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Pultrusion of commingled thermoplastic composite materials

Vijay Anand Ananthakrishnan
University of Nevada, Las Vegas

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UMI®
PULTRUSION OF COMMINGLED THERMOPLASTIC COMPOSITE MATERIALS

by

Vijay A. Ananthakrishnan

Bachelor of Science
University of Madras, Madras
1997

A thesis submitted in partial fulfillment of the requirements for the degree of

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Vijay Ananthakrishnan

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Examination Committee Chair

Dean of the Graduate College

Examination Committee Member

Graduate College Faculty Representative
ABSTRACT

Pultrusion Of Commingled Thermoplastic Composite Materials

by

Vijay A. Ananthakrishnan

Dr. Brendan J. O'Toole, Examination Committee Chair
Associate Professor of Mechanical Engineering
University of Nevada, Las Vegas

The work involves the analysis and verification of the pultrusion process. The pultrusion machine at UNLV was improved by modifying the consolidation and the cooling system. A comparative study of the material properties of the pultrusion process and the compression molding were done with the theoretical material properties. This involved a series of compression molding experiments of the commingled material followed by the pultrusion process itself. The results were then confirmed by a series of mechanical tests of the material obtained as a result of the pultrusion and the compression molding processes in the Material Test System (MTS) machine. The work also involved the setting up of a control system using LabView, instrumentation software. A data acquisition system and remote control system using serial communication was created for the pultrusion machine. The comparative study showed that compression molding samples were 32% stronger and had 52% higher modulus. All the data were plotted in a...
graph for better understanding. The changes in the consolidation did not improve the material properties but decreased the flexural strength by 60% and modulus by 75% from the previous dies. The material properties can be improved by changing the cooling system of the consolidation module. The flexural strength and the modulus of the pultrusion system were 10% and 15% better than the theoretically predicted values. The compression molding samples had flexural strength and the modulus were 20% better than the theoretically predicted.
# TABLE OF CONTENTS

ABSTRACT .......................................................................................................................... iii

LIST OF FIGURES .............................................................................................................. vii

LIST OF TABLES ............................................................................................................... viii

ACKNOWLEDGEMENT ........................................................................................................ ix

CHAPTER 1 INTRODUCTION ............................................................................................... 1
  1.1 Introduction .................................................................................................................. 1
  1.2 Composite Material ..................................................................................................... 5
  1.3 Pultrusion – Research and Advances ........................................................................ 11

CHAPTER 2 THE THERMOPLASTIC PULTRUSION SYSTEM ............................................. 16
  2.1 Overview ..................................................................................................................... 16
  2.2 Creel and Material Guide Module ............................................................................. 17
  2.3 The Preheat and Consolidation Module ..................................................................... 18
  2.4 The Cooling System and The Manual Control Module ........................................... 28
  2.5 The Pulling Mechanism and The Computer Data Acquisition Module .................. 31

CHAPTER 3 EXPERIMENTS ................................................................................................. 40
  3.1 The Compression Molding .......................................................................................... 40
  3.2 Pultrusion Process ....................................................................................................... 43
  3.3 Velocity Experiments .................................................................................................. 44
  3.4 Flexural Tests ............................................................................................................. 45
  3.5 Micro-Structural Analysis .......................................................................................... 47

CHAPTER 4 DISCUSSION ..................................................................................................... 49
  4.1 Interpretation of Results ............................................................................................. 49
  4.2 Comparison of Results .............................................................................................. 59

CHAPTER 5 CONCLUSION ................................................................................................. 63
  5.1 Summary ..................................................................................................................... 63
  5.2 Future Work ............................................................................................................... 64

APPENDIX I ......................................................................................................................... 66
LIST OF FIGURES

Figure 1.1 Polyetherethileketone ................................................................. 1
Figure 2.1 Creel Stand and Guides .............................................................. 2
Figure 2.2 The Preheat Oven ................................................................. 3
Figure 2.3 Preheat Oven and Thermocouples ........................................... 4
Figure 2.4 Consolidation Dies Phase 1 ...................................................... 5
Figure 2.5 Consolidation Dies Phase 2 ...................................................... 6
Figure 2.6 Cross-section at Point C of Figure 2.5 ...................................... 7
Figure 2.7 Consolidation Dies Phase 3 ...................................................... 8
Figure 2.8 Consolidation Dies Phase 5 ...................................................... 9
Figure 2.9 Phase 5 Consolidation Die ....................................................... 10
Figure 2.10 Reservoir and Plumbing ........................................................... 11
Figure 2.11 Cooling Die and Plumbing ..................................................... 12
Figure 2.12 Manual Control Box with Display ......................................... 13
Figure 2.13 Motor-Pulling Mechanism ..................................................... 14
Figure 2.14 Pulling Module ..................................................................... 15
Figure 2.15 SCXI System ......................................................................... 16
Figure 2.16 Consolidation Dies Phase 5 with Thermocouple Locations .... 17
Figure 2.17 Data Acquisition VI .............................................................. 18
Figure 2.18 Serial Communication VI ...................................................... 19
Figure 3.1 Flexural Test Set Up and Cross-section of the Sample .......... 20
Figure 4.1 Compression Molding ............................................................ 21
Figure 4.2 Flexural Test Data – Compression Molding ......................... 22
Figure 4.3 Flexural Test Data – Linear Section Compression Molding ...... 23
Figure 4.4 Flexural Test – Pultrusion – 180-375-3-2 .................................. 24
Figure 4.5 Flexural Test – Pultrusion – 180-375-3-2-Linear Section .......... 25
Figure 4.6 Consolidation of Commingled Tows at various Compaction Rates ... 26
LIST OF TABLES

Table 1.1  Thermo-Mechanical Properties of PEEK .................................................. 8
Table 1.2  IM7 Carbon Fibers Properties .................................................................. 11
Table 4.1  Flexural Strength and Modulus of the Compression Molding ............... 52
Table 4.2  Flexural Strength and Modulus of the Pultrusion Process .................... 56
Table 4.3  Flexural Strength and Modulus ................................................................. 61
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CHAPTER 1

INTRODUCTION

1.1 Introduction

Pultrusion is an automated process for manufacturing reinforced material into continuous constant cross-section profiles. The term pultrusion was derived from a similar process known as extrusion. Pultrusion can be thought of as the reverse to extrusion with similar results. In both the processes, drawing the material through a die or spinneret forms material.

With the pultrusion process, the material is pulled through the die where in the extrusion process the material is pushed through the die by pressure. Thousands of yards of finished structure can be made in a continuous process. The major limitation is that the finished product must be straight and of a constant cross section. It has taken a considerable amount of time for the pultrusion process to find its place in the spectrum of manufacturing processes for reinforced structures. Brant Goldworthy, of Goldworthy Engineering Inc., in the sixties first introduced the working model of pultrusion when he revealed his thermoset machine [1].

This method has the potential for cost reduction, but current technology is limited to constant cross sections and is restricted in fiber orientation. Pultrusion is not as popular as metal extrusions. Metal extrusions are attached to other structural members, such as
skins and webs, by hundreds and thousands of fasteners and rivets. This method of assembly is not acceptable for composites, where the strong trend is to eliminate fasteners.

The introduction of boron filaments in the early 1960s lead to the birth of advanced composites technology. High modulus, high strength continuous filaments, like boron and later carbon, have profoundly impacted today's aerospace airframe design and manufacturing. The application of boron/epoxy composites and the development of their manufacturing technology were limited by several factors: (1) the high cost of boron filament and no prospects for replacing expensive tungsten substrate, (2) the limitation on a bend radius of no less than 1", (3) the high cost of diamond tools required for machining, drilling, and trimming, (4) the fact that the applications were limited to one form of prepregs (i.e., they were only available in 3" wide tape) [4].

Carbon and aramid fibers and prepregs were introduced in the latter 1960s. These fibers have some distinct advantages over boron: (1) their extremely small diameter (6 to 10 microns) reduced the bend radius to less than 1/16", (2) traditional high-strength steel could be used instead of diamond-tip tools for cutting, trimming, drilling, and machining, (3) there was a greater potential for achieving low cost ($10/lb compared to $90/lb for boron in 1960s dollars), (4) they were available in a variety of strengths, stiffnesses, and other mechanical properties [4]. The introduction of carbon fibers drastically increased the variety of applications and changed the way airframe structures were manufactured.

Below is a list of manufacturing techniques and aids developed for carbon composites:
Many different kinds of fabrics and widths (some up to 60" wide) substantially increased the speed of hand lay-up processes. Drapeability was enormously helpful in the manufacturing of complex and concave products.

Tapes started to appear in 3, 6, 12, and 24-inch widths, which resulted in increased lay-up speeds.

Computer-controlled sophisticated ATLs (Automatic Tape Lay-up) began to appear in production. Improvements continue to this date. ATL can take different tape widths, and alignment becomes nearly perfect.

ACM (Automatic Cutting Machines) flooded the market. These included the Gerber Cutter, ultrasonic, laser, and water jet machines. Improvements continue to this date.

The one-shot co-cured techniques using expandable mandrels became popular. Several different kinds of washable mandrels were also introduced. Some large structural boxes were made this way.

Non-metallic (especially graphite or carbon) tools also became popular.

Filament winding and braiding became possible with carbon and aramid fibers.

Sophisticated multi-angled ply orientation pultrusion machines appeared.

Research and development yielded tow placement, RTM (resin transfer molding), injection molding, and other new manufacturing technologies.

Compression molding, diaphragm forming, hydroforming, magneforming, deepdrawing, stamping, etc., began to appear in abundance.

Preforms and stitched preplies appeared.
3-D and multi-directional weaving were used for improved damage tolerance and as potential replacements for joints and fittings.

Thanks to the introduction and continuous improvements in carbon fiber and prepreg technology, a quantum jump in progress was observed in advanced materials technology in general, and in manufacturing technology in particular. Pultrusion can be considered as one of the advancements that were achieved during this period.

The pultrusion process is more profound with thermosetting plastics and it was only in the eighties that, thermoplastic pultrusion was started. Among the reasons for this interest are that thermoplastic matrices, when compared to their thermoset relatives, potentially offer significant improvements in areas such as damage tolerance, postformability, processing rate, work environment, recyclability, etc. The aims of past research and development in thermoplastic pultrusion have been to realize these advantages through a purely technical approach. All along it has been more or less explicitly assumed that the excellent process economy of thermoset pultrusion would be transferable to its thermoplastic relation. Although the significant research and development efforts indeed have led to several demonstrator parts and a number of publications, the concrete results in economical terms have not been impressive. The commercial availability of pultruded thermoplastic composites is virtually non-existent and the expected goals remain as elusive as ever.

The following study is a report on thermoplastic pultrusion. The key elements of a thermoplastic pultrusion process are, a material handling system (referred to as creels), the heating system and the pulling system. Most commercial machines are capable of producing profiles at the rate of 1 in to 15 ft/min. The materials are pulled from a variety
of creel types. The materials are then preheated and are then made to enter a heated steel die that has been precision machined to the final shape of the part to be manufactured. The profile is continuously pulled and exits the mold as a cured, constant cross-sectional profile. The profile cools in ambient air or by water as it is continuously pulled by a mechanism that simultaneously clamps and pulls. The product emerges from the puller mechanism to be cut to the desired length.

1.2 Composite Material

Composites can be defined as materials made from two different materials that remain as different phases and where one of the materials is a binder or matrix and the other material is the reinforcement. The combination of dissimilar materials can have unique and advantageous characteristics, and can result in a material that is better in certain properties than either of the materials alone. The reinforcements and the matrix are usually very distinct types of materials with widely different physical properties. In most modern types of composites, the reinforcements are often fibers with high stiffness, high strength, and small diameters. Other reinforcements can be whiskers and particles. The matrix is less stiff and strong than the reinforcement but is easily formed into a complex shape. Typical matrix materials include polymers, ceramics, and metals. In addition to giving the part its shape, the matrix also protects the fibers from environmental damage and transfers impressed loads to the fibers. The choice of polymer, ceramic, or metal as a matrix material hinges on ease of fabrication, service conditions and specific properties that might be required for a particular application such as wear [5]. The pultruded material here is a composite material made from thermoplastic
material and carbon fibers. The composite material used in this thesis is a 12k tow commingled Carbon (IM7)/PEEK.

1.2.1 Thermoplastics

Plastics can be classified into thermosets and thermoplastics. The thermoplastics do not undergo any chemical changes during consolidation rather their changes are substantially physical. Thermoplastics are melt fusible and can be consolidated by the application of temperature and heat alone. They can be repeatedly softened by heating and hardened by cooling. Today’s thermoplastics are developed based on aromatic polymers rather than aliphatic chains (formerly used) [5]. The aromatic rings increase the intermolecular forces, thus restricting the movement of the backbone carbon chain. This enhances the mechanical properties, high temperature capabilities and solvent resistance. For the ease of processing, groups such as ether, carbonyl, thioether, amide, methylene, ester, isopropylidene and sulfones are incorporated between the aromatic rings to render the polymer chain more flexible. The thermoplastic used in this experiment is Poly Ether Ether Ketone (PEEK) (Figure 1.1).

All polyketones are very attractive because of their balanced properties. They exhibit excellent chemical resistance, excellent toughness, good strength and rigidity, good load bearing properties even at high temperatures and in harsh environments. PEEK has been the most investigated of all the thermoplastics and its composites are the most common and still much attention has been not devoted on the same. It is a semi-crystalline polymer for which the maximum achievable degree of crystallinity is around 50% [5]. Amorphous PEEK is produced if the melt is quenched. At normal cooling rates,
the crystallinity is between 30 and 35%. The presence of fibers in PEEK composites tends to increase the crystallinity to a higher level, since the fibers act as nucleation sites for crystal formation. Increasing crystallinity increases both modulus and high yield strength but it reduces its strain-to-failure [6].

Figure 1.1 PolyEther EtherKetone

It also exhibits a glass transition temperature (Tg) of 143° C and a crystalline melting point of 343° C [5]. Melt processing of PEEK requires a temperature range of 370-400°C. One can now consider IM7/PEEK as one of the foremost thermoplastic composite that can replace the epoxies in many aerospace composites. The outstanding property of PEEK is its high fracture toughness, which is 50-100 times higher than the epoxies. Another important advantage of PEEK is its low water absorption, which is less than 0.5% at 25°C compared to 4-5% for conventional aerospace epoxies. However, it may absorb solvents like methylene chloride. The amount of solvent absorbed decreases with increasing crystallinity [5]. The following table shows the thermo-mechanical properties of PEEK (Table 1.1) [5].
Table 1.1 Thermo-Mechanical Properties Of PEEK

<table>
<thead>
<tr>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Yield Strength</td>
<td>100 (MPa)</td>
</tr>
<tr>
<td>Tensile Modulus</td>
<td>3.1 (GPa)</td>
</tr>
<tr>
<td>Density</td>
<td>1.30 (g/cm³)</td>
</tr>
<tr>
<td>Poisson Ratio</td>
<td>0.42</td>
</tr>
<tr>
<td>Viscosity (at processing temperature)</td>
<td>34000 (poise)</td>
</tr>
<tr>
<td>Glass Transition Temperature $T_g$</td>
<td>143 (°C)</td>
</tr>
<tr>
<td>Melt Temperature $T_m$</td>
<td>343 (°C)</td>
</tr>
<tr>
<td>Processing Temperature $T_p$</td>
<td>370 – 400 (°C)</td>
</tr>
</tbody>
</table>

1.2.2 Fiber Reinforcement

The discovery of the extremely high stiffness and strength of single crystal filaments of materials, known as whiskers was made by Herring and Galt in 1952 [3]. This discovery could be said to mark the beginning of interest in high strength fiber-reinforced composites. A composite material typically consists of one or more fillers in a certain matrix. A carbon fiber composite refers to a composite in which at least one of the fillers is carbon fibers, either short or continuous, unidirectional or multidirectional, woven or non-woven. The matrix is usually a polymer, a metal, a carbon, a ceramic, or a combination of different materials. Carbon fiber fillers are usually three dimensionally
discontinuous, unless the fibers are three dimensionally interconnected by weaving or the use of a binder such as carbon.

The high strength and modulus of carbon fibers makes them useful as reinforcement for the polymers although they are brittle. Effective reinforcement requires good bonding between the fibers and the matrix, especially for the short fibers. For a unidirectional composite, the longitudinal strength is quite independent of the fiber-matrix bonding, while the transverse tensile strength and the flexural strength increases with the increasing fiber-matrix bonding. On the other hand, excessive bonding is also not good for the composite since it tends to make the matrix very brittle. Both chemical bonding as well as Van der Waal’s bonding, require the fibers to be in intimate contact with the matrix. The occurrence of a reaction between the matrix and the fiber improves the bonding between the two but at the same time excessive bonding may prove to be harmful to the fibers [7].

Carbon fibers are electrically and thermally conductive, in contrast to the non-conductive nature of the polymer matrices. Therefore, the carbon fibers can serve not only as reinforcements but also as an additive for the enhancement of the electrical and the thermal conductivity. Furthermore, carbon fibers have nearly zero coefficient of thermal expansion. Therefore, they can also serve as an additive for lowering the thermal expansion. The combination of high thermal conductivity and low thermal expansion makes carbon fiber composites useful for space structures that require dimensional stability.

Carbon fibers are built by long carbon-carbon molecular chains yielding very stiff fibers. The trends have driven development of carbon fibers in two directions; high-
strength (HS) fibers with very high tensile strength and a fairly high strain to failure (1-1.5%) and high modulus (HM) fibers with very high stiffness. Especially, the latter has found their use in advanced aerospace applications where the use of light weight materials with high stiffness is essential. Carbon fibers also have good friction properties, good X-ray penetration and are non-magnetic. The main drawback is the high cost and all carbon composites are relatively brittle. The polyacrylonitrile (PAN) fibers are stabilized in air (a few hours at 250°C) to prevent melting during subsequent higher temperature treatment. The fibers obtained after this treatment are heated slowly in an inert atmosphere to 1000-1500°C. Slow heating allows the high degree of order present in the fiber to be maintained. The rate of temperature increase should be low so as not to destroy the molecular order present in fibers.

The initial stretching treatment of PAN improves the axial alignment of the polymer molecules. During the oxidation treatment, the fibers are maintained under tension to keep the alignment of PAN while it transforms into rigid ladder polymer. In the absence of this tensile stress in this step, there will occur a relaxation and the ladder polymer structure will become disoriented. After the stabilization treatment, the resulting ladder type structure has high glass transition temperature so that there is no need to stretch the fiber during the next stage, namely carbonization. There still are present considerable quantities of nitrogen and hydrogen. These are eliminated as gaseous waste products during carbonization, that is heating to 1000-1500°C. The carbon atoms remaining after this treatment are in the form of a network of extended hexagonal ribbons. Although these strips tend to align parallel to the fiber axis, the degree of order of one ribbon with respect to another is relatively low. This can be improved by further
heat treatment at still higher temperatures (up to $3000^\circ$ C). This is called the graphitization treatment. The mechanical properties of the resultant carbon fiber may vary over a large range depending mainly on the temperature of the final heat treatment. Hot stretching above $2000^\circ$ C results in plastic deformation of fibers leading to an improvement in properties.

There have great advancements in the field of the PAN fibers especially in the intermediate modulus kind, which has higher specific tensile strengths than other types. One of the most important steps in improving the properties of the IM fibers are its final diameters, this reduces the probability of encountering critical flaw in a given test length. The following table shows the properties of the IM carbon fibers (Table 1.2) [6].

<table>
<thead>
<tr>
<th>Table 1.2 IM7 Carbon Fibers Properties</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tensile Modulus</td>
</tr>
<tr>
<td>Tensile Strength</td>
</tr>
<tr>
<td>Strain to Failure</td>
</tr>
<tr>
<td>Mean Fiber Diameter</td>
</tr>
<tr>
<td>Specific Gravity</td>
</tr>
</tbody>
</table>

1.3 Pultrusion – Research and Advances

Thermoplastic pultrusion is a promising continuous composite manufacturing process. The high viscosity of thermoplastics is the main reason for much narrower processing window possible for thermoplastic pultrusion than thermoset pultrusion.
Therefore, only a few companies have commercial production units [1]. Not until the nineties was much work done on the research done on thermoplastic pultrusion. Most work in the eighties was done by Tomas B. Astrom, etal, [8] who worked on a lot of mathematical models incorporating the processing parameters of the system. Most models developed by Astrom were developed for better consolidation of the material and also to make the process faster. In the nineties, there were some advances on the thermoplastic pultrusion process. One reason for the improvement in the field of thermoplastic composite materials was the concerns about environmental hazards in using thermosetting composites. The following is brief summary of papers on thermoplastic pultrusion.

1.3.1 Flow through Aligned Fiber Beds and its Application to Composite Processing

Thomas B. Astrom and R. Byron Pipes investigated the flow through aligned fiber beds, which represent the flow of thermoplastic resin over the carbon fiber bed. In the process modeling of continuous fiber composites, matrix flow through aligned fiber beds is explained by Darcy’s Law, which relates matrix flow rate to the matrix pressure gradient, matrix viscosity, and fiber permeability. The paper shows how previously all work related to flow over spherical bodies are related to Newtonian fluids and how the same can be incorporated for cylindrical beds and also how Darcy’s law can be applied on the same. The main objective to perform this study was to apply the study on composite processing [8].
1.3.2 A Modeling Approach to Thermoplastic Pultrusion

The following summary is a study of mathematical models on thermoplastic pultrusion. The whole paper is based on the fact that a typical die system of a thermoplastic pultrusion is a two-die system, the last of which is cooled. The heated die is shorter than the thermoset counterpart and also the taper of the cavity is tapered over a much greater portion of the die length to create back flow of the resin to achieve desired consolidation and compaction. Pulling Force and Pressure-Temperature Models were created to study the process better and achieve better consolidation of the material. The conclusions from the model stated that the matrix flow is caused by translation of the fiber bed through the taper of the heated die and by thermal expansion of the composite within the confines of the taper. The temperature model defined the temperature of the composite at any particular point and also yielded the composite cooling rate, hence explaining the crystallinity of the composite. The pulling force model helped determine the pulling force resistance as a function of the die length in terms of viscous, compaction and friction resistance [9].

1.3.3 Thermoplastic Pultrusion – A Cell Model Approach

The study adopts a cell model approach, which means that the taper section of the heated die is divided into self-similar cells. This arrangement is then geometrically arranged to solve for the flow field, which yields the pressure gradient. The force required to overcome resistance is then calculated. The paper also explains details about the shear thinning of the resin and the pressure drop of the same. Applying energy equations, the temperature dependent viscosity and the heat generated due to viscous
dissipation and hence the heat transfer in the die was determined. The model was parametrically applied and hence real process and the properties of the material were improved [10].

1.3.4 A Model For Consolidation And Void Reduction During Thermoplastic Tow Placement

This paper presents a model that is capable of predicting final void fraction and the final thickness of a composite part as a function of the processing speed, the consolidation pressure and the local temperature in the consolidation window.

During the in-situ composite processing heat and pressure are applied which causes the fiber in the tow to be transported with the matrix. The void content of the composite changes during the consolidation process. The voids may coalesce, get compressed due to the consolidation pressure or may grow due to temperature changes. All these changes and factors are taken into account in this model. The boundary conditions of the walls may vary from perfect to no-slip and the edges may be either a free or fully restricted surface.

The model has been developed based on the assumption that the composites can be modeled as a compressible continuum. Thus, the problem is solved as the squeeze flow of a compressible viscous fluid. In this model, three cases, namely, isothermal, incompressible, isothermal compressible and non-isothermal compressible have been solved. As a result of experiments, it has been seen that compaction pressure exhibited a linear increase with tow speed [11].
1.3.5 High Speed Pultrusion of Thermoplastic Matrix Composites

The difficulty in impregnating the fibrous reinforcements with high viscosity resins led to many manufacturing techniques, which are used to fabricate thermoplastic matrix composites. One method is to intermingle the polymer fibers with the reinforcing fibers by providing the matrix in a fiber form. Another technique is to impregnate the reinforcing tow with polymer powder particles, then melt, and fuse the particles in place. This paper models and characterizes these two techniques using molding techniques and applies these experiments to the on-line consolidation that occurs during the pultrusion process.

In this paper, a model has been developed to predict the consolidation time of a powder-impregnated fiber by considering the flow of molten particles along fiber axis driven by the applied pressure. The model predicts that the impregnation rate is controlled by the ratio of particle to fiber size, fiber content, matrix viscosity etc. The results show that the model accurately predicts the variation in void content during consolidation of laminates.

A model applied to the commingled fibers relates the impregnation time to pressure and velocity and this can be used to predict the optimum conditions for pultrusion processing. The results demonstrate that this method of pultrusion of commingled fibers is a promising technique in the development of thermoplastic composites [12].
CHAPTER 2

THE THERMOPLASTIC PULTRUSION SYSTEM

2.1 Overview

The pultrusion system at the University of Nevada, Las Vegas is an experimental thermoplastic pultrusion system. The machine has the capabilities to pull material of small constant dimension at varying speeds. The speeds vary from 1 in/minute to about 30 inches/minute. The main variable parameters are the number of tows of material that can be pulled through the machine, the pre-heat temperature, the consolidation temperature, the pressure and the consolidation time. The pressure of the system is a function of the number of tows and the opening of the cooling die. The consolidation time is a function of the pull speed of the pulling mechanism. The following is a comprehensive description of the original system and the changes that were incorporated to the same in order to improve the process. The pultrusion machine is setup in a modular fashion allowing for easy modification and expansion of the system. The system is made up of the creel and material guide module, preheat and consolidation module, cooling system and manual control module, pulling module, and computer with data acquisition module. Mr. Raymond C. Kozak originally developed the system and modifications were done on the same [1].

16

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2.2 Creel and Material Guide Module

The creel is an adjustable rack to hold rolls of material used by the machine. The whole system is adjustable both horizontally and vertically. The creel has three levels and two bays each able to carry ten rolls of material, thus making it capable of supporting sixty rolls. The material guide is mounted in front of the creel and was the initial guide for positioning the material to enter the preheat oven. The guide is adjustable in both the horizontal as well as the vertical direction.

![Creel Stand And Guides](image)

**Figure 2.1 Creel Stand And Guides**
2.3 The Preheat and Consolidation Module

2.3.1 The Preheat Oven

The preheat oven is a setup to make changes or repair in the system in case of failure of the material, a necessity for continuous material processing. This minimized the potential for degradation of the polymer in the event of processing interruption and also to heat the temperature to the glass transition temperature of the PEEK so as to increase the matrix flow over the fibers before the entry of the material into the consolidation die.

Figure 2.2 The Preheat Oven
This reduces sluffing, which is a process by which a part of material fed into the consolidation die refuses to enter the die and hence clogs the entrance and disrupts the flow of the material resulting in the failure of the process.

The Preheat oven is a convection type tubular oven (Figure 2.2). The preheat oven is constructed with horizontal steel platens positioned above and below the material to be heated. The plates are heated with eight horizontally placed 250-WATT cartridge heaters per platen, inserted in holes drilled through the platens. The entire setup is covered by alumina-silica insulation board all around. This prevents any heat loss.

The temperature of the preheat oven is determined using two systems. A non-contact thermometer and also by thermocouples placed at intervals along the length of the heater. The non-contact thermometer is an infrared temperature-sensing device, which is in turn connected to the closed loop manual control system. The sensor is placed right at the end of the heater, providing the temperature of the material just before it enters the consolidation die. The thermocouples are used to determine the temperature distribution across the length of the heating system. The temperatures from the thermocouples will be discussed in detail in a later section, section 2.5.2. The preheat was not modified a lot except for the introduction of the temperature distribution from the thermocouples (Figure 2.3).

2.3.2 The Consolidation Dies

The consolidation dies form the important part of the system, for this is the part, which provides the shape and properties intended for the material. The consolidation dies were the ones, which went through a lot of changes in the entire system from the
originally developed system. The main objective of this thesis is to improve the pultrusion process and main emphasis was put on the consolidation dies. The consolidation also includes the cooling system and hence the cooling system was also set to a lot of changes. The following is a comprehensive report on the consolidation system and the changes that were applied on the same.

2.3.2.1 The Consolidation Dies Phase I.

The first consolidation system was a simple two-part die system, the heating die and the cooling die separated by a narrow air gap, both mounted on a sliding table assembly (Figure 2.4). These dies were made in such a way that they could be assembled.
with an upper and lower half to facilitate pressure changes that would act on the material. The upper and lower parts of the heating die were a tongue and groove arrangement respectively whereas the cooling dies were just flat surfaces in contact. The entrances of the dies had tapers to induce back flow and reduce sluffing. The cooling die was cooled by circulating coolant through the holes drilled in the cooling die. The changes in the die resulted in the change in the cooling system and the coolant itself. These will be discussed in detail in the following paragraphs as we deal with the various consolidation dies used. The coolant used in the initial system was water.

![Diagram of Consolidation Dies Phase 1](image)

**Figure 2.4 Consolidation Dies Phase 1**

### 2.3.2.2 The Consolidation Dies Phase 2.

The phase 1 consolidation dies had problems with sluffing and hence a small amount of material was lost before consolidation. One of the reasons for the sluffing was the upper and the lower half assemblies of the dies. It was determined through
compression molding tests that the material consolidated well under constant pressures for longer periods of time and also when the material was subjected to pressure increments in steps rather than suddenly subjecting the same to the required pressure. Hence the phase 2 dies were developed by machining the required dimensions through solid rectangular blocks of steel. The dies were a three stage system with two heating dies and a cooling die. Another important feature incorporated in the dies is that there are no air gaps between the dies (Figure 2.5).

![Diagram of Consolidation Die Phase 2](image)

**Figure 2.5 Consolidation Die Phase 2**

![Diagram of Cross-Section at Point C of Figure 2.5](image)

**Figure 2.6 Cross-Section at Point C of Figure 2.5**
temperature and the material was below the glass transition temperature before it could enter the cooling die, (Point B, in Figure 2.5) where the final consolidation pressure was applied to the material. In order to facilitate the process the temperature of the second die was intended to be increased so that the temperature of the material will be higher than the melting temperature of the material. This was tried by changing the flow rate of the coolant. It was found that as the flow was reduced the temperature of the coolant increased which lead to pressure build ups in the die and also the vinyl plumbing tubes could not take more heat. This lead to phase 3.

2.3.2.3 The Consolidation Dies Phase 3

The phase 3 was a slight modification of the phase 2 consolidation dies (Figure 2.7). The cooling dies were separated from the heating dies and hence an half inch air gap was created between the dies. This helped increase the temperature of the second die but the temperature of the material fell below the glass transition temperature before it could enter the third die and hence the system failed. In addition, sluffing started to appear at the entrance of the cooling die.

![Figure 2.7 Consolidation Dies Phase 3](image_url)
2.3.2.4 The Consolidation Dies Phase 4

The separation of the heating and cooling dies proved futile as the material started sluffing again. To avoid sluffing, the air gap was removed and the dies were put back together. To increase the temperature of the second cooling die the heated dies were insulated with alumina-silica insulators. To maintain the temperature of the cooling die, the cooling system was modified. The following is a comprehensive report on the various changes that were applied to the cooling system.

The main reasons for making changes in the cooling die were that when the material enters the cooling die, the temperature of the die should be maintained above the melting temperature. The materials fail to enter at lower temperatures as it is not malleable. And the temperature should be lower than the glass transition temperature as at higher temperatures the material expands and opens up loosing the properties at the exit of the cooling die. The temperature distribution of the cooling die was so high that the water either absorbed all the heat from the die or was not able to take the heat. This was because when the flow rate of water was high, more heat was absorbed and hence the temperature of the die dropped rapidly. If the flow rate was controlled to maintain the temperature then the high temperature turned water into steam. Also water has a high thermal conductivity and the purpose of the coolant was more to maintain the temperature at the desired level rather than cooling it completely. Hence, more alternatives were sought for.

- The first alternative was to put additives in the water, hence increase its boiling point, and also reduce its thermal conductivity. Hence, common salt was added to water. It
is known that addition of common salt increased the boiling point of water. The tests revealed that the increase in boiling could not improve the needed temperature distribution. Also it was found that corrosion was increased to greater extent.

- The next alternative was to add commercial antifreeze. The commercial antifreeze is known to increase the boiling and the freezing points of water in the radiators of automobiles. It was also learned that the engine reaches high temperatures at continuous running. When antifreeze was added to water no improvement was observed, as the mixture improved the boiling point only by a few degrees. Later it was found that the rate at which the antifreeze is run through the automobile was high and at lower flow rates, it was no better than water.

- Heavy machining coolant was the next alternative that was investigated. The boiling point of the coolant increased considerably but still the temperatures at the entry and the exit of the cooling die were not maintained at the desired levels.

The failure of all water based coolants paved the way for other types. After intense study on coolants, four alternatives were investigated. The first one was Dowtherm a commercial heat exchanger fluid. Dowtherm is used in most solar farms where the heat from the solar panels are transferred to these fluids which in turn heat water. Dowtherm had a high boiling point far greater than the glass transition temperature of PEEK but had a low flash point and also was very expensive. The next alternative was paraffinic oil. Paraffinic oils also had high boiling points and was not expensive but had low flash point. Engine oil was tried next, engine oil did not have high boiling points compared to Dowtherm and paraffinic oils but was high enough but the problem still persisted with flash point. The final alternative was mineral oil.
Mineral oil had a high boiling point and a pretty high flash point. The flash was five degrees less than the glass transition temperature of PEEK. It was thought that a new cooling system, which would rapidly bring down the temperature of the coolant the moment it exits the dies and before it enters the reservoir, which was the only place where the coolant would be exposed to the accessible environment. The proposed design included a leak free plumbing and passing of the coolant through a freezing mixture before it would enter the reservoir.

2.3.2.5 The Consolidation Dies Phase 5

The failure with the coolants led to a total redesign of the dies. The new design involved the combination of the phase 1 and the phase 2 dies (Figure 2.9). The cooling die from phase 2 was removed and was replaced with the phase 1 dies. The air gap was reintroduced, as the faces of the die did not match. There was also a change in the first heating die. The failure in the phase 4 arrangement lead to the removal of one half of the first heating die and all four heating cartridges were placed in the second heating die (Figure 2.8).
The heavy machinery coolant and water was the coolant used. The problems with sluffing was reduced to negligible amounts and the material with better consolidation was pultruded by adjusting the taper in the cooling die. The cooling die were well adjusted so as to totally avoid sluffing. The combination die worked successfully and the material was pultruded for various parameters.

Figure 2.9 Phase 5 Consolidation Die
2.4 The Cooling System and The Manual Control Module

2.4.1 The Cooling System

The cooling of the consolidation system is achieved by circulating fluid through the cooling die. The cooling system is operated by circulating the coolant from a 33-gal reservoir through the dies and back to the reservoir (Figure 2.10). A turbine pump circulates the fluid and a throttle valve adjusts the flow rate. The reservoir has a pipe nipple at the bottom of the tank for fluid return. The fluid is supplied through a ¼" copper pipe and polypropylene tubing. The polypropylene tubing enhanced the movement of the die arrangement over the linear bearings.

Figure 2.10 Reservoir and Plumbing
With the changes in the coolant and the temperature that is to be maintained at the cooling die, the plumbing work for the cooling system was modified. Polypropylene had temperature ratings of 75°F and that was too low. Hence, the tubing had to be changed. The new tubing also needed to be flexible as the polypropylene tubing and at the same time needed to take a lot of heat. The only tubing that would serve the purpose was hard rubber tubing. But even that could not take the high temperatures at the contact point of the die and the tubing. Hence, a combination copper tubing and the hard rubber tubing were used (Figure 2.11). The hard rubber tubing or hoses were the typical ones used transferring steam at the dry cleaners. They had high temperature capabilities and at the same time also had insulation and protection to take high-pressure.

Figure 2.11 Cooling Die And Plumbing
2.4.2 The Manual Control

The manual control system was a two component system consisting of systems control and motor control (Figure 2.12). The systems control involves the temperature control of the heating system and also displays various parameters of the system. The temperature control system works on a simple feedback PID control system. The preheat system has an infrared sensor at its exit. The sensor sends feedback to the control unit located in the control box. The feedback is compared with the set point and the temperature is maintained. Similarly the temperature feedback from the consolidation die is obtained from a thermocouple and the temperature is hence controlled. The drawback with this system was that, only one temperature could be read and controlled at a time and there was no equipment to find the temperature distribution at the entire length of the preheat oven or the temperature at various points of the consolidation die.

The systems controls also had displays for the pull force and the pull speed of the system. A load cell located with die assembly at the end of the linear bearing determined the load that acted on the die as a result of the pulling of the material. The pull speed was determined by determining the rpm of the motor (Figure 2.13).

The motor control of the system was a simple set up with no feedback. It was a typical rheostat set up and the speed of the motor was varied with the change of the rheostat position. The manual control system also had other switches, to turn on/off the cooling system, the on/off switch for the air system and the power switches.
2.5 The Pulling Mechanism And The Computer Data Acquisition Module

2.5.1 The Pulling Mechanism

The pulling mechanism by itself consists of many components. The air- supply, the air handling and the motor and drive chain assembly. The pulling mechanism uses six pairs of rotating wheels, which grip and pull the consolidated material. The six upper wheels are free to rotate move vertically to accommodate the varying the thickness of the material. The vertical movements of these wheels were controlled by pneumatic cylinders, which cause the upper wheel to squeeze the consolidated material.
Figure 2.13 Motor – Pulling Mechanism

The pneumatic cylinders are controlled by the air supply and the air handling system (Figure 2.14). The air supply system is an air compressor system. The compressor system was a combination unit consisting of an air compressor and horizontal reservoir tank. The supply air is processed by passing the same through a combination filter, regulator and lubricator unit.

The air handling system controls the pinch rollers directly. The air from the air handling system is delivered to the cylinders through polypropylene tubing. Adjustable flow controls were employed at the cylinders to insure smoother consistent motion.
2.5.2 Computer Based Data Acquisition System

2.5.2.1 Temperature Control of the Die and Preheat System of the Pultrusion Machine from a Remote System.

All the heating in the machine is done by heating cartridges commercially manufactured Chromalox inc., The systems are already provided with control units. The control units make use of the PID control systems. The system also had the capability to display the temperature at one point of the die and at the exit of the preheat section of the machine.

The temperature from the pre-heat section of the machine was determined using an infrared sensor. This provided the temperature at the exit of the machine and also
served as the feedback for the PID control of the control system of the heating cartridges. The temperatures of the heating dies are obtained from a thermocouple that also provides a feedback for the control system.

The main objective of this endeavor is to obtain the temperatures from various sections of the pre-heat and the die-section and also to set the target temperatures of the die and the pre-heat sections of the machine. The following is a comprehensive report on the set up of the entire automation system. The entire automation system of the pultrusion was done using the products from National Instruments. The devices included:

1. A Data Acquisition Board (AT-MIO-16E-10): These boards are used for collecting data in general, by performing A/D conversions. The Board is sometimes also used to generate data. The AT-MIO-16E-10 boards are known for their versatility. These were among the first completely plug and play compatible multifunction analog, digital and timing input/output boards for PC AT and compatible computers. These boards are switches and jumpers free and can be calibrated using software. The boards have three timing groups that control analog input, analog output and general purpose counter timer functions. The AT-MIO-16E-10 boards can interface to a SCXI system so that one can acquire over 3,000 analog signals from thermocouples, RTDs, strain gauges, voltage sources, and current sources. One can also acquire and generate digital signals for communication and control.

2. Signal Conditioning eXtensions for Instrumentation (SCXI): The SCXI is a high-performance signal conditioning system, using an external chassis that contains input/output modules for signal conditioning, multiplexing, etc.
(Figure 2.15). The chassis is wired into a DAQ board in the PC. The SCXI can be used in two ways- as a front-end signal conditioning system for plug-in DAQ boards or as an external DAQ system. By using it as a front-end signal conditioning system for plug-in DAQ boards, one can combine the benefits of plug-in DAQ boards with external DAQ systems. The SCXI chassis houses a variety of signal conditioning and instrumentation modules. Analog signals are conditioned and passed back to a single plug-in board for acquisition directly into the PC memory. The plug-in DAQ board also controls and monitors the operations of digital I/O and analog output in the SCXI module. With a SCXI system one can monitor and control hundreds of signals from a single plug-in DAQ board.

3. Serial Communication Ports (Recommended Standard #232, RS 232): the RS 232 communication ports are standard proposed by the American Society for Serial Communications. The serial ports used here are used to communicate with the control systems for the heating cartridges. The heating cartridge controllers have certain modes in which they can be communicated from a remote computer. The main purpose of the system is to set the temperature for the dies in the control units.
Figure 2.15 SCXI System

Thermocouples: Thermocouples are one of the most frequently used temperature sensors. Thermocouples are very rugged and inexpensive and can operate over a wide temperature. A thermocouple is created whenever two dissimilar metals touch and the contact point produces a small open-circuit voltage as a result of temperature. There are several types of thermocouples classified on the basis of their temperature range and accuracy. The most common types are the J, K and the B type. The K type thermocouples are the ones made use of here. There are several thermocouples running along the length of the pre-heat and the die sections, the thermocouple positions in the consolidation dies are shown in Figure 2.16, in the figure, the numbers represents the thermocouple. These
thermocouples help us determine the temperature distribution in those sections. The manipulation of the data that is obtained is done using LABView. The data, which is otherwise of no use, is converted into its corresponding temperatures using the LABView that was developed. The program created had the capability to display all the temperature points on the die. The following is the Front Panel of the LABView Program.

Figure 2.16 Consolidation Dies Phase 5 with Thermocouple Locations

The Data Acquisition Program was created using LabView. The program had the capability to select what signals needed to be to be displayed and also which data acquisition board to be used. The Data acquired were displayed as an array of data and also plotted on a chart. There were options created in the program to store information to files and indicate when a temperature goes past a limit. The following is the Virtual Instrument developed (Figure 2.17).
An attempt was made to set up a remote system to operate (to change) the temperature from the computer. To achieve the same, the temperature controller was interfaced with the computer through a RS232 port. A RS232 board from National Instruments was installed and the two temperature controllers, the preheat and the consolidation die ones were connected to the computer.

It was found that the controllers responded to only one kind of protocol and it had provisions to change the set temperature of the material. A program was written to
communicate with the controller. The following is the communication VI created (Figure 2.18). The VI had problems with the system as the response time was long and the system timed before communication could be established.

Figure 2.18 Serial Communication VI
CHAPTER 3

EXPERIMENTS

3.1 The Compression Molding

Compression molding was developed in 1909 by Leo Bakeland to produce phenolic radio cabinets. Phenolic was originally named after Bakeland and called Bakelite. In the compression molding process, a thermoset plastic, phenolic, melamine, or thermoplastic, is molded in an enclosed mold between two heated platens. The plastics, in powder, pellet, liquid, or perform, are introduced into the mold in a partially cured condition. A perform is the exact amount of material required to mold the part pressed into a large pellet or plug. The materials are introduced into the mold just before molding. The platens that hold the two halves of the mold are heated to approximate processing temperature. Pressure in the range of 1,500 PSI is exerted on the material. The shearing action of the material being compressed together and the heat from the platens cause the plastic to become soft. The soft plastic fills the cavity and is compressed by the pressure. The contributory strength of the temperature and the pressure accelerates the curing of the plastic in approximately four minutes. The compression molding process produces a heavy and dense product. Thermoset products have the highest electrical, heat and chemical resistance of any plastic material.

Compression molds are machined from steel. Their cavities are designed to be full positive, semi-positive, landed positive or flash molds. The fully positive mold requires
that the exact amount of material be placed in the mold. The fully positive mold does not allow excess material to flow out between the parting line. Semi-positive molds allow for flash (material that flows out of the mold at the parting line) of excess materials just prior to the final closing of the mold. Landed positive molds allow for flashing in a prescribed area which forms a land or tab which can be removed and subsequently machine polished after molding. Flash molding allow for the elimination of excess materials during the final moments of the compression cycle. Products are more easily produced from flash mold since they do not require the exact amount of material. However, they must be machined and polished in post molding operations. Handles for cooking pots, housings for hot plates, electrical receptacles, and high voltage switch housing are common products. Melamine, which enjoys lighter colors, is used in inexpensive dinnerware, kitchen utensils, and high pressure laminated counter tops.

Matched metal compression molding was one of the oldest manufacturing techniques in the composites industry. Recent advancements in this field made compression molding process very popular for mass production of composite parts [3]. The principal advantage of the compression molding is its ability to produce parts of complex geometry in short periods of time. The compression molding operation begins with the placement of stacks of the charge on to the bottom half of the preheated mold cavity. The charge area is selected to cover up to 60-70% of the of the mold surface area. The mold is closed quickly after the charge placement, and the top half is lowered at a constant rate until the pressure of the charge reaches a preset pressure. With the increase in the pressure the charge starts to flow towards the cavity extremities, forcing the air out of the cavity. All the factors involved in the process, the temperature of the mold, the
pressure created in the die, the rate at which the pressure is applied or the speed of the ram play an important role in the final appearance of the composite. For instance, the rate at which the pressure is applied on the charge plays a vital role in the surface finish of the final product. The speed is often in the range of 4-12 mm/sec. Vacuum molding improves surface pressure for instance affects the porosity of the surface and starts to fill the cavity of the mold.

The Compression molding set up at the University of Nevada, Las Vegas is a typical experimental set up. The experiment is set up to simulate the pultrusion process. The dies developed match the tongue and groove type dies of the phase 1 consolidation dies [16]. The set up is also a process for thermoplastic composites unlike the conventional system, which is usually designed for the thermoplastic composites.

The main objective of the compression molding tests was to fabricate composites from commingled tows of Carbon and PEEK. The experiments were performed by varying the various processing parameters like the temperature, the pressure and the processing time. The experiments were performed on the MTS (Material Testing System) Machine. The MTS is a versatile testing machine. The machine is totally controlled by the computer. The compression die has eight slots and each of them house high wattage heating cartridges capable of reaching up to 1000 Celsius. The heating system is controlled to maintain the temperatures at a fixed temperature for periods of time. The dies are cooled by passing compressed air on the same. The experiments performed used 16 tows of material IM7 Carbon and PEEK. The procedures were modified for a processing temperature of 375°C and Pressures of 200 and 400 psi. The material is first held at a pressure of 100 psi and held until it reaches the processing temperature. On
reaching the processing temperature, the material is subjected to the processing pressure which here is 200 psi. This is followed by cooling at a pressure of 375 psi and held at that pressure until the material temperature goes below its glass transition temperature.

3.2 Pultrusion Process

The new dies paved the way for the pultrusion of the material and determination of the performance of the phase 5 dies. The material used was the commingled Carbon/PEEK. The process was performed by changing the various parameters of the machine.

The main parameters were

- The preheat temperature

The preheat temperature of the material in the preheat oven. The temperature is set such that the material is at the set temperature at exit. The preheat temperature enables the material to become more malleable and enhances the material to enter the consolidation die with more ease. The preheat temperature is usually maintained around the temperature where the matrix becomes a semi liquid forming spherical shapes over the fibers. The preheat temperatures used here are 165 and 180 C.

- The consolidation die temperature

The consolidation temperature is the temperature of the material when at the heated dies of the system. The material temperature is set at the entrance of the second consolidation die, the
thermocouple 3 position in figure 2.16. The consolidation temperature forms the important parameter of the process. The temperature is usually maintained between the processing temperature of the matrix. The processing temperature range is 375 – 400° C. The temperatures used were 375 and 395 degrees C.

- The pull speed.

The pull speed of the material is the speed in which the material is pulled through the die. The material pull speed varies around a wide range. The pull speeds used were three and ten inches per minute.

The materials were pultruded with all combination of the above parameters. The material samples were taken at intervals of time and lengths of 4 feet and labeled.

3.3 Velocity Experiments

An attempt was made to verify the work done A.H. Miller, N.Dodds, J.M. Hale and A.G.Gibson on the paper High Speed Pultrusion of Thermoplastic Matrix Composites [12]. The paper discussed the determination of the best parameters for the pultrusion process. The parameters were determined from a set of experiments called the velocity experiments. The experimental setup was similar to the compression molding ones but the experiment was performed by varying the ram velocity. The experiment also used 16 tows of material. The experiment was performed for various velocities but the data was not analyzed to determine the parameters.
3.4 Flexural Tests

The process of pultrusion has various parameters involved. The temperature, pressure and the pull-force are a few of them. The mechanical properties of the materials produced may vary with the change in the parameters. The variance in the material property has to be determined to determine the optimum processing parameters. The material properties can be determined using many methods like the mechanical testing methods, analyzing the microstructures, by chemical, and thermal methods. The testing for this work were done using the mechanical methods. The materials were subjected to flexural tests and hence their properties were determined.

3.4.1 Mechanical Testing – Flexural Tests

These test methods cover the determination of flexural properties of the composite material. There are two ways in which a flexural test can be performed, 1. The three-point loading system utilizing center loading on a simply supported beam. 2. A four point loading system utilizing two load points equally spaced from their adjacent support points. The pultruded material was tested using the three-point system. The objective of this experiment is to perform three point bend tests on the material prepared from the compression molding and the pultrusion process. The experiment also involves the calculation of the Elastic modulus of the samples. The samples used in the experiments were the ones molded at 200 psi from the compression molding tests and the pultrusion samples. The modulus of elasticity was calculated using the deflection method. The deflection and the loads were also plotted.
3.4.2 Experimental Setup

The experiments were performed on the MTS (Material Test System). The MTS is a versatile testing machine. The whole machine is controlled by a computer, which is run by Test-Star, the software that runs the system. The experimental setup is shown below (Figure 3.1). The specimens were placed on the setup and loaded until failure.

![Diagram of experimental setup](image_url)

Figure 3.1 Flexural Test Set Up and Cross-Section of the Sample
3.4.3 Procedure

1. The material is placed on the fixture and is loaded by a point load.
2. The load and displacement of the ram were noted every tenth of a second.
3. The material is loaded till failure.
4. The failed material is removed and steps 1 through 3 are repeated for the other samples.

The material obtained as a result of the compression molding tests and the pultrusion process were tested for their properties by performing flexural tests on the same. The flexural tests were performed in accordance to ASTM D790 [13].

3.5 Micro-Structural Analysis

The objective of this experiment was to determine the fiber volume fraction of the composite obtained from the compression molding process. The experiment also involved the preparation of the sample, which in turn involved the potting of the sample in epoxy, polishing the specimens, observation of the microstructure and determination of the fiber volume fraction.

3.5.1 Preparation of the Sample

The samples from the compression molding are cut into small thin pieces. Care should be taken that the edge of the specimen is perfect and not uneven. The specimens are then placed in a plastic mold, which is already cleaned and coated with mold release. The molds are then filled with the molding resin. The molding which was used was EPO-TEK. The epoxy is cured for about a day at room temperature. The potted specimens are then removed from the mold.
The specimens are then subjected to series of polishing. The specimen are first hand polished and then polished using polishing wheels of grit 3, 1 and 0.3 micron.

3.5.2 Microstructure Observation and Analysis

The specimen's microstructure were then observed under a microscope. The images of the microstructure were then captured by a camera and analyzed using Global Lab Image Software. The fiber volume percentage using software and also by using line and area methods. The average fiber volume fraction of the compression molding samples was 0.67.
CHAPTER 4

RESULTS AND DISCUSSION

4.1 Interpretation of Results

4.1.1 Compression Molding

The materials made from the compression molding experiments formed basis for the explanation of the material properties in the consolidation die of the pultrusion machine. The parameters pressure, temperature and displacement were plotted as a function of time (Figure 4.1). The data from the file were formatted to fit the graph, the displacement direction was changed and the values of the displacement data were made positive. The whole graph can be split into three sections; the contact pressure section, the consolidation pressure section and the cooling pressure section.

In the contact pressure section, the material is held at a nominal pressure to hold it in position while it is being heated to the processing temperature. At the consolidation section, the material heated is at the processing temperature and the consolidation pressure is applied. The heating system is then turned off and rapidly cooled at $26^\circ$ C per minute approximately. The system is held under pressure while being cooled. The displacement plot shows that yielding starts when the temperature reaches the melt temperature of the material. It was also noted that the yield is not prominent at any other
section of the graph. This information was used of in the pultrusion process. The material was maintained at its melt temperature when entering the cooling die and the failure at the consolidation system was minimized. The compression molding tests were performed for various pressures and consolidation times. The scope of this paper limits us to just one set of experiments. The processing parameters for the samples tested were 200 psi pressure and 1 minute consolidation time. The one minute consolidation samples were taken for the consolidation time in the pultrusion were also one minute.

4.1.2 Flexural Tests

The compression molding and the pultrusion process samples were tested for their flexural strengths and their flexural modulus. The tests were done by flexural tests methods ASTM D790. The samples from the compression molding were first tested and then compared with the pultrusion samples.

The data files for the tests were big and the properties of the sample would be erroneous if they were calculated without any data reduction. The following is the data reduction scheme followed for all the samples. The load versus displacement curves are plotted as shown in figure 4.2. The plot is analyzed for its linear section (Figure 4.3). A linear regression is then determined for the linear section and the data of that section is then used for calculations. The graph shows us the linear section of the load versus displacement curves of one sample. The flexural properties of the material were calculated using the formulae shown below and properties of the samples are tabulated in table 4.1. The parameters used in the formulae are shown in Figure 3.2.
Compression Molding at Pressure 200psi and 1 minute Consolidation time

Figure 4.1 Compression Molding
The flexural properties calculation.

- **Span length**: $L$ in
- **Width of the Specimen**: $b$ in
- **Thickness of the Specimen**: $h$ in
- **Load**: $P$ lb
- **Displacement**: $d$ in
- **Stress**: $P \cdot (3L)/(2b^2h^2)$ lb/in$^2$
- **Modulus**: $(L^3)/(4h^3b)(P/d)$ lb/in$^2$
- **Strain**: Stress/Modulus

Table 4.1 Flexural Strength and Modulus of the 200 psi Compression Molding Samples

<table>
<thead>
<tr>
<th>Sample</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Stress (ksi)</th>
<th>Modulus (msi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Specimen 1</td>
<td>0.286</td>
<td>0.039</td>
<td>50.7</td>
<td>1.4</td>
</tr>
<tr>
<td>Specimen 2</td>
<td>0.287</td>
<td>0.038</td>
<td>57.4</td>
<td>1.6</td>
</tr>
<tr>
<td>Specimen 3</td>
<td>0.287</td>
<td>0.0395</td>
<td>62.3</td>
<td>1.7</td>
</tr>
</tbody>
</table>

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Figure 4.2: Flexural Test Data - Compression Molding
The pultrusion samples were also tested to determine their flexural properties. The load versus displacement data were plotted as a graph (Figures through 4.10). The flexural strength and modulus of all the samples are tabulated below (Table 4.1). The sample names were given based on their processing parameters. The first three digits represent the preheat temperature, the next three digits represent the consolidation temperature and the last digits represent the pull speed.

Table 4.2 Flexural Strength And Modulus Of Pultrusion Samples

<table>
<thead>
<tr>
<th>Samples</th>
<th>Width (in)</th>
<th>Thickness (in)</th>
<th>Stress (ksi)</th>
<th>Modulus (msi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>165-375-03</td>
<td>0.220</td>
<td>0.065</td>
<td>25.7</td>
<td>1.0</td>
</tr>
<tr>
<td>165-375-10</td>
<td>0.225</td>
<td>0.067</td>
<td>19.4</td>
<td>0.6</td>
</tr>
<tr>
<td>165-395-03</td>
<td>0.214</td>
<td>0.068</td>
<td>25.8</td>
<td>1.0</td>
</tr>
<tr>
<td>165-395-10</td>
<td>0.227</td>
<td>0.066</td>
<td>19.1</td>
<td>0.7</td>
</tr>
<tr>
<td>180-375-03</td>
<td>0.222</td>
<td>0.066</td>
<td>12.8</td>
<td>0.4</td>
</tr>
<tr>
<td>180-375-10</td>
<td>0.225</td>
<td>0.061</td>
<td>21.9</td>
<td>0.7</td>
</tr>
<tr>
<td>180-395-03</td>
<td>0.223</td>
<td>0.064</td>
<td>16.7</td>
<td>0.5</td>
</tr>
<tr>
<td>180-395-10</td>
<td>0.223</td>
<td>0.063</td>
<td>16.7</td>
<td>0.5</td>
</tr>
</tbody>
</table>
Figure 4.4 Flexural Test – Pultrusion – 180-375-3-2
4.1.3 Velocity Experiments

The experiment was performed to obtain optimum parameters for the pultrusion process but could not be completed due to time constraints. The material was compressed at constant velocities and the graphs were plotted for the same (Figure 4.11).

4.2 Comparison of Results

The compression molding results showed that the material initially yields at a particular temperature, this is noted by the sudden drop of the load applied by the ram. The temperature at which the load drops, when compared with the material properties shows that the temperature correspond to the melting temperature of the resin. This helped designing the cooling system, that is, how much should the temperature of the material be when entering the cooling. This helps reducing the failure of the material on entering the cooling die.

The results from the flexural tests showed that the material consolidation was much better in the compression molding tests compared to the pultrusion process itself. The compression molding samples had flexural modulus 32% more than that of the pultrusion samples and had flexural strength 55% more than the pultrusion samples. This only proves that the pultrusion process is not complete with its design. This directly shows that more works needs to be done with the consolidation dies.
Figure 2.6 Consolidation of Comminuted Tows at Various Compaction Rates
The following table shows the flexural Strength of the samples from the pultrusion and compression molding tests.

Table 4.3 Flexural Strength and Modulus

<table>
<thead>
<tr>
<th>Experiment</th>
<th>Process</th>
<th>Flexural Strength (ksi)</th>
<th>Flexural Modulus (msi)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Pultrusion</td>
<td></td>
<td>25.8</td>
<td>2.9</td>
</tr>
<tr>
<td>Compression</td>
<td></td>
<td>62.3</td>
<td>1.7</td>
</tr>
<tr>
<td>Molding</td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Theoretical Analysis</td>
<td>Pultrusion</td>
<td>29.5</td>
<td>1.8</td>
</tr>
<tr>
<td></td>
<td>Compression</td>
<td>37.8</td>
<td>2.3</td>
</tr>
<tr>
<td></td>
<td>Molding</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Micro-Structure</td>
<td>Pultrusion</td>
<td>33</td>
<td>2</td>
</tr>
<tr>
<td>Analysis</td>
<td>Compression</td>
<td>47.3</td>
<td>2.9</td>
</tr>
<tr>
<td></td>
<td>Molding</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Previous Work [1]</td>
<td>Pultrusion</td>
<td>86.7</td>
<td>7.85</td>
</tr>
</tbody>
</table>
The pultrusion process pultruded materials at the required parameters. The results from the flexural tests showed us that the slower the process the better the consolidation. In addition, it was tested that for higher preheat temperatures above 190° C, the consolidation turned out to be bad. In addition, at low temperatures the same results were found usually with the failure of the material. The optimum range was found around 160° and 180° C. At this temperature the matrix forms into small beads all over the fiber. Better consolidation was observed at this temperature range.

From the overall results, it was found that the best consolidation results were found for the following set of parameters. Pull speeds of 3 in/min, preheat temperature of 165° C and a consolidation temperature of 395° C.
CHAPTER 5

CONCLUSION

5.1 Summary

Thermoplastic pultrusion is one of the engineering fields with only a few advancements. This is because of the fact that thermosetting plastics are still being used widely for its ease for manufacturing. The thermoplastics are more complex when it comes to manufacturing. The thermoplastic pultrusion system here at UNLV is one the most versatile available. The consolidation system and the cooling system of the die were modified, to try and improve the quality of the pultruded material. The result was a combination of the new dies and the original cooling die. The cooling system was considered for various alternatives. The coolants itself were changed and results were observed. The final system paved way for a new coolant and new plumbing, which could transfer coolants at higher temperatures compared to its predecessors.

An attempt was made to automate the entire machine. The machine was connected to a computer and was interfaced through data acquisition and serial communication boards. A program was created to display temperatures from the preheat and the consolidation dies. The temperatures were plotted and displayed in a graph. Another program was created to remotely access the temperature controller.
The machine was run with 16 tows of commingled Carbon/PEEK. The samples were made by changing the preheat temperature, the consolidation temperature and the pull speed. The material were then tested for their flexural modulus and their strengths.

The same material was created using the compression molding process. This process was more straightforward and no changes were made to the system. The samples were made at various processing temperatures and pressure. The samples were tested by flexural tests. The results from both the compression molding process and the flexural tests were compared to the pultrusion process and its samples. The results showed that at lower preheat temperatures and higher consolidation temperatures and lower pull speeds samples of high consolidation can be made.

5.2 Future Work

The pultrusion machine still needs to be improved overall. The most important of them are the consolidation die and the computer controls. The consolidation die still make use of the cooling die from the original system and hence there are still chance for sluffing and hence loss of material which results in lower grade material. The new cooling dies without the top and bottom assembly should be installed and hence a new cooling system should be designed.

The computer controls can be enhanced by setting up a control system to run the machine. A feedback control system can be created using pull force as the feedback and controlling the pull speed. This will partly automate the system and reduce the chances of failure of the system.
Another important field of study is the preheat temperature. It was noted during the experiment that as the temperature of the preheat went past the 195°C mark, the pull force drastically increased. Upon close studying, it was noted that a part of the matrix melted and vaporized before entering the consolidation temperature. It should be noted that the melting temperature of PEEK was well past that temperature and the glass transition temperature was much higher than this temperature. A study should be done on the and hence the right preheat temperatures for the system can be determined.
APPENDIX A

CALCULATION FOR THEORETICAL AXIAL MODULUS AND STRENGTH

Material Used IM7/PEEK

IM7 fiber Diameter = 5 μm
Area of IM7 fibers = 1.963*10^{-11} m^2

The material used is a 12 k Tow

Therefore Area of tow A_{tow} = 2.356*10^{-6} m^2

Number of Tows used = 16

Area of fiber A_{fiber} = 0.00584 in^2

Pultrusion Process

Average Dimensions

Width = 0.222 in
Thickness = 0.065 in
Total Area = 0.014 in^2

Fiber Volume Fraction V_f = A_{fiber} / Total Area = 0.41

Using rule of mixtures

Modulus = 1.8 msi
Strength = 29.5 ksi
Compression Molding Average Dimensions

- **Width** = 0.286 in
- **Thickness** = 0.039 in
- **Total Area** = 0.011 in²
- **Fiber Volume Fraction** \( V_f \) = \( \frac{A_{fiber}}{Total\ Area} = 0.53 \)

Using the rule of mixtures

- **Modulus** = 2.3 msi
- **Strength** = 37.8 ksi
APPENDIX B

CALCULATION FOR EXPERIMENTAL AXIAL MODULUS AND STRENGTH
FROM MICRO STRUCTURAL ANALYSIS

Compression Molding Average Dimensions

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Width</strong></td>
<td>0.286 in</td>
</tr>
<tr>
<td><strong>Thickness</strong></td>
<td>0.039 in</td>
</tr>
<tr>
<td><strong>Total Area</strong></td>
<td>0.011 in²</td>
</tr>
</tbody>
</table>

Fiber Volume Fraction $V_f$

| (from microstructural analysis) | 0.67 |

Using the rule of mixtures

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Modulus</strong></td>
<td>2.9 msi</td>
</tr>
<tr>
<td><strong>Strength</strong></td>
<td>47.3 ksi</td>
</tr>
</tbody>
</table>

Pultrusion Process

Average Dimensions

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Width</strong></td>
<td>0.222 in</td>
</tr>
<tr>
<td><strong>Thickness</strong></td>
<td>0.065 in</td>
</tr>
<tr>
<td><strong>Total Area</strong></td>
<td>0.014 in²</td>
</tr>
</tbody>
</table>
Percentage Change in Area from compression molding sample = 21 %

Hence Fiber Volume fraction = 0.46

Using rule of mixtures

Modulus = 2.0 msi

Strength = 33 ksi
REFERENCES


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VITA

Graduate College
University of Nevada, Las Vegas

Vijay Ananthakrishnan

Local Address:
4209 Grove Circle, #2
Las Vegas, Nevada 89119

Home Address:
6, Govardhan Street
Royapettah, Chennai 600 014

Degrees:
Bachelor of Science, Engineering, 1997
University of Madras, Chennai

Thesis Title: Pultrusion of Commingled Thermoplastic Composite Materials

Thesis Examination Committee:
Chairperson, Dr. Brendan J. O'Toole, Ph. D.
Committee Member, Dr. Robert Bhoeme, Ph. D.
Committee Member, Dr. Zhiyong Wang, Ph. D.
Graduate Faculty Representative, Dr. Clinton Richards, Ph. D.