samples made by the Hand-Lay-Up process, with and without the application of external pressure during the curing process. The microstructure of all samples will be analyzed for voids, resin-rich areas and resin-starved areas. A cost model will also be generated to understand the economics associated with the AFP process and the OOA curing technique.

Keywords: Out-of-Autoclave, Automated Fiber Placement, Thermoset Prepreg Material TC-250 Resin System, CYCOM 5320-1, Comparative Mechanical Characterization, Quality Control, Cost Model

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Dedicated to my DAD…

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1 INTRODUCTION

Over the years, composite materials have become an integral part of the aerospace industry. The never quenching demand of the aerospace industry has led to great advancements in the field of composite materials while maturing the manufacturing technologies and improving the materials themselves.

Today's aerospace industry is looking at composites and automated manufacturing industries to reduce the weight, cost and the processing times so as to cater to their demands [1]. The Automated Fiber Placement (AFP) process has been conceived over the years to overcome the limitations of traditional fabrication methods [2]. This automated manufacturing method addresses the industrial need to have a sophisticated and highly productive equipment that minimizes waste and human intervention.

In the complete process to make a composite structure, substantial cost reduction can be achieved by avoiding the Autoclave curing process. The Out-of-Autoclave (OOA) prepreg technology addresses this need [3]. The OOA prepregs were used initially only for non-structural and secondary structures in the aircrafts.

Recently, the Airbus 350 XWB jetliner became the first commercial aircraft to employ the MTM 44-1 OOA prepreg material to make their wing panels through automation [4], pushing the envelope of the percentage use of composites by weight to 53%, surpassing Boeing 787 and Airbus 380. Figure 1.1 shows the percentage of different materials used in the Airbus A350 XWB Aircraft [5].

Airbus further proved that the extensive use of these light-weight carbon-fibre components is a significant factor in the A350 XWB's 25% fuel savings when compared to the previous generation long-range competitors [6].

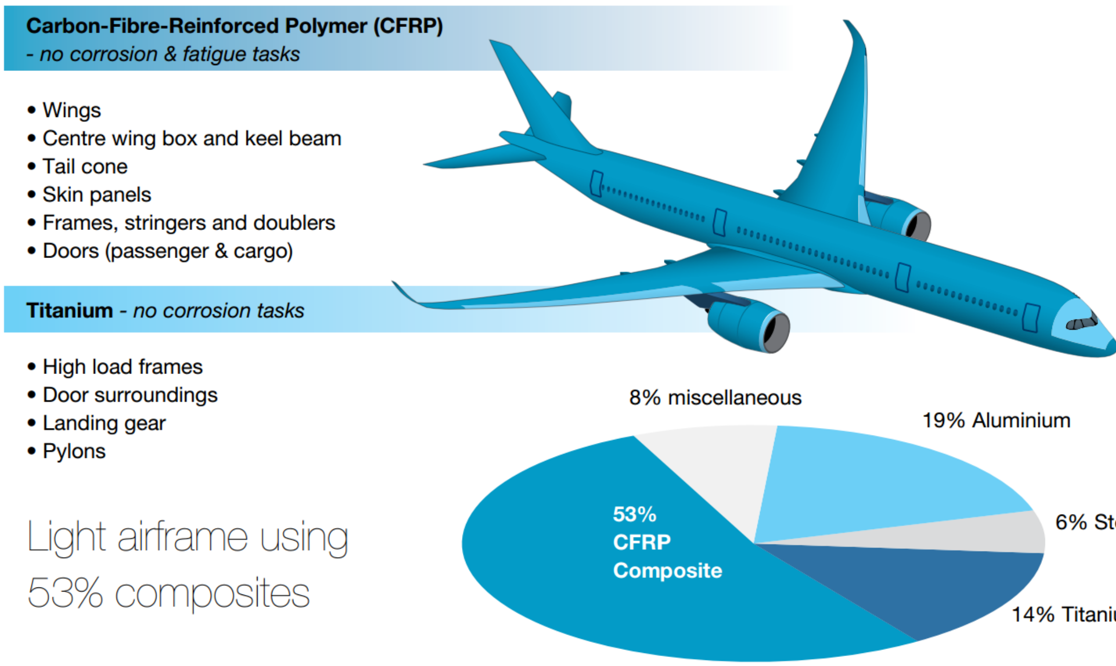


Figure 1.1 Airbus A350XWB Intelligent Airframe [5]

The present thesis deals with mechanical characterization of two different OOA Composite prepreg materials, manufactured by AFP and Hand-Lay-Up processes. The influence of the Autoclave curing cycle and Out-of Autoclave (or Vacuum Bag only) curing is studied and compared both mechanically and economically.

1.1 Thesis Organization

This dissertation has been divided into 6 chapters and a brief description of each chapter has been presented below.

Chapter 1 gives a brief introduction to AFP process and OOA curing technology. It outlines the main objectives and motivation behind this research project.

Chapter 2 highlights all the major work done to date in the fields of AFP and OOA prepreg technologies.

Chapter 3 deals with the material selection, manufacturing techniques, experimental manufacturing procedures and quality assurance aspects. This chapter includes the basic properties of the TenCate's TC-250 and the Cytec's CYCOM 5320-1 OOA prepreg materials. The Hand-Lay-Up and AFP processes are explained in detail. The issues with the initial laminates made by the Hand-Lay-Up and AFP processes are presented and discussed. The quality assurance aspects using the Digital Scanning Calorimetry, Scanning Electron Microscopy and Dual-loop temperature controller are considered.

Chapter 4 presents the experimental results for the Tensile, Shear and Compressive tests. The SEM analyses performed to study the resin rich areas and void content in the laminates manufactured by various processes are also presented in this chapter.

Chapter 5 presents the cost model for various manufacturing process combinations that are used in the present study. The assumed costs, comparison and other aspects pertaining to cost and manufacturing are discussed in detail.

Chapter 6 gives the conclusion of the work presented in this dissertation.

Chapter 7 presents the recommendations for future work in the field of OOA curing and AFP manufacturing.

1.2 Introduction to AFP Process

The aircraft industry has increased the use of high performance composites multifold in the past 15 years. There has been a tremendous amount of research and development on parts made up of Composites. The aircraft industry is expected to keep this growth up and make complex, intricate composite parts. Today the focus is on the composites industry to minimize the cost and maximize the production. All these things can be achieved by Automation. Automated manufacturing of composites has a lot to offer to the aerospace and mechanical industries. The following points summarize a few important reasons why Automated Manufacturing is an excellent way to lay up composites:

- With the high prepreg material costs, lesser is the scrap, greater will be the savings. The scrap rate for a semi-complex part such as that of a duct can be somewhere in between 30 to 50 % when laid up by traditional Hand-Lay-Up. This scrap rate can be brought down to under 5 % by the use of AFP process.
- Not only does automated manufacturing require less labour, but it also ensures a very clean and tidy manufacturing environment. The process ensures minimum human intervention and hence high quality parts are produced. This guarantees the quality and clean room standards are always maintained.
- Aircraft manufacturers usually make many parts of similar kind. The AFP process parameters namely, compaction force, Hot Gas Torch (HGT) temperature and the lay-up speed can be optimized for a particular type of prepreg and hence better compaction and laying parameters are obtained. This ensures high degree of uniformity in part processing from one batch to another.

- AFP is being used in the aerospace industry to make aircraft intake ducts, space launch vehicle components, engine nacelle components etc., and is considered superior to conventional methods, especially in terms of cost and quality.

Figure 1.2 shows a typical Automated Fiber Placement machine made by Automated Dynamics Corporation (ADC).

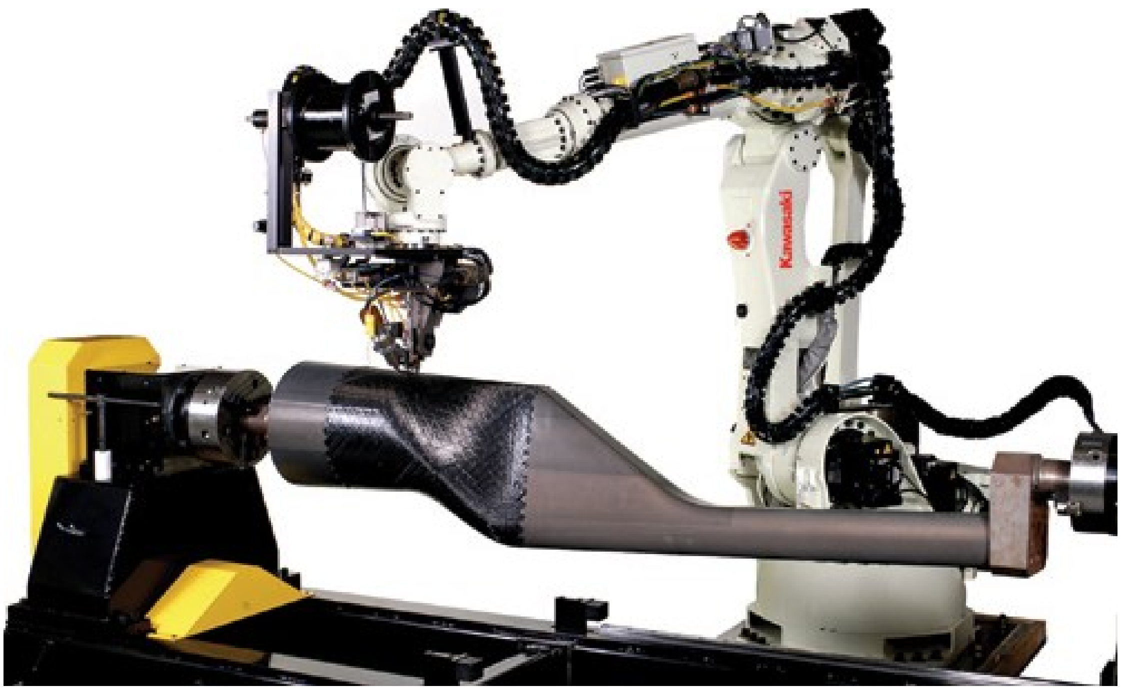
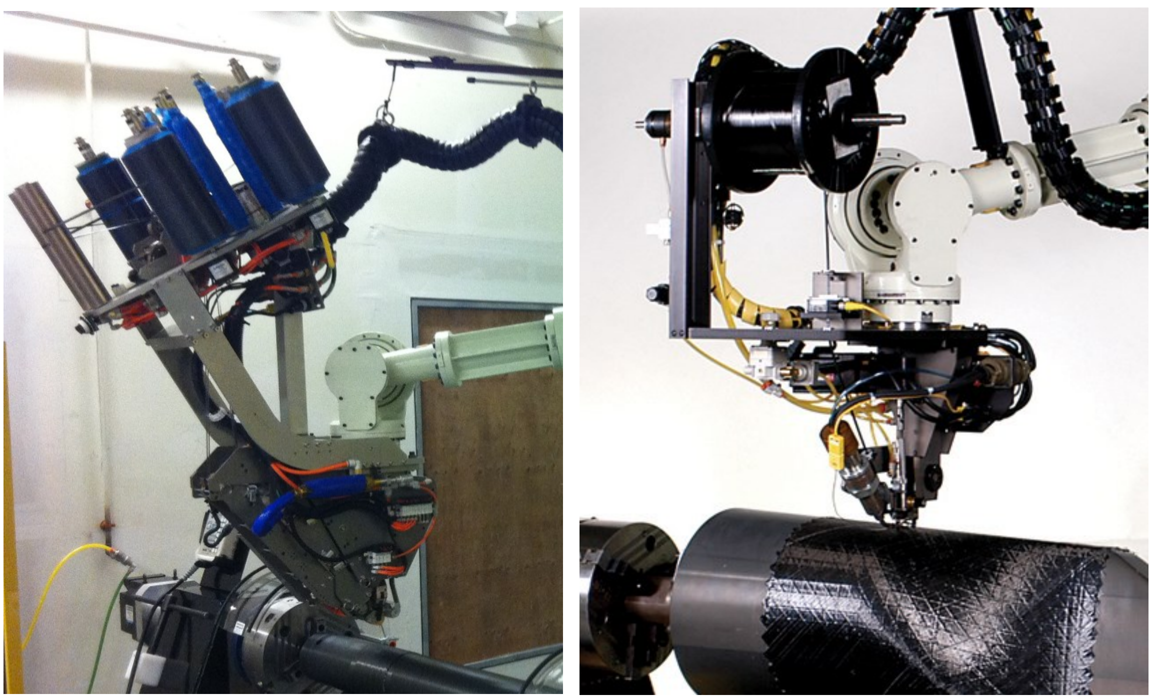


Figure 1.2 Automated Dynamics AFP Machine *(Courtesy: Automated Dynamics Corporation)*

The same machine has been used to manufacture the samples for the current project. This machine uses a Kawasaki robotic arm for the axial movement and can work in a cylindrical envelope of 10 feet in length by 6 feet in diameter. The AFP machine comes in various sizes and the working envelope is specific to a particular machine, for example, large gantry AFP machines manufacture the fuselages of aircrafts.

Figure 1.3 (a) shows the Thermoset head and Figure 1.3 (b) shows the Thermoplastic head. The flexibility of changing the head opens up more opportunities with the same machine.

The particular machine from ADC at Concordia Centre for Composites (CONCOM) has a thermoplastic head that supports only one prepreg material creel at a time whereas the thermoset head can support up to 4 prepreg material creels at the same time.



a) Thermoset Head **b) Thermoplastic Head**

Figure 1.3 Automated Fiber Placement Head *(Courtesy: Concordia University and Automated Dynamics Corporation)*

Figure 1.4 shows the schematic functioning of the AFP head while representing the layup direction, prepreg direction, compaction roller, prepreg cutter, HGT and the tool.

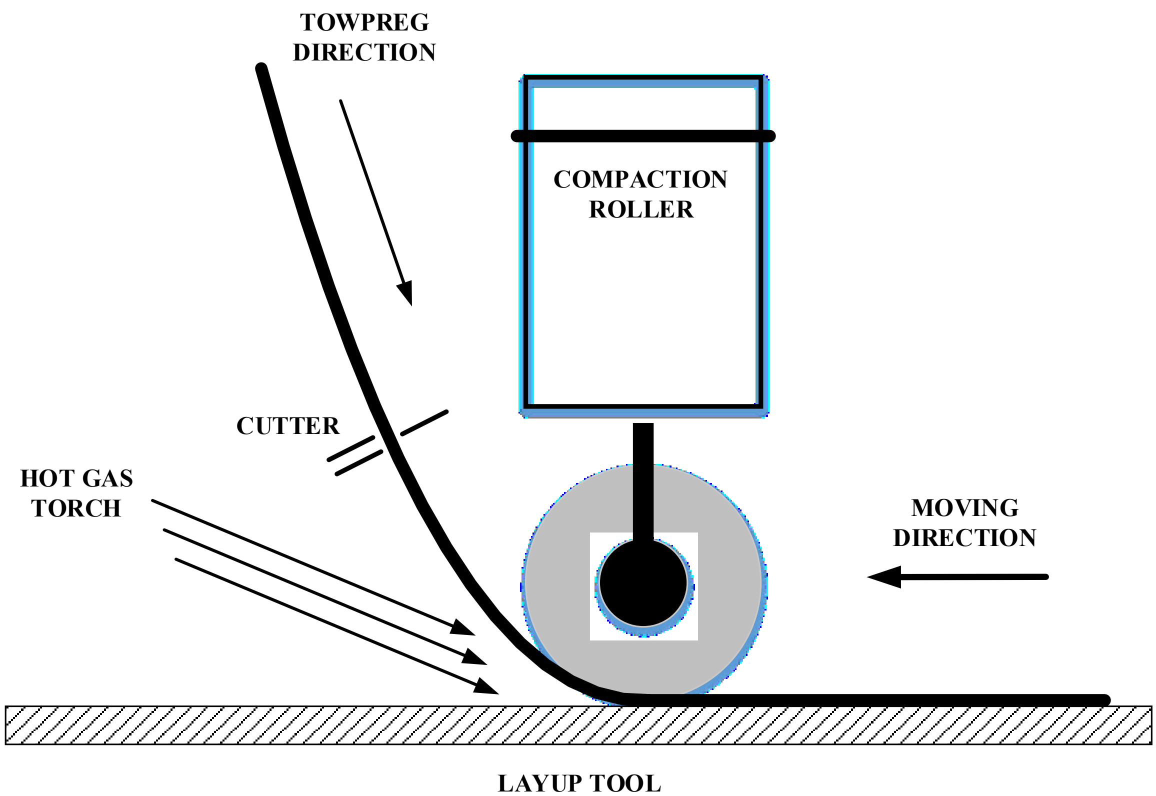


Figure 1.4 Functioning of the AFP Head

1.3 Introduction to Out-of- Autoclave (OOA) Manufacturing

Traditionally, while producing composite components for aerospace industry, autoclave curing is used to achieve the desired resin-to-fiber ratio and void-free components to produce light weight and strong parts. The traditional process involves laying up the fibres (prepregs), curing them in Autoclave by enclosing them in vacuum bag while applying temperature, pressure and vacuum. Currently, OOA technology is being used to make structural components at comparatively lower price [7].

In the OOA manufacturing process, a defect-free part is produced by the application of vacuum and temperature only. With the elimination of external pressure during the autoclave process, the costly autoclave is no longer necessary. The temperature application and vacuum application can be achieved by using a relatively smaller, simpler and portable oven. The investment cost of an oven is much less than that of an autoclave and hence it is more economical and in turn helps reducing the overall cost of the part.

The OOA manufacturing process utilizes special prepregs that are specially designed to remove the entrapped air during the lay-up process. The partially impregnated prepregs create a porous medium to ease the evacuation of any entrapped air before the resin becomes liquid and infuses the fibres [8].

Figure 1.5 shows the operational steps to obtain a defect-free part through OOA manufacturing process.

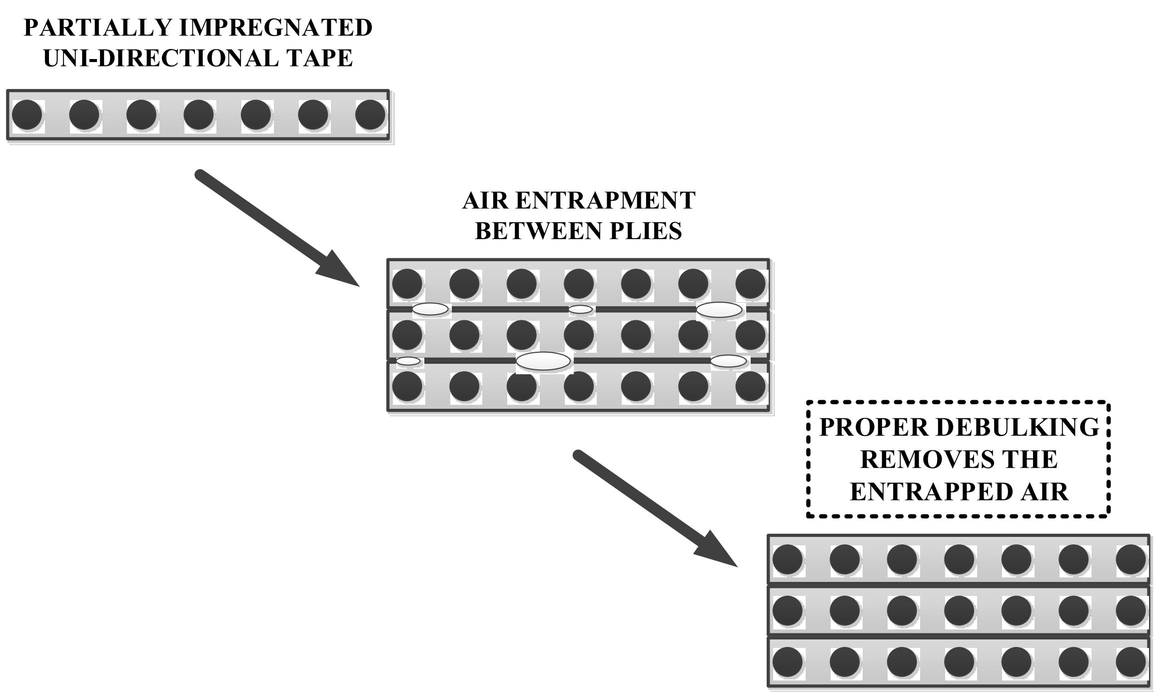


Figure 1.5 Steps to Obtain a Void-free Laminate

The main defects that affect the quality of a part made by OOA process are 'Voids' and 'Resin Rich Areas'. Both these defects significantly affect the load bearing capabilities of the part by hindering the transfer of loads from one fiber to another. Resin Rich areas typically will have less fibers (the load bearing component of the composite) and hence stress concentration will be high in this region, which will in turn result in failure. Inherently, the manufacturing process using OOA prepreg system requires more process control than traditional prepreg lay-up process, and hence proper debulking, experienced labour and technical knowledge do play crucial roles in manufacturing a high quality part.

1.4 Motivation

The main motivation behind this project has been to understand the influence of pressure during the curing cycle of a part made using the OOA prepreg materials. Apart from this, the recent advancements in the field of automated manufacturing of composites fueled the interest of the author. The combination of AFP and OOA technology can not only subsidise the cost of manufacturing but also helps deliver parts of superior quality. To study this further, various combination of samples were manufactured by AFP and Hand-Lay-Up processes and cured in Autoclave with pressure and cured Out-of-Autoclave (OOA) without pressure. These samples were then used to make coupons for mechanical testing and the results were then compared.

The other motivation has been the proven benefits of OOA processing like the reduction in capital expenditure, reduced overheads, increased production flexibility, facilitation of manufacture of very large structures (that cannot be accommodated in an Autoclave), reduced core-crushing and core stabilisation problems in sandwich panels and lower tooling costs.

1.5 Thesis Objectives and Challenges

The objective of this project is to demonstrate the capabilities of the AFP and OOA processes. The OOA processing technology presents some significant hurdles which are taken care of in the presented work.

The economics of using AFP and OOA technology shall be addressed. Most of the work carried out by various researchers in AFP process is focused on modeling and placement strategies. However, the combination of AFP process and OOA technology have not yet been addressed. The present research experimentally compares the AFP process with the Hand-Lay-Up process, and also helps in understanding the effects of pressure during the curing cycle of laminates made by the OOA prepregs.

A variety of concerns including the quality of the raw material (prepreg), void contents and resin rich areas in the samples have been addressed in this work. The importance of having stringent lay-up process with the OOA prepreg has been addressed because of its susceptibility to form laminates with high void content and resin rich areas.

Since the AFP process is majorly employed to make parts specific to the aerospace industry, only a few companies are currently using this process [9]. However, composites stand as main stream materials across many industries and the automation process for composites manufacturing can thus be widely used for various industrial applications.

The following challenges associated with the AFP process and OOA curing technology form the integral part of the present study:

- To find the optimum process conditions for laying up the OOA prepreg material using the AFP machine. The final quality of the laminate largely depends on the processing parameters i.e. lay-up speed, temperature of the Hot Gas Torch (HGT), the compaction force applied through the roller and the inter-band spacing between the bands of fibers. The combined effect of these parameters are to be practically analysed before fixing on an optimal combination.

- To characterize the mechanical properties (tensile, compressive and shear) of the laminates made by the combination of Hand-Lay-Up and AFP processes while curing with and without pressure. The laminates made by the OOA prepreg material must have acceptable void content, high fiber volume fraction while maintaining structural performance.
- To develop the cost model while comparing the costs associated with each combination of manufacturing and curing methodologies. From Industrial point of view, economic assessment plays a crucial role and hence, to address this need, the costs of various parameters are studied and analyzed.

2 LITERATURE REVIEW

Composites are ideal materials for aerospace applications where the use of high performance advanced materials directly enhances the capability of fuel-efficient aircraft and the development of new generation aerospace vehicles.

The structures made of advanced composites for aerospace applications have been majorly manufactured by Hand-Lay-Up of prepreg tapes to produce composite parts that are finalized by a consolidation and a curing process in an autoclave. However, widespread use of composite materials for aerospace applications has been limited due to high manufacturing costs, long processing times and size limitations of an autoclave. To achieve both desired quality and lower costs, for manufacturing high performance composite structures, the combination of automated manufacturing process with out-of-autoclave advanced prepreg systems has to be studied in detail.

Today, the use of composite materials is not only in the Aerospace industry but also in the Civil, Energy, Automotive and other Mechanical industries. All these industries have shown great interest in the integration and application of composite materials. In order to make a substantial increase in the use of composites in cost-sensitive markets such as the automotive and aerospace industries, it is desirable for the manufacture of composites to be fully automated. The important areas for developing affordable composites for aerospace applications were identified by Potluri and Atkinso. [10]; to replace prepregs with advanced materials and to automate the material deposition process. The replaced advanced prepreg materials must be of lower cost or should have the capability to be cured without the use of costly autoclave process.

2.1 Automated Manufacturing of Composite Materials

Automated manufacturing is now being widely used to manufacture advanced composite laminates from unidirectional prepregs. Automated Fiber Placement (AFP) and Automated Tape Laying (ATL) are the two main technologies that are used to make composite parts today. Both ATL and AFP, are processes that use computer-guided robots to lay one or several layers of carbon fiber tapes or tows onto a tool to manufacture a part. Typical applications include aircraft wing skins and fuselages.

ATL utilizes a single, wide, unidirectional slit tape to lay up simple, gentle contours or flat parts. Simple geometries over large surface areas can be laid by using typical prepreg tapes with widths of 152.40 mm to 304.80 mm (6 inch to12 inch). AFP is traditionally used on complex surfaces and smaller structures and utilizes single or multiple narrow, slit tapes or tows (generally of width in the range of 3.18 mm to 6.35 mm (0.125 inch to 0.250 inch)) to make up a given total prepreg band width.

For both ATL and AFP processes, the incoming prepreg tape is electrically heated using nitrogen for the case of both thermoset and thermoplastic processing. Prepreg material application and consolidation or debulking is achieved through a compaction roller medium that may be heated or cooled.

The need for individual tows as in AFP process or a single wide band as in the ATL process is a compromise between part surface geometry and manufacturing throughput. While this solution usually conquers the geometry challenge, it adds to machine complexity and cost and also increases the manufacturing time.

Over the years, research has been done in the field of Automated Manufacturing while improving the manufacturing process and adding several other features to it. However, most of the work done using automated manufacturing is mainly oriented towards Thermoplastic materials (in-situ fiber placement) even though the majority of the components manufactured today are made using Thermoset materials [10]. This is because the processing of thermoplastics results in challenging issues since the material is cured-on-the-fly and hence there is no autoclave curing involved. Generally, autoclave curing eradicates all the defects in the processing of a composite part and when this step is eliminated, various issues like voids, resin rich areas, degree of cure, compaction, etc. come into picture. However, for thermoset materials even though the part is manufactured using the automated manufacturing process, the curing of the part is mandatory using an autoclave. Hence, had there been any defect in the material laid by the AFP machine, they would have been solved while undergoing autoclave curing cycle.

2.1.1 Automated Fiber Placement Process

AFP system was developed in late 1970's to overcome the problem of laying material on a curved surface. The ATL process developed much before the AFP and used wider tapes and hence the challenges of conforming the wider tape to the curved surface surfaced. To solve this issue, the first AFP machine developed by Goldsworthy [12], was a modified version of ATL machine with an ability to slit down the wide tape. This was further modified in late 1980's by Evans et al. [13] by using separate individually driven bobbins.

The AFP systems were commercially available in the early 1990's and were described as a logical combination of ATL and Filament Winding (FW) [14]. The differential payout capability of AFP was taken from FW and the compaction and cut-restart capability from

ATL. The AFP incorporated all the lessons learnt during the development of ATL. The AFP system controls layup speed, pressure, temperature, and tape tension. To further enhance the productivity of the process, multiple tows were being used in the AFP Machine simultaneously. The tension in the tows, reliability, productivity and layup accuracy were the main unresolved issues in these early days of development.

The process parameters in AFP, namely, the torch temperature, fiber laying head speed, and fiber compaction force were analyzed by Tauseef Aized et al. [15]. Various experiments were performed by varying the process parameters and the results were presented. The experimental results showed that the quality of the product improved as the temperature was increased and the substrate also remained intact with an increase in temperature. The improvement of the quality as a result of an increase in temperature was observed up to a certain temperature limit beyond which a further increase in temperature caused thermal degradation, and hence, the quality deteriorated. The quality was deteriorated as the robot head speed was increased because fiber placement process required a certain amount of time for its completion and a greater head speed means that lesser process time was available. On the other hand, high speed of the robotic fiber head was necessary in order to improve the production rate. The quality was also improved with an increase in fiber compaction force, but the increase in quality was only significant up to certain level beyond which no significant impact was observed. The second phase of experimentation presented in the work done by Tauseef et al., was concerned with the performance maximization issue of the robotic fiber placement process using response surface method. The process performance could be improved according to constraints required. The important constraint in this study was to improve process throughput without compromising on the quality of the product.

Interesting results on the development process of a part with complex layup were presented by Measom and Sewell [16]. A comparison between the FW, Manual Lay-up and AFP was presented. The wastage was reduced from 62% to 6% and the productivity was improved by 450% using a 12.7 mm wide tape in the AFP process. In 1997, Pasanen et al. [17] reported 43% cost saving over the manual lay-up and supported the work done by Measom and Sewell.

In the late 2000's, two major parameters, namely, compressive force and prepreg heating associated with the AFP process, were identified as potential candidates to further improve the quality of the composite part. For better heating and compression methods, new heating alternatives and detailed analyses of the compaction process were suggested respectively. It was identified that each composite material had its own heat transfer model and heating requirements which could only be fulfilled by the use of specific tools. Thus, heat transfer models and corresponding process parameters changed with different composite materials, specifications and heating tools. In 2007, Calawa and Nancarrow [18], developed medium wave Infrared heater (Quartz heater) for high speed fiber placement and the factors that contribute to the choice as well as some of the details of the heater design were investigated. Other heating processes like that of Laser heating, Hot Gas heating were also extensively analyzed and studied.

2.2 Out-of-Autoclave Curing Technology

Out-of-Autoclave processing is a curing methodology in which the composite materials are cured without employing an autoclave. Only temperature control and vacuum suction with no external pressure are required in this technique. This curing process has been a source of phenomenal cost reduction in autoclave overheads, tooling costs and also has opened up paths to cure large structures that usually cannot be accommodated inside an autoclave. Flexible manufacturing is facilitated by the use of the Out-of-Autoclave curing process.

Although autoclave process has been an integral part of the manufacturing process used for almost all the composite parts in the aerospace industry, the present day economic situation requires more affordable solutions than the conventional autoclave processing without affecting quality of the final part [19]. Efforts to make parts of the same quality but with less expensive methods such as vacuum-assisted resin transfer molding (VARTM) [20], resin infusion techniques [21], and resin transfer molding (RTM) techniques [22]. The main issues of using these methods are no temperature and pressure control during the curing process, and non-uniform processing throughout the part, which results in voids [19]. To overcome these issues, the OOA prepreg materials were developed. The main reasons why prepregs are used in the aerospace industry this commonly are: they are pre-impregnated with resin, hence they facilitate void reduction, avoid resin handling, and facilitate swifter manufacturing with uniform quality throughout the finished structure.

2.2.1 Out-of-Autoclave Prepreg Materials

Prepreg materials that do not require autoclave processing are called Out-of-Autoclave (OOA) prepregs. They can also be addressed as Vacuum Bag Only (VBO) prepregs. These prepreg materials are designed for processing at much lower pressure without the use of autoclave. This factor can result in great cost savings for the aerospace industry.

These advanced prepregs developed in late 1990's support in reducing the costs, but have suffered in the past because of high percentage of voids in parts made by them. Voids present in composite structures have a direct impact on the mechanical properties [23]-[28]. Voids in the final parts can be a result of either moisture dissolved in the resin and/or can be a result of entrapped air and volatiles [29], [30]. In the case of OOA prepregs where a maximum of 1 atm. pressure is applied through vacuum suction, the void content in the final part can be much higher and it can be challenging to make void-free complex laminates [31]. Also, resin rich areas are found in the final part as there is no external pressure used in the curing process.

A lot of research work has been performed to study the effects on the laminate caused by the presence of moisture content, entrapped air and volatiles in the prepreg, however, very limited amount of research has been done in the area to understand the effect of pressure during the curing cycle of these OOA prepregs [32]. The key lies in the evacuation of entrapped air, moisture and other volatiles prior to the resin gelation. The viscosity of the OOA prepregs during the cure cycle (Rheology) is critical i.e. the prepreg must remain breathable for sufficient time.

The new generations of these prepregs with tailored resin rheology are intended for OOA processing are designed and formulated to reduce void formation from volatiles and entrapped air. For example, these prepregs are now manufactured by a hot-melt process [33], and thus contain negligible solvent content [34]. Furthermore, the present day OOA prepregs are designed with engineered vacuum channels to facilitate air removal [33], [35].

3 MATERIAL SELECTION AND FABRICATION OF SAMPLES

This chapter describes the selected OOA prepreg materials in detail, the manufacturing techniques employed, the quality assurance aspects that are considered while manufacturing the samples and the challenges related to AFP manufacturing.

3.1 Material Selection

The materials selected for this project were TenCate Advanced Composites' TC -250 resin system and the Cytec Advanced Materials' CYCOM 5320-1. Both of these are OOA materials and are being used in today's aerospace industry [36]. The reason for selecting materials from two different suppliers is to understand the importance of having material tailored to a given manufacturing process. CYCOM 5320-1 is a new generation material tailored specifically for automated manufacturing as an OOA prepreg material whereas TC-250, one of the oldest OOA resin systems, has optional pressure application during the curing operation. The influence of this variation in prepreg manufacturing will be comparatively analyzed in the present work. The quality of both these prepreg materials was examined and is presented in the Quality Control section in this chapter.

3.1.1 CYCOM 5320-1 Resin System

CYCOM 5320-1 is a toughened carbon/epoxy resin prepreg system designed for OOA manufacturing of structural components. Its lower temperature curing capability makes it suitable for prototyping where low cost tooling or Vacuum-bag-only (VBO) curing is applicable. The wet glass transition temperature of this material is 163°C. The nominal resin content in the prepreg is 33% by weight of the resin [37]. The prepreg material was provided in 6.35 mm (0.25 inch) wide slitted tapes from Cytec Advanced Materials.

3.1.2 TC-250 Resin System

TC-250 resin system is a member of the TenCate family of toughened matrices for structural advanced composite applications. It is a 130°C cure epoxy OOA resin system. This carbon/epoxy resin system can be initially cured at 82°C and post-cured free standing at 130°C or 177°C for prototyping with inexpensive tooling [38].

This prepreg was available in 6.35 mm (0.25 inch) wide tapes and also in 304.8 mm (12 inch) wide rolls. The quarter inch wide tape was used to make laminates using AFP Process and the 12 inch wide rolls were used for the Hand-Lay-Up process.

3.2 Fabrication of Samples

This section explains all the manufacturing steps used in making the samples using the OOA materials. The initial sequence of operation in ***Hand-Lay-Up process*** is as follows:

a. The uni-directional prepreg tape is cut according the size of the laminate to be manufactured. The number of prepreg layers to be stacked depends on the thickness of the laminate to be used for testing. It was determined that 8 layers of prepreg stacked one above the other would result in about 1 mm thick laminate. This material was left at room temperature for 24 hours so as to increase the tackiness of the prepreg.
b. The mold plate was cleaned and a thin layer of release agent was applied on the mold and was left to dry for 15 minutes. Then the prepreg tape was laid on the mold using a roller to remove the entrapped air. After every layer of prepreg stacked the entire lay-up was enclosed in a vacuum bag and a pressure of about 80 kPa was applied for 15-20 minutes. This pressure is necessary for proper debulking of the prepreg and the elimination of air and moisture.

c. After the required number of prepreg layers have been stacked the entire assembly is placed in an autoclave or an oven and the curing process is begun as per the curing cycle suggested by the prepreg manufacturer.

The vacuum bag assembly for the autoclave consolidation of the laminates and the sequence of materials used in the bagging process is shown schematically in Figure 3.1. The curing cycles used for both the sets of laminates employed the same conditions except for conditions with and without external pressure. The cross sectional view is expanded so as to make it clearer.

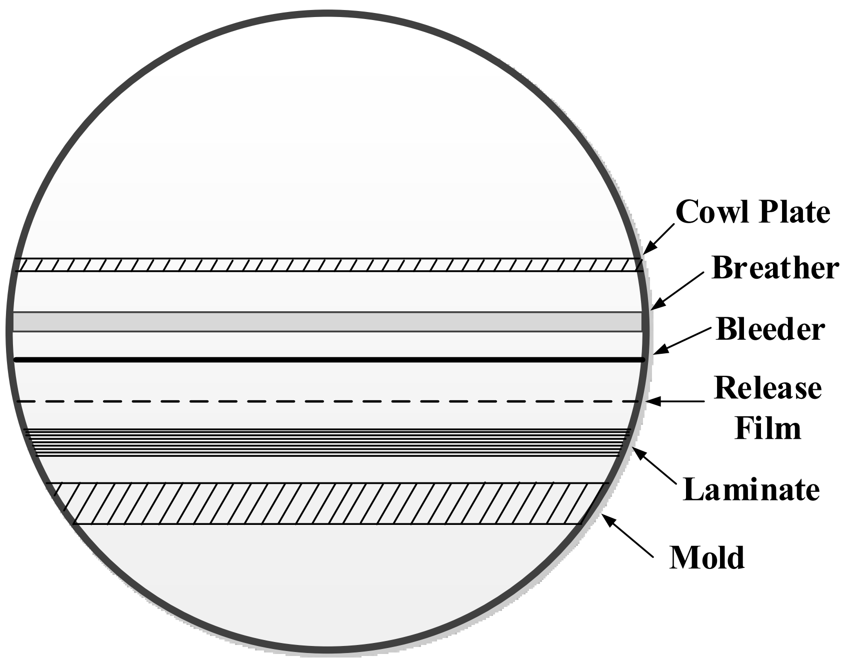


Figure 3.1 Expanded Cross Sectional view of Vacuum bagging process

The initial sequence of operation in ***Automated Fiber Placement process*** is as follows:

a. The prepreg tape is loaded onto the head of the machine after being left at room temperature for about 3-4 hours. The tows are supplied from the creels on the head to the compaction roller, passing through the HGT torch, as shown in the schematic Figure 1.4.

b. The program, which describes the size of the part and stacking sequence for making the part is developed using the AFP machines' custom CAD software.

c. The processing parameters for the AFP used while making the laminates were: HGT Torch Temperature: 190°C, Layup-Speed: 50.8 mm/sec (2 inch/sec) and Roller head Pressure: 130 N (29 lbf).

The vacuum bagging process and the curing process for these laminates is similar to that of the parts made by Hand-Lay-Up process.

3.2.1 Fabrication of laminates using CYCOM 5320-1

CYCOM 5320-1 was used to make laminates using the AFP machine at Concordia Centre for Composites (CONCOM) at Concordia University. The material was available in 6.35 mm (0.25 inch) wide tapes. The average thickness of the raw material tape was nearly 0.237 mm (It is to be noted that the prepreg material gets compacted in the thickness direction while undergoing the debulking and curing processes).

Various laminates were made for the mechanical characterization of this material. Two sets of all the laminates were laid using the AFP machine. The first set of laminates (identified as 1A, 2A, 3A and 4A) was cured in autoclave and the second set (identified as 1B, 2B, 3B and 4B) was cured without the application of external pressure, i.e. the first set was cured with external pressure in the autoclave and the second set was cured using the vacuum bag only technology. Table 3.1 shows the identifications, measured properties, dimensions and stacking sequences. Figure 3.2 shows the curing cycle employed for the CYCOM 5320-1 prepreg material [37].

Table 3.1 Identifications, Dimensions and Stacking Sequence of all laminates made by CYCOM 5320-1 Material

Laminate Identification	Number of Laminates	Measured Property	Laminate Length / Width (mm)	Stacking Sequence
1A and 1B	2	Tensile Modulus and Strength (0°)	304.8/304.8	$[0]_8$
2A and 2B	2	Tensile Modulus and Strength (90°)	228.6/304.8	$[90]_{16}$
3A and 3B	2	In-Plane Shear Modulus and Strength	304.8/304.8	$[0,90]_{10s}$
4A and 4B	2	Compressive Modulus and Strength (0°)	190.5/304.8	$[0]_{12}$

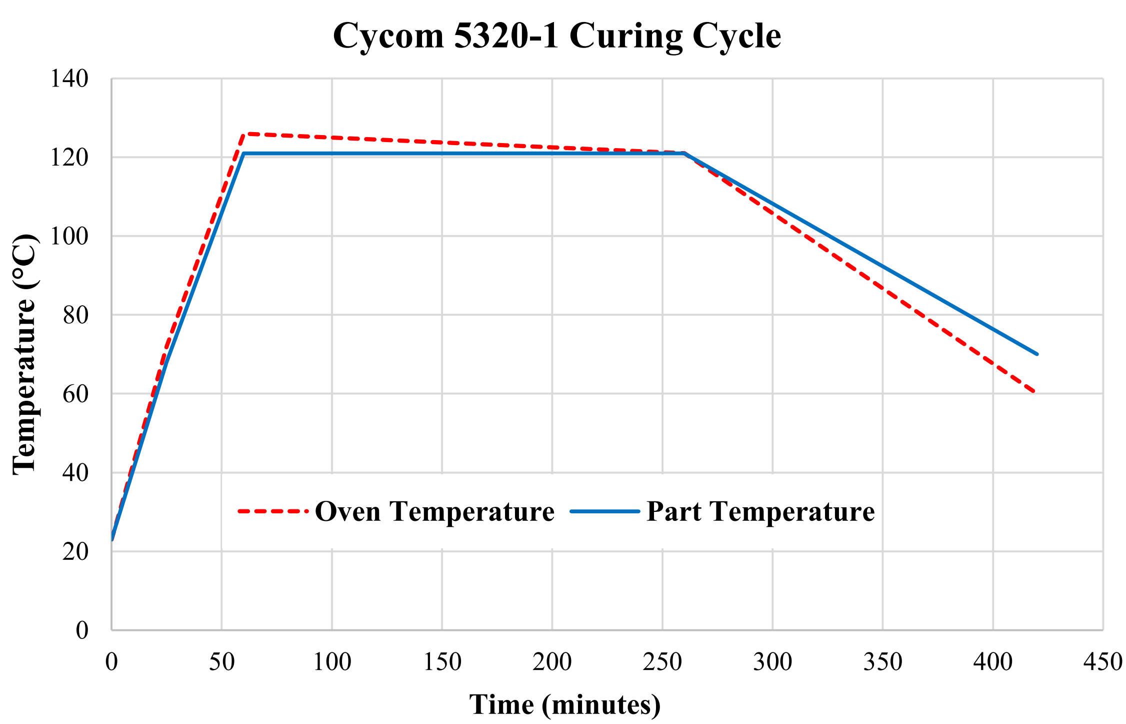


Figure 3.2 Curing Cycle of CYCOM 5320-1 Material [37]

Prior to heating, a vacuum hold within 68 mbar (2 inch of Hg) was ensured. The vacuum was held for the first 350 minutes of the curing cycle at around 950 mbar (28 inch of Hg). After the curing cycle, the laminates were post cured at 177°C under free standing conditions in a gravity convection oven.

Apart from all the curing conditions discussed above, the first batch of the laminates identified as x'A' also had the application of external pressure in their autoclave curing cycle. The pressure was applied at the 60 minute mark and was maintained for 3 hours at 7000 Pa (62 psi). However, no external pressure was applied on the laminates identified as x'B' (where x is 1, 2, 3 and 4).

3.2.2 Fabrication of laminates using TC-250

TC-250 resin system was used to make laminates by both Hand-Lay-Up and AFP Processes. The prepreg available in 6.35mm wide tapes was fed to the AFP machine and the 304.8mm wide roll was used in the Hand-Lay-Up process.

To ensure proper debulking in the Hand Lay-Up process, vacuum was applied after stacking each new ply of prepreg over the existing plies. There were two sets of laminates made by each process and the first one was cured in autoclave with pressure and the other one was cured without pressure.

The material datasheet of the TC-250 material states that the pressure application during curing is optional. Both these sets of laminates have been used to make coupons for the mechanical testing and the findings have been compared.

The slitted TC-250 material tapes were used to manufacture parts using the AFP process. Unlike Hand-Lay-Up process, there has been no vacuum application while stacking the prepreg plies. The compaction in this case has been achieved by the pressure applied by the compaction roller.

Table 3.2 shows the Identifications, Dimensions and Stacking Sequences of all the laminates made by the TenCates' TC-250 material.

Table 3.2 Identifications, Dimensions and Stacking Sequence of all the laminates made by TC-250 Material

Laminate Identification	Number of Laminates	Measured Property	Laminate Length / Width (mm)	Stacking Sequence
1P, 1Q, 1R and 1S	4	Tensile Modulus and Strength (0°)	304.8/304.8	$[0]_8$
2P, 2Q, 2R and 2S	4	Tensile Modulus and Strength (90°)	228.6/304.8	$[90]_{16}$
3P, 3Q, 3R and 3S	4	In-Plane Shear Modulus and Strength	304.8/304.8	$[0,90]_{10s}$
4P, 4Q, 4R and 4S	4	Compressive Modulus and Strength (0°)	190.5/304.8	$[0]_{12}$

The curing cycle employed for the laminates made by this material is shown in Figure 3.3 [38]. Unlike the CYCOM 5320-1's curing cycle, this curing cycle has a 2-step curing process. Prior to heating, a vacuum hold within 68 mbar (2 inch of Hg) was ensured. The vacuum was held for the first 350 minutes of the curing process at around 950 mbar (28 inch of Hg). After the curing cycle, the laminates were post cured at 177°C under free standing conditions. Apart from all the curing conditions discussed above, the first batch of the laminates x'P' and x'Q' also had application of external pressure in their autoclave curing. The pressure was applied at the 55 minute mark and was maintained for 3 hours at 7000 Pa (62 psi). However, no external pressure was applied on the laminates identified by x'R' and x'S' (where x is 1, 2, 3 and 4).

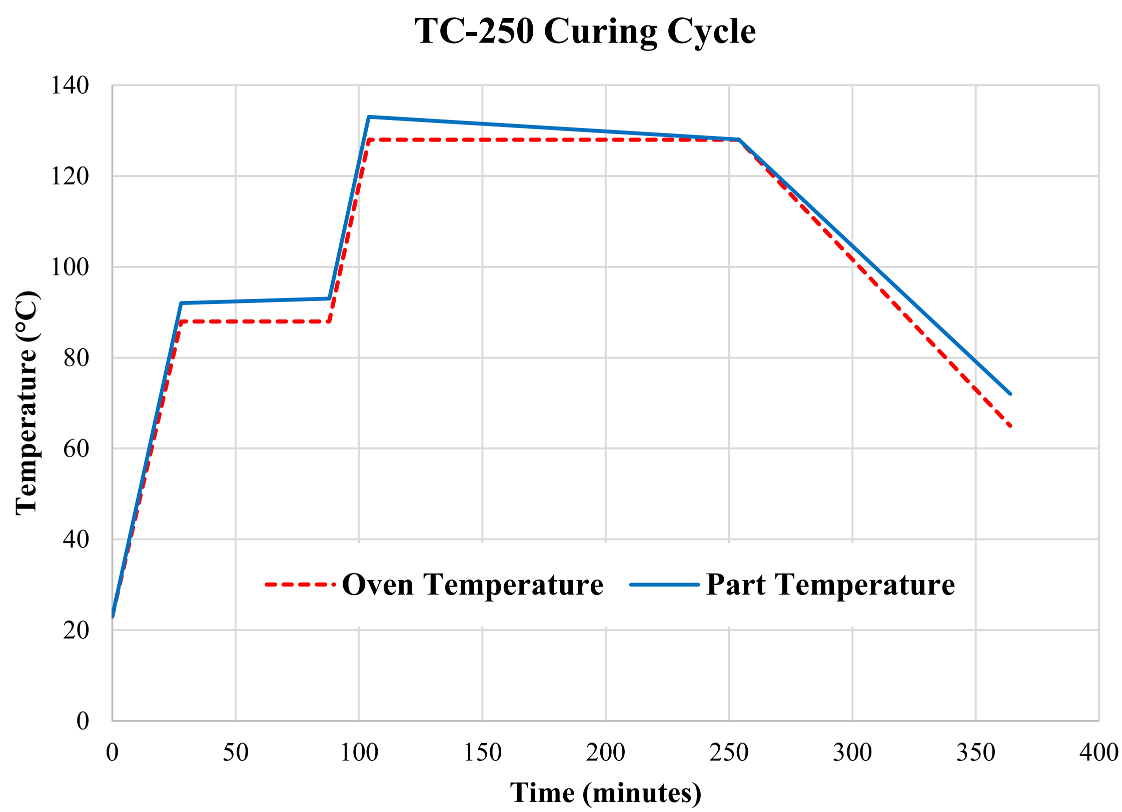


Figure 3.3 Curing Cycle of TC-250 Material [38]

3.2.3 Summary of all Samples Manufactured

Table 3.3 summarizes all the samples manufactured by AFP and Hand-Lay-Up processes using both the prepreg materials. Each laminate is identified based on the manufacturing technique and the curing process used. Each of these laminates has been used to make coupons for mechanical testing.

Table 3.3 Summary of Samples Manufactured

Identification	Material	Manufacturing Process	Curing Methodology
x A	CYCOM 5320-1	Automated Fiber Placement	With Pressure
x B	CYCOM 5320-1	Automated Fiber Placement	Without Pressure
x P	TC-250	Automated Fiber Placement	With Pressure
x Q	TC-250	Hand-Lay-Up Process	With Pressure
x R	TC-250	Automated Fiber Placement	Without Pressure
x S	TC-250	Hand-Lay-Up Process	Without Pressure

3.3 Quality Control

Quality control plays a crucial role in making defect-free parts using the OOA prepreg technology. The two major issues with OOA prepregs are voids and resin rich areas. These defects, to a large extent, can directly be addressed by ensuring high quality while manufacturing the parts. The quality assurances used while making the laminates for this study are presented in the following sections.

3.3.1 Dual-loop Temperature Control

In order to control the temperature inside the oven accurately, a controller has been used. In order to select a proper temperature controller, the whole system should be analyzed and the criteria for the selection are as follows:

I. Type of the input temperature sensor and the range of temperature to be measured
II. Type of the output required
III. Control algorithm required
IV. Number and type of output required

For the process at hand, it is required to control the temperatures of the plate on which the sample is being cured and also the ambient temperature inside the oven. Since there are two temperatures to be controlled, a dual-loop temperature controller (Proportional, Integral and Derivative (PID)) has been used. The temperature inside the oven is sensed using a thermocouple which relays the information to the PID controller; the controller then minimizes the difference in the desired and measured temperatures. The desired temperature is the temperature of the part which is measured by placing a thermocouple directly on the part.

The proportional part of the PID controller helps in controlling the temperature by decreasing the average power supplied to the heater as the temperature approaches the desired temperature point. This action slows the heater down so that the temperature does not rise above the set point and helps in maintaining a stable temperature. This is accomplished by turning the controller output on or off for short time intervals in order to control the temperature. If the temperature is below the set point, the output will be on for a longer time; if the temperature is too high, the output will be off for a longer time. As stated above, the PID controller provides proportional with integral and derivative controls. The controller has two extra adjustments, integral and derivative, apart from the proportional control that helps the unit to compensate for the changes in the system. Though the adjustments are expressed in time-based units, they can also be referred to by

Reset and Rate integrals that are for the integral and derivative controls respectively. The proportional, integral and derivative terms are individually adjusted or “tuned” to a particular system using trial and error. The system thus provides accurate control. In simple words, the working principle of PID controller is as follows: The set temperature is fed in the ‘PID controller’; the output of the controller is fed to the ‘process’ which has to be controlled. The disturbances present inside the oven due to the presence of non-uniform air currents inside the oven influence the process. The output of the process is provided as feedback to the ‘feedback sensor’ which in turn is fed to the’ PID controller’. This is diagrammatically represented in Figure 3.4.

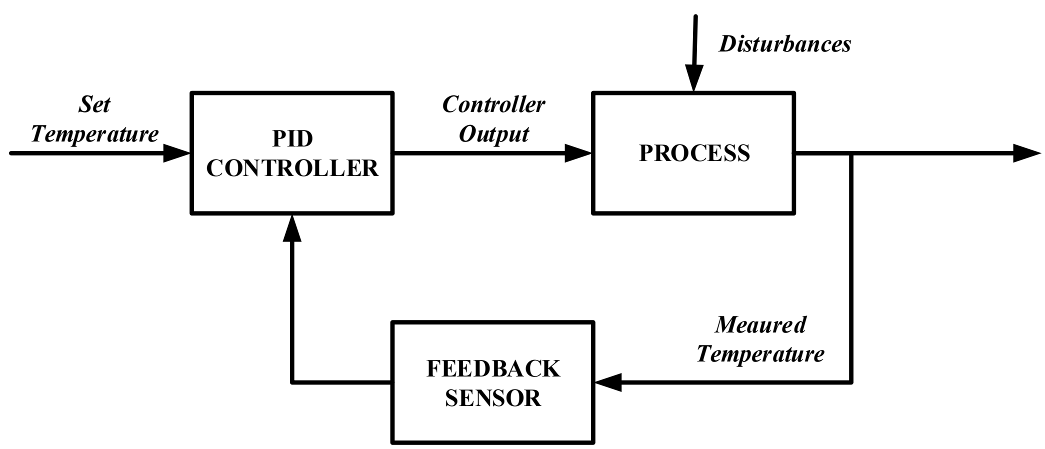


Figure 3.4 Working of PID Controller

3.3.2 Differential Scanning Calorimeter (DSC) Analysis

DSC analysis is a thermo-analytical technique in which the difference in the amount of heat required to increase the temperature of a sample and reference material is measured as a function of temperature. Both the sample and the reference material are maintained at the same temperature throughout the experiment. The temperature program for DSC analysis is designed such that the sample holder temperature increases linearly as a function of time. The equipment utilized for the DSC analysis of samples made by Hand-Lay-Up and AFP Processes for this project was TA Instruments' DSC Q10 as shown in Figure 3.5.

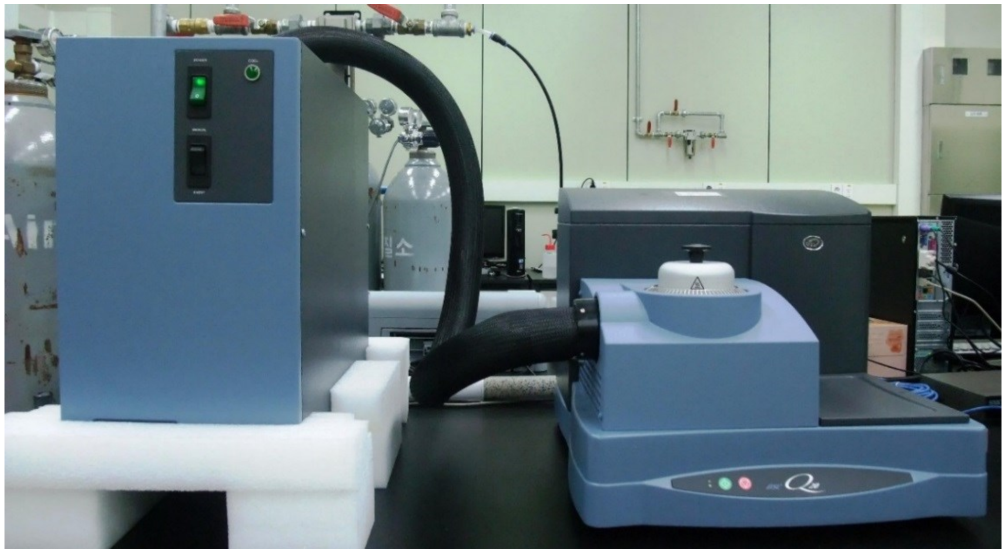


Figure 3.5 TA Instruments DSC Q10

For the samples manufactured using the carbon/epoxy OOA prepregs, Hot-Cold-Hot program cycle was used. This means that the temperature was initially raised to 250°C and then brought back to room temperature and again reheated to 250°C. A sample which is cured properly does not liberate heat, i.e. no exothermic reaction should occur during this Hot-Cold-Hot cycle. Graphically, this reaction can be seen as difference in the first and second heating cycles (It is to be noted that, though ideally the paths should be overlapping for both the heating cycles, but realistically we must aim to have them as close as possible). The parameters used for the analyses are as follows: Nitrogen Gas: 50.0 ml/min, Pan: Aluminum, Set Point Temperature: 240°C / 250°C and Temperature gradient: 5°C/min. Figure 3.6 to Figure 3.8 shows the DSC curve for the CYCOM 5320-1 material manufactured by the AFP Process, TC-250 material manufactured by Hand-Lay-Up process and the TC-250 material manufactured by AFP process respectively.

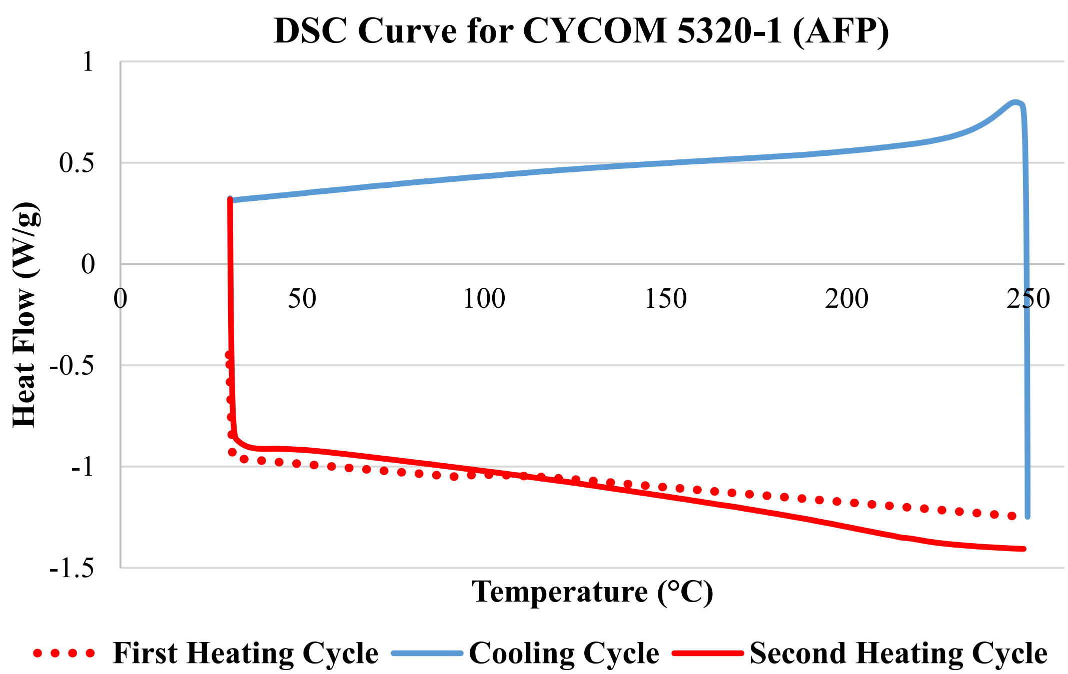


Figure 3.6 DSC Curve for CYCOM 5320-1 Material made by AFP Process

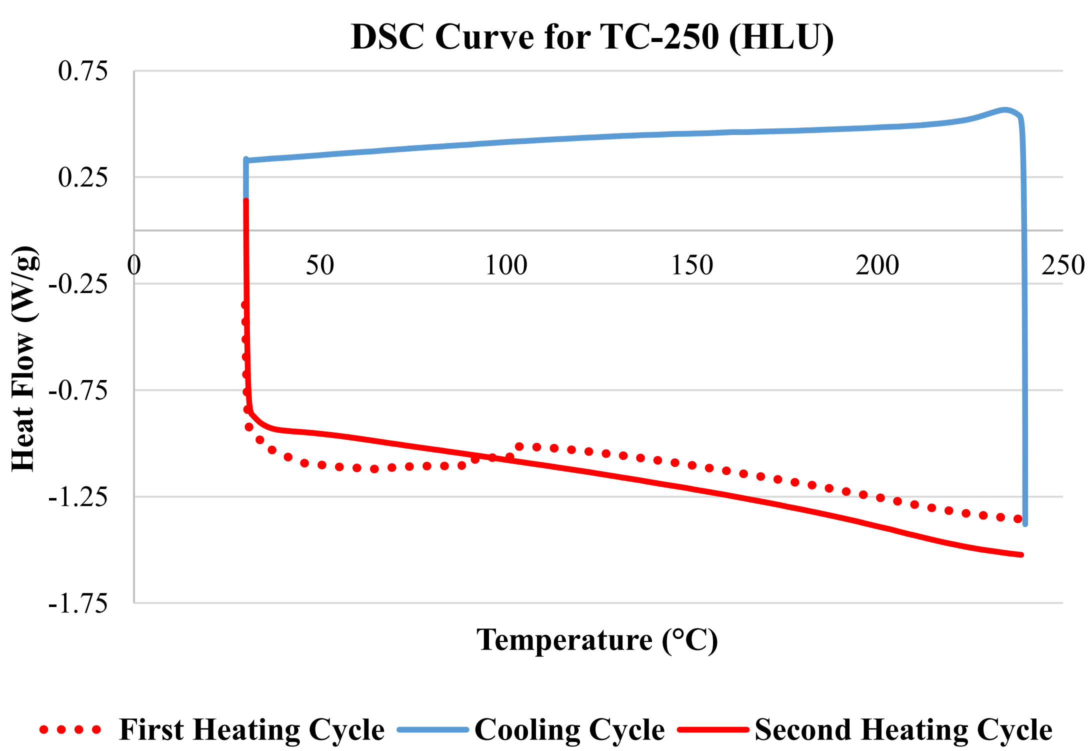


Figure 3.7 DSC Curve for TC-250 Material made by Hand-Lay-Up

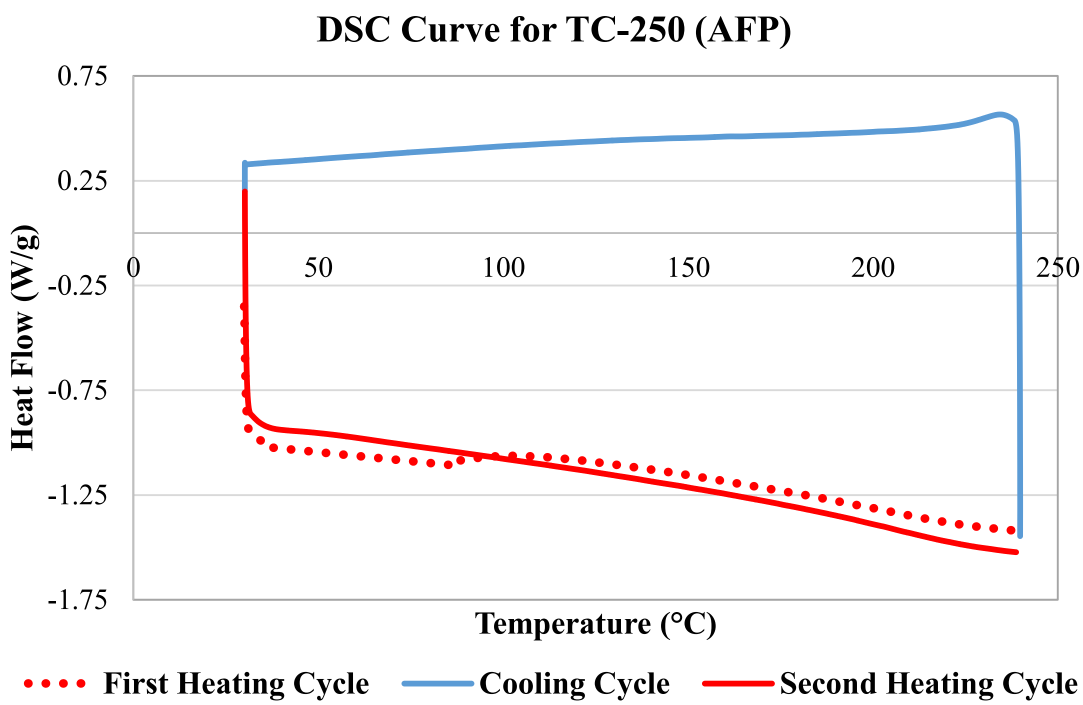


Figure 3.8 DSC Curve for TC-250 Material made by AFP Process

Pressure application during the curing cycle had no effect on the DSC analysis, validating the work done on 'Curing Pressure Influence of Out-of-Autoclave Processing on Structural Composites for Commercial Aviation' by Drakonakis et al. [19]. The samples cured with pressure in autoclave and the samples cured without pressure produced similar DSC results for both the Hand Lay-Up and the AFP Processes.

In the DSC curves presented in Figure 3.6 to Figure 3.8, it can be seen that the first heating cycle curve crossed the second heating cycle curve. However, the deviation in these two heating cycle curves is not large, but it still denotes a small amount of heat transfer that occurred in the first heating cycle. A few samples were initially made using the OOA materials by Hand-Lay-Up and AFP processes. These samples were cured in autoclave without employing the dual-loop temperature controller. The DSC analysis of these initial samples had resulted in graph where the paths followed by the first and second heating cycles are widely different as shown in Figure 3.9. The area between these paths signifies the occurrence of exothermic reaction in the sample, which in turn signifies improper curing of the laminate.

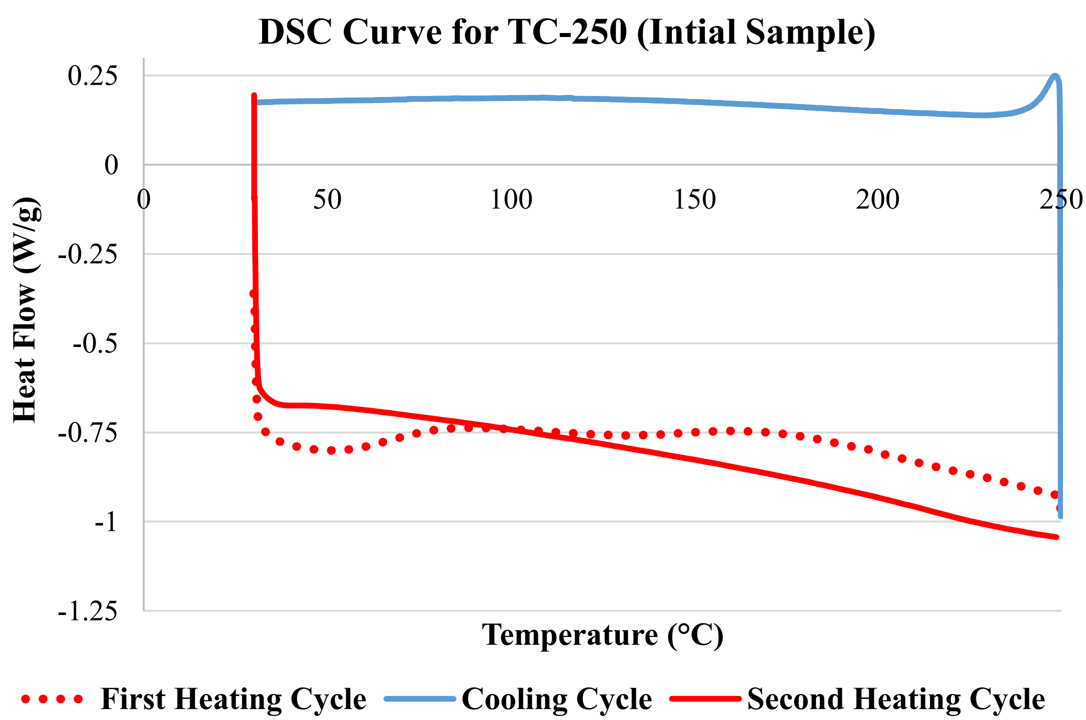


Figure 3.9 DSC Curve for TC-250 Material made by AFP Process (Initial Sample)

The reason behind the improper curing was the temperature of the part during the curing cycle in the autoclave. The initial controller did not take into account the temperature of the part and considered only the oven temperature. This was rectified by the use of Dual-loop controller which operated on two thermocouples, one measuring the ambient temperature of oven and the second thermocouple measuring the part temperature. This minimized the variation between the paths followed by the heating cycles.

3.3.3 Scanning Electron Microscope (SEM) Analysis of the Prepreg material

SEM is used to observe small objects and fine structure of the specimen surface at a high magnification. The sample surface is scanned with a finely converged electron beam in a vacuum and the information produced from the sample at that time is detected. The enlarged image of the sample surface can thus be seen on the monitor screen. The sample is irradiated with an electron beam in a vacuum. Figure 3.10 shows a schematic diagram of a working SEM.

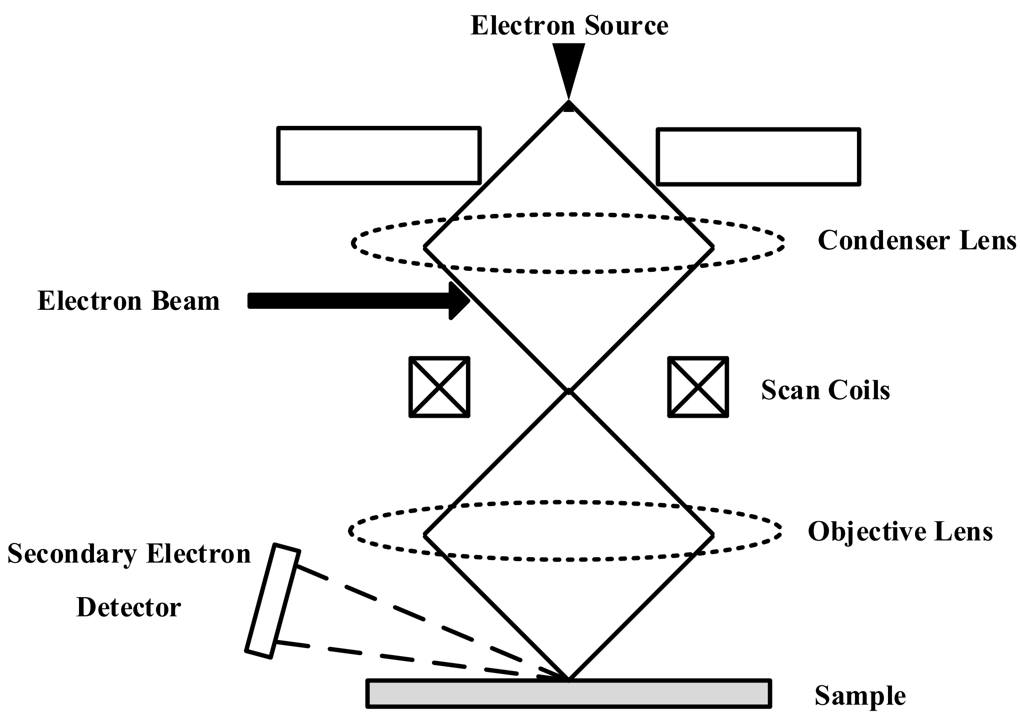


Figure 3.10 Working Principle of SEM

The SEM mainly utilizes secondary electron or backscattered electron signals to form an image. Secondary electrons (SE) are produced near the sample surface, and the SE image, obtained upon detecting these electrons, reflects the fine topographical structure of the sample. The Hitachi S-3400N used for examining the samples for this project (shown in

Figure 3.11), is a powerful, yet user-friendly SEM through newly developed electron optical and automated functions. It is equipped with Wavelength Dispersive Spectrometer (WDS) and Energy Dispersive Spectrometer (EDS) systems for element analysis [39]. In this section, the images of both the TC-250 and CYCOM 5320-1 prepreg materials seen under the microscope for the resin and fiber distribution are presented. SEM has also been used to preform microscopy on the samples fabricated by various manufacturing methods to determine the quality of the laminate by the determination of voids and resin-rich areas [40]. These microscopic images will be presented and discusses in Chapter 4.

Figure 3.11 Hitachi S-3400N SEM

Quality of the Prepregs: Prepreg material manufacturers use different techniques to make prepregs. The following two methods for making the prepregs namely, Hot Melt Process and Solvent Dip Process, are most predominantly used. In the 'Hot Melt Process' both fabric and unidirectional prepregs can be produced. In the first stage, a thin film of the heated resin is coated on a paper substrate. The reinforcement material and the resin are allowed to interact in the prepreg machine. On application of pressure and heat, the resin is impregnated into the fiber resulting in the final prepreg which is ultimately wound on a core. The 'Solvent Dip process' involves dissolving the resin in a solvent bath and dipping the reinforcing fabric in the resin solution. Using a drying oven, the solvent is then evaporated off the prepreg. However, these processes are customized by each manufacturer to suit their needs based on the resin rheology and the reinforcement properties. The microscopic image of the CYCOM 5320-1 prepreg material shown in Figure 3.12 was taken at 100x magnification. For each of the samples, 5 specimens of size 25.4mm x 6.35mm were examined (The total area examined is about 800 mm^2). As seen in Figure 3.12, the fiber and resin distribution in CYCOM prepreg is found to be uniform. Voids/Air Bubbles were seen in the prepreg material in a few places. Figure 3.13 and Figure 3.14 show images of voids at 100x and 350x magnifications.

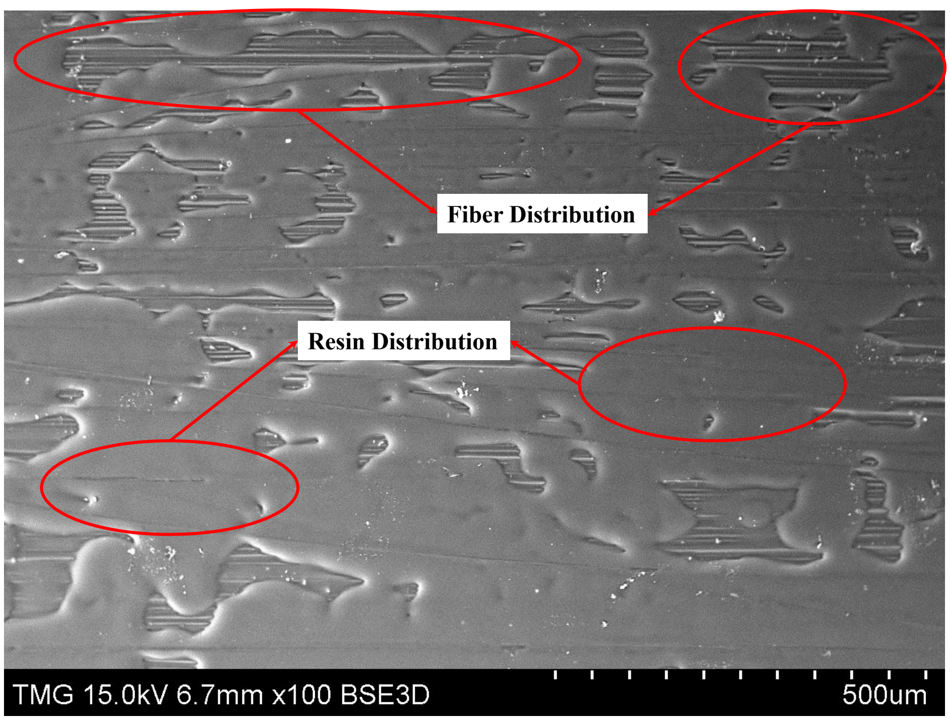


Figure 3.12 SEM Image of CYCOM 5320-1 Prepreg Material (100x)

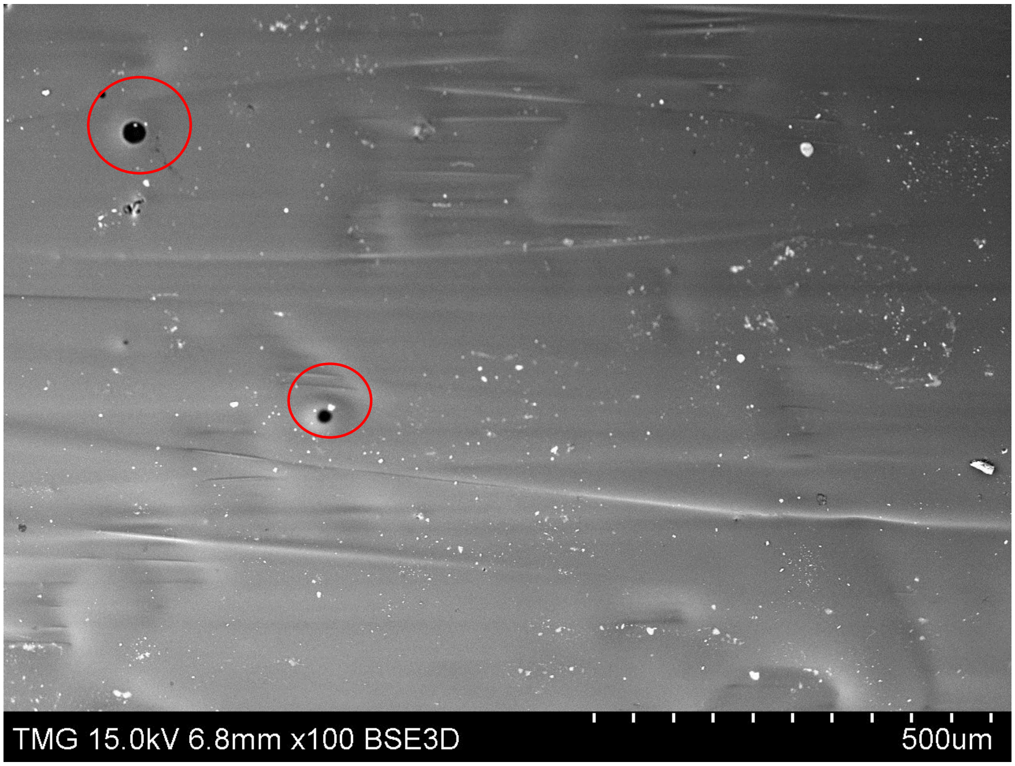


Figure 3.13 Voids in the CYCOM 5320-1 Prepreg Material (100x)

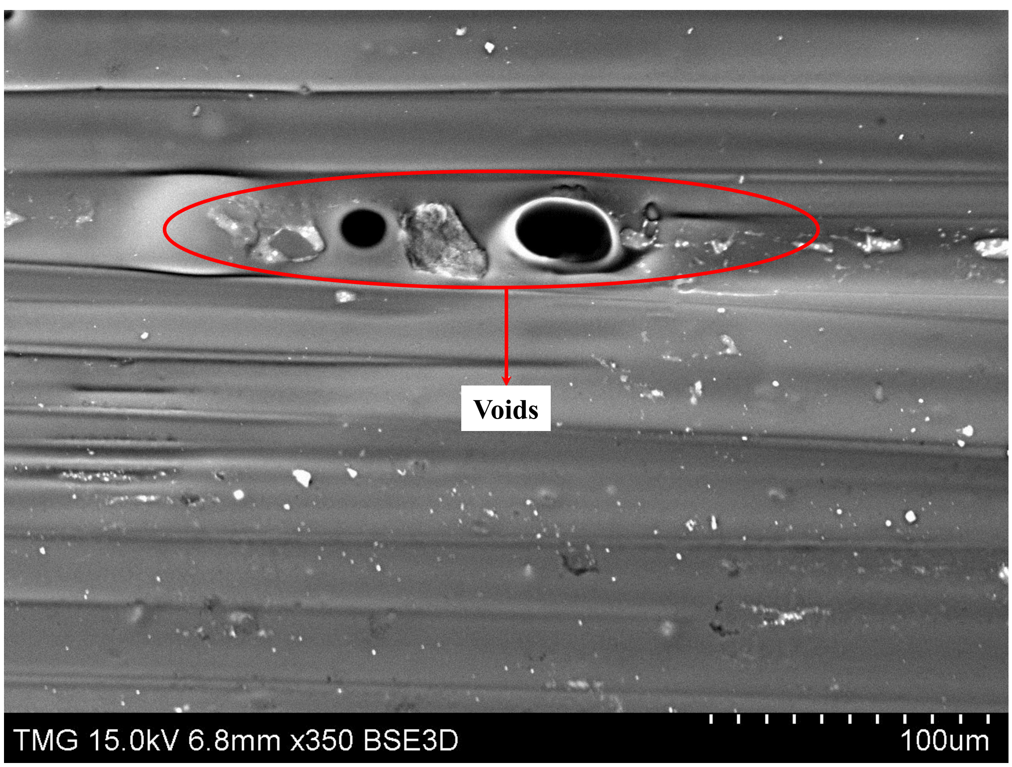


Figure 3.14 Voids in the CYCOM 5320-1 Prepreg Material (350x)

The microscopic images of the TC-250 resin system are shown in Figure 3.15 to Figure 3.17. The images have been taken at 50x, 100x and 350x magnifications respectively. The fiber and resin distribution is shown in Figure 3.16.

Figure 3.17 shows a typical resin-starved area in the prepreg raw material. These resin-starved areas have been observed in all the 5 different samples that were observed under the microscope for the TenCate prepreg material. These samples were cut from different places in the prepreg material tape. The percentage of these resin-starved areas (the ratio of the area of the 'resin starved areas' over the total area of the sample under observation) in a sample of size 25.4mm x 6.35mm was nearly 15%. The influence of this high percentage of resin-starved areas will be observed in the laminates made by this material.

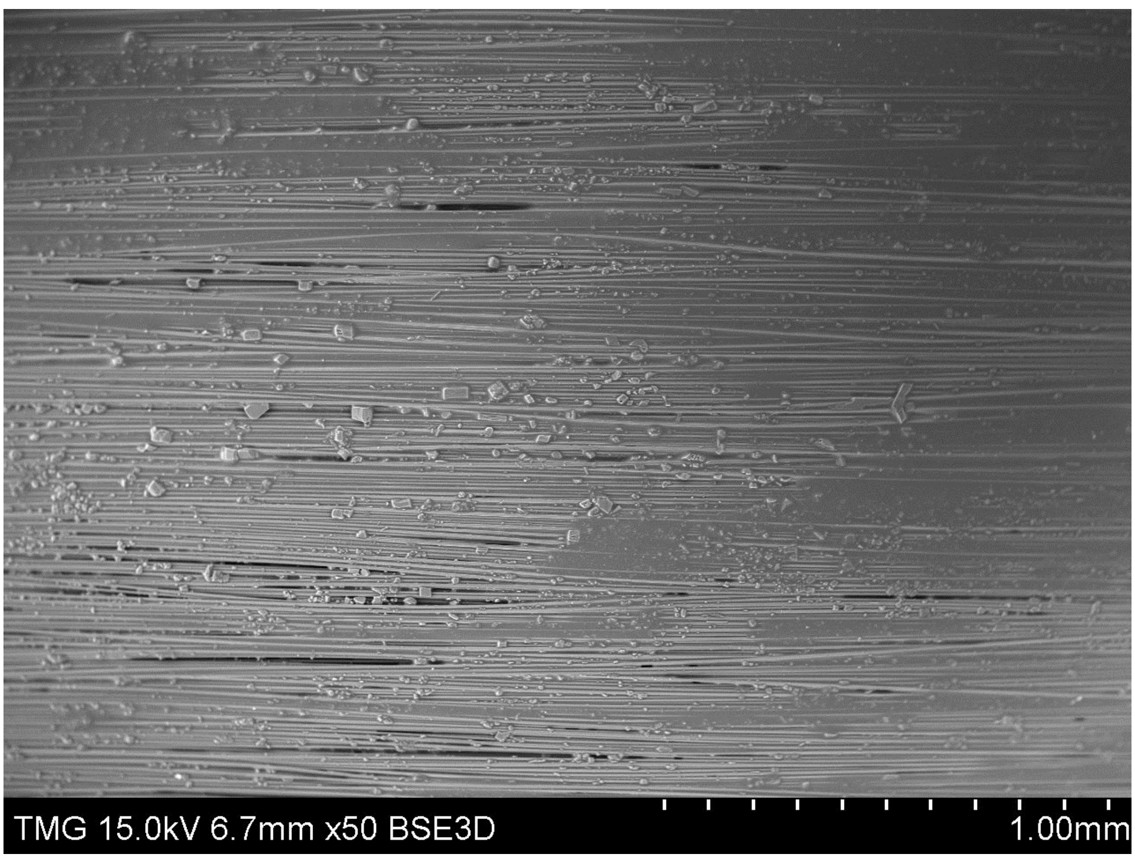


Figure 3.15 SEM Image of TC-250 Prepreg Material (50x)

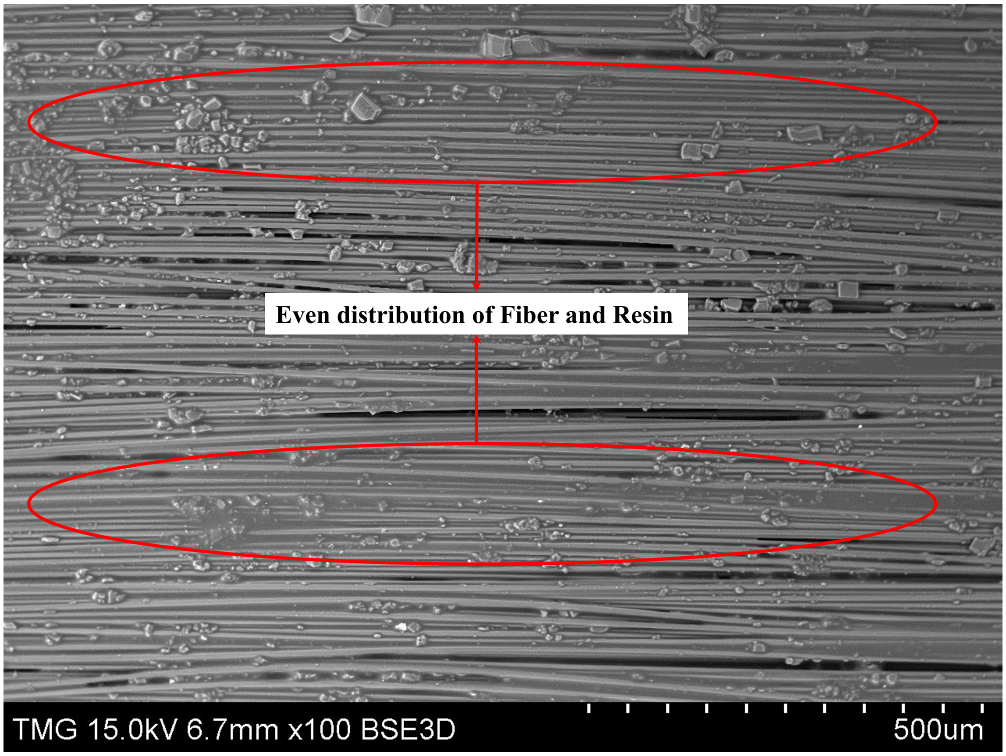


Figure 3.16 SEM Image of TC-250 Prepreg Material (100x)

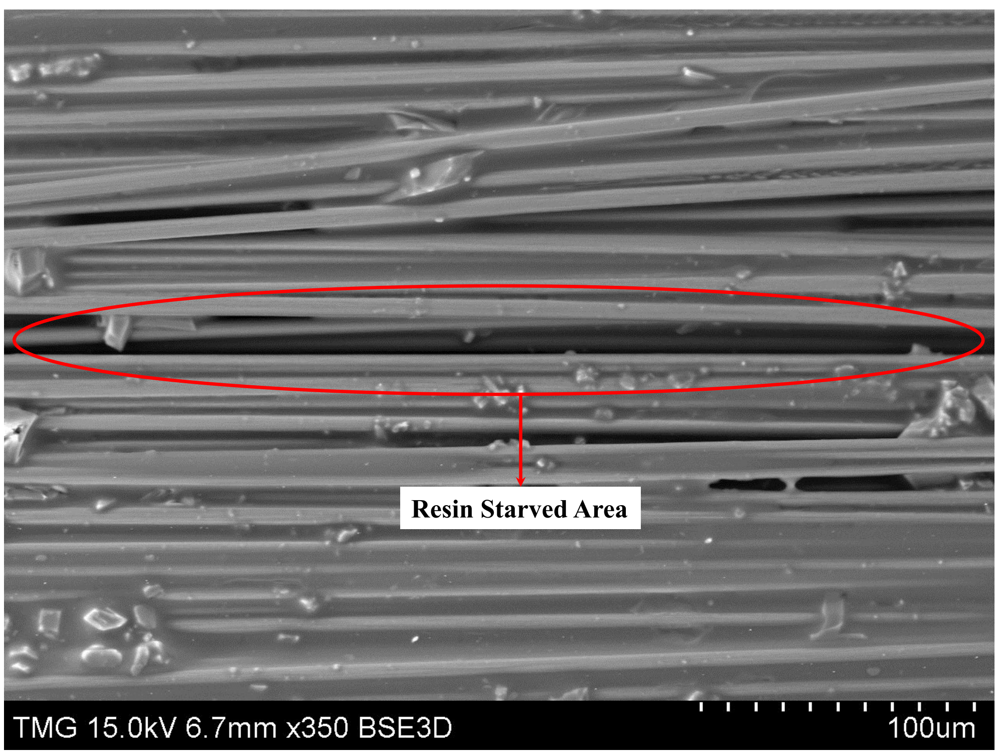


Figure 3.17 Resin Starved Area in the TC-250 Prepreg Material (350x)

The main motivation behind performing the DSC analyses, SEM analyses and the use of dual-loop temperature controller in the autoclave, is to give high importance to the quality control aspect because of the susceptibility of the OOA materials to form voids and resin rich areas. Following, these quality assurances, concrete and reliable results from the mechanical testing as discussed in Chapter 4, can be obtained.

3.4 Challenges in AFP Manufacturing Process

While manufacturing laminates for mechanical testing, the thickness of the laminate has to be uniform. All the samples made by Hand-Lay-Up process using the TC-250 OOA prepreg materials for the tensile, compressive and shear tests were perfectly flat and had no variation in thickness, width and length. A sample made using 16-layers of TenCate's TC-250 material is shown in Figure 3.18.

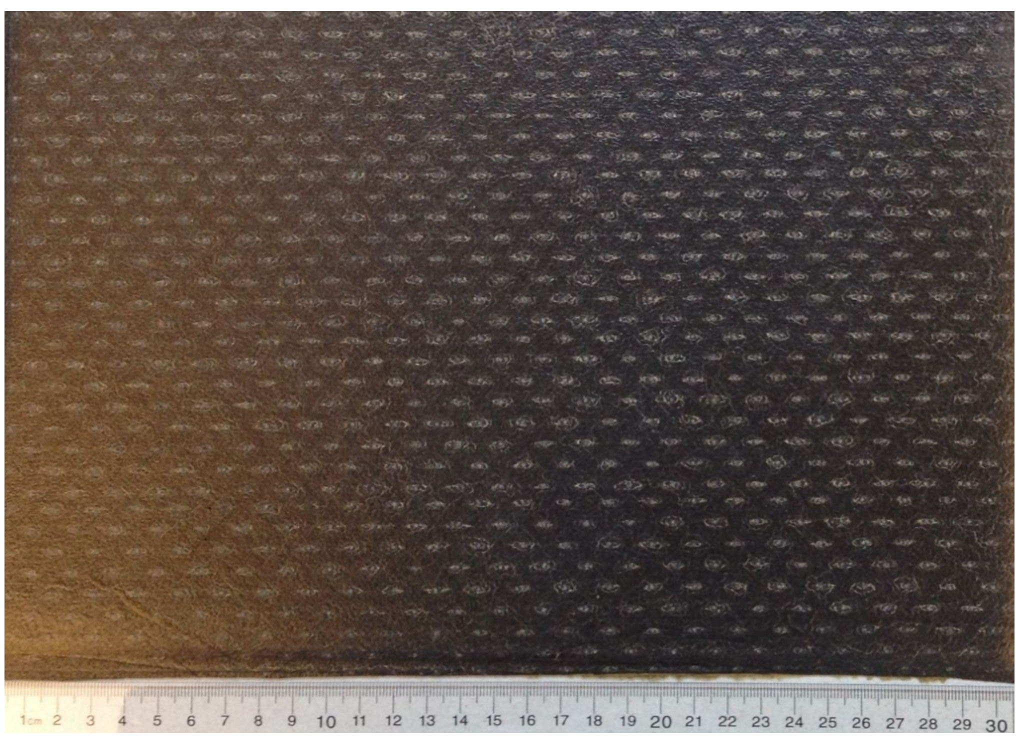


Figure 3.18 Sample made using TC-250 Material by Hand-Lay-Up Process

However, in the case of laminates made by the AFP process (using both TC-250 and CYCOM 5320-1 material), the task of making perfectly flat laminates has been challenging. This issue is further discussed in the Section 3.4.1.

The samples made by the AFP Process using both TenCate's and Cytec's OOA prepreg material have resulted in laminates with non-uniform thickness. This section presents the fabrication of the initial samples, the steps taken to improve the samples by changing the processing conditions while understanding the reason for this non-uniform thickness in the samples. To illustrate this further, images of samples, comparative graphs and SEM images have been presented in the following sections.

3.4.1 Initial Samples Manufactured by the AFP Process

The following process parameters were used in the AFP machine to manufacture the initial samples: HGT Torch Temperature: 200°C, Layup-Speed: 76.2 mm/sec (3 inch/sec) and Roller head Pressure: 135 N (30 lbf). It is to be noted that the temperature of the HGT torch reflects the temperature at the tip of the torch and not the temperature of the prepreg tow. When the HGT torch was at 200°C, the temperature of the incoming tape was measured in the range of 110°C -115°C using an Infrared laser thermometer.

Figure 3.19 and Figure 3.20 shows the initial samples which were made by the AFP process using the prepreg tape. These samples had a considerable degree of non-uniformity in thickness. In the first sample shown in Figure 3.19, the maximum and minimum thickness observed were 2.51 mm and 1.91 mm respectively, which accounts for nearly 31% difference in the thickness. The sample shown in Figure 3.20 is a $[0,90]_{10S}$ laminate, cut along the diagonal. This laminate also has similar variation in thickness as seen in the 0° laminate.

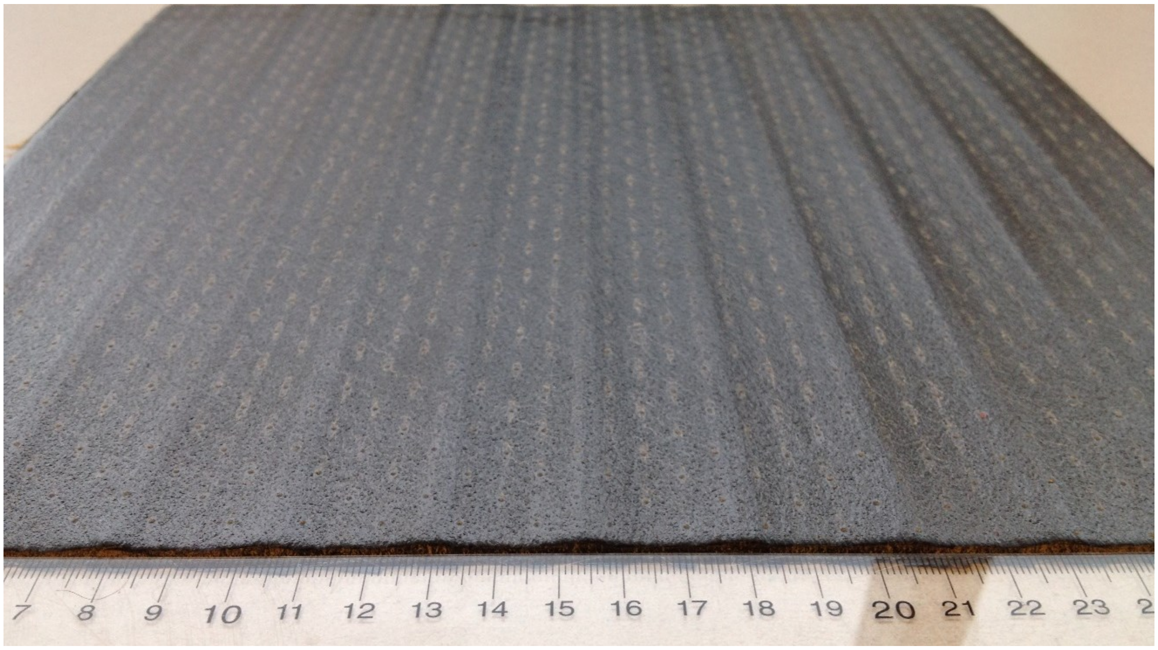


Figure 3.19 Initial Samples made by the AFP Process ($[0]_{16}$) using TC-250 Material

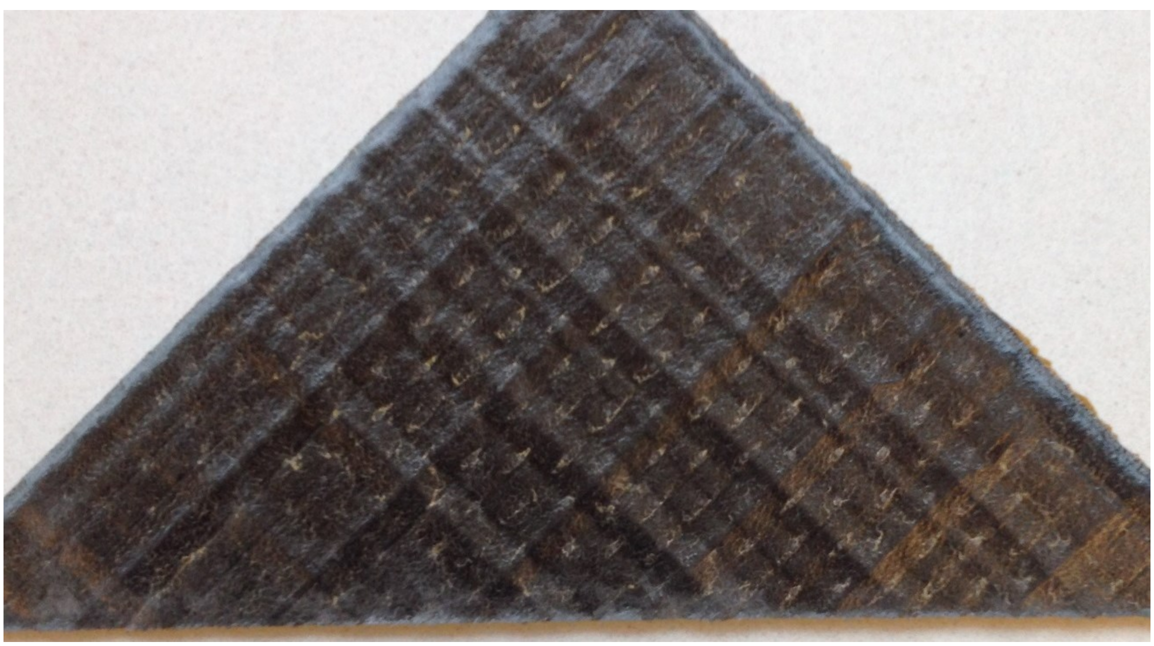


Figure 3.20 Initial Samples made by the AFP Process ($[0, 90]_{10s}$) using TC-250 Material

On careful observation, it was seen that the 4 tows (Band-1) which were getting laid by the AFP machine head on the tool had a total width of 26.5 mm (~1.05 inch). There was a slight gap between each tow, upon measurement it was found to be around 0.4 mm (the width of each towpreg is 6.35 mm).

This gap between each tow is for accommodating the increase in the tow-width due to heat exerted by the HGT torch, and compression roller pressure. Initially, the machine was programmed in a way so as not to leave any gap between the two adjacent bands (inter-band), i.e. there was no gap between the fourth tow of the first band and the first town of the second band. This is illustrated in Figure 3.21.

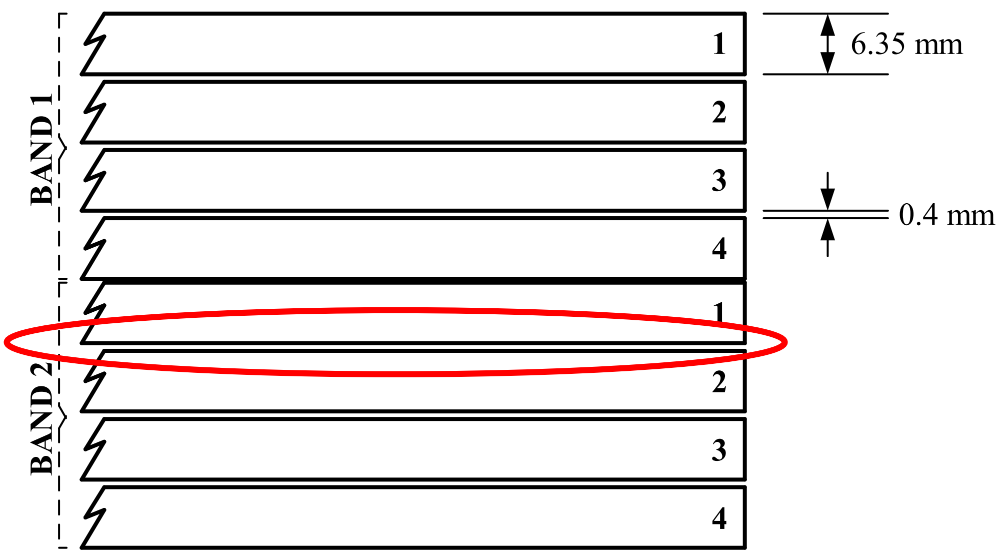


Figure 3.21 Initial Tow Placement Set-up

Since there was no gap between the fourth tow of the first band and the first tow of the second band, the material in between these towpregs had physically no area to undergo lateral expansion. Changing the gap between the bands (inter-band spacing) was the first iterative measure to the AFP process to achieve laminates with uniform thickness.

3.4.2 Changing AFP Process Parameters to Improve the Samples

A few iterations while changing the AFP process parameters resulted in lesser variations in the thickness. The lay-up speed was reduced from 76.2 mm/sec to 50.8 mm/sec, and the temperature of the HGT torch was also reduced to 190°C from 200°C. Even by reducing the temperature of the torch, the tackiness of the tape (required for the towpregs to stick to the mandrel or previous layers) was maintained because of the lower lay-up speed.

The new set of processing parameters for the AFP machine used while making the new set of laminates were: HGT Torch Temperature: 190°C, Layup-Speed: 50.8 mm/sec (2 inch/sec) and the Roller head Pressure: 135 N (30 lbf).

Also, a gap of 0.4 mm (equivalent to the gap between each towpreg) was specified between each band. This is illustrated in Figure 3.22.

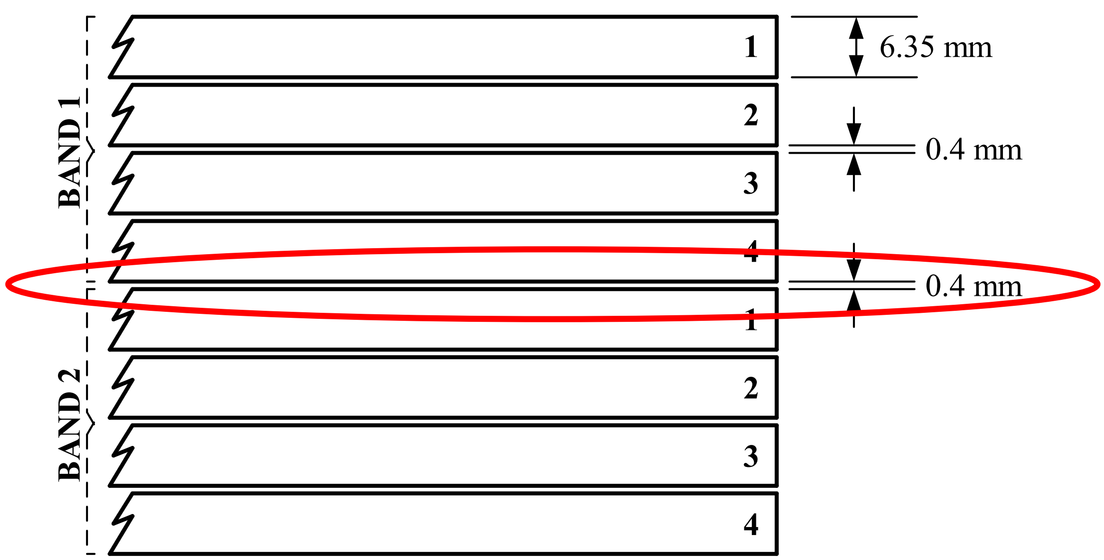


Figure 3.22 Final Tow Placement Set-up

Additionally, the second layer of the prepreg material was laid by an offset of 3.175mm, i.e. half the width of the prepreg tow to normalize the effects of these gaps. This was not done in the samples manufactured before. This further reduced the thickness variation in the laminates. This is illustrated in Figure 3.23.

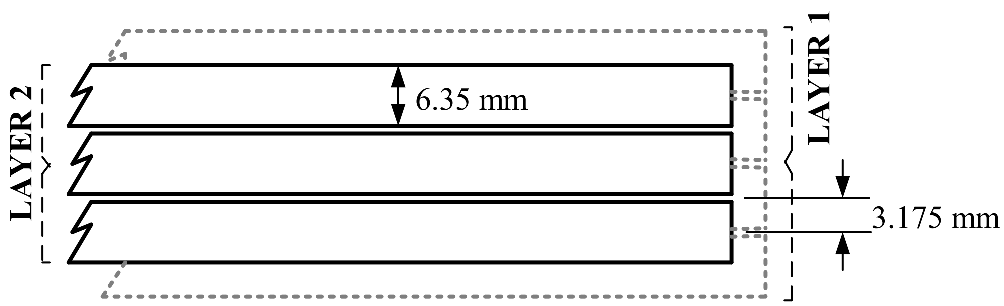


Figure 3.23 Offset between each Layer of laid Prepreg Material

The new set of samples were manufactured using the new AFP processing parameters, considering the gap between each band laid and offsetting each layer of the material stacked. These were the new AFP parameters used for making the plate. Any further increase (or decrease) in the gap between bands will only result in more variation in thickness. Also the layer-offset resulted in less thickness variation at 3.175 mm offset, which is half the width of the prepreg tow.

These samples had about 15% difference in thickness as compared to that of 31% in the samples manufactured initially (It is to be noted that when a coupon of 12.7 mm is cut from this sample, the thickness variation seen in that coupon is less than 8%). The thickest section measured 2.38 mm and the thinnest section was 2.09 mm. The images of $[0_{16}]$ laminate are shown below in Figure 3.24 and the images of $[0/90]_{10s}$ laminate cut along the diagonal, manufactured with the new AFP parameters is shown in Figure 3.25.

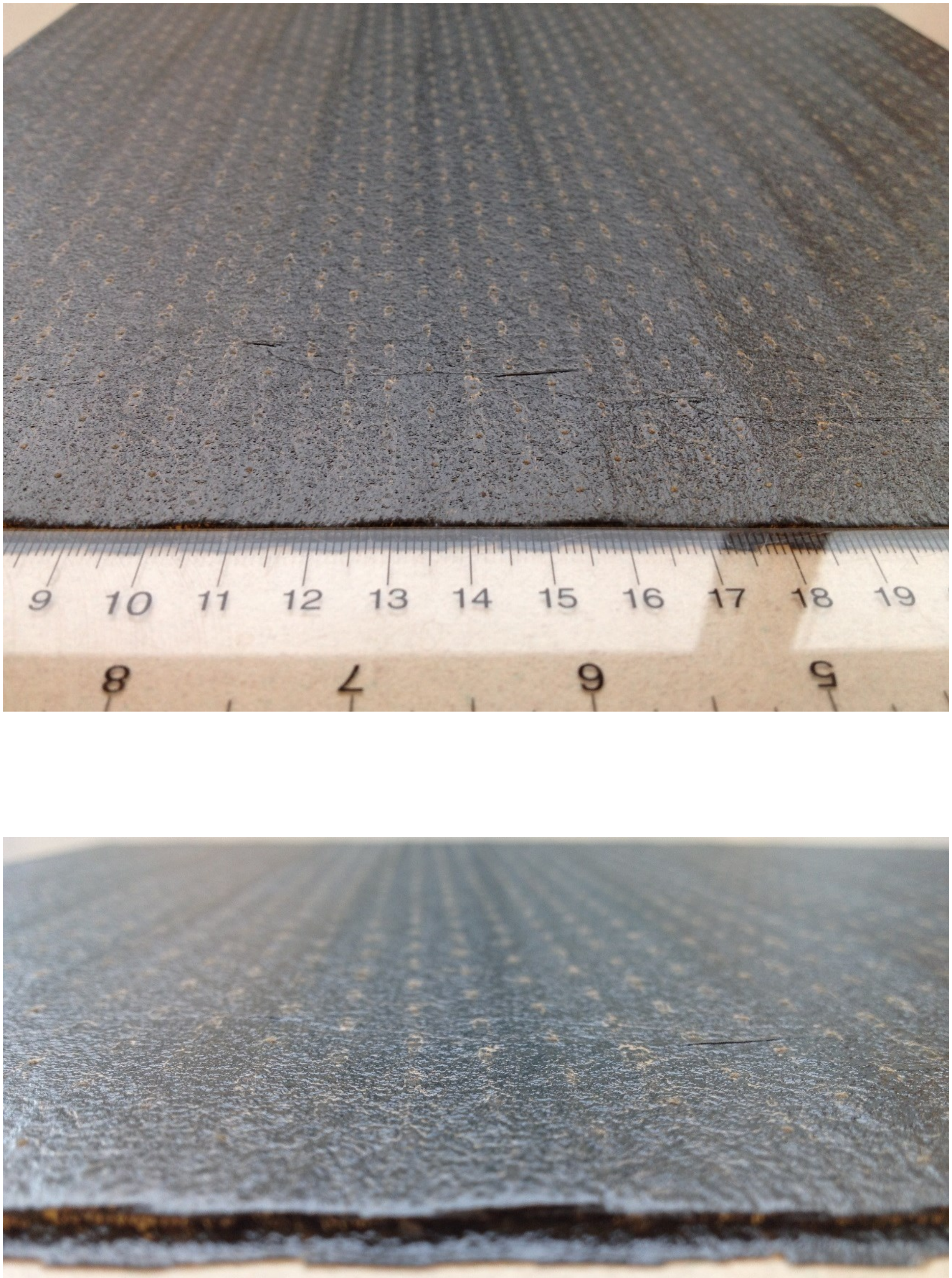


Figure 3.24 Final Sample made by the AFP Process ($[0]_{16}$) using TC-250 Material

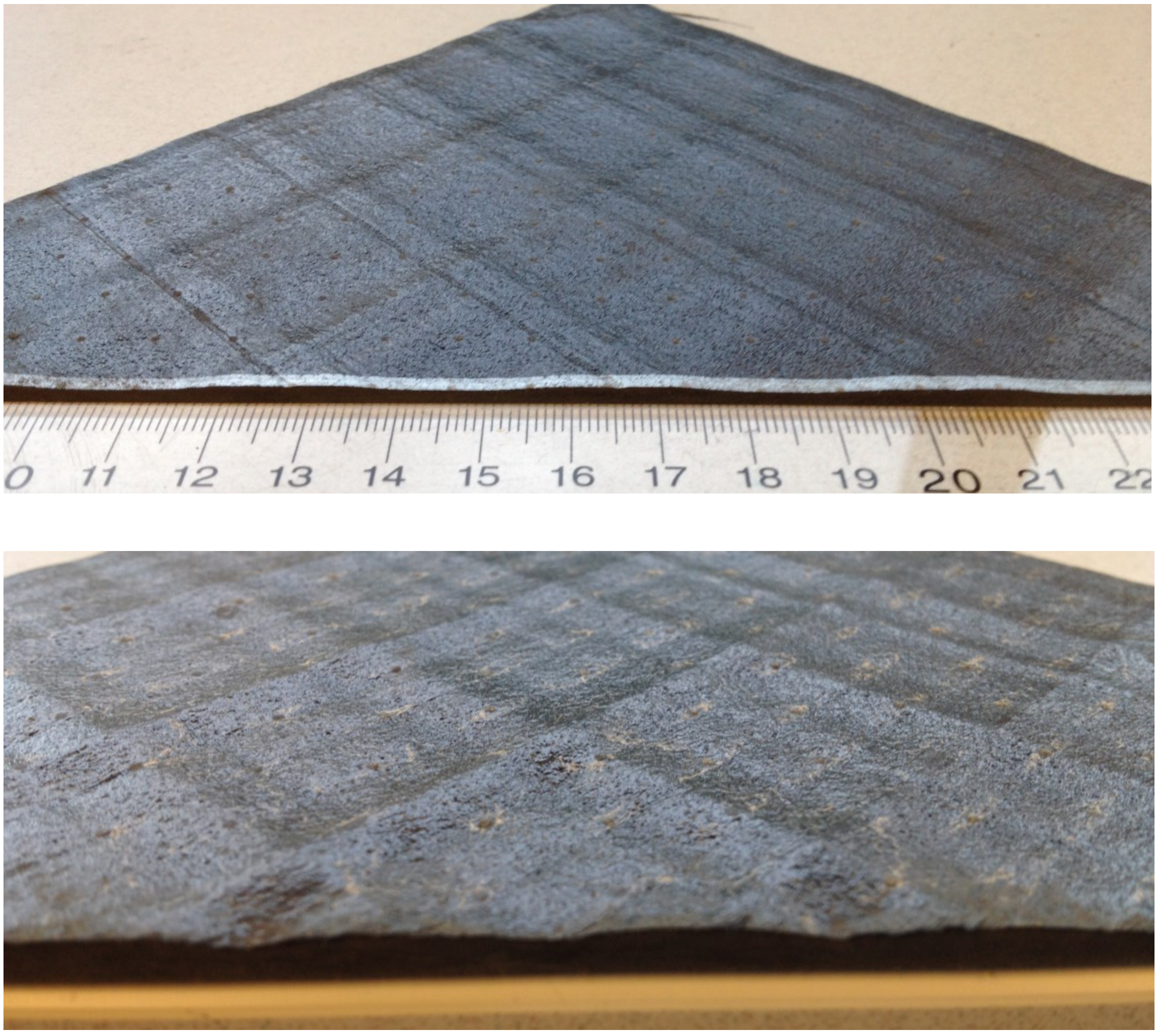


Figure 3.25 Final Sample made by the AFP Process ($[0, 90]_{10s}$) using TC-250 Material

3.4.3 Graphical Representation of Thickness Variation

To understand the differences in the thickness variation in the initial and final samples, the thickness of these samples were measured at various locations in between two crests (thickest section of the sample).

Measurements were taken at 5 different areas in each $[0_{16}]$ sample. The data was then approximated with an error ±7%, to make average curves showing the thickness variation in initial and final samples. It was observed that the pattern repeats itself after every 25.5

mm (~1 inch). Figure 3.26 represents the variation in thickness of initial and final samples graphically.

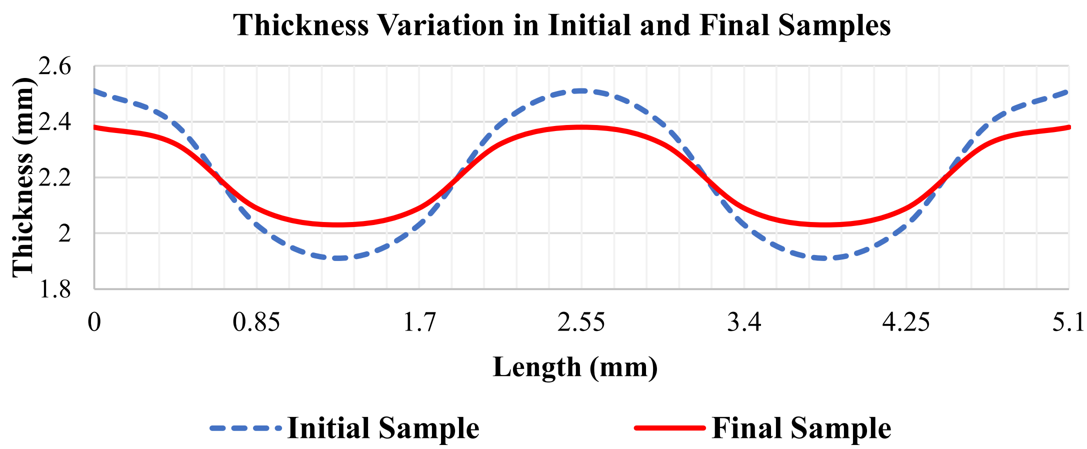


Figure 3.26 Thickness Variation in Initial and Final Samples

To further validate these above curves, the surface profile of the samples were analyzed using the 'Mitutoyo Surftest SJ 400' profiliometer. The image of the profiliometer with the samples is shown in Figure 3.27.

Figure 3.27 Mitutoyo Surftest SJ 400 Profilometer

The images were taken while using profilometer along various sections in the composite samples. The maximum distance measured by the profilometer is 12.5mm. Figure 3.28 shows the schematic diagram of various cross section where this profile analysis is performed on the sample and Figure 3.29 to Figure 3.31 shows images of the profile curve taken at these sections while doing analyses using profilometer.

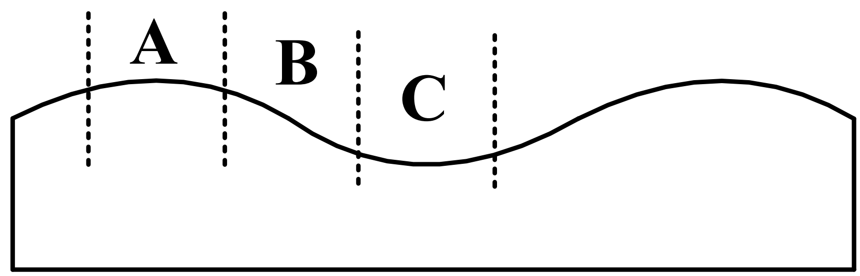


Figure 3.28 Schematic Diagram showing the Cross-section of the Sample

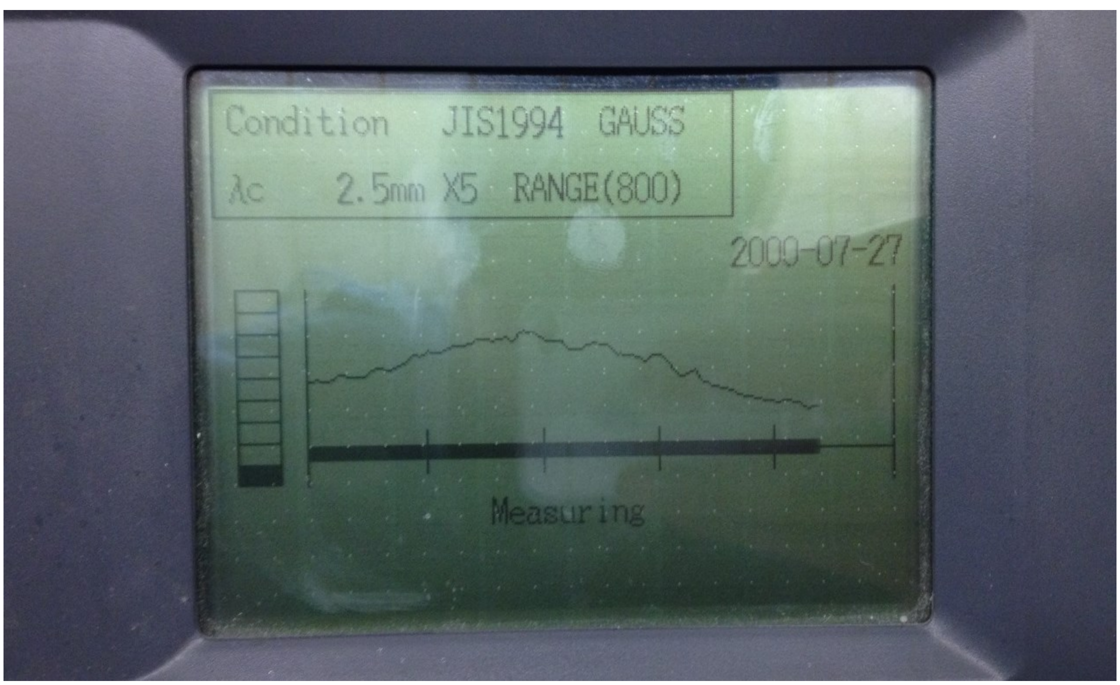


Figure 3.29 Image from the Profilometer at Section 'A'

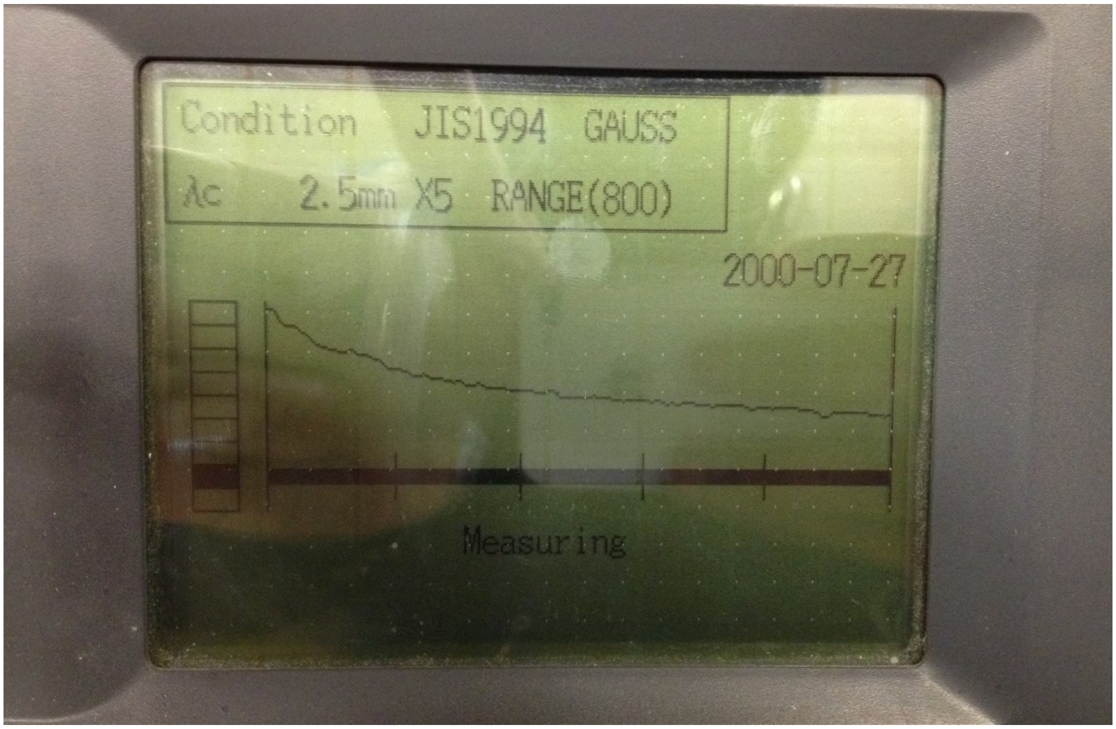


Figure 3.30 Image from the Profilometer at Section 'B'

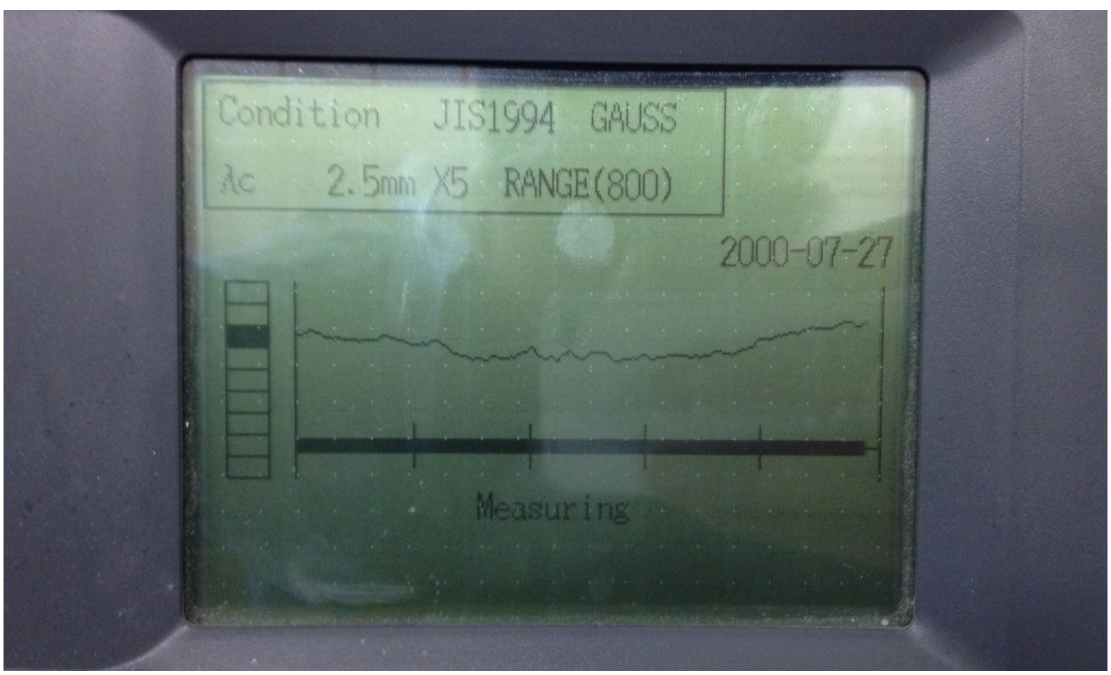


Figure 3.31 Images from the Profilometer at Section 'C'

To further study the effects of these variations in thickness at the microscopic level, SEM analyses have been performed to see the distribution of fibers in the samples.

3.4.4 SEM Analyses of the Samples

SEM images were taken at various segments in the samples manufactured by AFP. The SEM images of the initial samples made by AFP is presented in Figure 3.32, taken at 50x magnification to show the flow of fibers in the initial set of samples.

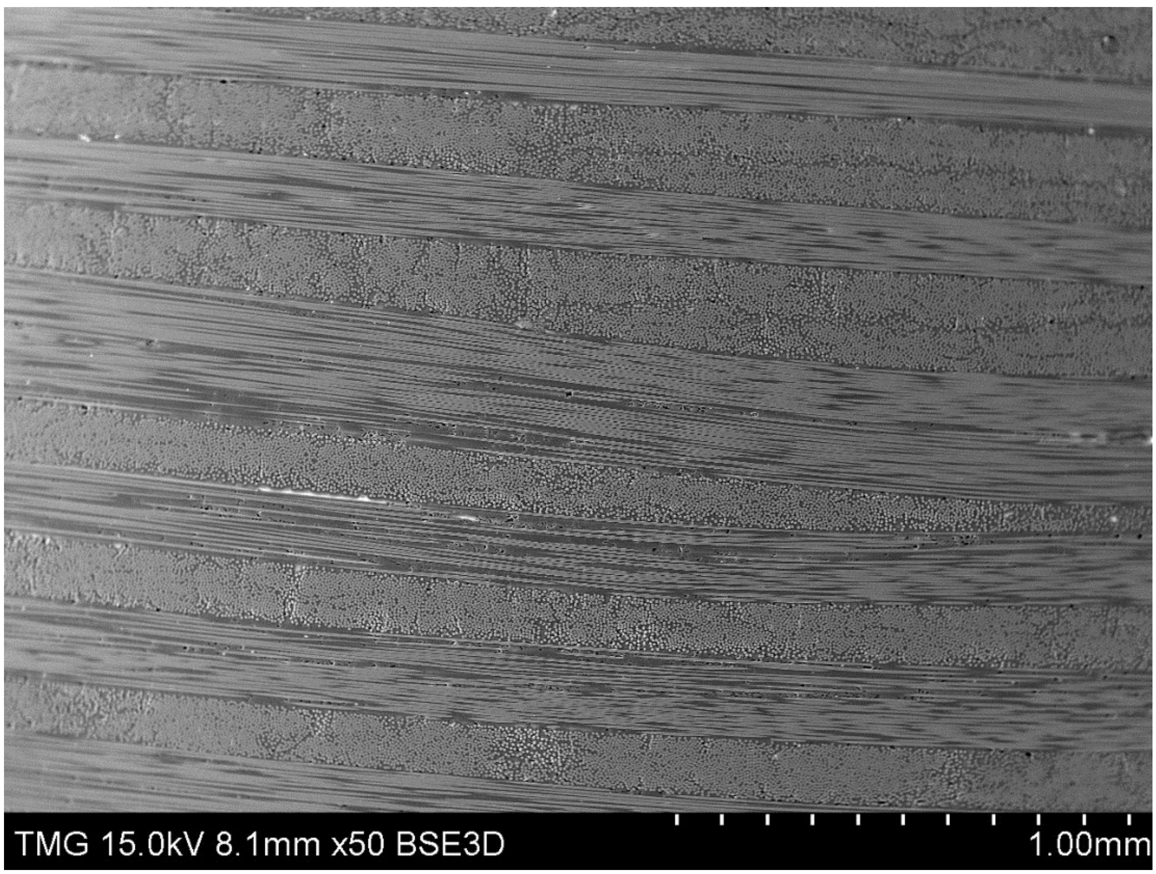


Figure 3.32 Thickness Variation in Initial Sample at Cross-section 'B'

The variation in thickness of each layer of in the initial sample can be seen in the SEM image in Figure 3.32. The varying thickness of the layers is the primary reason why the thickness of overall sample is varying. It is to be noted that the image is take in the middle of a [0, 90]$_{10S}$ sample and hence the thick band of fibers seen in the middle of the image. In Figure 3.33, another segment of the intial sample is seen. The waviness of layers at the micro-level is evident from these images. Although there are no voids and resin starved areas, the improper variation of fiber orinetation in a layer can impact the strength of the samples. It will be seen in the following sections in this chapter.

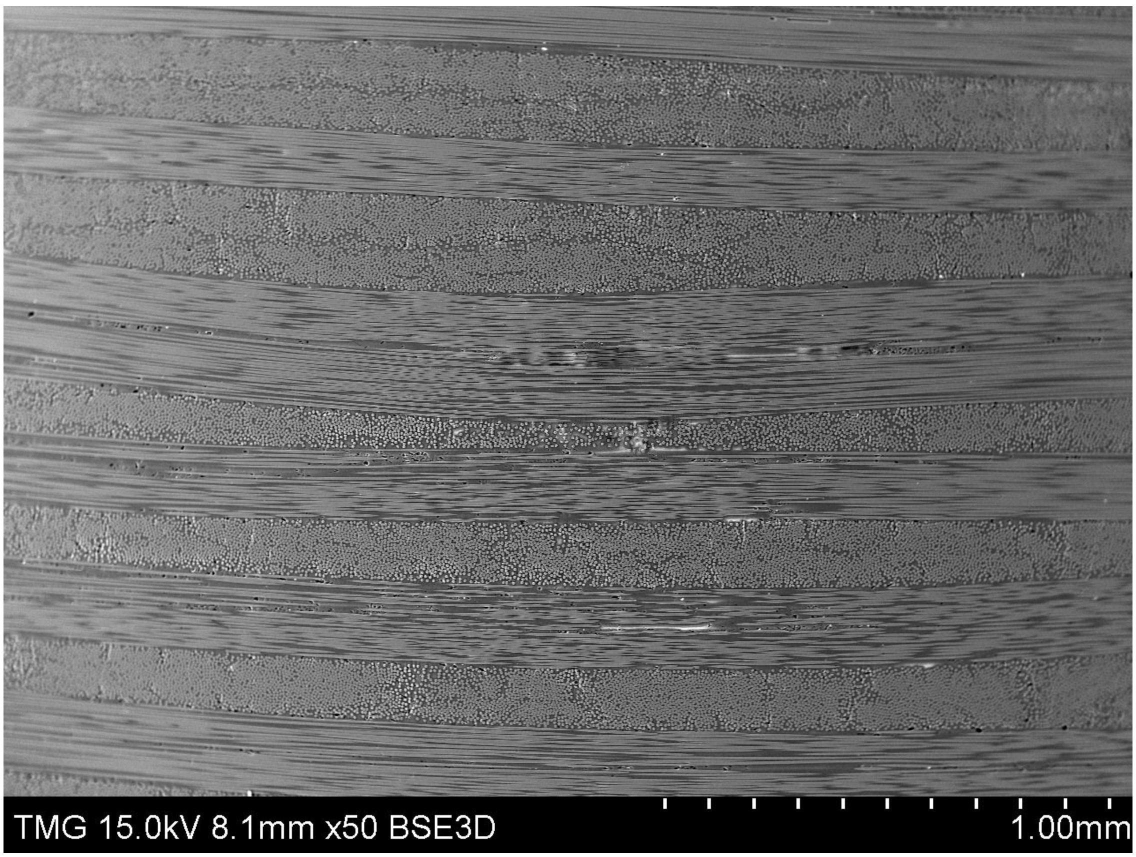


Figure 3.33 Thickness Variation in Initial Sample at Cross-section 'A'

SEM images were also taken for both the initial and final samples that were manufactured using AFP machine to calculate average variation in thicknesses of the layers in each of these samples. The SEM images of the initial sample is presented in Figure 3.34, taken at 100x magnification. The SEM images of the final sample is presented in Figure 3.35, taken at 100x magnification.

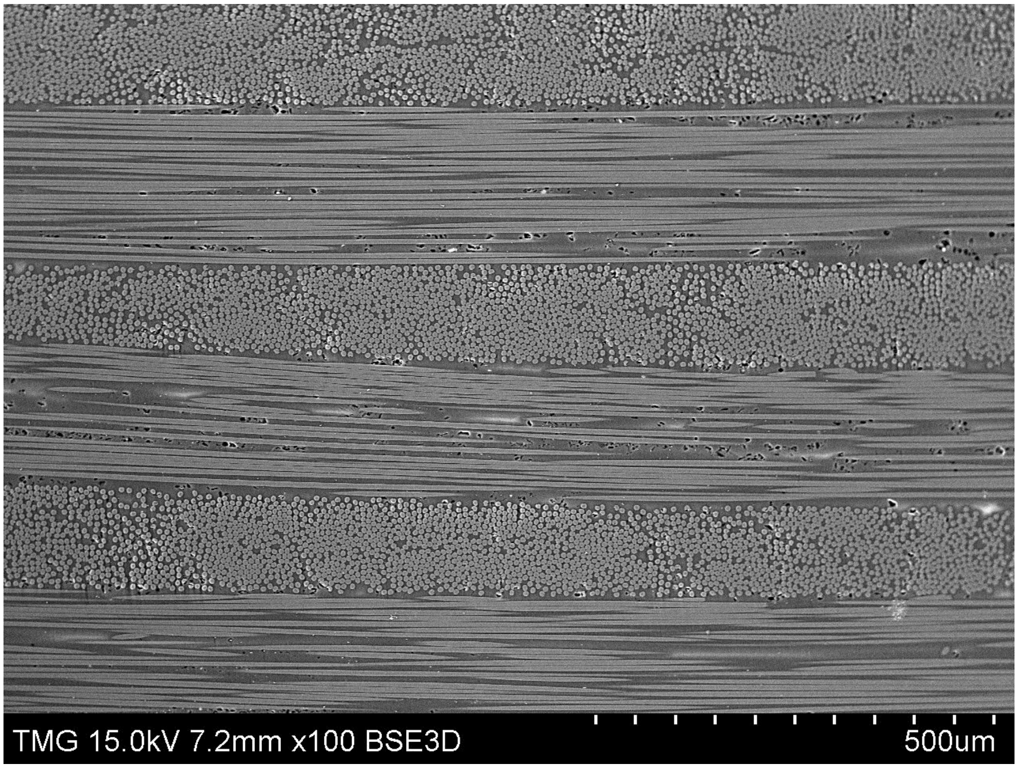


Figure 3.34 Thickness Variation in each layer of the Initial Sample

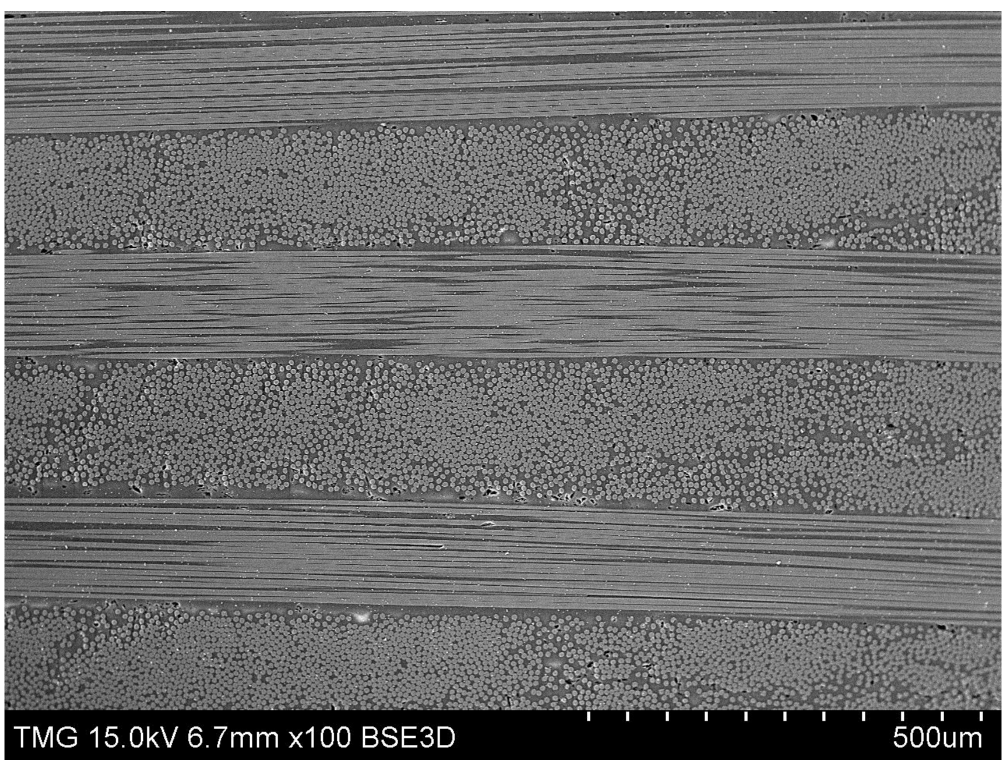


Figure 3.35 Thickness Variation in each Layer of the Final Sample

From the SEM images, it can be observed that the fiber distribution and the thickness of layers in the final sample is comparatively more uniform than those in the initial sample. Quantification of the thickness variations at the microscopic level present 30% maximum variation in thickness in the initial sample and 18% maximum thickness variation in the final sample. These values were obtained by taking the average of the maximum thickness variation of 5 different layers in each microscopic sample. These values are close to that observed in the physical measurement of the entire cross section of the sample. Due to the constraints presented by the AFP machine (Overall band width cannot be changed), no further modification was possible to adjust the gap between each tow (and each band of material). Increasing the gap between the bands will also result in non-uniform distribution of fibers and the issues pertaining to thickness variation will prevail. Furthermore, by decreasing the temperature of the HGT torch below 190°C, the necessary tackiness of the prepreg tows was lost, which is required to keep the material sticking in each layer and to the mold.

Apart from the gap between the tows, another possibility that could have caused the variation in thickness is the cross section of the prepreg tape. Garnich and Kymshyn [42] had considered a non-rectangular cross-section of the prepreg tape while working on stamp forming of woven composite. This will be presented in Section 3.4.5.

3.4.5 Analysis of the Cross-Section of the Prepreg Tape

The cross-section of the prepreg tape plays an important role in the surface profile of a cured laminate. In Figure 3.36 the first image shows a 3-D model of prepreg that has rectangular cross-section. The thickness of the prepreg tape is measured as 0.25 mm and the width of the towpregs is 6.35 mm. In the second image, the prepreg tape considered is curved at the edges, for simplicity, it was assumed to be a semi-circle in this case.

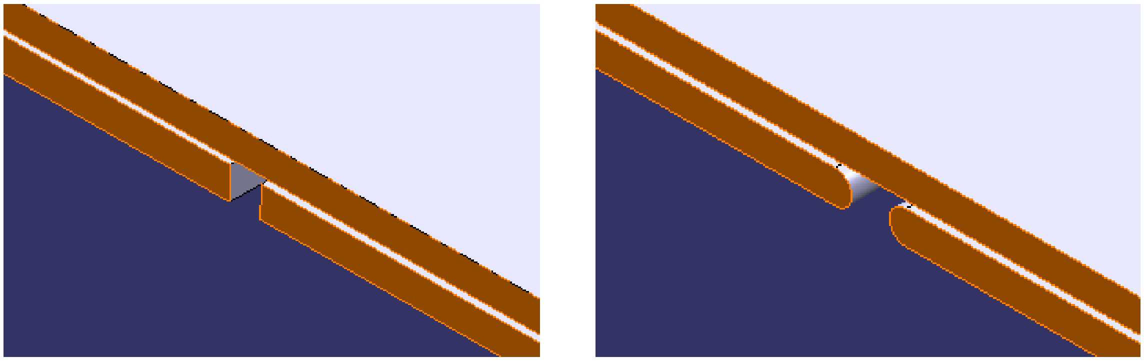


Figure 3.36 Cross-Sections of the Prepreg Tape

To further validate this, images of the cross-section of the prepreg tape were taken. To take the images of the cross-section, the prepreg material was cut, this was done by a tungsten carbide coated knife which was used to cut the cross-section of the towpregs swiftly. Images of the cross-section were obtained which showed the cross-section of the prepreg tape to be curved. Figure 3.37 show the microscopic set-up and the optical image of the cross-section of the prepreg tape (towpreg) that has a curved edge.

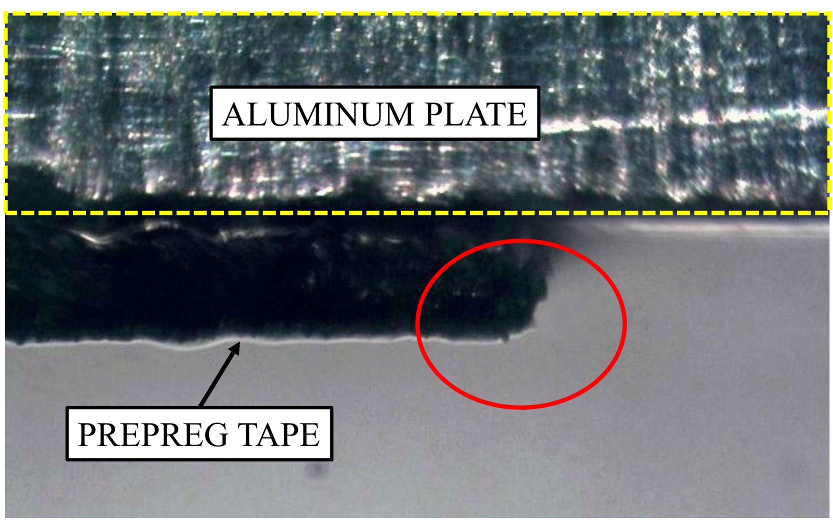


Figure 3.37 Optical Image of the Towpregs Cross-Section Edge

From Figure 3.37, it can be observed that the edge of the towpreg is curved and does not have a rectangular cross-sectional area. This cross-sectional area of the prepreg tape is another major factors contributing to the variable thickness issue present in the laminates, along with the towpreg gap issues, as discussed. For the purpose of characterization of these materials using AFP process, samples were made using the modified AFP process parameters.

3.4.6 Analysis of Tow-Placement in a band from guide-shoot

To further understand the variation in thickness of AFP samples, the intra-band gap between the tows was carefully observed. After repeated tests, it was observed that the gap between the tows is not same. In fact, it was observed that the gap between Towpreg-2 and 3 in a band is always larger than the gap between Towpreg-1 and 2 and the gap between Towpreg-3 and 4; as seen in Figure 3.38.

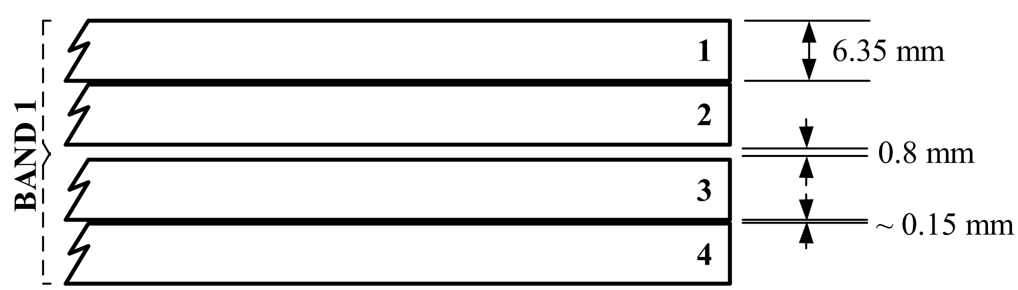


Figure 3.38 Intra band width between Towpregs

Also, the gap between Towpreg-3 and 4 was variable and showed values higher than an average of 0.4 mm. This sort of variation can be a cause of non-uniform thickness in the laminate. Also, this variation in gap between the towpregs in uncontrollable. It majorly depends on the path traversed by towpreg in the guide shoot on the AFP machine head.

Figure 3.39 shows the picture of four towpregs in a band. It can be clearly seen that the gap between the towpregs in the band is non-uniform and particularly the gap between Towpreg-2 and 3 is larger than the others. This large gap between the Towpreg-2 and 3 is at the same place where the sample has the least thickness. This largely influences the thickness variation in the samples.

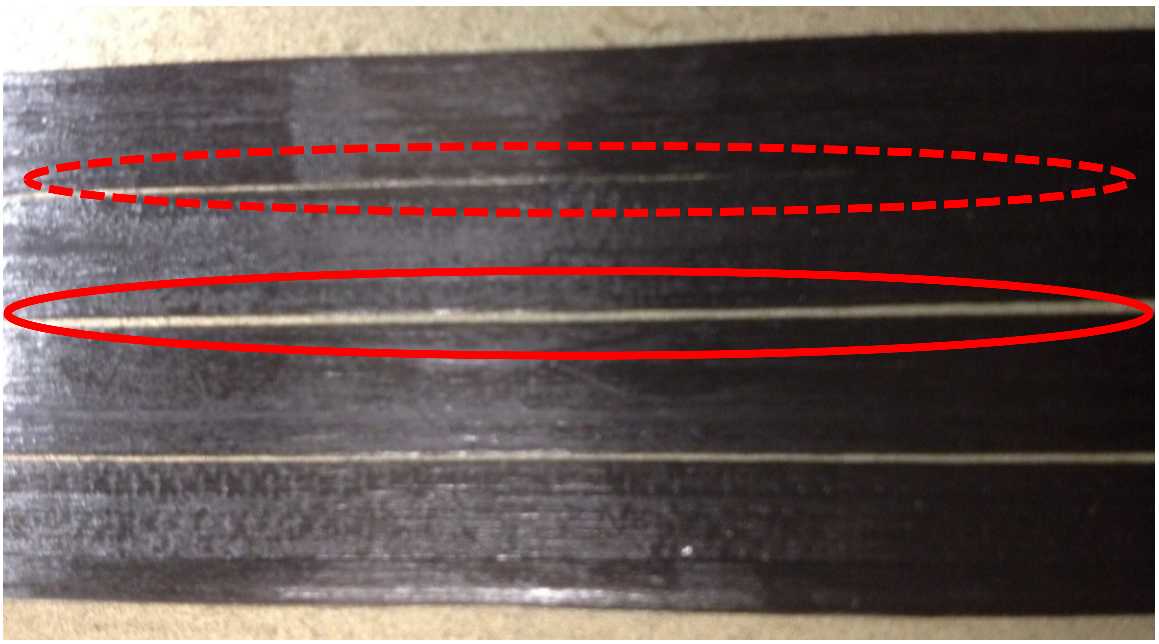


Figure 3.39 Non-uniform gap between the Towpregs in a Band

3.4.7 Conclusion

From the optical image, it is clear that the cross-section of the prepreg tape is curved and is not rectangular. Now, considering that the gap between two consecutive prepregs to be 0.4 mm, the areal gap between each consecutive prepreg tow is 0.1 mm^2 for the rectangular cross-section case and nearly 0.113 mm^2 for the case with semi-circular edges. Assuming that, the rectangular cross-section prepregs produces perfectly flat laminates, the 13% more area in the other case, has to be filled in by the prepreg above these layers or will result in voids. This can be the reason why the layers in the laminate made using AFP have non-uniform thickness, which in-turn causes the thickness variation in the manufactured sample.

4 TESTING, RESULTS AND DISCUSSIONS

Mechanical tests to determine the tensile, compressive and in-plane shear properties have been performed on the coupons made by different combinations of manufacturing techniques and curing cycles. These tests are important to understand the behaviour of real complex structures under multi-axial loads [41].

Table 4.1 shows the mechanical properties measured with their associated American Society for Testing and Materials (ASTM) standards and the size of coupons along with their respective stacking sequences. In order to normalize the data and to get accurate results, 7 to 13 coupons were cut and tested for each sample laminate.

Table 4.1 ASTM Standard, Measured Property, Coupon Dimensions and Stacking Sequence

ASTM Test Method	Measured Property	Coupon Length/ Width (mm)	Stacking Sequence
D3039	Tensile (0°)	254/12.7	$[0]_8$
D3039	Tensile (90°)	177.8/25.4	$[90]_{16}$
D3518	In-Plane Shear	254/25.4	$[+45/-45]_{10s}$
D3410	Compressive (0°)	139.7/12.7	$[0]_{12}$

The $[0, 90]_{10s}$ laminate has been used to cut coupons along the diagonal resulting in coupons with $[+45/-45]_{10s}$ orientation.

4.1 Mechanical Testing

The present section discusses the mechanical tests performed on the coupons, for both the prepreg materials. The results obtained from the mechanical tests will be discussed and the mode of failure is also presented. A picture of the most prominent failure mode is also shown in every test section.

In the chapter, a coupon identified as 'HLU+AC' refers to a coupon cut from a laminate made by Hand-Lay-Up process and cured in autoclave with pressure and 'HLU+OOA' refers to a coupon cut from a laminate made by Hand-Lay-Up process and cured without any external pressure. Similarly, 'AFP+AC' refers to a coupon cut from a laminate manufactured by AFP process and cured in autoclave with pressure and 'AFP+OOA' refers to a coupon cut from a laminate manufactured by AFP process and cured without any external pressure. Also, the TenCate's TC-250 resin system will be addressed as 'TC-250' and the Cytec's CYCOM 5320-1 will be addressed as '5320-1'.

4.1.1 Tensile Test (0°)

ASTM D3039 was employed to determine the tensile strength and modulus. Static loads have been introduced into the specimen by a hydraulic grips at a standard head displacement rate of 1.27 mm/min, until the load dropped significantly [43]. The tensile moduli and strengths were calculated from the slopes of the stress-strain curves for the 0° samples. Table 4.2 shows the average Tensile Moduli along the fiber direction (0°). Both the prepreg materials utilized to make the coupons are presented below and the testing conditions employed were consistent for both the materials.

From Table 4.2, it can be seen that in case of TC-250 material, the samples made by AFP process present better tensile modulus results along the fiber directions, than the ones made by Hand-Lay-Up process. The reason behind this can be the tension in the prepreg tape in AFP Lay-Up ensures the fibers laid are straighter and better compaction (can be concluding from the thickness values of the sample) than the Hand-Lay-Up counterpart, and thus resulting in high modulus along 0°. Also the curing of samples in autoclave with external pressure has resulted in comparatively higher tensile modulus, due to better compaction (seen in the coupon thickness variation). The difference in moduli of the samples AFP+AC and HLU+OOA is nearly 20%. In case of CYCOM 5320-1 material, the samples cured in autoclave with pressure are seen to produce slightly better (2% higher) tensile modulus results than the ones cured without any external pressure.

Table 4.2 Average Tensile Modulus, Standard Deviation and Coupon Thickness along 0°

Coupon Identification	Prepreg Material	Tensile Modulus (GPa)	Standard Deviation	Coupon Thickness
HLU+OOA	TC-250	140.2	1.82	1.13 mm
HLU+AC	TC-250	149.5	1.46	1.12 mm
AFP+OOA	TC-250	158	1.73	1.1 mm
AFP+AC	TC-250	167.2	2.00	1.1 mm
AFP+OOA	5320-1	159.7	1.15	1.09 mm
AFP+AC	5320-1	162.2	1.21	1.09 mm

Table 4.3 shows the average Tensile Strength along the fiber direction (0°) which follow the same trend as in the case of tensile modulus. For the TC-250 material, the samples made by AFP process have shown better tensile strength results than the ones made by Hand-Lay-Up process. The samples cured in autoclave present higher tensile strength. The percentage difference in strength between the samples AFP+AC and HLU+OOA is around 9%. In the case of CYCOM 5320-1 material, the samples cured in the autoclave with external pressure produced better tensile strength results than the ones cured without pressure. The percentage difference in tensile strengths is around 5%. The tensile curve of the AFP+OOA specimen made using TC-250 material is shown below in Figure 4.1.

Table 4.3 Average Tensile Strength, Standard Deviation and Coupon Thickness along 0°

Coupon Identification	Prepreg Material	Tensile Strength (MPa)	Standard Deviation	Coupon Thickness
HLU+OOA	TC-250	1697.2	7.94	1.13 mm
HLU+AC	TC-250	1754.6	7.12	1.12 mm
AFP+OOA	TC-250	1786.2	7.12	1.1 mm
AFP+AC	TC-250	1829	7.51	1.1 mm
AFP+OOA	5320-1	2342.4	5.78	1.09 mm
AFP+AC	5320-1	2462.3	6.12	1.09 mm

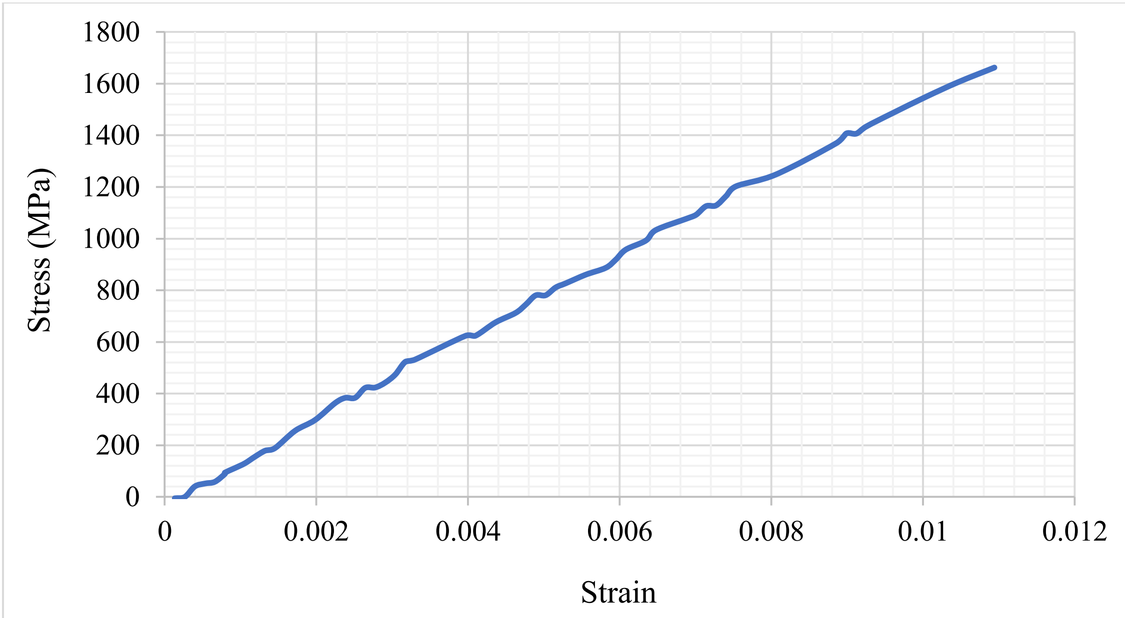


Figure 4.1 Tensile Curve of TC-250 Coupon (0°) made by AFP+OOA

The samples failed under the SGM (Splitting-Gage-Middle) and XGM (eXplosive-Gage-Middle) modes of failure. Figure 4.2 shows failed coupon made up of TC-250 material under the XGM and SGM modes respectively.

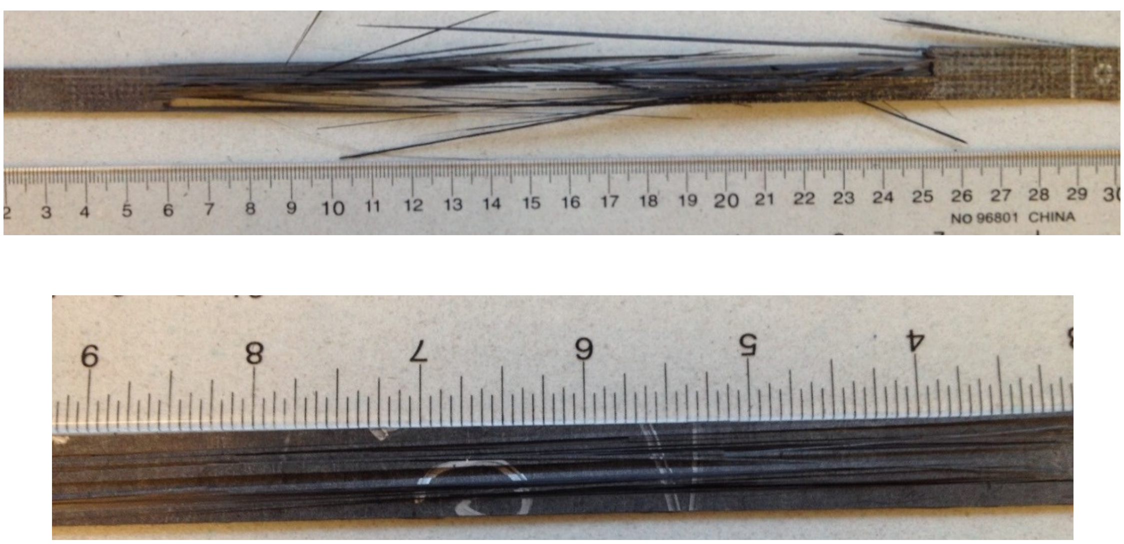


Figure 4.2 Tensile Test coupons (0°) failed under the XGM and SGM Failure Modes respectively

4.1.2 Tensile Test (90°)

ASTM D3039 was employed to determine the tensile strength and modulus. Static loads were introduced into the specimen by hydraulic grips at a standard head displacement rate of 1.27 mm/min, until the load dropped significantly [43]. The tensile moduli and strengths has been calculated from the slopes of stress-strain curves for the 90° samples. Table 4.4 shows the average Tensile Modulus perpendicular to the fiber direction (90°).

Table 4.4 Average Tensile Modulus, Standard Deviation and Coupon Thickness along 90°

Coupon Identification	Prepreg Material	Tensile Modulus (GPa)	Standard Deviation	Coupon Thickness
HLU+OOA	TC-250	8.7	0.6	2.26 mm
HLU+AC	TC-250	9.8	0.19	2.24 mm
AFP+OOA	TC-250	10.1	0.3	2.21 mm
AFP+AC	TC-250	11.2	0.36	2.21 mm
AFP+OOA	5320-1	11.7	0.18	2.17 mm
AFP+AC	5320-1	11.9	0.42	2.17 mm

As can be seen from Table 4.4, in case of TC-250 material, the samples made by AFP process and cured in autoclave show better tensile modulus results along 90°, than the ones made by Hand-Lay-Up process. The curing of samples in autoclave with external pressure resulted in comparatively higher tensile modulus. The autoclave curing process ensures better consolidation and resin-fiber distribution which in turn accounts for better load bearing capacities of the laminates. The AFP processing also ensures minimum gap (Thickness direction) in between layers which in turn helps in bearing the load. The difference is moduli of the samples AFP+AC and HLU+OOA is nearly 28%. In the case of CYCOM 5320-1 material, the samples cured in the autoclave with pressure produced better tensile modulus results than the ones cured without external pressure. However, the difference in the moduli is about 4%. The Table 4.5 shows the average Tensile Strength perpendicular to the fiber direction (90°).

Table 4.5 Average Tensile Strength, Standard Deviation and Coupon Thickness along 90°

Coupon Identification	Prepreg Material	Tensile Strength (MPa)	Standard Deviation	Coupon Thickness
HLU+OOA	TC-250	36.6	3.21	2.26 mm
HLU+AC	TC-250	43.4	2.30	2.24 mm
AFP+OOA	TC-250	46.6	2.07	2.21 mm
AFP+AC	TC-250	48.9	1.81	2.21 mm
AFP+OOA	5320-1	72.4	1.67	2.17 mm
AFP+AC	5320-1	76.2	2.34	2.17 mm

From Table 4.5, in case of TC-250 material, the samples made by AFP process and cured in autoclave show higher tensile strengths. The percentage difference in strengths of the samples AFP+AC and HLU+OOA is around 9%. In case of CYCOM 5320-1 material, the samples cured with pressure produced better tensile strength results than the ones cured without pressure. The percentage difference is tensile strengths is around 5%. The tensile curve of the AFP+OOA specimen made using TC-250 material is shown in Figure 4.3.

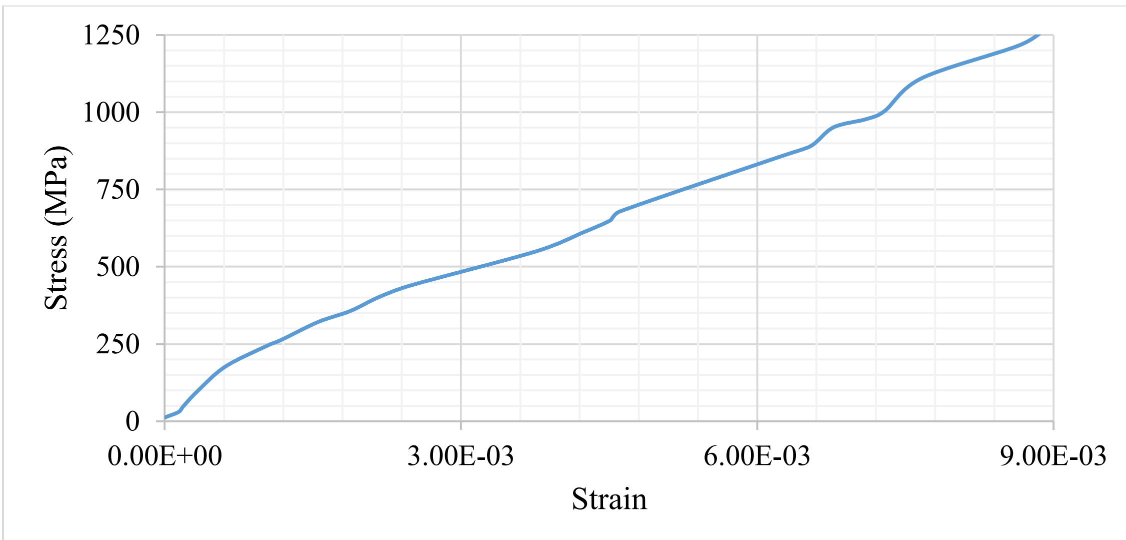


Figure 4.3 Tensile Curve of TC-250 coupon (90°) made by AFP+OOA

The samples failed under the LGM (Lateral-Gage-Middle) modes of failure. Figure 4.4 shows failed coupon made up of TC-250 material under the LGM modes.

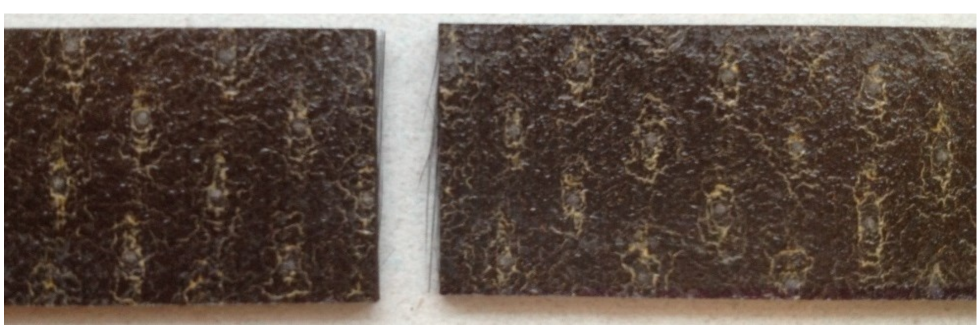


Figure 4.4 Tensile Test Coupon (90°) Failed under the LGM mode

4.1.3 In-plane Shear Test

ASTM D3518 was adopted to examine the In-plane shear modulus and strength. The testing has been conducted in accordance to the ASTM D3039 tensile test standard. Instead of having unidirectional fiber layout, the coupons had a symmetrical lay-up of $[+45/-45]_{10}$. All the other test conditions were maintained similar to that of the ASTM D 3039 standard [44]. Table 4.6 shows the Average In-Plane Shear Modulus for all the samples.

Table 4.6 Average In-Plane Shear Modulus, Standard Deviation and Coupon Thickness

Coupon Identification	Prepreg Material	Shear Modulus (GPa)	Standard Deviation	Coupon Thickness
HLU+OOA	TC-250	4.51	0.13	2.83 mm
HLU+AC	TC-250	5.05	0.17	2.82 mm
AFP+OOA	TC-250	5.29	0.13	2.80 mm
AFP+AC	TC-250	5.76	0.15	2.79 mm
AFP+OOA	5320-1	5.14	0.16	2.73 mm
AFP+AC	5320-1	5.41	0.11	2.73 mm

From Table 4.6, in case of TC-250 material, comparatively better results can be seen for the in-plane shear modulus test performed on the sample made by AFP process and cured in autoclave. The percentage difference between AFP+AC and HLU+OOA is nearly 27%. Both AFP processing and Autoclave curing plays crucial role in obtaining high strength laminates. For the CYCOM 5320-1 material, the sample made by AFP and cured with pressure has shown nearly 5% higher modulus than that made by AFP and cured without pressure. Table 4.7 shows the In-plane shear strength of the samples.

Table 4.7 Average In-Plane Shear Strength, Standard Deviation and Coupon Thickness

Coupon Identification	Prepreg Material	Shear Strength (MPa)	Standard Deviation	Coupon Thickness
HLU+OOA	TC-250	72.96	1.52	2.83 mm
HLU+AC	TC-250	79.08	0.87	2.82 mm
AFP+OOA	TC-250	81.56	1.02	2.80 mm
AFP+AC	TC-250	84.84	1.15	2.79 mm
AFP+OOA	5320-1	88.74	1.05	2.73 mm
AFP+AC	5320-1	92.4	0.63	2.73 mm

From Table 4.7, in case of TC-250 material, better In-plane shear strength can be seen in the sample made by the AFP process and cured with pressure in an autoclave. The percentage difference in the strengths of the samples AFP+AC and HLU+OOA is around 16%. In the case of samples made by CYCOM 5320-1 material, the shear strengths are comparable with one another and the percentage difference is around 4%. Figure 4.5 shows the shear curve for the TC-250 coupon made by TC-250 material using AFP+OOA process.

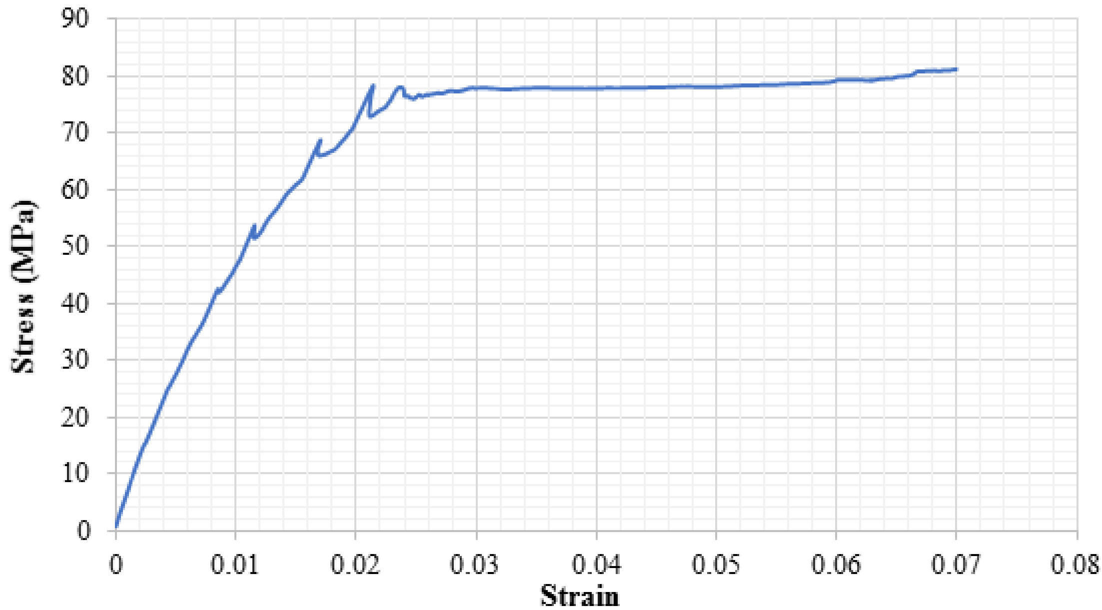


Figure 4.5 Shear Stress Curve of TC-250 Coupon made by AFP+OOA

The failure of most of the samples occurred at the middle of the coupon and is shown in Figure 4.6.

Figure 4.6 In-Plane Shear Test Coupon Failure

4.1.4 Compressive Test

ASTM D3410 was used to evaluate the compressive strength and modulus. The Illinois Institute of Technology Research Institute (IITRI) compression test fixture in accordance with the standard test method was used to provide stability in an un-notched compression testing [45].

Static loads were introduced progressively into the specimen via the fixture at a standard head displacement rate of 1.27 mm/min until the loads dropped significantly. Table 4.8 show the average compressive modulus of the samples.

Table 4.8 Average Compressive Modulus, Standard Deviation and Coupon Thickness along 0°

Coupon Identification	Prepreg Material	Compressive Modulus (GPa)	Standard Deviation	Coupon Thickness
HLU+OOA	TC-250	103.7	1.89	1.7 mm
HLU+AC	TC-250	114.6	4.03	1.69 mm
AFP+OOA	TC-250	113.4	5.66	1.67 mm
AFP+AC	TC-250	125.6	2.80	1.66 mm
AFP+OOA	5320-1	128.4	2.47	1.64 mm
AFP+AC	5320-1	132.4	3.15	1.64 mm

From Table 4.8, in case of samples made by TC-250 prepreg material, the samples cured with pressure showed better compressive moduli results than the samples cured without pressure.

Also, the samples made by AFP process resulted in higher moduli than those made by Hand-Lay-Up process. A marked observation from the test results is that the HLU+AC sample produced better result than the AFP+OOA sample. In other words both the samples cured in autoclave outperformed other samples.

Table 4.9 Average Compressive Strength, Standard Deviation and Coupon Thickness along 0°

Coupon Identification	Prepreg Material	Compressive Strength (MPa)	Standard Deviation	Coupon Thickness
HLU+OOA	TC-250	1223.4	6.02	1.7 mm
HLU+AC	TC-250	1252.6	6.50	1.69 mm
AFP+OOA	TC-250	1248.4	6.91	1.67 mm
AFP+AC	TC-250	1276.4	7.7	1.66 mm
AFP+OOA	5320-1	1359.6	6.23	1.64 mm
AFP+AC	5320-1	1424.8	5.1	1.64 mm

In the case of CYCOM 5320-1 material, slightly higher compressive moduli can be seen for samples cured with pressure. The percentage difference in the moduli however is around 3%. Table 4.9 shows the average compressive strength of the samples. From Table 4.9, in the case of TC-250 material, better results for compressive strength were obtained for the samples made by AFP process and cured in autoclave with pressure. In either manufacturing process, the samples cured in autoclave have shown better compressive strengths. The percentage difference in the compressive strengths of the samples HLU+AC and AFP+OOA is nearly 4%. In the case of CYCOM 5320-1 material, the compressive strength for the sample cured in autoclave with pressure is 5% better than the one cured without pressure. Figure 4.7 shows the compressive test curve for the TC-250 coupon made by AFP+OOA process.

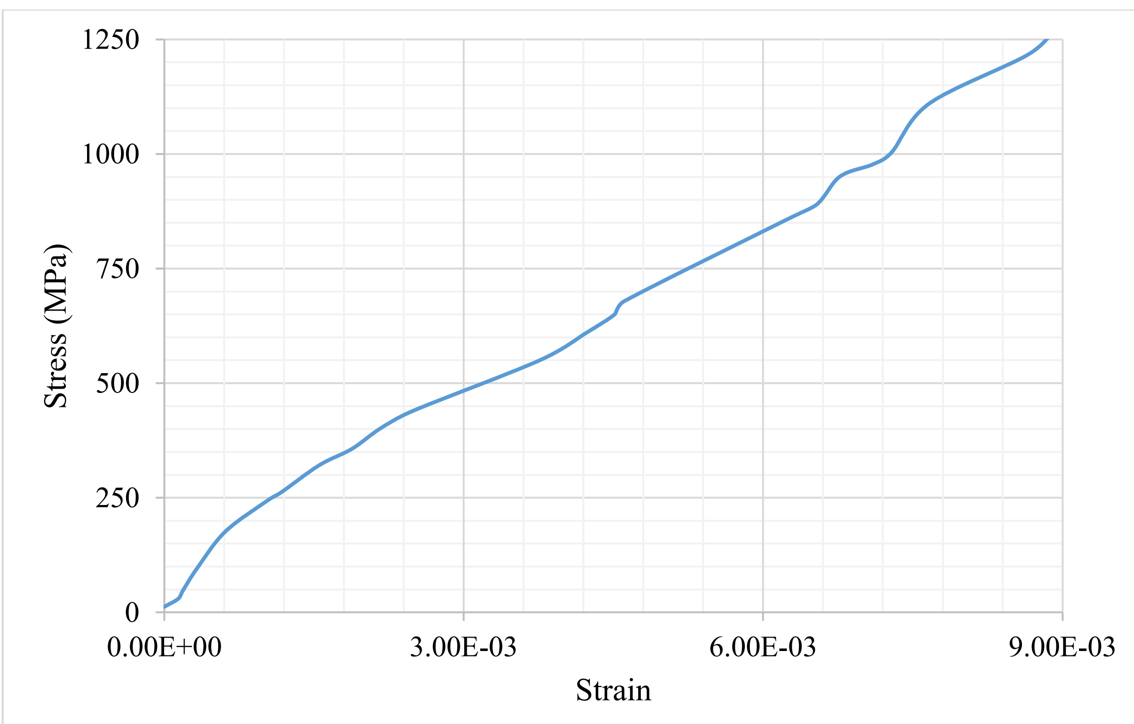


Figure 4.7 Compressive Stress Curve of TC-250 Coupon made by AFP+OOA

Most of the failed specimen under the compressive loads, failed according to long.- Splitting-Gage-Various (SGV) and Transverse shear-At grip/tab-Top (TAT) failure modes. Figure 4.8 shows the failed specimen by the SGV and TAT modes.

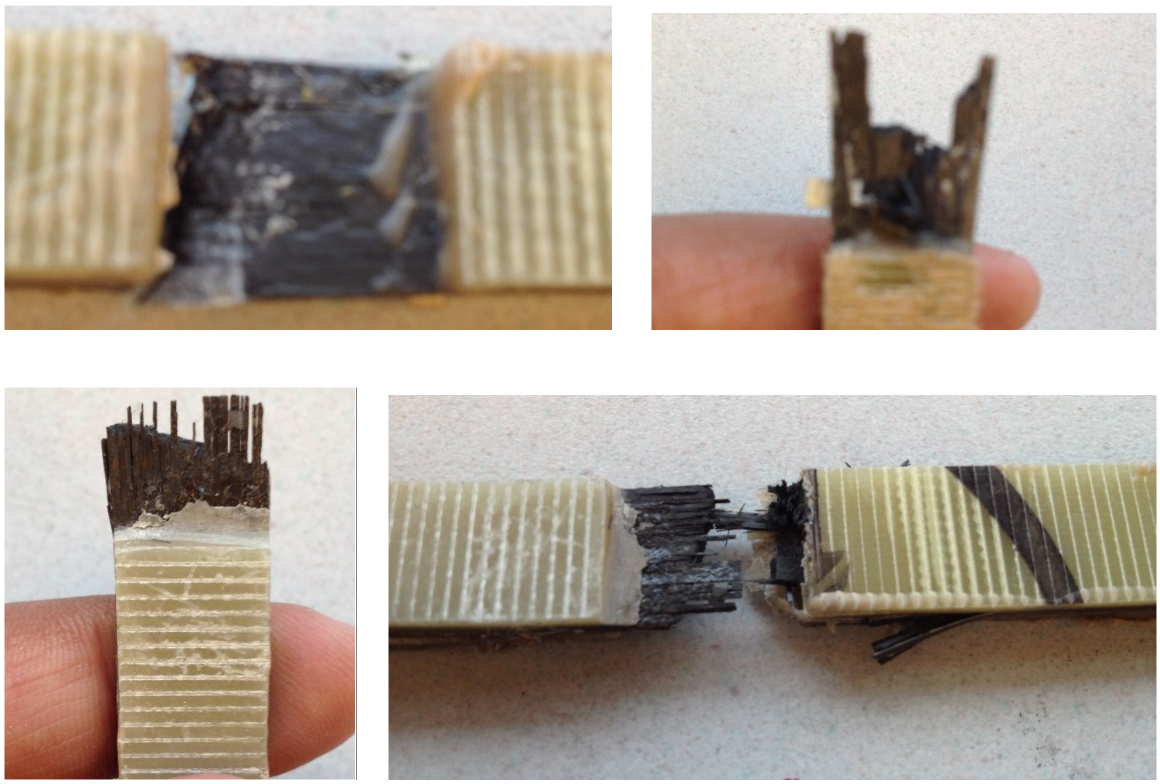


Figure 4.8 Compressive Test Coupons Failed under the SGV and TAT Failure Modes respectively

4.2 Summary of the Mechanical Tests Results

The following conclusions can be drawn from the mechanical tests performed on the TC-250 and CYCOM 5320-1 samples:

- The best results were obtained for the samples made by AFP process and cured with pressure. These samples had better compaction resulted from AFP manufacturing and the pressures in Autoclave resulted in better consolidation.

- In case of CYCOM 5320-1 material, the results obtained for samples cured with pressure in autoclave and the samples cured without pressure produced very similar test results. In every mechanical test, the sample cured without pressure lagged the sample cured with pressure by a mere 3-5%.
- In case of TC-250 material, the AFP+OOA samples outperformed the HLU+AC samples in every test except for the compressive test where the Hand-Lay-Up samples cured with pressure showed slightly better strengths and moduli.

Table 4.10 shows the test summary for the samples made using the TC-250 material.

Table 4.10 Summary of Mechanical Tests performed on Samples made using the TC-250 Material

Identification		TENSILE (0°)	TENSILE (90°)	SHEAR	COMPRESSIVE
HLU+OOA	E	140 GPa	9 GPa	4.5 GPa	104 GPa
	σ	1700 MPa	37 MPa	72 MPa	1223 MPa
HLU+AC	E	149 GPa	10 GPa	5 GPa	115 GPa
	σ	1755 MPa	43 MPa	79 MPa	1253 MPa
AFP+OOA	E	158 GPa	10 GPa	5.3 GPa	113 GPa
	σ	1786 MPa	46 MPa	82 MPa	1248 MPa
AFP+AC	E	167 GPa	11 GPa	5.8 GPa	125 GPa
	σ	1829 MPa	49 MPa	85 MPa	1276 MPa

The Figure 4.9 represents the tensile, shear and compressive moduli in bar chart format so as to show the variation for the TC-250 material.

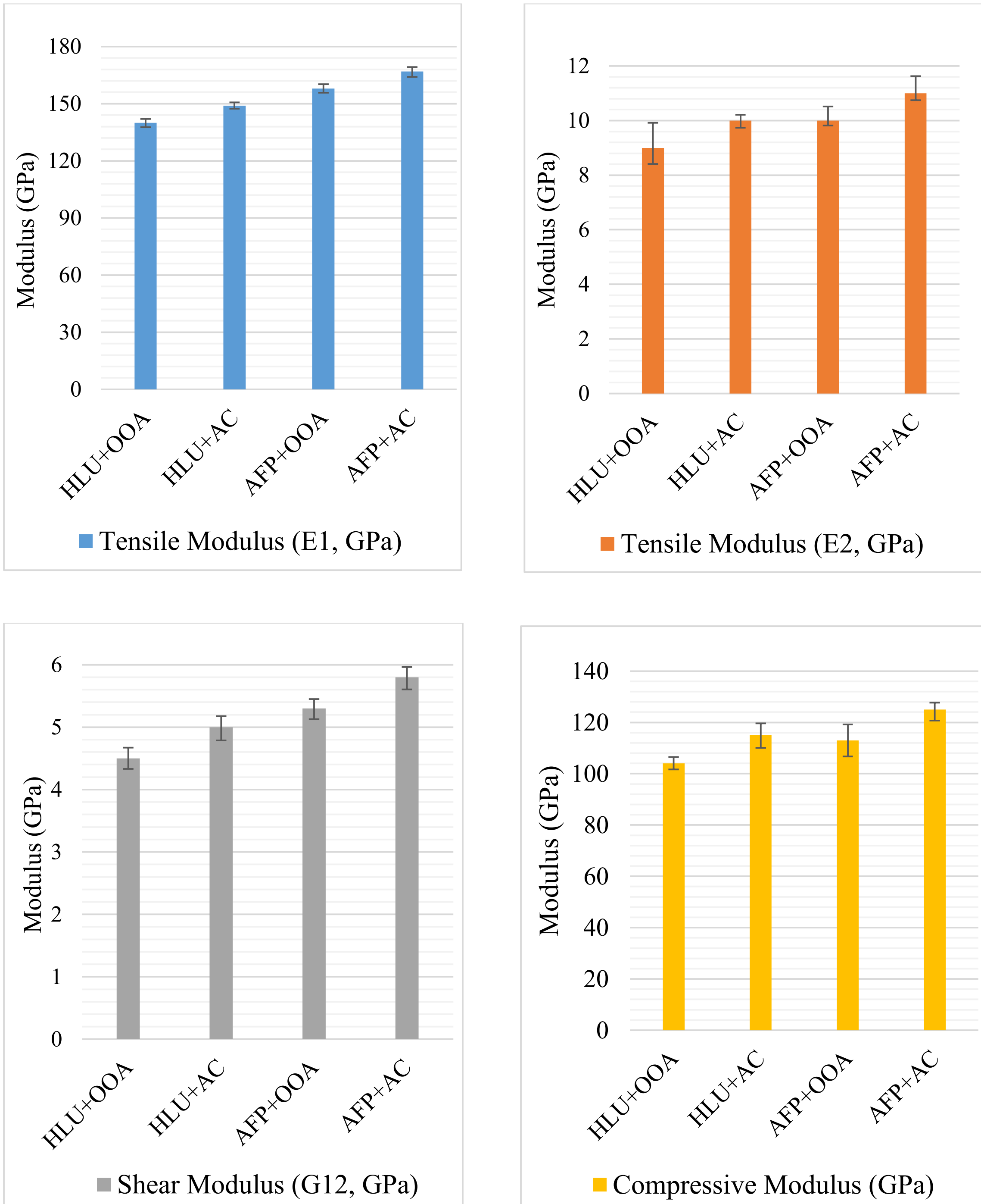


Figure 4.9 Comparison of Tensile, Shear and Compressive Moduli for TC-250 Material

Figure 4.10 represents the tensile, shear and compressive moduli in bar chart format so as to show the variation for CYCOM 5320 material.

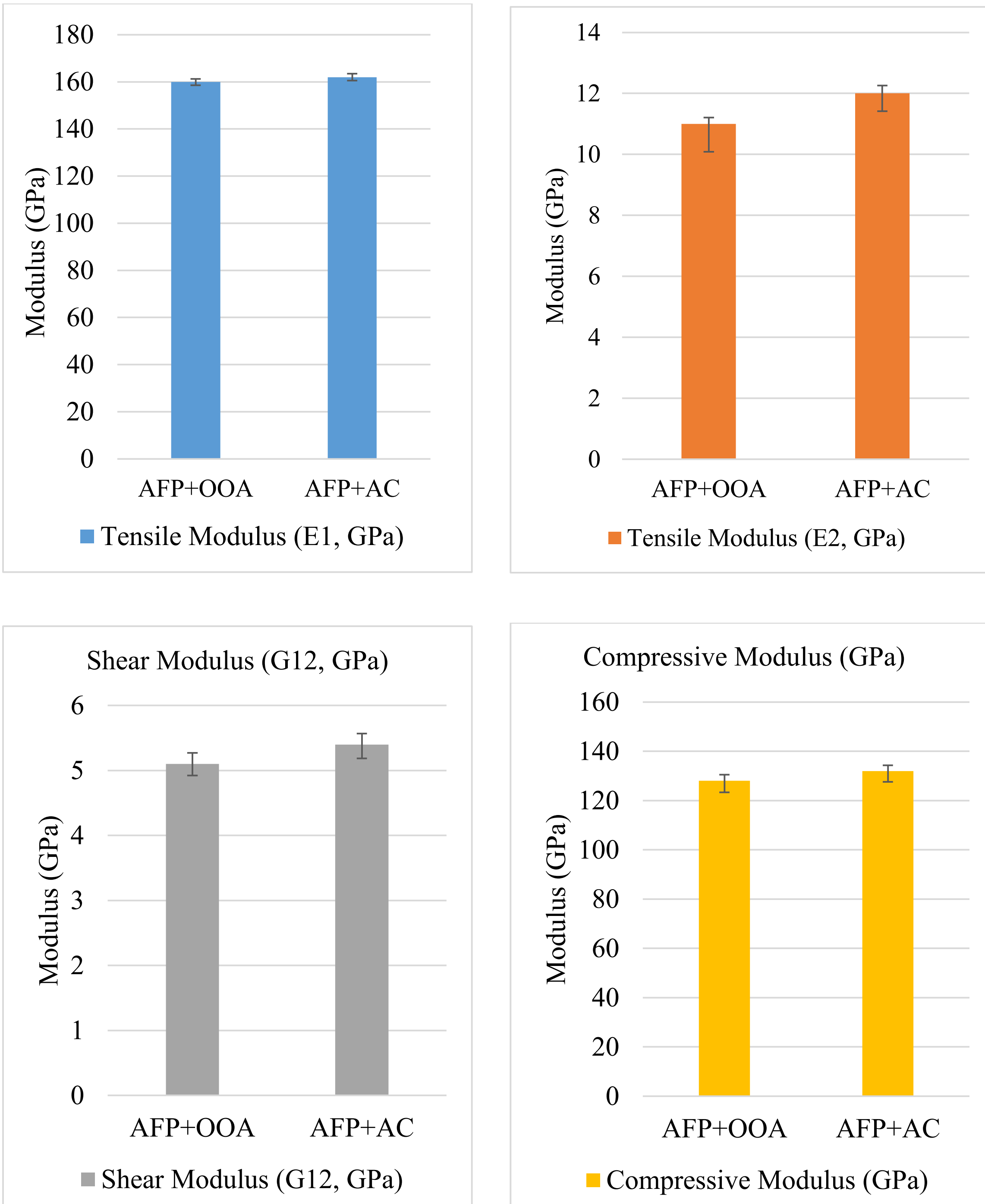


Figure 4.10 Comparison of Tensile, Shear and Compressive Moduli for CYCOM 5320-1 Material

Table 4.11 shows the test summary for the samples made using the CYCOM 5320-1 material.

Table 4.11 Summary of Mechanical Tests performed on Samples made using the CYCOM 5320-1 Material

Identification		TENSILE (0°)	TENSILE (90°)	SHEAR	COMPRESSIVE
AFP+OOA	E	160 GPa	11 GPa	5.1 GPa	128 GPa
	σ	2342 MPa	72 MPa	89 MPa	1360 MPa
AFP+AC	E	162 GPa	12 GPa	5.4 GPa	132 GPa
	σ	2362 MPa	76 MPa	92 MPa	1425 MPa

4.3 SEM Analysis of TC-250 and CYCOM 5320-1 Samples

The SEM analysis, as done for the raw materials in Chapter 3, has also been performed on the samples cut from the cured laminates made by the Hand-Lay-Up and AFP processes. This section presents the SEM analyses of the samples. Various defects like resin-rich areas, resin-starved areas and voids will be addressed.

4.3.1 SEM Analysis of Samples made by the Hand-Lay-Up Process for the TC-250 Prepreg Material

The SEM image of the samples made by the Hand-Lay-Up process using the TC-250 resin system are presented below. Figure 4.11 shows the sample that was cured with pressure and Figure 4.12 shows the sample that was cured without any external pressure.

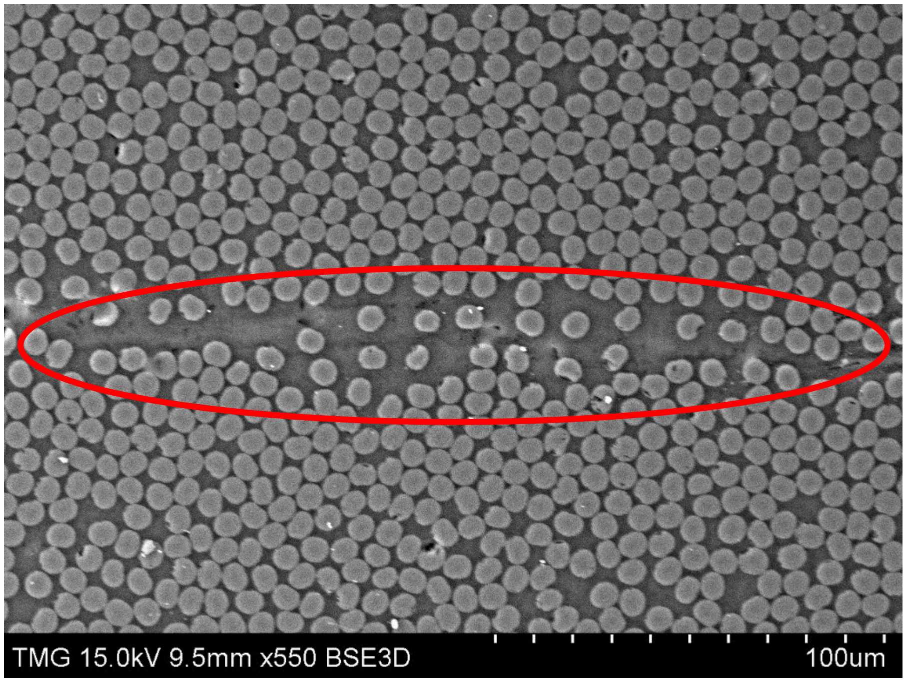


Figure 4.11 SEM Image for the TC-250 sample made by Hand-Lay-Up Process and cured with 550 kPa external Pressure (Magnification x550)

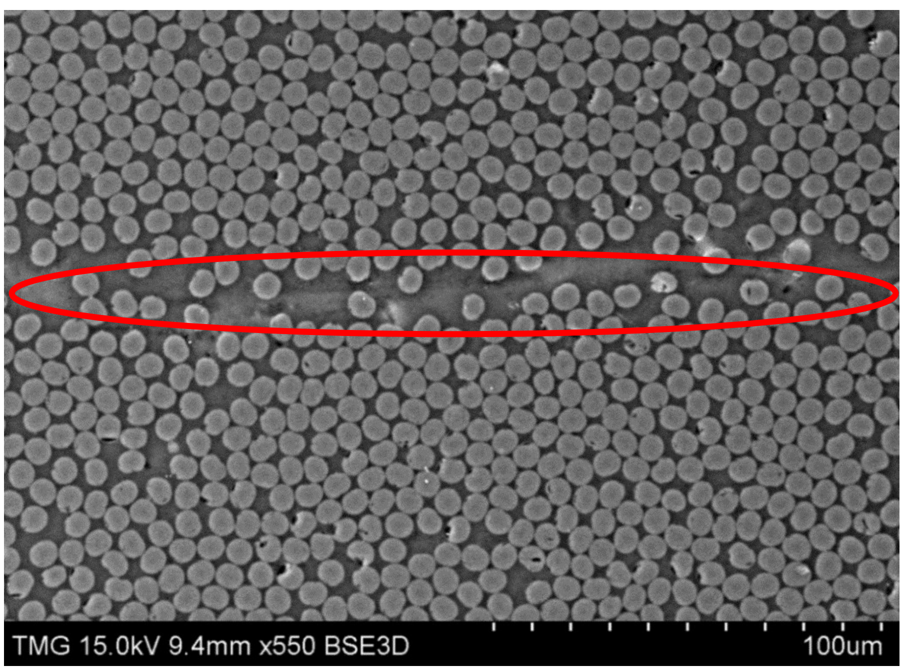


Figure 4.12 SEM Image for the TC-250 sample made by Hand-Lay-Up Process and cured without external Pressure (Magnification x550)

The images (Figure 4.11 and Figure 4.12) were taken at the interface of two successive prepreg plies in the sample. Resin-rich areas were observed in the interface region for both the samples, as highlighted in the figures. However, no voids and resin starved areas were seen in either case. The sample cured in autoclave with pressure didn't show any significant compaction than the one cured without pressure.

The fiber volume fraction estimated for the sample manufactured by Hand-Lay-Up process and cured with pressure in an autoclave is around 52% and is nearly 50% in case of the sample manufactured by Hand-Lay-Up process and cured without external pressure. These percentages have been calculated using the areal analysis process, in which the total area of the observed features in calculated. From the SEM image, the percentage fiber volume is obtained by dividing the light grey area (fiber area) over the total area. This 2% difference in fiber volume fraction is the cause why the Autoclave cured samples had demonstrated slightly better mechanical properties than the others.

4.3.2 SEM Analysis of Samples made by the AFP Process using the TC-250 Prepreg Material

The SEM images of the samples manufactured by AFP process using the TC-250 resin system prepreg are shown below. Figure 4.13 shows the sample cured in autoclave with pressure and Figure 4.14 shows the sample cured without any external pressure.

Similar to what was observed in the samples manufactured using the Hand-Lay-Up process, the sample cured with pressure didn't show any significant reduction in resin-rich areas and hence the compaction in the case of AFP process. Both the samples, cured with and without pressure presented similar microstructure.

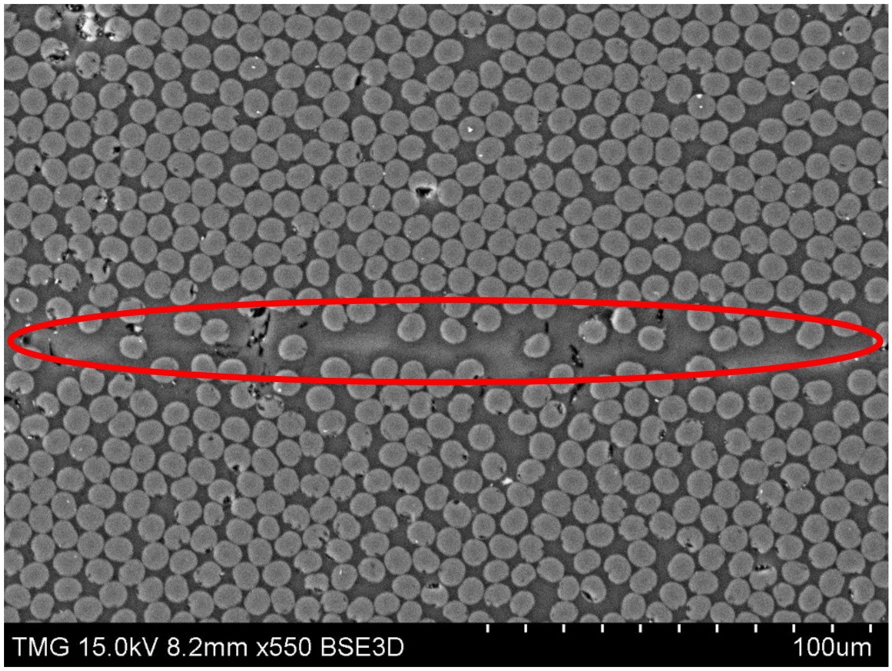


Figure 4.13 SEM Image for the TC-250 sample made by AFP Process and cured with 550 kPa external Pressure (Magnification x550)

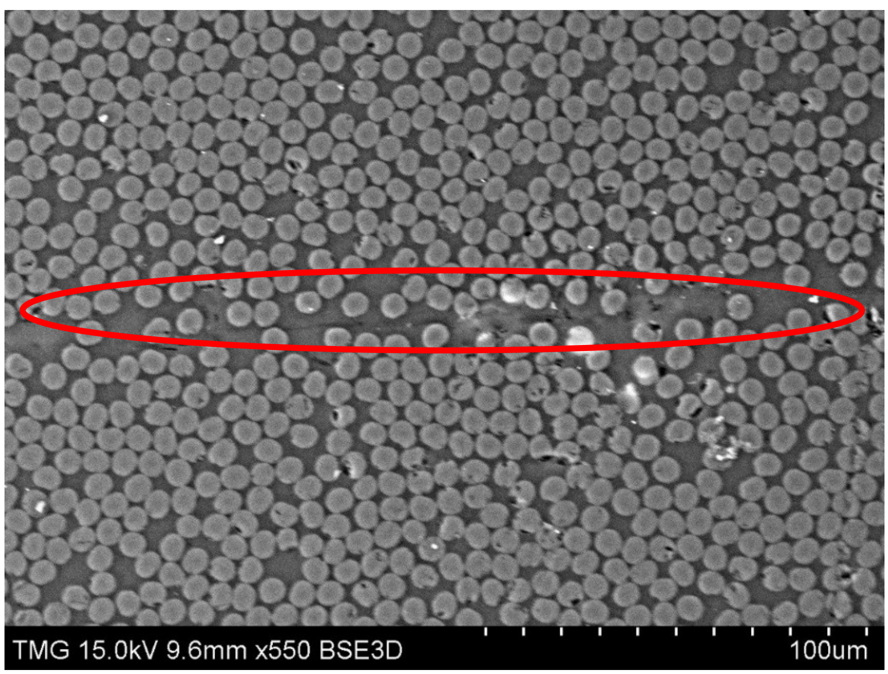


Figure 4.14 SEM Image for the TC-250 sample made by AFP Process and cured without external Pressure (Magnification x550)

No voids and resin-starved areas were seen in either of the samples. The fiber volume fraction for the sample cured with pressure and without pressure were around 54% and 56% respectively, calculated using the areal analysis technique. The samples with higher fiber volume fraction (cured in Autoclave with pressure) demonstrated better mechanical properties than the samples cured without pressure.

4.3.3 SEM Analysis of Samples made by the AFP Process using the CYCOM 5320-1 Prepreg Material

The SEM images of the samples manufactured by AFP process for the CYCOM 5320-1 material are presented. Figure 4.15 and Figure 4.16 show the images of sample cured with pressure and Figure 4.17 and Figure 4.18 show the samples cured without pressure.

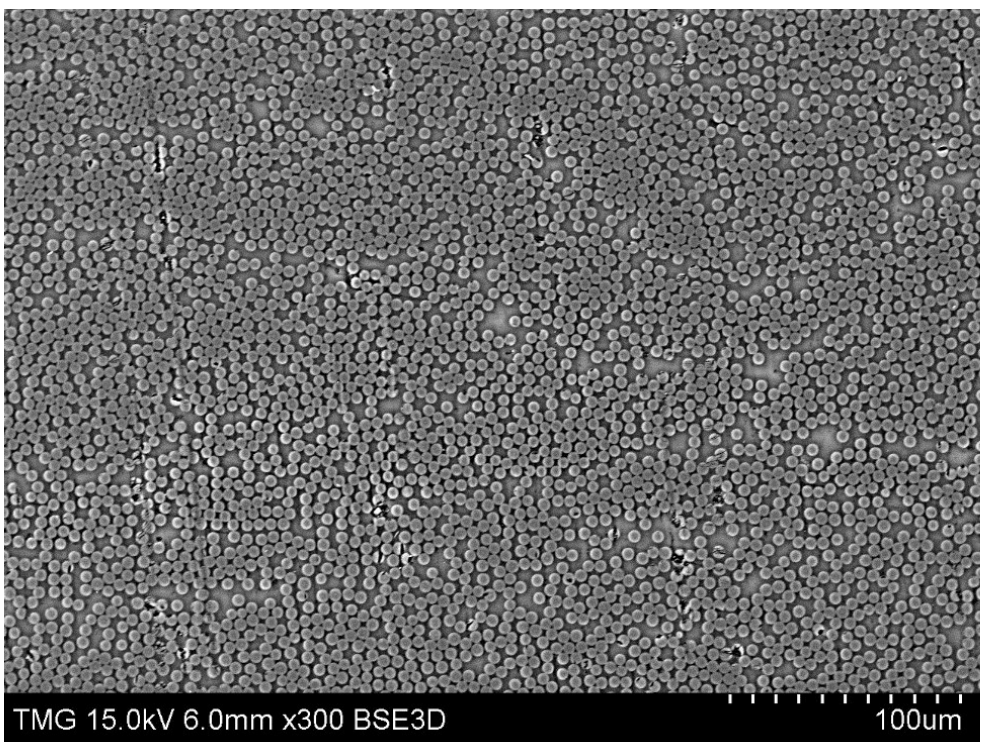


Figure 4.15 SEM Image for the Sample made by AFP Process and cured with 550 kPa external Pressure (Magnification x300)

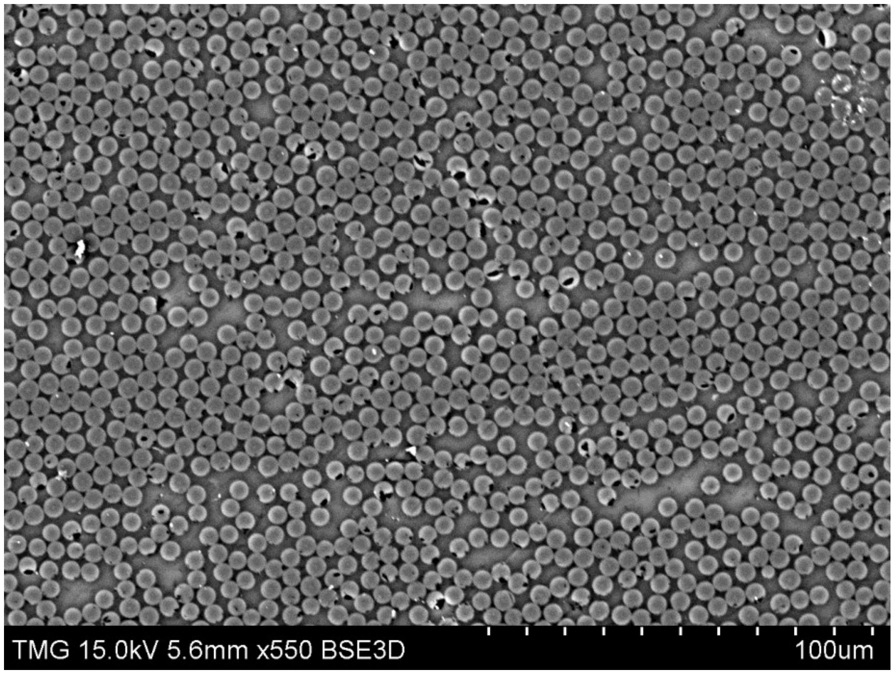


Figure 4.16 SEM Image for the Sample made by AFP Process and cured with 550 kPa external Pressure (Magnification x550)

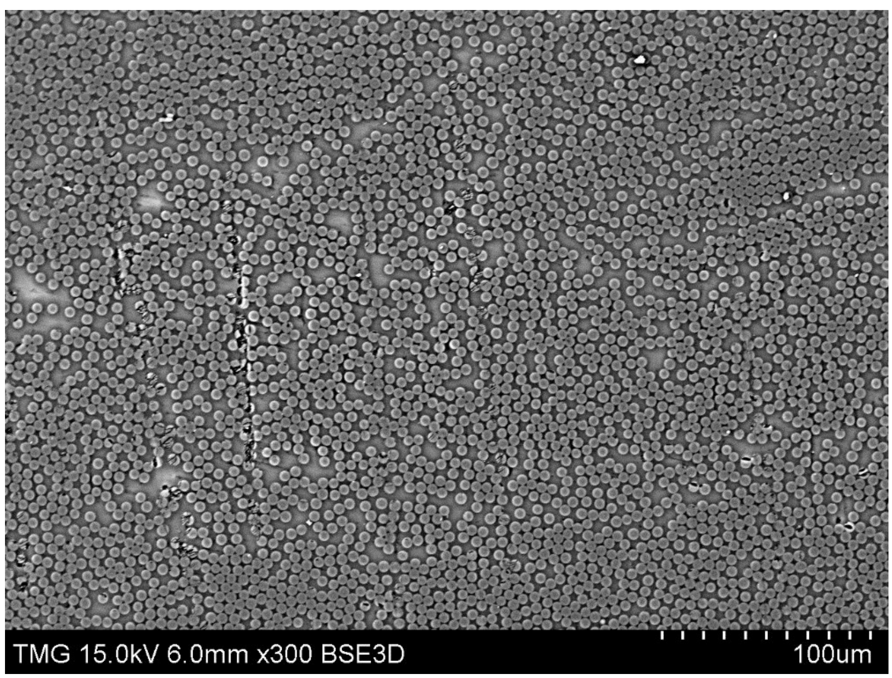


Figure 4.17 SEM Image for the Sample made by AFP Process and cured without external Pressure (Magnification x300)

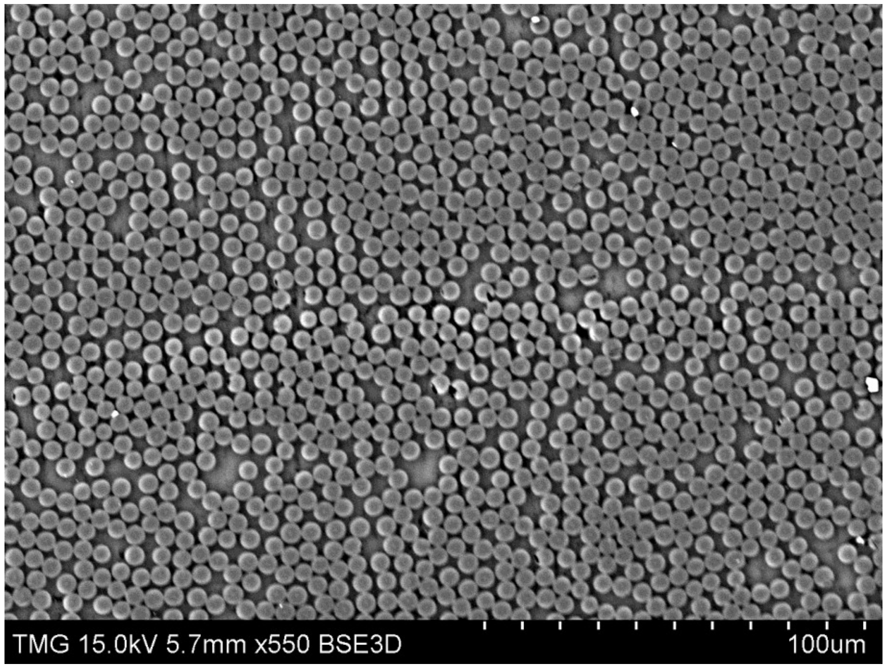


Figure 4.18 SEM Image for the Sample made by AFP Process and cured without external Pressure (Magnification x550)

After performing the SEM analysis on the samples made up of CYCOM 5320-1 material using AFP Process, two more samples of the same material were made using the Hand-Lay-Up process and cured with and without external pressure. Microscopic analysis was performed on these samples so as to see if the uniformity in fiber-matrix distribution will be similar to that seen in the samples made by AFP process. This microscopic analysis will also help understand the impact of the AFP manufacturing process on the lay-up of the prepregs.

The microscopic images of samples made by Hand-Lay-Up process and cured in Autoclave with pressures is shown in Figure 4.19. The microscopic images of samples made by Hand-Lay-Up process and cured in Autoclave with pressures is shown in Figure 4.20. These images were taken at 550x magnification.

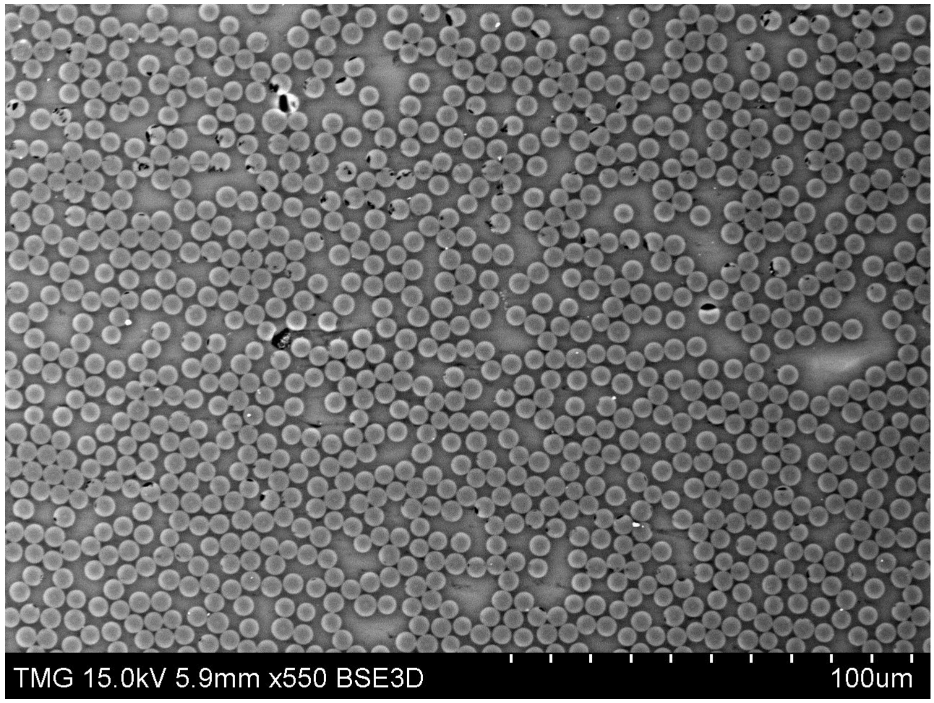


Figure 4.19 SEM Image for the Sample made by Hand-Lay-Up Process and cured with external Pressure (Magnification x550)

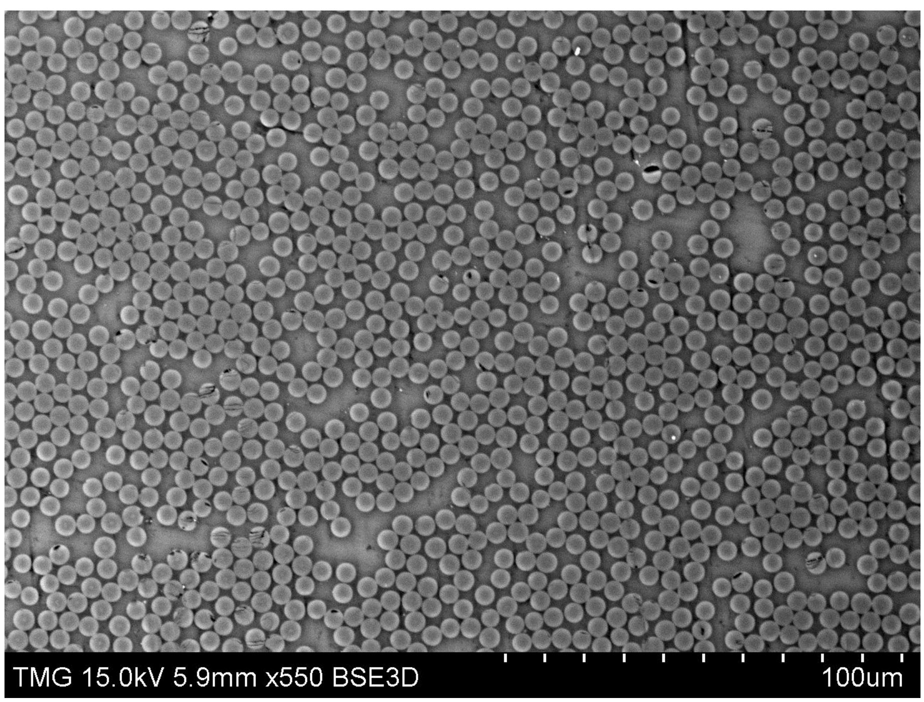


Figure 4.20 SEM Image for the Sample made by Hand-Lay-Up Process and cured without external Pressure (Magnification x550)

From Figure 4.19 and Figure 4.20 it is evident that the CYCOM material irrespective of the process used to manufacture, irrespective of the curing method employed produces uniform and defect free laminates. Also another important observation made from these microscopic images is about the diameter of carbon fiber in CYCOM material is around 5-6 μm, which is about 30% smaller than the carbon fiber in the TC-250 material.

4.4 Summary of the Microscopic Analysis Results

For the TC-250 material, it is evident from these microscopic images that the samples made by Hand-Lay-Up process and cured in autoclave with pressure did not show any significant consolidation compared to the samples cured without pressure. The average gap between the consecutive layers in the samples cured with pressure was 6.9 μm while for the sample cured without pressure was 7.1 μm. The pressure applied in the autoclave along with high temperature accounted for slightly better uniform distribution of resin and also proper consolidation of the laminate, validating the work done by Drakonakis et al. on 'Curing Pressure Influence of Out-of-Autoclave Processing on Structural Composites for Commercial Aviation' [19].

Similar to the trend observed in the TC-250 prepreg samples made by Hand-Lay-Up process, the TC-250 samples made using the AFP process which were cured in the autoclave with pressure had relatively better consolidation than the ones cured without pressure (can be observed in thicknesses of the coupons), evident from the fiber volume fraction obtained from the microscopic images. The average gap between consecutive layers in the samples cured with external pressure was 6.2 μm compared to 6.4 μm for the samples cured without pressure.

Comparatively, the AFP samples had insignificant resin-rich areas compared to HLU samples. From the SEM analyses, it is clear that the compaction pressure applied while manufacturing using AFP machine plays an important role in debulking and compaction of the prepregs. In Hand-Lay-Up, this debulking is achieved by vacuum bagging the stacked prepregs after every layer for 15-20 minutes.

Second important observation is that the external pressure applied by the Autoclave had no significant impact on the compaction. However, in either of the samples no major resin rich areas, no resin-starved areas and no voids were observed in the OOA cured samples, thus validating the work presented by Dang et al. in the "Mechanical Comparison of Out-of-Autoclave Prepreg Part to conventional Autoclave Prepreg Part" [46].

For the CYCOM 5320-1 material, the microscopic images for the samples cured with and without pressure had insignificant differences. Due to the perfect consolidation, the demarcation between the consecutive layers at the interface were not visible even in the microscopic images. The similar microstructure of the samples cured with and without pressure also supports the results observed in the mechanical testing of these samples which show the variation in results in the range of 3-5%.

The sample manufactured by Hand-Lay-Up using the same material possessed similar microstructure to that of laminates made by the AFP processes. As seen in the TC-250 material, the roller compaction pressure in the AFP process for these CYCOM material had no significant effect on the consolidation of the prepregs. In this case, proper resin rheology in the material accounts for this uniform distribution of fiber-matrix. Also there was no difference in the thickness of either of the samples, cured with and without pressure.

4.5 Discussion

Table 4.12 shows the comparative results obtained from all the tests for the TC-250 prepreg material. It ranks each process in the order of performance; where I, II, III and IV are in the decreasing order of performance. Tensile, Shear and Compressive test results are obtained from the mechanical tests, whereas the percentage fiber volume results are obtained from the SEM analyses using areal method. In all the tests, the samples manufactured by using the AFP process and cured with external pressure out-performed all the other combinations of manufacturing and curing processes.

Table 4.12 Manufacturing Process Rankings for TC-250 Material

Manufacturing Technique	Percentage Fiber Volume	Tensile Tests	Shear Tests	Compressive Tests
HLU+OOA	IV	IV	IV	IV
HLU+AC	III	III	III	II
AFP+OOA	II	II	II	III
AFP+AC	I	I	I	I

However, the samples made by AFP and cured without pressure also produced promising results. In almost all the cases, these samples produced better results than the samples made by Hand-Lay-Up and cured with pressure. However, the latter showed slightly better results in the compressive tests. Also, the samples made by AFP process could have produced even superior results had they not had the flatness issue while manufacturing.

The uneven distribution of fibers in the material because of this also would have impacted the load bearing capacity of the material.

For the CYCOM 5320-1 prepreg material, the mechanical tests and the SEM analyses showed insignificant differences in the samples cured with and without external pressure. However, the samples cured in Autoclave had produced better results than the ones cured without external pressure.

An important observation was made from the microscopic images of the CYCOM 5320-1 material. The diameter of carbon fibers used in this material are smaller than those used in the TC-250 material. The diameter of carbon fibers in TC-250 material is around 8-9 µm, whereas the diameter of carbon fibers in the CYCOM 5320-1 material are 5-6 µm (Figure 4.14 for TC-250 material and Figure 4.16 for YCOM 5320-1 material).

5 COST MODEL DEVELOPMENT

The phenomenal cost savings associated with the OOA prepreg technology is one of the main driving forces for the advancements in the last few years. OOA prepreg materials were introduced in the early 1990's with the aim to reducing the cycle times and the costs associated with the manufacturing process. To demonstrate these savings, a comparative cost model has been developed to study the fabrication cost of a part made by Hand-Lay-Up and AFP Processes, when cured with and without the application of external pressure. These four scenarios will be presented and studied in detail in the following sections.

5.1 Cost Model

Figure 5.1 shows the schematic representation of the whole manufacturing process in making a part.

- The initial step requires the preparation of prepreg and the tool which is common to both the manufacturing processes.
- The next step 'Ply Collation' is a major step in which the AFP process differs from the conventional Hand-Lay-Up process. In the Hand-Lay-Up manufacturing process, Ply Collation consumes the maximum time and requires high amount of precision and skill. The prepreg is first cut according to the shape of the part and the desired fiber orientation, and each piece of the prepreg is then stacked upon one another to make the whole part. After stacking each layer of prepreg material, the whole assembly is placed in a vacuum bag and the vacuum is applied for 15 minutes to ensure no entrapped air is present in the part and proper debulking is achieved. All these steps increase the time and also the cost

of the Hand-Lay-Up Process. In case of AFP manufacturing, 'Ply Collation' sequence can be completed much faster than the Hand-Lay-Up. The AFP head lays the prepreg and the compaction roller does the debulking and eliminates the air entrapments. No further vacuum bagging operation is thereby required while stacking the prepregs.

- The next sequential step in both the manufacturing options is vacuum bagging and it consumes an equivalent amount of time, labour and material in either of the manufacturing options.
- In the curing phase, the cost of operation varies largely based on the curing path chosen i.e. curing with pressure or without pressure. Curing with pressure is done in the Autoclave which has high operation and overhead costs, whereas curing without pressure can be achieved in a Heat oven which has low operation and overhead costs.
- The final operations of 'Testing' and 'Machining and Assembly' are identical for both the operations.

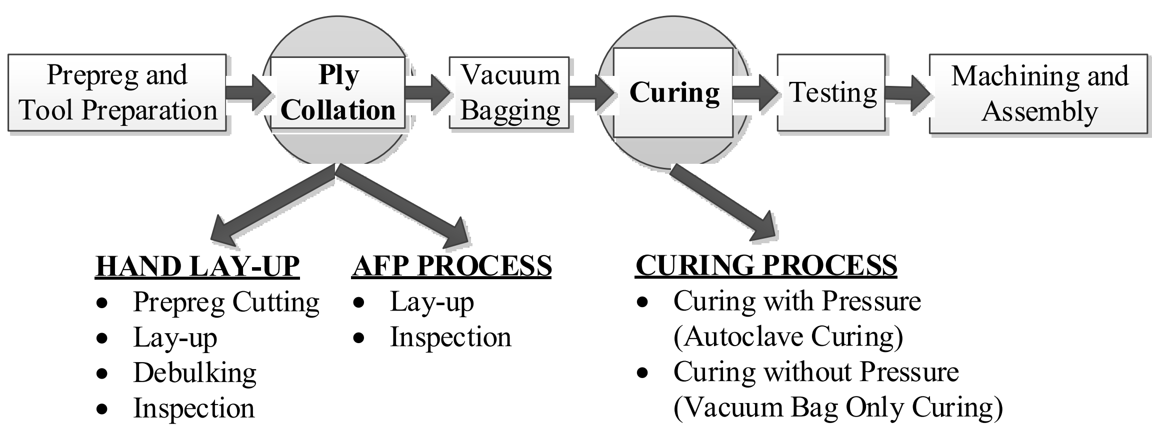


Figure 5.1 Schematic Representation of the Manufacturing Process

5.2 Cost Model Assumptions

In order to have a real cost comparison after developing the cost model, some assumptions have been made. The costs of the materials mentioned in these assumptions have been procured from the suppliers and a general trend has been developed for the cost model.

The overhead costs (hourly costs for using the machine) have been calculated by normalizing the cost of the equipment for 15 years and then dividing the amount by the number of hours the equipment shall be used. The inspection and miscellaneous costs are same for all the processes and hence do not reflect any changes in the cost-model comparison.

The following parameters have been considered for the cost model development:

I. ***Part Geometry -*** The part geometry plays a crucial role in the cost of the part. It directly influences the raw material wastage, the tooling and the labour time in making the part. For the purposes of demonstrating the comparative cost model, a part with flat geometry has been considered. The length and width of the flat plate under consideration are 450 mm and 320 mm respectively. A total of 16 layers has been considered with the stacking sequence of $[0_2, 90_{2,} {+45}_{2,} -45_2]_s$.

II. ***Prepreg Material Cost -*** The cost of the raw material varies largely from one supplier to the other. For example, the cost of TC-250 material slit tape is around \$167/kg (\$76/lb) and the cost of CYCOM 5320-1 material slit tape is around \$299/kg (\$136/lb). These costs vary with the order quantity, lead time and the demand of the material in the industry.

For the cost model, the cost of the prepreg used for the Hand-Lay-Up process is \$133/kg (\$60/lb) and the cost of the slit-tape for the AFP machine is \$177/kg

($80/lb). With the given part dimensions and considered wastage at 35%, the weight of the prepreg needed to make this part using the Hand-Lay-Up process is 1.85 kg. The weight slit prepreg required to make the same part using AFP is 1.45 kg considering 7% wastage.

III. ***Skilled Labor / Technician Cost -*** The cost of skilled labour and technician is assumed to be $75/hr. It has been considered that the technician shall require at least 8 hours to cut the prepreg and lay-up this part manually. Debulking time of 15-20 minutes has been considered and debulking is performed after each layer of prepreg is stacked. This increases the time of lay-up, and hence the labour cost is significantly affected. However, the same task using AFP machine shall be around 1.5 hours. While curing process, an additional 2 hours of labour is needed in either case.

IV. ***AFP Layup Rate -*** Though the AFP machines are capable of laying fibers at 2.72 kg/hr (6 lb/hr), using a pessimistic approach for the cost model, a layup rate of 1.36 kg/hr (3 lb/hr) has been considered. Therefore, time required to make a part including the machine set-up and warming-up times shall be around 1.5 hours.

V. ***Autoclave / Oven Cycle Time -*** The curing cycle time depends on the cycle as suggested by the prepreg manufacturer. For the cost model, 6 hours for curing and 2.5 hours for post-curing shall be considered.

VI. ***Autoclave Overhead Cost*** - A medium-sized autoclave with an internal compartment size of 6 feet length and 3 feet diameter has an overhead cost of $180/hr.

VII. ***AFP Machine Overhead Cost -*** The AFP machine available in the CONCOM laboratory at Concordia University was made my Automated Dynamics Corporation. The overhead cost of the machine is around $250/hr.

VIII. ***Heat Oven Overhead cost -*** A medium size gravity convection oven will serve the purpose of curing and post curing the part. The oven must have a provision for vacuum suction during the curing cycle. The heat oven overhead cost is around $75/hr.

IX. ***Inspection and Miscellaneous Costs -*** The cost of inspecting the part is considered to be $250/hr. Miscellaneous costs of $100 is considered for the energy consumption, bagging film, release agent, bleeder film, breather film and other consumables used in the vacuum bagging. These costs are common for all the different scenarios presented.

5.3 Cost Model Development

The objective of developing this cost model is to do a comparative analysis between the two manufacturing techniques i.e. Hand-Lay-Up and AFP processes and between the two curing methods that have been used i.e. autoclave with pressure and vacuum bag only (without pressure). Table 5.1 shows the individual cost of each parameter for the combination of various processes.

The term ‘AFP + AC’ used in Table 5.1 refers to the part made by AFP process and cured in Autoclave with external pressure and the term ‘AFP + OOA’ refers to the part made by AFP process and cured without pressure.

Similarly, the term ‘HLU + AC’ refers to the part made by Hand-Lay-Up process and cured in Autoclave with pressure and ‘HLU + OOA’ refers to the part made by Hand-Lay-Up process and cured without pressure.

Table 5.1 Details of the Cost associated with each Parameter

Parameter	AFP+AC	AFP+OOA	HLU+AC	HLU+OOA
Prepreg	$257	$257	$246	$246
Labour / Technician	$263	$263	$750	$750
Cost of Tooling	$450	$150	$450	$150
AFP Overhead	$375	$375	$0	$0
Autoclave Overhead	$1,080	$0	$1,080	$0
Oven Overhead	$188	$638	$188	$638
Inspection Cost	$250	$250	$250	$250
Miscellaneous	$100	$100	$100	$100
Total Cost	$2,962	$2,032	$3,064	$2,134

Figure 5.2 shows the differences in costs for each parameter affecting the cost- model. The Inspection costs and the miscellaneous costs are same for all the different combinations of processes and curing methods and hence are not shown in the comparison of costs. Figure 5.3 shows the comparison of the total cost of all the processes combinations.

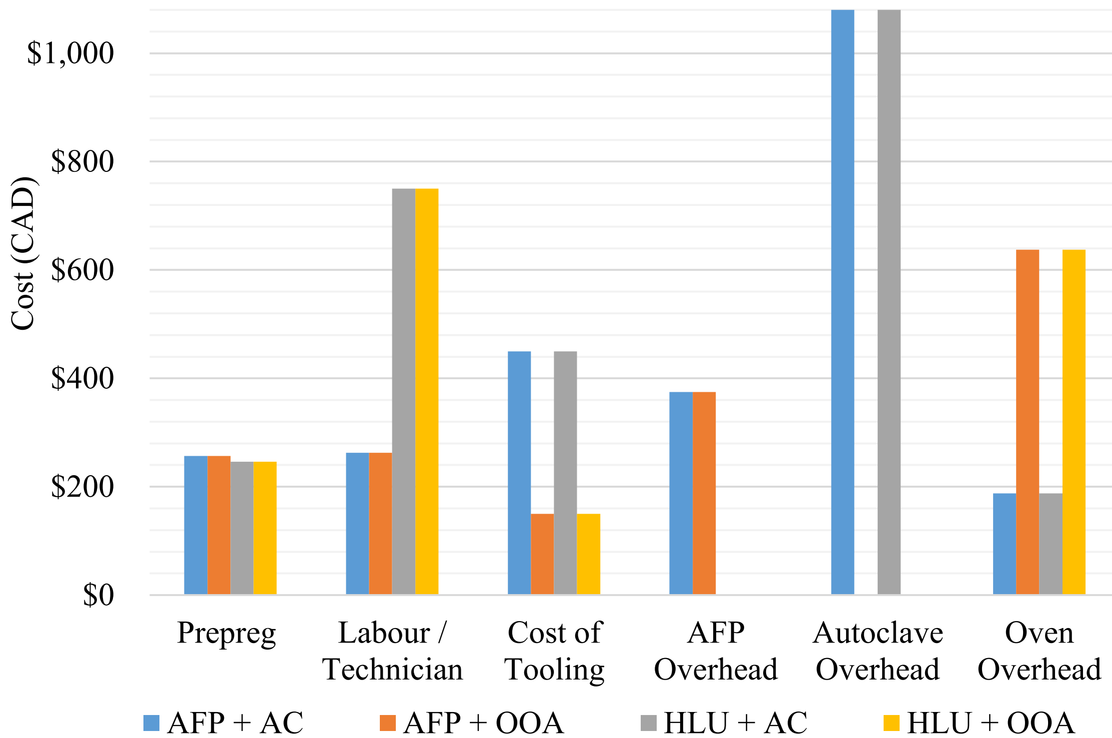


Figure 5.2 Break-up of Cost Comparison

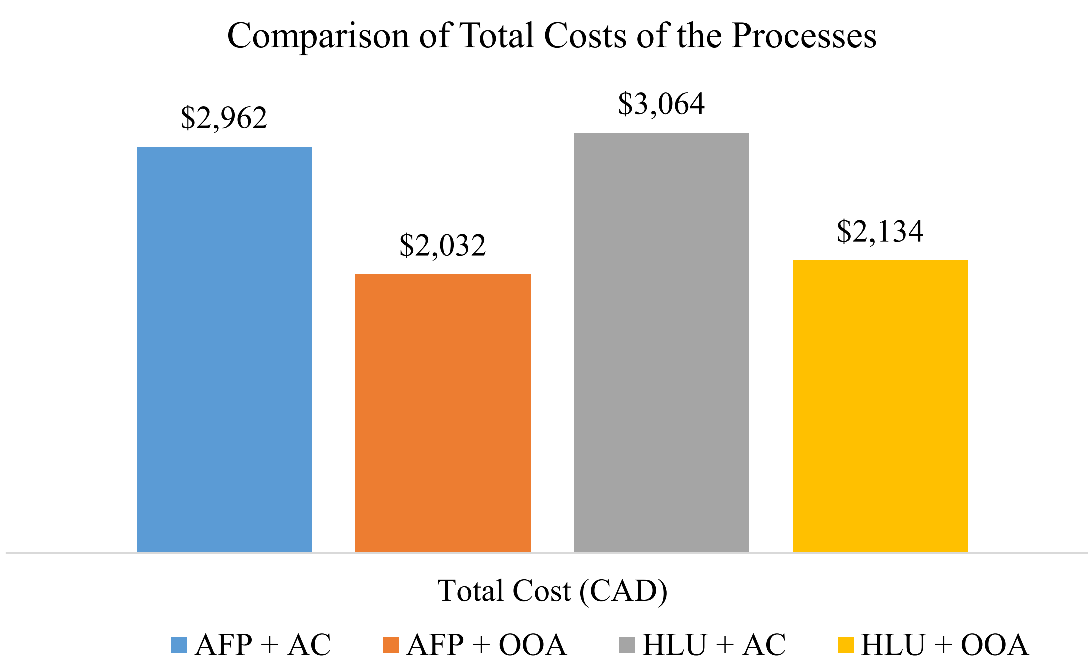


Figure 5.3 Total Cost Comparison

5.3.1 Comparison of Costs involved in Hand-Lay-Up and AFP Processes

The prepreg costs involved in the AFP process are higher than that of the one used in Hand-Lay-Up because of the costs incurred during the slitting process. Additionally, this is totally dependent on the material supplier. For e.g. TenCate's slitted prepreg costs around 30% more than the wide tape used for the Hand-Lay-Up process, whereas the cost of the CYCOM 5320-1 prepreg is less for the slitted tape by almost 15% . Also, the amount of raw material wastage is minimized in the AFP process. Practically, the maximum wastage associated with AFP process is 7% for the current part, where as a minimum of 35% wastage may be seen in the Hand-Lay-Up process. This factor also influences the cost of the prepreg required during the Hand-Lay-Up process. For the AFP process, the cost incurred during slitting the prepreg is compensated by the minimized wastage, whereas in the Hand-Lay-Up process though the prepreg cost is lower, the wastage associated increases the overall cost of the required prepreg.

Hand-Lay-Up is a highly labour intensive process. Comparatively, it requires higher labour hours when compared to the AFP Process. For example, the labour hours required to accomplish the tasks of cutting, laying-up and debulking the prepregs for manufacturing the current part using the Hand-Lay-Up process shall be almost 5 times more than that of the typical time required when the AFP machine is used. The labour involved during the curing process will be same for either process. Therefore, the overall cost associated with the labour in the Hand-Lay-Up process is almost 2.5 times the cost involved in AFP Process.

The AFP process utilizes a sophisticated robotic arm that performs the cutting, laying-up and debulking operations automatically. Therefore, the overhead cost of the AFP machine is high. The present cost of the machine normalized over 15 years is around $ 250 per hour, provided the machine is used 40 hours a week. With increasing popularity and the growing demand in the automation of composites, there shall be a decline in the overhead cost associated with the AFP machine. Also, the speed of lay-up does play a crucial role in deciding the time for making the part. Though the AFP machine is capable of laying up fibers at the maximum rate of 2.72 kg/hr (6 lb/hr), the assumed lay-up rate for the cost model is 1.36 kg/hr (3 lb/hr) in order to present the most accurate comparison. With an increase in the lay-up rate, the lay-up time reduces and hence reduces the cost of using the machine. The lay-up rate also depends on the degree of complexity of the part. A part that has a constant or no curve will be faster to fabricate than that which has a complex geometry. Generally, there is no constant or standard value for the lay-up rate and therefore it is decided experimentally.

5.3.2 Comparison of Costs involved in Curing with and without Pressure

The cost of tooling associated in making a part also does affect the total cost of the part. The type of tooling used is dependent on many features like the cost of tooling, cycle time, life expectancy of the tooling, compatibility in the coefficient of thermal expansion (CTE) of the tooling and the part and the operating conditions (temperature, pressure, etc.).

The tooling needed for a part that is being cured in Autoclave with pressure has to be made of a strong material like Invar Steel so as to sustain the pressure, whereas the tooling needed for a part that is cured in an oven with no external pressure need not be of high strength, for example, it can be made up of Aluminum 6061 or 7075. The cost of Aluminum tooling is much less than that of the Invar Steel. Also, the weight of the tooling will be greatly reduced when Aluminum is used. This will ease the process of moving the tool in and out of the oven after the lay-up and thus reduces the layover time, therefore, reducing the overall cost of the process.

Autoclave helps make an almost defect-free laminate with void content less than 1%. However, autoclave is an expensive equipment. The cost of autoclave also varies widely depending upon the operating conditions and the size of the part. Autoclaves are capable of applying large amount of external pressure so as to consolidate the laminates properly. Also, this external pressure limits the use of some of the honeycombs and inserts which cannot sustain this high pressure. The overhead cost of autoclave can vary from anywhere between $ 180/hour to $ 400/hour. The cost incurred in using the autoclave also depends upon the autoclave curing cycle. Most of the cycles vary from 5 hours to 10 hours. The time to cool the autoclave before the part can be removed safely is also taken into account. The overhead cost of a gravity convection oven can be around $ 75/hour. Hence, by employing the oven for the curing process, the curing cost can be reduced to about one-third the cost incurred when an autoclave is used. This difference has been demonstrated in Figure 5.2 in the cost model development. The post curing operation is accomplished by using the oven in either case.

5.4 Summary of Results obtained in the Cost Model

The total cost of making a part using the AFP process when cured without the application of external pressure turns out to be $ 2,032 as compared to $ 2,962 for the part made by AFP process cured in the autoclave with pressure. There is a 30% cost increment so as to gain 5.8% more Tensile Modulus, 20% more Shear Modulus and 10.8% more Compressive Modulus. In other words, to gain 1 GPa of Tensile Modulus by using the Autoclave curing process, a cost of $ 86.4 is involved (difference in costs involved in the processes /

difference in the tensile moduli of the samples made by the two processes). Therefore, depending on the mechanical constraints and strength requirements, the process can be chosen.

From the cost model, the cost of autoclave is one of the major factors in determining the cost of manufacturing a composite part. The example discussed in this chapter is for a part with simple geometry, however, with increasing size and complexity of the part, the cost of using the autoclave, labour costs and wastage shall be increased.

Major cost reduction can be obtained on autoclave overhead, tooling costs, and the costs associated when moving the inventory, by using the out-of-autoclave curing process. OOA technology enables the manufacturing of the composite part by eliminating the costs associated with the operation of the autoclave.

6 CONCLUSIONS

Conclusions drawn from the research work presented in the preceding Chapters are summarized below:

I. From the point of view of manufacturing, it can be concluded that the OOA prepreg material requires greater process control during lay-up (proper control of curing, lay-up speed, HGT torch temperature, Compaction force, etc.). Some of the initial samples that were manufactured contained voids, resin-rich areas and also absorbed heat during the DSC analyses. These issues were overcome by vacuum debulking the laminates after every new layer was stacked. To ensure that both the part and the oven temperatures reflects the temperature in the curing cycle, a dual-loop temperature controller was utilized. This minimized the scope of error resulting from the difference in the oven and the part temperatures.

II. It can also be concluded that high quality of laminates made by AFP process depends on the processing parameters fed to the AFP system. Moreover, the compaction force applied during the lay-up process plays a crucial role in achieving proper debulking which, in turn, helps in producing defect-free laminates using the OOA prepreg materials. The optimum process conditions for manufacturing parts using the CYCOM 5320-1 and TC-250 OOA carbon/epoxy prepregs are:

- Compaction force (pressure applied by compaction roller): 135 N
- Lay-up rate: 50.8 mm/sec (2 inch/sec)
- Temperature of the HGT: 190°C

III. From the microscopic analyses, no voids were seen in any of the manufactured samples. However, resin-rich areas were observed in the samples made by TC-250 prepreg material. However, the resin-rich areas in the AFP samples were comparatively lesser than the resin rich areas in the Hand-Lay-Up samples. The laminates made by AFP and cured without pressure had microscopic structure similar to that of the laminate cured in autoclave with pressure. In case of the samples made by CYCOM 53201-1 prepreg material, no significant differences were seen in the microstructure of samples cured with external pressure and those cured without external pressure. The fiber and resin distribution was highly consistent throughout the samples.

IV. The tensile, shear and compressive properties of the samples made using the TC-250 prepreg material by the AFP process and cured in autoclave had better mechanical properties, followed closely by the samples made by AFP cured without pressure. The results obtained from the Hand-Lay-Up samples were comparatively inferior to the ones obtained from the AFP samples. In case of CYCOM 5320-1 prepreg material, the tensile, shear and compressive properties of the samples cured with and without external pressure presented no significant differences. However, these samples cured in autoclave have shown slightly better mechanical properties than the ones cured without external pressure.

V. The better mechanical properties of samples made by the AFP process and also the samples that were processed with pressure in autoclave can be attributed to the thickness of the coupons. The thickness of the coupons made from the sample that was manufactured using AFP process was nearly 0.2 mm less than that of the sample made by Hand Lay-Up process for 8-layer unidirectional laminate. Similarly, slender difference in thickness was seen in the samples processed with pressure in autoclave and the ones cured without external pressure. The thickness variation in turn corresponds to better consolidation which is responsible for better mechanical properties.

VI. High cost savings can be obtained by avoiding the costs associated with the autoclave and further cost reduction in tooling, inventory transportation and operator costs can also be achieved with the elimination of autoclave. The economics of curing the samples without the use of autoclave are promising. The need of using an autoclave to achieve parts with slightly superior strength is totally dependent on the stress requirements of the part. This has been demonstrated in the cost model. The economic aspects of using the AFP machine for laying up the prepreg has been discussed and compared for various process combinations in the cost model.

VII. The cost model developed in this project is quite sensitive to the geometry and features of the part, properties of the prepreg material and the automated manufacturing machine. However, it gives a thorough and detailed understanding about the economics involved in using an AFP machine. The general trend discussed in the cost model will hold good for majority of the cases.

Overall, for the TC-250 material, the AFP processing outperformed Hand-Lay-Up process in every test and analysis. The samples made by AFP machine and cured without pressure produced results comparable to those of the samples cured with pressure resulting in substantial cost reductions. The resin-rich areas observed were least for the samples made by AFP process and cured with pressure.

However, for the CYCOM 5320-1 material, the samples made by the AFP process and cured without pressure produced excellent properties in all the tests and analyses. The results obtained were close to those obtained from the samples cured with pressure. The microscopic analyses performed on these two samples produced images with insignificant differences presenting no voids, resin-rich areas or resin-starved areas.

7 FUTURE WORK RECOMMENDATIONS

The following are the recommendations for the future work:

- Many parts used in aircraft industry are sandwich structures. Over the years, a great challenge for researchers has been to make high quality sandwich structures that do not deform or buckle under the external pressure in the autoclave curing cycle. Automated laying up of OOA prepregs on various honeycombs can be an interesting solution.
- In certain applications in the aerospace industry, the use of thick composites is desired. It will be important to evaluate the properties of thick parts / laminates that are made using the OOA prepreg materials by automated manufacturing.
- Evaluating the effects of using different tooling for making the parts using AFP machine and OOA prepregs can be of great interest to the automotive industry. The issues pertaining to the Coefficient of Thermal Expansion (CTE) can be addressed.

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