Project #: E-25-M62  Cost share #:  Rev #: 3
Center #: 10/24-6-R7252-0A0  Center shr #: OCA file #:  Project: AMENDMENT
Prime #:  Contract #: 60NANB1D1144  Mod #: ADMIN.
Subprojects #: N  Main project #: OCA file #:  03/23/92

Cost share I:
Center #: 10/24-6-R7252-0A0  shr 1: 10/24-6-R7252-0A0
Rev #: 3
OCA file #: 03/23/92

Active

Project unit:  MECH ENGR
Project director(s): COLTON J S  MECH ENGR
(404)894-7407

Sponsor/division names: US DEPT OF COMMERCE  / NATL INST OF STDS & TECH
Sponsor/division codes: 110  / 005

Award period: 910701 to 920630 (performance)  920930 (reports)

Contract #:
Prime I:
Subprojects #: N
Main project #: 60NANB1D1144

Sponsor amount
Contract value New this change Total to date
0.00 28,237.00
Funded 0.00 28,237.00
Cost sharing amount 0.00

Does subcontracting plan apply ?: N

Title: MODELING AND TESTING OF THERMOPLASTIC COMPOSITE FILAMENT WINDING ...

PROJECT ADMINISTRATION DATA

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Sponsor technical contact  Sponsor issuing office
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(301)975-3440  (301)975-6327

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NIST
ACQUISITION AND ASSISTANCE DIVISION
GRANTS UNIT
BLDG. 301, ROOM B128
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Security class (U,C,S,TS): U
Defense priority rating : N/A
Equipment title vests with: Sponsor
ONR resident rep. is ACO (Y/N): N
N/A supplemental sheet
GIT X

Administrative comments -
ADMINISTRATIVE CORRECTION - O/H RATE WAS INCORRECTLY STATED AS 50.5% ON LATEST FUNDING INCREMENT. O/H RATE HAS BEEN CORRECTED TO READ 61.5%.
GEORGIA INSTITUTE OF TECHNOLOGY
OFFICE OF CONTRACT ADMINISTRATION

NOTICE OF PROJECT CLOSEOUT

Closeout Notice Date: 06/09/92

Project No. E-25-M62
Center No. 10/24-6-R7252-0A0

Project Director: COLTON J S
School/Lab: MECH ENGR

Sponsor: US DEPT OF COMMERCE/NATL INST OF STDS & TECH

Contract/Grant No.: 60NANB1D1144
Contract Entity: GTRC

Prime Contract No.

Title: MODELING AND TESTING OF THERMOPLASTIC COMPOSITE FILAMENT WINDING

Effective Completion Date: 920630 (Performance) 920930 (Reports)

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Comments:

Subproject Under Main Project No.

Continues Project No.

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NOTE: Final Patent Questionnaire sent to PDPI.
Quarterly Performance Report

Modeling and Testing of Thermoplastic Composite Filament Winding Consolidation Mechanisms

to

National Institute of Standards and Technology

Program Manager: Richard Norcross
Building 220, Room B127

Contract Number: 60NANB1D1144

Date: October 30, 1991

by

Georgia Institute of Technology

Dr. Jonathan Colton
School of Mechanical Engineering
Introduction

A general deformation model describing the mechanical processes that occur during the filament winding of thermoplastic towpregs using a single-roller, on-line consolidation mechanism (Figure 1) is proposed. The basis for the model is that the primary deformation mechanisms are resin flow parallel to the fibers and fiber bed compaction. These two mechanisms are assumed to occur when the material passes through the nip region and the formation of intimate contact and the removal of entrapped air occurs. Some of the components of this model are derived from a pultrusion model developed by Astrom and Pipes [1,2]. Stated below is the proposed deformation model modified for application to a filament winding process.

Deformation Model

The model assumes the process is steady-state, that no transverse flow occurs, and that the nip temperature is constant through the cross-section of the tow. A simplified lay-up geometry is used in the analysis of the nip region (Figure 2).

Applying fiber continuity and assuming the tow is transversely isotropic, the fiber volume fraction at a position, x, in the nip can be obtained as

\[ \frac{v_f(x)}{v_L} = \frac{h_L}{h(x)} \]

where \( h(x) \) is the height of the tow at a position, \( x \), within the nip. The terms \( h_L \) and \( v_L \) refer to the final tow height and fiber volume fraction, respectively.

With geometric compatibility between the compaction roller and the towpreg, the fiber volume fraction distribution in the nip may be written as

\[ v_f(x) = \frac{h_0v_0}{h_L + R - \sqrt{R^2 - (x - L)^2}} \]

where \( L \) refers to the length of the nip and \( R \) refers to the radius of the compaction roller.

The matrix flow rate is obtained by applying continuity to the resin material. As the matrix travels at the same velocity as the fibers at the end of the nip, the matrix flow rate may be written in terms of the winding speed as

\[ q(x) = \left( \frac{h_L}{h_0v_0} - 1 \right)Uv_f(x) \]

where \( U \) refers to the winding speed.

The matrix pressure gradient can be related to the matrix flow rate using Darcy's law modified with the Kozeny-Carmen expression for
permeability and the Carreau model for shear-thinning fluids [2]. The flow rate/pressure drop relationship can be written as

\[ \frac{\Delta p_m}{L} = \frac{4K_0K_1^2\eta_a q(x)}{r_f^2} \frac{v_f^2}{(1 - v_f)^2} \]

with

\[ \eta_a = \eta_0 (1 + (\lambda \dot{\gamma})^2)^{\frac{n-1}{2}} \]

where the term \( K_0K_1^2 \) refers to the Kozeny constant and the symbol, \( \eta_0 \), refers to the zero-shear-rate viscosity. The time constant, \( \lambda \), refers to the shear rate at which shear-thinning effects become important. The matrix pressure distribution is obtained by integrating the above equation with respect to position in the nip (Figure 3).

The fiber bed pressure is obtained from Gutowski [3] and can be written as

\[ p_f = \frac{C \frac{\sqrt{v_f}}{v_o} - 1}{(\frac{\sqrt{v_\infty}}{v_f} - 1) \frac{4}{4}} \]

where \( C \) is a spring constant and \( v_\infty \) refers to the highest obtainable fiber volume fraction. The fiber bed pressure is relatively low compared to the matrix pressure since the fibers are well aligned when they enter the nip.

The applied load necessary to achieve a given percent height reduction or final fiber volume fraction can be obtained by integrating both the matrix and fiber pressure distributions over the contact length of the nip and multiplying this by the width of the tow. This is written as

\[ P_{appl} = \int_0^L \left[ p_m(x) + p_f(x) \right] dx \]

where \( w \) refers to the width of the tow.

**Preliminary Model Verification**

A Microsoft Excel spreadsheet was developed to evaluate the deformation model at various process conditions (Figure 4). Preliminary verification of the model was carried out using data obtained from NIST ring specimen #11. This particular sample was wound from 1/4 inch wide APC-2 towpreg at a speed of 2 inches per second and underwent a 28 percent reduction in height. The consolidation pressure was provided by a single compaction roller with a diameter of 1 inch. Material constants for APC-2 at its processing temperature of 750°F were also entered into the...
spreadsheet. The model predicted that an applied load of 55 pounds would be required to achieve the percent height reduction for this sample, as compared to the actual applied load of 48 pounds.

**Conclusions**

Preliminary results indicate that the proposed deformation model may sufficiently account for the effects of winding speed and applied load on the total amount of deformation that occurs during the filament winding of APC-2 towpreg. However, further experimental verification is needed to evaluate the model's predictive ability.

**References**


Figure 1: Experimental Set-up
R = radius of roller
L = length of contact zone
$h_0 = h(x=0) = $ height of incoming tow
$h_L = h(x=L) = $ height of deformed tow
$x = $ lay-up direction
Figure 3:
Matrix Pressure Distribution Within Nip for NIST Specimen #11

Matrix Pressure (MPa)

Height Reduction = 28%
Nip Temperature = 400 C
Winding Speed = 0.06 m/s

x-position / nip length
## Figure 4: Deformation Model Worksheet (NIST Specimen #11)

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<th>D</th>
<th>E</th>
<th>F</th>
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**Calculation Summary:**

- **Initial:**
  - Radius: 0.0127 m
  - Apparent vfo: 0.5
  - Height: 0.0001778 m

- **Final:**
  - Radius: 0.013187 m
  - Apparent vfo: 0.5
  - Height: 0.00013187 m

**Inputs:**

- **x-position (m)**
- **vf**
- **dP/dx (Pa/m)**
- **Pm(x) (Pa)**
- **Pf(x) (Pa)**
- **Pm(x) (MPa)**

**Outputs:**

- **Radius:** 0.0127 m
- **Apparent vfo:** 0.5
- **Height:** 0.0001778 m
- **Final:** 0.013187 m
- **Apparent vfo:** 0.5
- **Height:** 0.00013187 m

**Other Values:**

- **Kozeny:** 0.00026374
- **Viscosity:** 0.00032967
- **Fiber:** 0.00036264
- **Radius (m):**
  - Initial: 0.0127 m
  - Final: 0.013187 m
- **Time:** 0.00049451
- **Constant (s):** 0.04
- **Viscosity:** 0.00056044
- **Exponent:** 0.8
- **Fiberpress:** 0.44
- **Towwidth:** 0.00635 m
- **Load:** 62592946 N
- **CO:** 1.4728E+11
- **c1:** 9.1636E+13
- **c2:** -2.597E+20
- **c3:** -3.267E+23
- **c4:** 4.2705E+26

**Additional Values:**

- **Input:**
  - 2 x-position (m): 0.49983536
  - 3 dP/dx (Palm): 0.50705374
  - 4 Radius (m): 0.0127
  - 5 vf= 0.49983536
  - 6 vf= 0.50705374

**Additional Calculations:**

- **Final:**
  - Radius: 0.013187 m
  - Apparent vfo: 0.5
  - Height: 0.00013187 m

**Additional Output:**

- **Pm(x) (Pa):**
  - Initial: 1.47E+11
  - Final: 2.53477902E+11

**Other Details:**

- **Additional calculations and results related to deformation model and specimen characteristics are detailed in the table.**
Quarterly Performance Report

Modeling and Testing of Thermoplastic Composite Filament Winding Consolidation Mechanisms

to

National Institute of Standards and Technology

Program Manager: Richard Norcross
Building 220, Room B127

Contract Number: 60NANB1D1144

Date: January 13, 1992

by

Georgia Institute of Technology

Dr. Jonathan Colton
School of Mechanical Engineering
Introduction

An experimental investigation of consolidation in thermoplastic composite filament winding is presented. A $2^2$ factorial design was used to investigate the effects of winding speed and applied load on the void percentage, fiber volume fraction, and final thickness of the consolidated part. An experimental on-line consolidation mechanism was constructed and installed on a filament winding machine. Composite rings were fabricated and prepared for testing. The ring specimens currently are being measured and tested. Once sample testing is complete, the data will be statistically analyzed and used to determine the validity of the proposed deformation model. The model will then be modified, if necessary.

Experimental Set-up

The development of the experimental apparatus (Figure 1) involved the design of an on-line consolidation device which could simultaneously apply heat and pressure to the nip over a range of winding speeds. Heat to the nip is supplied by two Sylvania hot air guns fed by regulated air and open-loop voltage controllers. A Series III, 4000 watt hot air gun is used to heat the incoming tow to 400 °C. A Series I, 2000 watt hot air gun is used to heat the surface of the substrate (preprocessed layers) directly beneath the nip to 300 °C. This substrate temperature was chosen, as it promotes diffusion bonding without significantly altering the substrate; it has a relatively small heat-affected zone. The actual tow and substrate temperatures were determined at each winding speed by applying thin coatings of Omega temperature indicating paint to each surface and adjusting the air pressure and/or applied voltage until the paint transformed. Pressure within the nip is generated by a single 28.6 mm diameter compaction roller maintained at constant applied load using a diaphragm air cylinder and a servo-controlled regulator. The roller is cooled actively and contains Molycoat high-temperature lubricant. A calibration curve of applied load versus cylinder air pressure was obtained using a compression scale. The mandrel consisted of a 178 mm diameter collapsible aluminum cylinder filled with insulation and sealed on the ends with Kapton polyimide film. The winding speed was determined from the measured rotational velocity and the mean diameter of the mandrel.

Material

The composite material studied is 6 mm wide APC-2 AS-4 12 K prepreg tow supplied by ICI. This particular form of PEEK/carbon fiber is recommended by the manufacturer for filament winding. The prepreg tow has an initial fiber weight fraction of 37.9 percent (reported by manufacturer) and an initial average height of 0.254 mm. The tow height was measured with a micrometer and therefore represents an upper bound. Further height and fiber volume fraction measurements will be made using optical microscopy techniques.
**Experimental Description**

A total of five experiments were performed (Table 1). The first four experiments constitute the $2^2$ factorial design and can be represented geometrically as the corners of a square (Figure 2). The last experiment corresponds to the center of the square and represents ring specimens run at an intermediate winding speed and applied load. The results from this particular experiment will be used to aid in the verification of the proposed deformation model and will not be contained in the statistical analysis since they are not part of the full factorial design. Three ring specimens were fabricated at each of the five experiment points for a total of 15 rings produced. Each ring consisted of 50 layers of prepreg tow with an inside diameter of 178 mm.

Table 1: Experimental Design

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<th>Winding Speed (mm/s)</th>
<th>Winding Speed (in/s)</th>
<th>Applied Load (N)</th>
<th>Applied Load (lbf)</th>
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<td>89 (-)</td>
<td>20 (-)</td>
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<td>RS2-i</td>
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<td>0.5 (-)</td>
<td>222 (+)</td>
<td>50 (+)</td>
</tr>
<tr>
<td>RS3-i</td>
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<td>2.0 (+)</td>
<td>222 (+)</td>
<td>50 (+)</td>
</tr>
<tr>
<td>RS4-i</td>
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<td>89 (-)</td>
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<td>35</td>
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</tbody>
</table>

Independent variables studied: winding speed, applied load (or pressure)

Dependent variables studied: void percentage, final fiber volume fraction, total height reduction

Figure 2: The $2^2$ factorial design
The range for each parameter studied was determined from preliminary experiments in which rings were fabricated at various loads and speeds and then qualitatively tested for degree of consolidation using an acoustic response technique. This technique involves striking the rings with a metal rod and listening for the quality of the sound produced. A "bell-like" sound generally indicates a consolidated part.

Sample Testing and Preliminary Results

The thickness of each consolidated ring was measured at ten equally-spaced positions on the ring using callipers. Preliminary results of the thickness test (Table 2) indicate that there is some effect of the various parameters studied on the thickness of the consolidated part. The relatively large variance in experiments RS1 and RS2 is due to the difficulty in maintaining tow alignment at the slower winding speed.

Table 2: Preliminary Thickness Results

<table>
<thead>
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<th>Experiment # (i=1,2,3)</th>
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<th>Average Final Thickness (in)</th>
<th>Coefficient of Variation</th>
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<td>1.82</td>
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<td>0.877</td>
</tr>
<tr>
<td>RS4-i</td>
<td>6.43</td>
<td>0.253</td>
<td>0.779</td>
</tr>
<tr>
<td>RS5-i</td>
<td>6.43</td>
<td>0.253</td>
<td>0.594</td>
</tr>
</tbody>
</table>

The fiber volume fraction of each consolidated ring will be determined using optical microscopy techniques. The percent fibers by volume is equivalent to the percent fibers by cross-sectional area. The latter can be obtained using image analysis software in conjunction with an optical microscope.

The void percentage of each ring will be determined using a relative density technique as outlined in ASTM standards D792-86 and D2734-70. However, the fiber volume fraction and the crystallinity of the ring specimens must be known in order to achieve a representative density of the sample. The percent crystallinities of ring specimens from each experiment were determined using DSC techniques. An average weight percent crystallinity of 20 percent will be used to calculate the density of the resin.

Once sample testing is complete, the data will be statistically analyzed using analysis of variance to determine the main effect of each independent variable as well as the interactions between them. The fiber volume fraction and height reduction data also will be used to determine the validity of the proposed deformation model for this particular application.
Conclusions

Preliminary thickness results indicate that the parameters studied affect the quality of the consolidated part. The actual prediction of part quality may later be determined from a master curve of winding speed and applied load versus degree of consolidation (i.e., void percentage). However, the determination of a dimensionless consolidation number that takes into account both the winding speed (shear rate, resin flow), applied load (pressure gradients), and the nip temperature (resin viscosity) will provide significant aid in predicting part quality. These goals will be addressed during the continuation of this research.
Figure 1: Experimental Set-up
Quarterly Performance Report

Modeling and Testing of Thermoplastic Composite Filament Winding Consolidation Mechanisms

to

National Institute of Standards and Technology

Program Manager: Richard Norcross
Building 220, Room B127

Contract Number: 60NANB1D1144

Date: April 28, 1992

by

Georgia Institute of Technology

Dr. Jonathan Colton
School of Mechanical Engineering
**Introduction**

Presented in this report are the results from a theoretical analysis and an experimental investigation of consolidation in thermoplastic composite filament winding. A single roller, on-line consolidation mechanism was studied (Figure 1). A one-dimensional, semi-empirical process model was developed to describe the mechanical deformation and flow phenomena that occur during the on-line consolidation process. The model was used to predict the applied load required to achieve a given fiber volume content of the wound laminate. A $2^2$ factorial design was used to investigate the effects of winding speed and applied load on the void percentage, fiber volume content, and final thickness of the consolidated part. An experimental on-line consolidation mechanism was constructed and installed on a filament winding machine. Composite rings were fabricated, tested, and statistically analyzed. Three rings were consolidated at each experiment level with each ring consisting of 50 layers of APC2 AS4 12K towpreg. The model's predictions were compared to the experimental results in order to assess the adequacy of the proposed model for describing the consolidation process in thermoplastic filament winding.

**Theoretical Results**

The proposed model was used to predict the applied load required to achieve the reported final fiber volume content for each of the five experiment sets conducted in the experimental investigation. The winding speed used in each experiment and the average measured fiber volume content of the three ring specimens from each experiment served as inputs to the model. The material constants used in the model were for APC2 at 400°C. The model also requires two empirically determined parameters. These were obtained using the results of the experimental investigation. Figure 2 shows the predicted matrix pressure distribution generated in the nip for each experiment. All five profiles show an expected steady pressure rise due to the backflow of matrix material out the nip entrance. Figure 3 shows the predicted fiber bed pressure distribution generated within the nip for each experiment. The variation in the maximum (final) fiber volume content among the five experiment sets is reflected in the relative location of the peaks of each profile. The maximum fiber bed pressure is much lower than the maximum matrix pressure. There is little load transferred to the fiber bed during the on-line consolidation process studied because the range of fiber volume content is sufficiently less than the highest fiber volume content obtainable, the time at pressure is very short, the matrix viscosity is relatively high, the fibers are well aligned, and the winding tension is negligible.

The predicted applied load required to achieve the final fiber content in each experiment was obtained by integrating both the matrix and fiber bed pressure distributions over the length of the nip and multiplying this by the width of the towpreg. The applied load predictions are listed in Table 1. Figure 4 provides a graphical representation of the relationships between the predicted applied load and the fiber volume content for each of the three winding speeds studied in the experimental investigation. The figure indicates that the required applied load increases with increasing fiber volume content for a constant winding speed.
This is because more load must be applied to achieve the increased deformation corresponding to the higher fiber volume content. Figure 4 also indicates that the applied load decreases with increasing winding speed for a constant fiber volume content. This is due to the shear-thinning behavior of the molten PEEK polymer matrix. The increase in shear rate with winding speed results in a decrease in the matrix viscosity.

**Experimental Results**

The experimental results were obtained from composite ring specimens filament wound at various applied loads and winding speeds and include the results of the statistical analyses performed on the process parameters. The experimental conditions are listed in Tables 2 and 3. The experimental results are summarized in Table 4. It should be noted that the values listed in Table 4 represent an average of the three ring specimens fabricated in each experiment. An analysis of variance (ANOVA) statistical technique was used to determine the effects of applied load and winding speed on the thickness, fiber volume content, and void content of the ring specimens. The ANOVA results are summarized in Table 5. The winding speed was found to significantly affect all three of the dependent parameters studied. The applied load was found to significantly affect the void content and the thickness of the ring specimens. The effect of the speed/load interaction was found to be statistically insignificant.

**Comparison of Theoretical and Experimental Results**

The theoretical predictions for the applied load required to achieve the fiber volume content in each experiment were compared to the actual (measured) applied load in order to assess the adequacy of the proposed model for describing the mechanical phenomena that occur during the on-line consolidation process. Figure 5 provides a graphical representation of the correlation between the theoretical (predicted) applied load and the actual (measured) applied load. The average deviation in the model’s predictions for the required applied load was approximately 23 percent. In general, the experimental results follow the same trends predicted by the model: the required applied load increases with increasing fiber volume content and decreases with increasing winding speed.

**Conclusions**

A semi-empirical, consolidation process model was developed and found to describe reasonably well the mechanical phenomena that occur during the on-line consolidation process in thermoplastic filament winding. The ramifications of the theoretical and experimental results in addition to the application of the model to other processes and material systems will be addressed in the final report.
Table 1: Model Predictions for the Required Applied Load

<table>
<thead>
<tr>
<th>Experiment #</th>
<th>Winding Speed (mm/sec)</th>
<th>Final Fiber Content (% by volume)</th>
<th>Predicted Applied Load (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>12.7</td>
<td>55.6</td>
<td>131</td>
</tr>
<tr>
<td>RS2</td>
<td>12.7</td>
<td>56.0</td>
<td>166</td>
</tr>
<tr>
<td>RS3</td>
<td>50.8</td>
<td>60.5</td>
<td>196</td>
</tr>
<tr>
<td>RS4</td>
<td>50.8</td>
<td>58.5</td>
<td>84.9</td>
</tr>
<tr>
<td>RS5</td>
<td>31.8</td>
<td>58.9</td>
<td>193</td>
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</tbody>
</table>

Table 2: Experimental Design

<table>
<thead>
<tr>
<th>Experiment (i=1,2,3)</th>
<th>Winding Speed (mm/sec)</th>
<th>Winding Speed (in/sec)</th>
<th>Applied Load (N)</th>
<th>Applied Load (lbf)</th>
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</thead>
<tbody>
<tr>
<td>RS1-i</td>
<td>12.7</td>
<td>0.5</td>
<td>89</td>
<td>20</td>
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<tr>
<td>RS2-i</td>
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<td>0.5</td>
<td>222</td>
<td>50</td>
</tr>
<tr>
<td>RS3-i</td>
<td>50.8</td>
<td>2.0</td>
<td>222</td>
<td>50</td>
</tr>
<tr>
<td>RS4-i</td>
<td>50.8</td>
<td>2.0</td>
<td>89</td>
<td>20</td>
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<tr>
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<td>31.8</td>
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Table 3: Constant Parameters and Values

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<tr>
<th>Parameter</th>
<th>Value</th>
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<tr>
<td>Type of material</td>
<td>PEEK matrix / AS-4 carbon fibers</td>
</tr>
<tr>
<td>Material system</td>
<td>6 mm wide preimpregnated 12K tow</td>
</tr>
<tr>
<td>Process temperatures</td>
<td></td>
</tr>
<tr>
<td>- Incoming towpreg</td>
<td>400 °C</td>
</tr>
<tr>
<td>- Substrate surface</td>
<td>300 °C</td>
</tr>
<tr>
<td>Number of plies wound</td>
<td>50 plies</td>
</tr>
<tr>
<td>Lay-up angle</td>
<td>0 degrees</td>
</tr>
<tr>
<td>Size of mandrel</td>
<td>178 mm OD x 305 mm</td>
</tr>
<tr>
<td>Size of compaction roller</td>
<td>28.6 mm OD x 9.5 mm</td>
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<td>Winding tension</td>
<td>250 grams</td>
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Table 4: Experimental Results Used in Statistical Analysis

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<th>Experiment (i=1,2,3)</th>
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<th>Fiber Content (% by vol.)</th>
<th>Void Content (% by vol.)</th>
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<tr>
<td></td>
<td>Wind. Speed</td>
<td>Appl. Load</td>
<td>Speed/Load Interaction</td>
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<td>RS1-i</td>
<td>-</td>
<td>-</td>
<td>+</td>
<td>38.6</td>
</tr>
<tr>
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<td>-</td>
<td>+</td>
<td>-</td>
<td>40.8</td>
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<td>+</td>
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<td>+</td>
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<td>n/a</td>
<td>n/a</td>
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Table 5: ANOVA Results - Significance Level (%)

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<th>Fiber Content</th>
<th>Void Content</th>
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<td>Winding Speed</td>
<td>99.9</td>
<td>99.1</td>
<td>99.9</td>
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<td>Applied Load</td>
<td>99.8</td>
<td>70.6</td>
<td>94.3</td>
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<tr>
<td>Speed/Load Interaction</td>
<td>72.5</td>
<td>54.2</td>
<td>17.3</td>
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Figure 1: FILAMENT WINDING SET-UP
Figure 2: Matrix Pressure Distribution Within Nip for Experiments RS1 - RS5

RS1 (final Vf=0.556, wind.sp.=12.7 mm/s)
RS2 (final Vf=0.560, wind.sp.=12.7 mm/s)
RS3 (final Vf=0.605, wind.sp.=50.8 mm/s)
RS4 (final Vf=0.585, wind.sp.=50.8 mm/s)
RS5 (final Vf=0.589, wind.sp.=31.8 mm/s)
Figure 3: Fiber Bed Pressure Distribution Within Nip for Experiments RS1 - RS5

- RS1 (final Vf = 0.556, wind.sp. = 12.7 mm/s)
- RS2 (final Vf = 0.560, wind.sp. = 12.7 mm/s)
- RS3 (final Vf = 0.605, wind.sp. = 50.8 mm/s)
- RS4 (final Vf = 0.585, wind.sp. = 50.8 mm/s)
- RS5 (final Vf = 0.589, wind.sp. = 31.8 mm/s)
Figure 4
Predicted Relationships Between Applied Load and Fiber Volume Content for the Winding Speeds Used in the Experimental Investigation

- U = winding speed used to obtain predicted applied load curve
- U = 12.7 mm/s
- U = 31.8 mm/s
- U = 50.8 mm/s

fiber content (% by volume)
Figure 5
Comparison of Predicted and Measured Applied Loads for Experiments RS1 - RS5

- experimental data for wind.sp. = 12.7 mm/s
- experimental data for wind.sp. = 31.8 mm/s
- experimental data for wind.sp. = 50.8 mm/s

U=12.7mm/s
U=31.8mm/s
U=50.8mm/s

required applied load (N)

fiber content (% by volume)
Modeling and Testing of Thermoplastic Composite Filament Winding Consolidation Mechanisms

Final Report to National Institute of Standards and Technology

Contract Number: 60NANB1D1144

Program Manager: Richard Norcross
Building 220, Room B127

Jonathan Colton
Charles Carpenter

School of Mechanical Engineering
Georgia Institute of Technology
Atlanta, GA 30332-0405

June 15, 1992
SUMMARY

A science-based understanding of the on-line consolidation process in thermoplastic filament winding was developed. This was accomplished through a theoretical analysis and an experimental investigation. The theoretical analysis involved the development of a one-dimensional, semi-empirical, on-line consolidation process model that describes the deformation and flow phenomena that occur during the filament winding of thermoplastic composite towpregs using a single-roller, on-line consolidation mechanism. The model was used to predict the applied load necessary to achieve a desired fiber volume content of filament wound thermoplastic composite rings consolidated at various processing conditions. The model also was used to provide an estimate of the winding speed and applied load necessary to achieve a given degree of consolidation. The experimental investigation involved the fabrication, testing, and analysis of filament wound thermoplastic composite rings consolidated at various process conditions. The rings were fabricated using a single-roller, on-line consolidation mechanism. A "design of experiments" methodology was used to investigate the effects of winding speed and applied load on the void content, fiber content, and final thickness of the consolidated composite rings. The results of the experimental investigation were compared to the deformation model's predictions and the two were found to agree reasonably well. General guidelines for the application of the deformation model to material systems and on-line consolidation mechanisms not studied in this research were presented and discussed. In addition, general comments on the thermoplastic filament winding process with on-line consolidation were presented.
ACKNOWLEDGMENT

This work was funded in part the National Institute of Standards and Technology contract # 60NANB1D1144. Richard Norcross was the program manager. This funding and support is gratefully acknowledged.
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<table>
<thead>
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<th>Definition</th>
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<tbody>
<tr>
<td>a</td>
<td>weight of composite in air</td>
</tr>
<tr>
<td>Ac</td>
<td>contact area</td>
</tr>
<tr>
<td>Ar</td>
<td>area fraction of fibers</td>
</tr>
<tr>
<td>A_total</td>
<td>total image area</td>
</tr>
<tr>
<td>a0</td>
<td>initial height of rectangle</td>
</tr>
<tr>
<td>b0</td>
<td>initial width of rectangle</td>
</tr>
<tr>
<td>C</td>
<td>spring constant of fiber bed</td>
</tr>
<tr>
<td>Dau</td>
<td>degree of autohesion</td>
</tr>
<tr>
<td>df</td>
<td>fiber diameter</td>
</tr>
<tr>
<td>Dic</td>
<td>degree of intimate contact</td>
</tr>
<tr>
<td>d̄</td>
<td>mean diameter of wound ring specimen</td>
</tr>
<tr>
<td>k</td>
<td>temperature-dependent constant</td>
</tr>
<tr>
<td>K</td>
<td>Kozeny constant</td>
</tr>
<tr>
<td>L</td>
<td>length of nip region</td>
</tr>
<tr>
<td>L_l</td>
<td>longitudinal flow path length</td>
</tr>
<tr>
<td>L_t</td>
<td>transverse flow path length</td>
</tr>
<tr>
<td>n</td>
<td>power-law index</td>
</tr>
<tr>
<td>N</td>
<td>number of fibers within image area</td>
</tr>
<tr>
<td>P_appl</td>
<td>applied gauge pressure</td>
</tr>
<tr>
<td>P_appl</td>
<td>applied load</td>
</tr>
<tr>
<td>P_avg</td>
<td>average applied pressure</td>
</tr>
<tr>
<td>p_r(x)</td>
<td>fiber bed pressure at a x-position</td>
</tr>
<tr>
<td>p_m(x)</td>
<td>matrix pressure at a x-position</td>
</tr>
</tbody>
</table>
\[ q \] = fluid flow rate  
\[ q_l \] = longitudinal matrix flow rate  
\[ q_t \] = transverse matrix flow rate  
\[ q(x) \] = matrix flow rate at a x-position  
\[ R \] = radius of compaction roller  
\[ r_f \] = fiber radius  
\[ S \] = permeability  
\[ S_l \] = longitudinal permeability  
\[ S_t \] = transverse permeability  
\[ t \] = time  
\[ t_a \] = time elapsed from start of bonding process  
\[ t_L \] = final towpreg thickness  
\[ t_m \] = maximum local towpreg thickness  
\[ t_{\text{reduction}} \] = percent reduction of towpreg thickness  
\[ t_s \] = minimum local towpreg thickness  
\[ t(x) \] = towpreg thickness at a x-position  
\[ t_0 \] = initial towpreg thickness  
\[ t_1 \] = towpreg thickness measured with Method 1  
\[ t_2 \] = towpreg thickness measured with Method 2  
\[ U \] = winding speed  
\[ V_f(x) \] = fiber volume fraction at a x-position  
\[ V_L \] = final fiber volume fraction  
\[ V_v \] = volume fraction of voids  
\[ V_x \] = volume fraction of crystalline matrix
$V_0$ = initial fiber volume fraction of towpreg
$V_{\infty}$ = highest fiber volume fraction obtainable
$w$ = weight of composite in water
$w_{tow}$ = width of towpreg
$w_0$ = initial spacing between rectangle elements
$W_x$ = weight fraction of crystalline matrix
$x$ = position in nip in the lay-up direction
$\Delta H_{r,\text{max}}$ = heat of fusion for 100% crystalline polymer
$\Delta H_{r,\text{meas}}$ = heat of fusion from DSC analysis
$\Delta p$ = pressure drop
$\Delta p_m$ = matrix pressure drop
$\Delta t$ = average thickness variation
$\gamma$ = specific gravity of composite
$\dot{\gamma}$ = matrix shear rate
$\eta_a$ = apparent matrix viscosity
$\eta_0$ = zero shear rate matrix viscosity
$\lambda$ = time constant at which shear-thinning effects become important
$\mu$ = fluid viscosity
$\mu_{mf}$ = viscosity of fiber/matrix mixture
$\rho$ = density of composite
$\rho_a$ = density of amorphous matrix
$\rho_f$ = density of fiber
\( \rho_M \) = measured composite density

\( \rho_T \) = theoretical composite density

\( \rho_{\text{water}} \) = density of water

\( \rho_x \) = density of crystalline matrix

\( \omega \) = rotational velocity of mandrel
CHAPTER 1

INTRODUCTION

1.1 Background

The development of continuous fiber-reinforced thermoplastic matrix composites has provided substantial benefits to designers, engineers, and manufacturers. In general, these composites offer improved damage tolerance and repairability characteristics, increased design and manufacturing flexibility, improved material handling and storage characteristics, and in some cases increased mechanical performance when compared to thermoset matrix composites [1]. However, the widespread acceptance of thermoplastic matrix composites has been hindered by their relatively high manufacturing cost. This is due to a lack of well-defined design guidelines and a lack of high productivity manufacturing technology.

The development of manufacturing technology for the fabrication of thermoplastic matrix composites is an area of intense research. A science-based understanding of their behavior during manufacturing is essential for the development of processes that can efficiently produce quality thermoplastic composite structures. Unlike thermoset matrix composites which are cured (or cross-linked) to achieve their final state, thermoplastic
matrix composites exhibit melting behavior in their final state and therefore can be melted and reshaped (or repaired) without degradation of the matrix material. As thermoplastic composites only need to be melted and not cured to achieve their final shape, manufacturing techniques with much shorter processing times can be used to produce thermoplastic composite structures. This can lead to potential cost savings in manufacturing. However, thermoplastic composites require special manufacturing equipment and methods because their relatively high melting points and melt viscosities require the use of high processing temperatures and pressures. This drawback is magnified when continuous processing techniques, such as filament winding, are employed.

Thermoplastic filament winding is a continuous process in which a thermoplastic composite material (usually in the form of a tape) is wound onto a mandrel and consolidated on-line in order to achieve the desired shape. The continuous processing of thermoplastic composites with on-line consolidation can lead to potential cost savings in manufacturing because no secondary processing is needed to complete the consolidation process. Consolidation is a processing step in which heat and pressure are applied to the thermoplastic composite in order to force out any entrapped air and thereby compress and bond the composite laminate into a void-free, finished part. Continuous thermoplastic composite manufacturing processes require that full consolidation be achieved in a short period of time in order to produce a quality, cost-efficient, finished part. However, consolidation of thermoplastic composites is difficult to complete in such short process
times due to the relatively high melt viscosity and high specific heat of the thermoplastic matrix material.

The majority of thermoplastic composite processes involving on-line consolidation rely on manufacturing experience and "rules of thumb" to produce composite parts. This experience is gained from costly and time-consuming trial-and-error production and varies for different material systems and processes. To alleviate this deficiency, a complete science-based understanding of the on-line consolidation process is needed for the optimization and design of mechanisms that can achieve full consolidation in the short times required for efficient, economic, continuous processing.

1.2 Project Description

The goal of this research is to obtain a science-based understanding of the on-line consolidation process in thermoplastic filament winding. This is to be accomplished through a theoretical analysis and an experimental investigation. The theoretical analysis of the process will involve the development of an on-line consolidation process model that describes the deformation and flow phenomena that occur during the filament winding of thermoplastic composite towpregs using a single-roller, on-line consolidation mechanism. The model will be used to predict the applied load necessary to achieve a given fiber volume content of the wound laminate for various process conditions. The model's predictions will be compared to the results of the experimental investigation in order to assess the accuracy of the proposed deformation model for describing the
deformation and flow phenomena that occur during the on-line consolidation process. The model also will be used in conjunction with the results of the experimental investigation to provide an estimate of the process conditions required to achieve a given degree of consolidation.

The experimental investigation will involve the analysis of filament wound thermoplastic composite rings consolidated at various process conditions. The composite rings will be fabricated using a single-roller, on-line consolidation mechanism installed on a filament winding machine. The on-line consolidation mechanism consists of electric hot air guns for applying the consolidation heat and a single, pneumatically-controlled, compaction roller for applying the consolidation pressure. The "design of experiments" statistical analysis method will be used to investigate the effects of winding speed and applied load on the void content, fiber content, and final thickness of the consolidated composite rings.

1.3 Overview

Chapter Two describes thermoplastic filament winding and consolidation and provides a review of previous research conducted in the area of the on-line consolidation modeling of thermoplastic composites. The theoretical analysis is presented in Chapter Three and includes the development of the on-line consolidation deformation model. The experimental investigation is presented in Chapter Four and includes descriptions of the equipment, material, test procedures, and analysis techniques used in the experimental study. The theoretical and
experimental results are presented, compared, and discussed in Chapter Five. Conclusions are drawn from the results and recommendations are made for further research in Chapter Six.
CHAPTER 2

BACKGROUND

2.1 Introduction

A description of thermoplastic filament winding and a discussion of consolidation are presented in this chapter. In addition, previous research in the area of on-line consolidation modeling is presented.

2.2 Thermoplastic Filament Winding

Thermoplastic filament winding is a continuous manufacturing process in which continuous fiber-reinforced thermoplastic composite material is wound onto a mandrel and consolidated on-line thus building up the desired shape in a layer-by-layer fashion. The continuous processing of thermoplastic composites with on-line consolidation can lead to potential cost savings in manufacturing because no secondary processing, such as autoclave / bleeder ply molding, is needed to complete the consolidation process. The filament winding process is used primarily to fabricate axisymmetric composite structures, such as pressure vessels, pipes, and drive shafts. A typical thermoplastic filament winding set-up, as shown in Figure 2.1 [2], consists of a winding machine and carriage...
Figure 2.1: Typical Thermoplastic Filament Winding Machine [2]
with one or more winding axes, a creel system with tensioner, a towpreg delivery system, a mandrel, an on-line consolidation mechanism, and process monitoring and control equipment. The thermoplastic composite material used for filament winding is generally in the form of a tape (or towpreg) consisting of a fiber tow preimpregnated with the matrix material. This particular material form facilitates accurate towpreg placement and lay-up control because of its uniform width.

2.2.1 Process Parameters

As with most composites manufacturing processes, the process parameters are important in determining the quality of the finished product. The process parameters for thermoplastic filament winding can be categorized into five groups: material, geometry, consolidation pressure, consolidation temperature, and winding speed. The material parameters are the physical form of the towpreg material and the tooling material. The geometric parameters include the dimensions of the towpreg material, the dimensions of the tooling, the lay-up pattern, and the winding angle. The consolidation pressure includes the force supplied by external compaction devices in addition to the winding tension. The consolidation temperature parameters refer to the temperatures of the towpreg and the substrate at the point of consolidation. The winding speed is obtained from the rotational velocity of the mandrel, the mandrel geometry, and the lay-up pattern. The consolidation pressure, the consolidation temperature, and the winding speed have the greatest impact in the thermoplastic filament
winding process and are generally designated as the controllable (or independent) process variables.

2.2.2 On-Line Consolidation Mechanism

One of the most important components of a thermoplastic filament winding machine is the on-line consolidation mechanism. The on-line consolidation mechanism is used to apply heat and pressure to the point of contact between the incoming towpreg and the substrate. The mechanism must be able to achieve the consolidation pressures and temperatures required for complete consolidation to occur in the relatively short time the mechanism is in contact with the composite material. Various devices have been used to apply heat to the point of contact and include heated shoes, heated rollers, lasers, focused infrared heaters, exposed flame heaters, and flameless hot gas torches (or guns) [3]. Of these, the hot gas torches provide the most simple, cost-efficient, versatile, and controllable means of applying large amounts of heat to a localized area. Hot gas torches are convective heat sources that blow a stream of high temperature gas (usually air or nitrogen) onto the towpreg or substrate material in order to achieve melting of the matrix material. While hot gas torches do not have the power density capabilities of lasers or focused infrared heaters, they are more versatile and easier to control. With the convective heat transfer mode employed by hot gas torches, there is little risk of overheating or degrading the composite material. However, care must be taken when determining the gas flow rates through the torches to ensure that the
molten matrix does not get blown off the fibers in the towpreg when impinged by the hot gas stream.

Various devices also have been developed to apply the required consolidation pressure to the point of contact. These include compaction shoes, air-bearing devices, needle-scalers, conforming bands or tracks, multiple compaction rollers, and single compaction rollers [4]. Winding tension also has been used without the presence of an external compaction device to achieve the required consolidation pressure. Of these, the single compaction roller is the most widely-used device for achieving the required consolidation pressure. This is because of its simplicity, compactness, versatility, and ease of control. Single compaction rollers have the ability to apply pressure evenly over complex shapes with double-curvatures and sharp changes in geometry. The compaction roller brings the melted towpreg into intimate contact with the heated substrate and forces out any entrapped air as the towpreg passes underneath the roller (see Figure 2.2). With a single-roller, on-line consolidation mechanism, consolidation takes place in the nip (or the region of contact between the roller, the incoming towpreg, and the substrate).

2.3 Consolidation of Thermoplastic Composites

Consolidation is a processing step in which heat and pressure are applied to the thermoplastic composite in order to force out any entrapped air and thereby compress and bond the composite laminate into a void-free, finished part. Improper consolidation can lead to voids, residual
Figure 2.2: Typical Single-Roller Compaction Device for Applying Consolidation Pressure
stresses, warpage and, in some cases, premature mechanical failure of the composite part. There are several critical functions of the consolidation process that must be achieved in order to obtain a quality composite laminate with optimum properties [5]. The critical consolidation functions include complete bonding between the plies (or layers) of the composite laminate, prevention and removal of voids, attaining the desired fiber volume content in the part, attaining a uniform distribution of fibers and matrix within the part, attaining the desired dimensions of the part, and prevention of material degradation during the process.

2.3.1 Consolidation Mechanics

The consolidation process can be divided into three mechanisms [6,7]: bulk consolidation, matrix flow, and fiber network deformation. These components occur as the composite laminate thickness is decreased during the deformation (compaction) process (see Figure 2.3). The first stage in the consolidation process is bulk consolidation which describes the elimination of spatial gaps between the composite layers. This is accomplished through matrix flow along the surfaces of the composite layers and is completed when intimate contact is achieved. Once the layers of the composite are coalesced, diffusion bonding can occur across the interface. The second stage of consolidation is deformation due to matrix flow within each composite layer. This matrix flow is necessary to transport the voids away from the interface and is the result of matrix pressure gradients established during the compaction process. The majority of the load applied to deform and compact the composite laminate
Figure 2.3: Variation in Composite Thickness During Consolidation
is borne by the matrix during the first two stages of the consolidation process. The final stage of the consolidation process is elastic deformation of the fiber network. This occurs when the deformation is such that the matrix pressure rapidly decreases due to excess matrix flow. If this occurs, the elastic fiber network can be compressed to the point where it starts to take up a significant portion of the load. If the load were suddenly removed while the matrix was still molten, then the elastic deformation of the fiber network would be recovered thus resulting in an increase in the thickness of the composite laminate and possible reformation of voids. This elastic behavior is more significant in composites consisting of woven or multi-directional fiber arrangements.

2.3.2 On-Line Consolidation

The consolidation mechanisms described up to this point were developed for post-layup processes, such as compression molding. With such processes, consolidation occurs after the layers of composite material have been laid up in the desired arrangement. In order to describe consolidation in continuous processes, such as thermoplastic filament winding, the motion of the fibers and the matrix during processing must be considered. This involves describing the transfer of heat, mass, and momentum that occur during the on-line consolidation process.

Continuous thermoplastic composites manufacturing processes require that full consolidation be achieved in a short period of time in order to produce a quality, cost-efficient, finished part. However, consolidation of thermoplastic composites is difficult to complete in such short process
times. This is due to the relatively high melt viscosity and high specific heat of the thermoplastic matrix material. The elimination of the difficulty in achieving on-line consolidation of thermoplastic composites is the motivation for this research. The development of a science-based understanding of on-line consolidation in thermoplastic filament winding will allow for the optimization and design of on-line consolidation mechanisms that can achieve full consolidation in the short process times required for the efficient and continuous fabrication of quality thermoplastic composite parts.

2.4 Previous Work in On-Line Consolidation Modeling

Recently, thermoplastic composites processing research has been focused on on-line consolidation modeling. This is because the continuous processing of thermoplastic composites with on-line consolidation can lead to potential cost savings in manufacturing because no secondary processing is needed to complete the consolidation process. The basis of consolidation modeling is the prediction of the quality of the consolidated, finished part based upon the process and material parameters used to fabricate the part. This would allow for the determination of the optimum process parameters without the need for costly and time-consuming, trial-and-error experimentation. In order to obtain a robust consolidation model, the model must be developed from the fundamental analysis of the phenomena associated with consolidation in the process studied. However, it should be noted that the development of a consolidation model applicable
to all thermoplastic composites processing, both continuous and discontinuous, would be extremely difficult as the consolidation mechanisms and phenomena are strongly dependent on the manufacturing process. Of the continuous processes involving on-line consolidation, thermoplastic filament winding is the most studied. Presented in this section are brief overviews of on-line consolidation models for thermoplastic composites that currently exist, described in terms of the manufacturing process for which they were developed.

2.4.1 Calhoun [8]

Calhoun developed an empirical model of the on-line consolidation process in thermoplastic filament winding. The on-line consolidation device studied consisted of an infrared spot-heater and a hot nitrogen torch for applying the necessary heat to the consolidation point, and winding tension and a compaction roller for applying the required consolidation pressure. The model consisted of a mathematical function that was fit to the response data of a $3^5$ full factorial experimental design and allowed for the prediction of response using previously untried process settings. The model was used to obtain the optimum processing parameters for filament winding APC-2 towpreg and focused mainly on the heating mechanisms associated with the on-line consolidation process. However, as the consolidation model was based completely on empirical data, it only is applicable to the on-line consolidation mechanism and material used to obtain the response data and cannot be extended to other material systems or on-line consolidation mechanisms.
2.4.2 Roychowdhury [5]

Roychowdhury developed a semi-empirical, one-dimensional flow model for consolidation in thermoplastic filament winding. The particular winding process studied used winding tension and an infrared, nip point heater to achieve the on-line consolidation. The winding tension was used to apply the consolidation pressure and no external compaction devices were used. The model was developed to predict the distribution and time-varying nature of the fiber volume fraction and matrix pressure as a function of material and processing parameters in filament wound rings. The basis of the model is that radial matrix flow is the dominant deformation mechanism and governs the extent of consolidation. As the melted towpreg is wound onto the mandrel, the winding tension causes the molten matrix material to flow from the interface through the thickness of the towpreg and to the surface thus causing intimate contact and void elimination. However, with the on-line consolidation mechanism used, a nonuniform fiber volume fraction distribution occurred between the inner and outer layers of the composite part because the matrix flowed radially away from the mandrel surface. Roychowdhury suggested that, in order to derive a complete understanding of the on-line consolidation process in thermoplastic filament winding, the use of a single-roller, on-line consolidation mechanism should be investigated.

2.4.3 Springer, et al. [9,10]

Springer and co-workers developed a model to simulate the entire thermoplastic filament winding process. One of the subcomponents of the
model dealt with consolidation and bonding and was based upon a microscopic model developed by Dara and Loos [11]. Springer suggests that consolidation and bonding occur in two steps: intimate contact followed by autohesion. Intimate contact refers to the smoothing of the ply surfaces where the surface irregularities are assumed to be a series of rectangles. The degree of intimate contact is determined as a function of the deformation of these rectangles and can be expressed in terms of the applied pressure as

\[ D_{ic} = \frac{1}{(1 + w_0/b_0)} \left[ 1 + \frac{5p_{appl}}{\mu_{mf}} \left( 1 + \frac{w_0}{b_0} \right) \left( a_0 \right)^2 t \right]^{1/5} \]  

(2.1)

where \( t \) is the time, \( p_{appl} \) is the applied gauge pressure, \( \mu_{mf} \) is the viscosity of the fiber-matrix mixture, and \( w_0 \) is the initial spacing between the rectangle elements. The terms \( a_0 \) and \( b_0 \) refer to the initial height and width of each rectangle, respectively. The initial dimensions of the rectangle elements are measured from photomicrographs of the cross-section of an uncompacted ply. Once the rectangles have deformed such that the surfaces of the plies come into intimate contact, bonding between the interfaces starts by an autohesion process. The degree of autohesion is represented by the expression

\[ D_{au} = k t_a^{1/4} \]  

(2.2)
where $t_a$ is the time elapsed from the start of the bonding process and $k$ is a temperature-dependent constant. Consolidation is complete when both $D_{ic}$ and $D_{au}$ approach unity.

This consolidation model will be applied to a thermoplastic filament winding process employing a single-roller, hot gas torch, on-line consolidation mechanism. However, this particular on-line consolidation model does not take into account the deformation of the fiber network or the variations in the thickness of the composite plies (or layers) caused by the nonuniform distribution of fibers and matrix within each layer. The model also only considers matrix flow on the surface of the composite layers and does not account for matrix flow within each layer during the consolidation process. In thermoplastic filament winding, matrix flow within each layer is required to transport the voids trapped within the layer away from the consolidation interface, even after intimate contact has been achieved. In addition, as the model requires a difficult to measure fiber-matrix viscosity instead of a simple matrix viscosity, employing the effects of strain rate on the viscosity and subsequent deformation would be difficult.

2.4.4 Pipes, et al. [12,13,14]

Pipes and co-workers developed a process model for thermoplastic composite pultrusion. Thermoplastic pultrusion is a continuous manufacturing process, similar to filament winding, in which layers of preheated composite towpreg are pulled through a heated die and consolidated on-line (see Figure 2.4). The composite layers are compacted and brought into intimate contact as they pass through the die taper. The
Figure 2.4: Typical Thermoplastic Composite Pultrusion Process
entrapped air and voids between the layers and within the layers are removed in the tapered region. Once the compressed layers leave the tapered region, they pass through a cooled, constant cross-section region where the bonding process is completed and where solidification of the matrix takes place.

The one-dimensional process model developed by Pipes and co-workers predicts the matrix pressure and flow as well as the temperature within a thermoplastic composite during the pultrusion process. The governing equations used in the model were stated in general terms and include continuity of the fiber and matrix material, fiber volume fraction / thickness reduction relationships, matrix flow rate / pressure drop relationships, matrix viscosity / shear rate / temperature relationships, fiber deformation, and transient heat transfer and thermal expansion equations. It should be noted that the model takes into account the effects of shear rate and temperature on the matrix viscosity. Unlike post-layup consolidation processes, such as compression molding, which generally involve low shear rates and isothermal conditions, continuous manufacturing processes employing on-line consolidation, such as pultrusion and filament winding, produce relatively high shear rates and temperature gradients during the consolidation process.

While the pultrusion process model does not formally address the on-line consolidation process, the pressure and flow analysis used to develop the model are directly applicable to the analysis of the various stages of consolidation in the continuous processing of thermoplastic composites. The basic assumptions of the pressure and flow submodels are that the load
applied to the consolidation region is borne by the fibers and the matrix, that the primary deformation mechanisms are matrix flow parallel to the fiber direction and fiber bed compaction, and that the matrix and fiber bed pressures are functions of the fiber volume fraction. The deformation and flow processes that the towpreg undergoes during pultrusion are very similar to those experienced during filament winding with a single-roller, on-line consolidation mechanism. Much like the linear taper at the entrance to the pultrusion die, the curvature of the compaction roller on the filament winder is used to compact and deform the towpreg in order to achieve intimate contact and void elimination. The pressure and flow submodels developed by Pipes and co-workers, extended to a thermoplastic filament winding process with on-line consolidation, is the basis of this research and is presented in Chapter 3.

2.5 Summary

Thermoplastic filament winding is a continuous process in which continuous fiber-reinforced thermoplastic composite material is wound onto a mandrel and consolidated on-line thus building up the desired shape in a layer-by-layer fashion. Consolidation is a processing step in which heat and pressure are applied to the thermoplastic composite in order to force out any entrapped air and thereby compress and bond the composite laminate into a void-free, finished part. The consolidation process can be divided into three components: bulk consolidation, matrix flow, and fiber network deformation. Continuous manufacturing processes, such as
filament winding, require that consolidation be completed on-line in order to produce a quality, cost-efficient, finished part. In order for complete consolidation to occur in such short process times, the process parameters affecting consolidation must be determined such that both part quality and manufacturing efficiency are optimized. A robust on-line consolidation process model developed from fundamental principles would allow for the determination of these optimum process parameters without the need for costly and time-consuming, trial-and-error experimentation. Several on-line consolidation models have been developed. Of these, the model developed by Pipes, et al. [12,13,14] provides the most comprehensive and fundamental analysis of the deformation and flow processes that occur during the on-line consolidation process in thermoplastic pultrusion. The application and extension of this model to thermoplastic filament winding using a single-roller, on-line consolidation mechanism is the basis of this research.
CHAPTER 3

THEORY

3.1 Introduction

The major obstacle to the wide-spread usage of continuous processes for thermoplastic composites is the lack of a science-based understanding of the phenomena associated with on-line consolidation. The majority of processes involving on-line consolidation rely on manufacturing experience and "rules of thumb" to produce composite parts. This experience is gained from costly and time-consuming trial-and-error production and varies for different material systems. In order to overcome these obstacles, a robust, on-line consolidation process model is needed to determine the optimum process conditions required for the efficient and continuous production of thermoplastic composite parts.

Any consolidation model needs to take into account the mechanical, thermal, and chemical aspects of the process in order to predict adequately the overall performance and quality of the consolidated composite part. This involves determining the effects of the various process conditions on the uniformity, the void content, the degree of bonding, the final dimensions, and the fiber content of the consolidated part. The goal of this chapter is to develop a model that describes the mechanical phenomena
that occur during the on-line consolidation process. The thermal and chemical aspects of the on-line consolidation process will not be modeled in this study.

3.2 Proposed On-Line Consolidation Deformation Model

A one-dimensional model that describes the mechanical deformation and flow processes that occur during the filament winding of thermoplastic towpregs using a single roller, on-line consolidation mechanism is proposed. The objective of the deformation model is to predict the applied load necessary to achieve a given fiber content of the wound laminate for various process conditions and material systems. Ultimately, the model will be used in conjunction with experimental results to provide an estimate of the applied load and winding speed necessary to achieve a given degree of consolidation.

A flow diagram of the model is shown in Figure 3.1. Components of the model are derived from a consolidation model developed by Gutowski [15] and from a thermoplastic composite pultrusion model developed by Pipes and Åström [12,13,14]. The formulation of the proposed deformation model for thermoplastic filament winding with on-line consolidation is presented and discussed in this chapter.

3.2.1 Preliminary Assumptions

The basic assumption of the model is that the primary deformation mechanisms are matrix flow parallel to the fibers and fiber bed
**INPUTS**
- Process Conditions
- Material Properties
- Desired Fiber Volume Fraction of Wound Laminate
- Boundary Conditions

**SIMULATION**
- Towpreg Height Distribution
- Fiber Volume Fraction Distribution
- Matrix Pressure Distribution
- Fiber Bed Pressure Distribution

**OUTPUTS**
- Required Applied Load
- Final Laminate Thickness
  - Area of Contact
- Residence Time in Nip
- Average Applied Pressure

Figure 3.1: Flow Diagram of Deformation Model
compaction. These two mechanisms are assumed to occur during the formation of intimate contact as the towpreg passes through the nip region (the nip is the region of contact between the compaction roller and the incoming towpreg). The model also assumes that deformation due to consolidation only takes place in the towpreg being laid down and that the substrate is unaffected by the compaction process. Therefore, because each ring consists of consecutive layers of consolidated towpreg, the fiber content and thickness reduction of the deformed towpreg should represent that of the wound laminate. The preliminary assumptions for the deformation model are outlined below:

- The applied load is carried by the fibers and the matrix.
- Towpreg deformation is the result of matrix flow parallel to the fiber direction and fiber bed compaction.
- The matrix pressure and the fiber bed pressure are functions of the fiber volume fraction.
- Deformation only takes place in the towpreg and not in the roller or the substrate beneath the nip region.
- The fiber content and thickness reduction of the deformed towpreg is representative of the wound laminate.
- The fibers are continuous and well-aligned.
- No transverse matrix flow occurs.
- There is negligible change in towpreg width during consolidation.
- The nip region can be represented by a simplified lay-up geometry.
- The elastic recovery of the deformed towpreg is negligible.
- The matrix temperature is constant throughout the nip region.
- The process is quasisteady-state.
It should be noted that while many of the parameters presented in this chapter are tensors, the tensor indices will not appear because only the components corresponding to the towpreg lay-up direction will be used in the model.

### 3.2.2 Development of the Deformation Model

#### 3.2.2.1 Determination of Nip Length

The proposed deformation model requires that the length of the nip region be known in order to obtain the desired pressure distributions. The contact length between the compaction roller and the towpreg being laid down can be determined using geometric compatibility relationships. These relationships often are used in "cushion-nip" analyses of polymer calendering processes where deformation takes place in the feedstock and not in the compaction rollers [16,17].

The lay-up geometry shown in Figure 3.2 assumes that the diameter of the mandrel is much greater than that of the roller and that the towpreg enters the nip region at a direction perpendicular to the direction of applied load. The length of the nip region, \( L \), is obtained from the radius of the compaction roller and the reduction in towpreg thickness and can be written as

\[
L = \sqrt{(t_0 - t_L)[2R - (t_0 - t_L)]}
\]

where \( t_0 \) and \( t_L \) refer to the initial and final towpreg thicknesses, respectively, and \( R \) refers to the radius of the compaction roller.
R = radius of compaction roller
L = length of nip region
\( t_0 = t(x=0) \) = thickness of incoming towpreg
\( t_L = t(x=L) \) = thickness of deformed towpreg
\( V_0 = V_f(x=0) \) = initial fiber volume fraction
\( V_L = V_f(x=L) \) = final fiber volume fraction
\( x \) = direction of towpreg lay-up
\( y \) = direction of applied load

Figure 3.2: Lay-up Scheme Used for Pressure/Flow Analysis of Nip Region
In order to calculate the average applied pressure, the contact area is required. The contact area can be determined from the arc length corresponding to the portion of the roller that is in contact with the nip region. Fortunately, for small deformations such as those experienced in thermoplastic filament winding, the contact arc length is equivalent to the nip length. Thus, the contact area can be determined by multiplying the towpreg width by the nip length.

3.2.2.2 Fiber Volume Fraction Distribution

The basic assumption of the deformation model is that the matrix pressure and the fiber bed pressure are functions of the fiber volume fraction. Applying fiber continuity and assuming the towpreg is transversely isotropic, the fiber volume fraction at a position, \( x \), in the nip, \( V_f(x) \), can be obtained as

\[
\frac{V_f(x)}{V_L} = \frac{t_L}{t(x)}
\]

where \( t(x) \) is the thickness of the towpreg at a position, \( x \), within the nip. The terms \( t_L \) and \( V_L \) refer to the final towpreg thickness and fiber volume fraction, respectively.

Again, using the geometric compatibility between the compaction roller and the towpreg, the fiber volume fraction distribution in the nip may
be written in terms of the nip length as

$$V_l(x) = \frac{t_L V_L}{t_L + R - \sqrt{R^2 - (x - L)^2}}$$  \hspace{1cm} (3.3)

3.2.2.3 Determination of Matrix Pressure

As the towpreg passes through the nip region, large matrix pressure gradients are established along the length of the nip and across the width of the towpreg due to compaction by the roller. The matrix pressure gradients are essential for the formation of intimate contact and for the removal of voids because they induce matrix flow that fills in gaps and transports entrapped air out of the nip. Both longitudinal (parallel to the fibers) and transverse (perpendicular to the fibers) matrix flows are possible within the nip because pressure gradients exist in both directions.

An order-of-magnitude analysis determined the contributions of longitudinal flow and transverse flow to the overall matrix flow that occurs during the on-line consolidation process. A relative comparison of the matrix flow rates in each direction was obtained using a generalized form of Darcy's law (Equation 3.4) [18]. Darcy's law is a widely-used expression for describing viscous flow through a porous medium. Darcy's law states that the flow rate per unit area is directly proportional to the pressure gradient. This is written as

$$q = \frac{S \Delta p}{\mu L}$$  \hspace{1cm} (3.4)
where $q$ refers to the matrix flow rate, $S$ refers to the permeability, $\mu$ refers to the fluid viscosity, $\Delta p$ refers to the pressure drop, and $L$ refers to the flow path length. The constant of proportionality is $S/\mu$ and the pressure gradient is $\Delta p/L$.

It is presumed that the maximum matrix pressure occurs at the end of the nip at a position halfway across the width of the towpreg. In addition, it is presumed that the minimum matrix pressure occurs at the nip entrance and on the sides of the nip where the pressure goes to zero (or atmospheric). Therefore, as the maximum pressure drop is the same in both the longitudinal and the transverse directions and as the average nip length is approximately three times less than the half-width of the towpreg, the longitudinal matrix pressure gradient is approximately three times greater than the transverse matrix pressure gradient. The average nip length and towpreg width used in this analysis were obtained from the results of the experimental investigation presented in Chapter Five. In addition, the permeability of an aligned fiber bed is approximately ten times greater for flow parallel to the fibers as compared to flow perpendicular to the fibers [19]. Assuming a uniform matrix viscosity, the ratio of longitudinal flow to transverse flow can be estimated from the flow path lengths and permeabilities associated with both directions. This can be written as

$$\frac{q_l}{q_t} = \frac{S_l}{S_t} \frac{L_t}{L_l} = 30$$
where the subscript "l" refers to the longitudinal direction and the subscript "t" refers to the transverse direction. The comparative analysis indicates that the transverse matrix flow is approximately 30 times less than the longitudinal matrix flow for the process studied. Therefore, the proposed deformation model will consider only matrix flow parallel to the fibers along the length of the nip and will assume that the contributions of transverse matrix flow are negligible. It should be noted that although microscopic transverse matrix flow takes place on the surface of the towpreg due to the localized deformation of surface irregularities, the macroscopic transverse matrix flow within the towpreg is negligible when compared to the macroscopic longitudinal matrix flow.

As the matrix pressure is higher at the exit of the nip than at the nip entrance, a backflow of matrix material parallel to the aligned fibers will occur. The proposed matrix flow is shown in Figure 3.3. It should be noted that the molten matrix is assumed to be completely viscous with no elastic response. Researchers have indicated that the viscoelastic effects of the molten polymer matrix are negligible for consolidation processes in which matrix flow occurs parallel to the fiber direction [14].

3.2.2.4 Matrix Flow Rate

The matrix flow rate is obtained by applying continuity to the matrix material. As the matrix travels at the same velocity as the fibers at the exit to the nip, the matrix flow rate per unit area may be written in terms of the
Figure 3.3: Schematic of Matrix Flow in the Nip
winding speed as

\[ q(x) = \left( \frac{1 - V_L}{V_L} \right) U V_r(x) \]  

(3.5)

where \( U \) refers to the winding speed. The matrix flow that occurs during the bulk consolidation of the towpreg is included in Equation 3.5 because the surface contours are accounted for in the measurement of the initial towpreg thickness, \( t_0 \) (see Section 4.4.1). Bulk consolidation describes the elimination of spatial gaps between the surface of the towpreg and the compaction roller or substrate [6]. The spatial gaps are the result of irregularities and contours that exist along and across the surface of the towpreg. As the towpreg passes through the nip region, the surface peaks and asperities deform and flow until intimate contact is achieved. The deformation of these surface irregularities involves the flow of matrix as well as fibers in both the longitudinal and transverse directions. However, it is assumed that the dominant mechanism for achieving intimate contact within the nip is matrix flow parallel to the fibers. Whereas intimate contact may occur before the exit of the nip, further matrix flow is needed to transport entrapped voids back out the nip entrance.

3.2.2.5 Matrix Pressure Gradient

The matrix pressure gradient along the length of the nip can be related to the matrix flow rate using Darcy's law modified by the Kozeny-Carmen expression for permeability and by the Carreau model for shear-thinning fluids [14]. Darcy's law and the Kozeny-Carmen expression for
permeability provide an expression for the flow of Newtonian fluids through beds of spherical particles. In order to describe the flow of non-Newtonian fluids through aligned fiber beds, the hydraulic radius associated with spherical particles was replaced with that for aligned fiber beds. In addition, the Newtonian fluid viscosity was replaced with a viscosity model that describes the non-Newtonian behavior of the thermoplastic matrix studied. Unlike low shear rate consolidation processes such as compression molding, thermoplastic filament winding generates comparatively higher shear rates within the polymer matrix during the online consolidation process. A dynamic viscosity model that takes into account the effect of these higher shear rates on the matrix viscosity is essential for modeling matrix flow in this process. The Carreau dynamic viscosity model (Equation 3.7) was chosen over the widely used power-law model because the former accounts for the shear-thinning behavior of the thermoplastic polymer melt at intermediate shear rates. The relationship between the matrix flow rate and the matrix pressure drop can be written as

$$\frac{\Delta p_m}{L} = \frac{4K_0 q(x)}{r_f^2} \frac{V_f(x)^2}{[1 - V_f(x)]^3}$$

(3.6)

where $r_f$ refers to the radius of the fibers. The symbol, $\eta_0$, refers to the zero-shear-rate viscosity and the time constant, $\lambda$, refers to the shear rate at

$$\eta_a = \eta_0 (1 + (\lambda \dot{\gamma})^\eta)^{n-1}$$

(3.7)
which shear-thinning effects become important. Both $\eta_0$ and $\lambda$ depend on the process temperature. The power-law index, $n$, refers to the degree of shear thinning. These three viscosity model constants can be determined from viscosity data obtained from simple flow experiments. The term $\dot{\gamma}$ refers to the average shear rate and is obtained from Newton's friction law for average conditions at the walls of a flow capillary [14]. The term $K$ refers to the Kozeny constant, which is an indicator of the permeability of the fiber bed. The Kozeny constant is determined empirically and depends on the process conditions, the matrix pressure gradient, the fluid and fiber materials, the fiber arrangement, and the fiber volume fraction among others. For the proposed one-dimensional deformation model, the Kozeny constant associated with flow parallel to the fiber direction will be used.

It should be noted that the largest pressure gradient actually occurs at the exit of the nip where the matrix pressure goes from maximum to minimum over a very short distance. However, it is assumed that the matrix material solidifies upon exiting the nip region and thereby prevents matrix flow out of the exit of the nip.

3.2.2.6 Matrix Pressure Distribution

The matrix pressure distribution is obtained by substituting Equations 3.5 and 3.7 into Equation 3.6 and then integrating the resulting expression with respect to position in the nip. It should be noted that the matrix pressure is assumed to return to zero (or atmospheric) immediately after the towpreg exits the nip.
3.2.2.7 Fiber Bed Pressure Distribution

The fiber bed pressure distribution is obtained from Gutowski [20] and can be written as

$$p_f(x) = \frac{C}{\sqrt{\frac{V_f(x)}{V_0}} - 1} \left( \sqrt{\frac{V_\infty}{V_f(x)}} - 1 \right)^4$$

(3.8)

where C is a spring constant for the fiber bed and \(V_\infty\) is the highest fiber volume fraction obtainable. The term \(V_0\) refers to the initial (or undeformed) fiber volume fraction of the towpreg. The value of \(V_0\) cannot be obtained directly from the reported weight fractions and densities of the constituent materials because internal voids and external surface irregularities exist in the towpreg material. Therefore, Equation 3.2 was used to obtain an estimate for \(V_0\) based upon the fiber volume fraction of the deformed towpreg and the initial and final towpreg thicknesses.

Gutowski suggests that the fiber bed acts as a deformable, porous, elastic network. As the fibers are compacted, the fiber bed stiffens and creates resistance to matrix flow across and along the fibers. The load-bearing contribution of the fiber bed increases as the fiber volume fraction approaches \(V_\infty\).

3.2.2.8 Predicted Applied Load

The applied load necessary to achieve a given final laminate thickness or final fiber volume fraction can be obtained by integrating both
the matrix and fiber pressure distributions over the length of the nip and multiplying this by the width of the towpreg. This can be expressed as

\[ P_{\text{appl}} = w \int_0^L [p_m(x) + p_f(x)] \, dx \]  

(3.9)

where \( P_{\text{appl}} \) refers to the predicted applied load and \( w \) refers to the width of the towpreg.

### 3.2.3 Deformation Model: Method of Solution

An Excel™ spreadsheet was developed to evaluate the deformation model at various process conditions (Figure 3.4). The parameters, simulation, and verification procedures associated with the deformation model are presented in this section.

#### 3.2.3.1 Model Input and Output Parameters

Inputs to the deformation model include the tool geometry, the material constants, the winding speed, the thickness of the undeformed towpreg, and the desired fiber volume fraction of the deformed towpreg. Outputs include the required applied load, the load taken by the matrix, the load taken by the fiber bed, the nip length, the contact area, the average applied pressure, and the residence time in the nip. The residence time in the nip is simply the nip length divided by the winding speed. The residence time is important for determining the time the composite laminate is under consolidation pressure. It should be noted that the
<table>
<thead>
<tr>
<th>INPUTS:</th>
<th>OUTPUTS:</th>
<th>Nip Position x-position (m)</th>
<th>FiberVolFrac Vf(x)</th>
<th>PressGradient dP/dx (Pa/m)</th>
<th>MatrixPress Pm(x) (Pa)</th>
<th>FiberBedPress Pf(x) (Pa)</th>
<th>MatrixPress Pm(x) (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Roll radius (m) =</td>
<td>Initial Vf</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Initial Towpreg Thickness (m) =</td>
<td>Final Towpreg Thickness (m) =</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Desired</td>
<td>Thinning Vf</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Final Vf =</td>
<td>Winding</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Speed (m/s) =</td>
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<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Zero-Shear Viscosity (Pa sec) =</td>
<td>Fiber Contact Area (m²) =</td>
<td></td>
<td></td>
<td></td>
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<td></td>
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<tr>
<td>Time</td>
<td></td>
<td></td>
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<td></td>
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<td></td>
</tr>
<tr>
<td>Constant (s) =</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
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</tr>
<tr>
<td>Viscosity</td>
<td>Exponent</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Exponent =</td>
<td>Fiber Spring</td>
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<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Constant (Pa) =</td>
<td>Obtainable Vf</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Highest</td>
<td>Obtaining Fiber Vf</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td>Tow width (m) =</td>
<td>Regress Consts matrix fiber</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>c0=c0</td>
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<tr>
<td></td>
<td>c1=c1</td>
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<tr>
<td></td>
<td>c2=c2</td>
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<td></td>
<td>c3=c3</td>
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<td></td>
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<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>c4=c4</td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
</tr>
<tr>
<td></td>
<td>c5=c5</td>
<td></td>
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<td></td>
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</tr>
</tbody>
</table>

Figure 3.4: Deformation Model Spreadsheet
thickness of the deformed towpreg also is an output because it can be used to predict the final thickness of a laminate consisting of layers of consolidated towpreg simply by multiplying the number of layers by the predicted thickness of the deformed towpreg. A complete listing of the model parameters is presented in Table 3.1.

3.2.3.2 Empirically Determined Model Parameters

The deformation model also contains several empirically determined parameters. These consist of the final thickness of the deformed towpreg, the Kozeny constant, and the void content. These parameters depend on the actual inputs to the model and to one another and are obtained from the correlation relationships created by the regression analysis of empirical data. It should be noted that the parameter values obtained from the correlation equations are applicable only to the materials and process conditions used to create the empirical database.

3.2.3.3 Model Simulation

The program simulates the fiber volume fraction, fiber bed pressure, matrix pressure gradient, and matrix pressure at positions along the length of the nip. The matrix pressure gradient distribution is obtained by plotting Equation 3.6 with respect to position in the nip and fitting the resulting curve with a fifth-order polynomial using CricketGraph™. A fifth-order curve fit provides a coefficient of determination or "$R^2$" equal to unity. The matrix pressure gradient distribution then can be expressed in
Table 3.1: On-Line Consolidation Deformation Model Parameters

<table>
<thead>
<tr>
<th><strong>INPUT PARAMETERS</strong></th>
<th>Symbol</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Tool Geometry</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>• Radius of compaction roller</td>
<td>$R$</td>
<td>m</td>
</tr>
<tr>
<td><strong>Process Conditions</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>• Winding speed (or lay-up rate)</td>
<td>$U$</td>
<td>m/s</td>
</tr>
<tr>
<td><strong>Material Parameters</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>• Desired fiber volume fraction of wound laminate</td>
<td>$V_L$</td>
<td>dim.less</td>
</tr>
<tr>
<td>• Initial thickness of towpreg</td>
<td>$t_0$</td>
<td>m</td>
</tr>
<tr>
<td>• Width of towpreg</td>
<td>$w_{tow}$</td>
<td>m</td>
</tr>
<tr>
<td>• Zero-shear rate matrix viscosity</td>
<td>$\eta_0$</td>
<td>Pa•s</td>
</tr>
<tr>
<td>• Time constant at which shear-thinning effects become important</td>
<td>$\lambda$</td>
<td>s</td>
</tr>
<tr>
<td>• Viscosity model exponent</td>
<td>$n$</td>
<td>dim.less</td>
</tr>
<tr>
<td>• Fiber spring constant</td>
<td>$C$</td>
<td>Pa</td>
</tr>
<tr>
<td>• Highest fiber volume fraction obtainable</td>
<td>$V_\infty$</td>
<td>dim.less</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th><strong>OUTPUT PARAMETERS</strong></th>
<th>Symbol</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>• Required applied load</td>
<td>$P_{appl}$</td>
<td>N</td>
</tr>
<tr>
<td>• Load carried by the matrix</td>
<td>$P_m$</td>
<td>N</td>
</tr>
<tr>
<td>• Load carried by the fiber bed</td>
<td>$P_f$</td>
<td>N</td>
</tr>
<tr>
<td>• Length of nip region</td>
<td>$L$</td>
<td>m</td>
</tr>
<tr>
<td>• Area of contact between the roller and the towpreg</td>
<td>$A_c$</td>
<td>m²</td>
</tr>
<tr>
<td>• Residence time in the nip</td>
<td>$t_{nip}$</td>
<td>s</td>
</tr>
<tr>
<td>• Average applied pressure</td>
<td>$p_{avg}$</td>
<td>MPa</td>
</tr>
<tr>
<td>• Initial fiber volume fraction of the undeformed towpreg</td>
<td>$V_0$</td>
<td>dim.less</td>
</tr>
</tbody>
</table>

**Parameters Obtained from Empirical Database**

<table>
<thead>
<tr>
<th></th>
<th>Symbol</th>
<th>Units</th>
</tr>
</thead>
<tbody>
<tr>
<td>• Thickness of the deformed towpreg</td>
<td>$t_L$</td>
<td>m</td>
</tr>
<tr>
<td>• Void content</td>
<td>$V_v$</td>
<td>volume %</td>
</tr>
<tr>
<td>• Kozeny constant</td>
<td>$K$</td>
<td>dim.less</td>
</tr>
</tbody>
</table>
terms of the constants of regression as

$$\frac{dp_m(x)}{dx} = c_0 + c_1x + c_2x^2 + c_3x^3 + c_4x^4 + c_5x^5$$  \hspace{1cm} (3.10)

where the term, \(x\), refers to the position in the nip relative to the nip entrance. Using the constants of regression obtained from the curve-fit, the matrix pressure distribution can be determined simply by integrating Equation 3.10 with respect to position in the nip. The matrix pressure distribution can be expressed as

$$p_m(x) = c_0x + \frac{c_1}{2}x^2 + \frac{c_2}{3}x^3 + \frac{c_3}{4}x^4 + \frac{c_4}{5}x^5 + \frac{c_5}{6}x^6$$  \hspace{1cm} (3.11)

where \(p_m(x)\) refers to the matrix pressure at a position \(x\) within the nip. The fiber bed pressure distribution was obtained in a similar manner using the constants of regression from a fifth-order polynomial curve-fit of the fiber bed pressure data. The predicted applied load necessary to achieve a given final fiber content then can be determined from the matrix and fiber pressure distributions using Equation 3.9.

3.2.3.4 Model Verification

The adequacy of the proposed on-line consolidation deformation model for describing the mechanical phenomena that occur during the on-line consolidation process should be determined for each material system and manufacturing process studied. The first step in verifying the proposed deformation model is to obtain experimental data from filament
wound composite ring specimens. Regression analyses must then be performed on the experimental data in order to obtain the empirical model parameters. The materials and parameters used in the experiments are then input to the model's spreadsheet. Verification of the deformation model involves comparing the theoretical predictions for the applied load to the actual (measured) applied load required to achieve the final fiber volume content determined for each experiment.

3.2.4 Model Application

The proposed deformation model was used to predict the required applied load for winding speeds between 12.7 and 50.8 mm/sec and fiber volume contents of 55, 58, and 61 percent. The material, equipment, and process parameters used in the experimental investigation presented in Chapter 4 served as inputs for the model. The material constants for APC-2 (PEEK and AS4 carbon fibers) at a temperature of 400°C [12] were used in the simulation and are listed in the spreadsheets contained in Appendix A. The empirically determined model parameters were obtained from experimental data and are derived in Chapter Five.

The results of the simulation for the filament winding of 6 mm wide APC-2 towpreg using a single roller on-line consolidation mechanism are presented in Figure 3.5. The figure shows a predicted decrease in the required applied load with increasing winding speed for a constant fiber volume content. This is due to the shear-thinning behavior of the molten PEEK polymer matrix. The increase in shear rate with winding speed results in a decrease in the matrix viscosity. The lower viscosity reduces
Figure 3.5: Predicted Applied Load Versus Winding Speed for Various Fiber Volume Contents

Vf = fiber volume content used to obtain predicted applied load curve.
the viscous resistance of the matrix material and thus reduces the load required to compact the towpreg to the given fiber volume fraction. Figure 3.5 also shows a predicted increase in the required applied load with increasing fiber volume content for a constant winding speed. This is because more load must be applied to the towpreg in order to achieve the increased deformation corresponding to the higher fiber volume content. In addition, the applied load appears to approach some minimum value as the winding speed is increased for each of the fiber volume content profiles. One explanation for this is that there is a minimum load that must be applied to the compaction roller in order to maintain contact with the towpreg, no matter what winding speed is used.

3.3 Summary

A one-dimensional model describing the mechanical deformation processes that occur during the filament winding of thermoplastic towpregs using a single roller, on-line consolidation mechanism was developed. The basis for the model is that the primary deformation mechanisms are matrix flow parallel to the fibers and fiber bed compaction. The on-line consolidation deformation model predicts the applied load necessary to achieve a given fiber volume fraction of the wound laminate for various winding speeds, process conditions, and material systems. A spreadsheet was developed to evaluate the deformation model for various inputs. The model will be verified using the results of the experimental investigation.
CHAPTER 4

EXPERIMENTAL PROCEDURE

4.1 Introduction

An experimental investigation of on-line consolidation in thermoplastic composite filament winding is presented. A $2^2$ factorial design was used to investigate the effects of winding speed and applied load on the void content, fiber content, and final thickness of consolidated composite ring specimens. A single-roller, on-line consolidation mechanism was constructed and installed on a filament winding machine. Composite rings were fabricated at various process conditions and prepared for testing. The rings were measured and tested for thickness, fiber content, density, crystallinity, and void content. Descriptions of the experimental set-up, the material, the procedures used to fabricate and test the ring specimens, and the experimental design are presented in this chapter.

4.2 Experimental Set-up

The experimental set-up consisted of a filament winding machine outfitted with a creel system, a delivery system, a mandrel, an on-line
consolidation mechanism, power and air controllers, and a process monitoring system (Figure 4.1). This section describes in detail the equipment used in the experiments.

4.2.1 Filament Winding Machine

A two-axis McClean-Anderson filament winding machine (Model W-1) was used for the experiments (Figure 4.2). The basic components of the winding machine were an electric motor, a variable hydraulic transmission for changing the winding speed, a mechanically-driven carriage with variable crosshead speed, and an adjustable mandrel bed. As only rings were fabricated, the carriage was placed in neutral so that the crosshead would not traverse back and forth. A position lock was attached to the carriage to ensure that the proper crosshead location would be maintained during the fabrication of each ring. A rotary counter was installed on the drive spindle to determine the number of layers wound.

4.2.2 Creel and Delivery Systems

The creel system consisted of a let-off wheel assembly, a friction brake for applying tension, and a spool containing the towpreg material. The delivery system consisted of free-spinning square groove rollers that guided the towpreg from the creel to the on-line consolidation mechanism. Nylon guide rollers with a groove width of 10 mm directed the 6 mm wide towpreg from the creel to the towpreg placement roller. The extra-wide grooves accommodated any misalignment that occurred as the towpreg was unwound from the spool. A grooved metal guide roller consisting of a
Figure 4.1: Experimental Set-up
Figure 4.2: Filament Winding Set-up
6.5 mm wide steel ball bearing with steel flanges was placed at the entrance to the on-line consolidation mechanism to ensure accurate towpreg placement underneath the compaction roller and onto the mandrel. The bearing in the towpreg placement roller was lubricated with Molycoat high temperature lubricant to protect it from the heat generated by the on-line consolidation mechanism.

4.2.3 Mandrel

The mandrel consisted of a collapsible aluminum cylinder filled with glass wool insulation and sealed on the ends with Kapton polyimide film (Figure 4.3). The aluminum mandrel had a diameter of 178 mm, a length of 305 mm, and a wall-thickness of 12.7 mm. Aluminum ends fastened to the mandrel accommodated the support spindles and drive pin of the filament winder.

4.2.4 Thermoplastic On-Line Consolidation Mechanism

The development of the experimental apparatus also involved the design of an on-line consolidation device that could simultaneously apply heat and pressure to the nip over a range of winding speeds (Figure 4.4). The equipment and methods used to apply the heat and pressure required for on-line consolidation are presented in this section.

4.2.4.1 Heat Application

The heat in the nip was produced by two electrical resistance hot air guns fed by regulated air and open-loop voltage controllers. A GTE-
Figure 4.3: Schematic of Collapsible Mandrel
Figure 4.4: On-Line Consolidation Mechanism

- Diaphragm Air Cylinder
- Hot Air Guns
- Swing Arm
- Compaction Roller
Sylvania Series III, 4,000 watt, hot air gun was used to heat the incoming towpreg. A GTE-Sylvania Series I, 2,000 watt, hot air gun equipped with a deflection nozzle was used to heat the surface of the substrate (preprocessed layers) beneath the nip. The compactness and versatility of the GTE-Sylvania hot air guns allowed them to be placed in close proximity to the nip entrance.

The amount of heat applied to the incoming towpreg and substrate was controlled by adjusting the nozzle exit temperatures and air flow rates of the hot air guns. The voltage supplied to the heating elements in the hot air guns was controlled with phase-controlled power modules (GTE-Sylvania #057081). The nozzle exit temperatures were set by adjusting the applied voltage and were monitored with K-type high temperature thermocouples positioned in the center of the air stream at each nozzle exit. Air flow through the guns was controlled with air pressure regulators (Arrow Type 1612-1L) and filtered with two in-line filters (Fulflo Filter Type 33). The air flow rate was set by adjusting the supply air pressure for each hot air gun. The supply air pressure refers to the pressure indicated on the regulator placed before a flow restrictor contained in the inlet to each hot air gun. The flow restrictor was used to reduce the inlet air pressure to within the relatively low pressure range recommended by the manufacturer. With the flow restrictor in place, higher supply pressures can be used thus reducing the sensitivity of the air flow rate to changes in the supply pressure.
4.2.4.2 Pressure Application

Pressure within the nip was generated by a single compaction roller maintained at constant applied load using a diaphragm air cylinder (Bellofram Super Cylinder Type 4-F) and a servo-controlled regulator (Bellofram Type 10). The air cylinder was attached to a custom-made aluminum swing arm that housed a clevis-mounted compaction roller. Both the air cylinder and the swing arm were mounted to the carriage of the filament winder. A purge valve was placed between the air cylinder and the regulator to allow the swing arm to be lifted up during the set-up and removal of the wound specimens. The compaction roller consisted of a fully-complimented, steel ball-bearing with a diameter of 28.6 mm and a width of 9.5 mm. The roller was actively cooled with compressed air and contained Molycoat high-temperature lubricant to protect it from the heat applied to the nip region. The amount of force delivered to the compaction roller was controlled by adjusting the cylinder air pressure.

4.2.5 Process Monitoring

Temperature data supplied by the thermocouples placed at the exits of the hot air guns were monitored "real time" with an Apple Macintosh II running Workbench™ I/O software (Strawberry Tree Inc. Version 3.0.3). The software provided temperature readouts and profiles of both hot air guns and was programmed to trigger an alarm if the nozzle exit temperatures exceeded a specified maximum (i.e., if a sudden disruption in air flow or applied voltage occurred). The software also was used to
monitor the temperature of the mandrel during the mandrel preheat stage via a thermocouple attached to the mandrel surface.

4.3 Calibration of Experimental Equipment

In order to perform an accurate experimental investigation of the online consolidation process, the actual process temperatures, applied pressure, and winding speed must be known. This section describes the measurement techniques and calibration methods used for each critical process condition.

4.3.1 Applied Heat

The actual towpreg and substrate temperatures were determined at each winding speed using Omega temperature indicating paint. Thin coatings of the paint were applied to the top and bottom surfaces of the incoming towpreg and to the top surface of the substrate. The air flow and voltage supplied to the hot air guns were adjusted until the paint changed color. The temperature indicating paints corresponding to the desired incoming towpreg and substrate process temperatures were used during the calibration. The air pressure settings and the nozzle exit temperatures necessary to transform the paints at each winding speed were recorded and used to set the heat inputs for each experiment.
4.3.2 Applied Load and Applied Pressure

The amount of force delivered to the compaction roller was controlled by adjusting the pressure in the diaphragm air cylinder. A compression scale was used to measure the load applied to the compaction roller for various cylinder air pressures. The scale was mounted to the filament winding machine in place of the mandrel and positioned directly underneath the compaction roller. The cylinder air pressure and corresponding applied load data were recorded and used to obtain a correlation equation. The correlation equation was used to set the cylinder air pressure for the applied load specified in each experiment.

An average applied pressure, $P_{\text{avg}}$, can be obtained from the applied load and the contact area using the simple relationship

$$P_{\text{avg}} = \frac{P_{\text{appl}}}{A_c}$$  \hspace{1cm} (4.1)

where $P_{\text{appl}}$ is the applied load and $A_c$ is the contact area. The contact area is determined from the width of the towpreg and the nip length and can be expressed as

$$A_c = w_{\text{tow}} \cdot L$$  \hspace{1cm} (4.2)

where $w_{\text{tow}}$ is the nominal towpreg width and $L$ is the nip length (i.e., the length of contact between the compaction roller and the towpreg). The latter is obtained from the geometric compatibility relationships that were discussed in detail in Section 3.2.2.1. It should be noted that the contact
length is a function of the total amount of deformation (or reduction in thickness) that the towpreg undergoes as it passes underneath the roller. Therefore, the applied pressure is not an independent or controllable process parameter.

4.3.3 Winding Speed

The winding speed (lay-up rate), $U$, can be determined from the relationship

$$U = \pi \bar{d} \omega$$  \hspace{1cm} (4.3)

where $\bar{d}$ refers to the mean diameter of the wound specimens and $\omega$ refers to the measured rotational velocity of the mandrel. The mean diameter of the wound specimens is used to calculate the winding speed because the diameter increases as the towpreg is wound onto the mandrel. The error in calculating the winding speed with an average diameter is negligible because the change in diameter is small when compared to the overall ring diameter. The rotational velocity of the mandrel is obtained by counting the number of revolutions that occur over a set amount of time. The winding speed (or lay-up rate) was set by adjusting the hydraulic transmission in the filament winder until the rotational velocity of the mandrel matched that necessary to achieve the winding speed specified for each experiment.
4.4 Material Description and Characterization

The composite material studied was 6 mm wide APC-2 preimpregnated tow (towpreg) supplied by ICI Fiberite. The thermoplastic matrix is poly (etheretherketone) (PEEK) polymer and the reinforcement is AS-4 PAN-based continuous carbon fibers manufactured by Hercules. The towpreg is fabricated by melt impregnating a single 12 K tow of carbon fibers (i.e., a collection of approximately 12,000 continuous carbon filaments) with the PEEK matrix. This particular form of APC-2 is recommended by the manufacturer for filament winding because its uniform width facilitates accurate towpreg placement and lay-up control. A list of several general specifications of the towpreg is presented in Table 4.1 [21].

Table 4.1: General APC-2 Towpreg Specifications

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Constituent Materials</td>
<td>PEEK matrix / AS-4 carbon fibers</td>
</tr>
<tr>
<td>Material Form</td>
<td>melt impregnated 12K tow</td>
</tr>
<tr>
<td>Process Temperature</td>
<td>400°C</td>
</tr>
<tr>
<td>Fiber Content</td>
<td>62.1 percent by weight</td>
</tr>
<tr>
<td>Width</td>
<td>6 mm</td>
</tr>
<tr>
<td>Average Thickness</td>
<td>0.237 mm</td>
</tr>
</tbody>
</table>

The 400°C processing temperature is recommended by the manufacturer for filament winding APC-2 towpreg. The towpreg width of 6 mm was reported by the manufacturer and was verified by measurements.
The fiber content of 62.1 percent by weight also was reported by the manufacturer and was obtained from the lineal weight of the composite towpreg and the lineal weight of the fiber tow. The reported fiber content is slightly lower than usually reported for APC-2 towpreg indicating the presence of a slight amount of excess matrix within the towpreg. The initial thickness of the towpreg was measured using techniques described in the following section.

4.4.1 Initial Towpreg Thickness

An accurate initial towpreg thickness is important in predicting the thickness of a consolidated composite laminate. Unfortunately the towpreg studied had considerable surface contour variations. In an attempt to compensate for these surface irregularities, the towpreg thickness was measured using two distinctly different methods. Method 1, discussed below, involves the measurement of towpreg thickness using a conventional flat anvil micrometer. Method 2, also discussed below, involves the use of optical microscopy techniques to determines towpreg thickness [22].

In Method 1, a conventional hand held, flat anvil, digital micrometer (Starrett No. 216) was used to measure the towpreg thickness, $t_1$. Incoming towpreg thickness measurements were taken at the beginning and end of each composite ring fabricated. A total of 30 measurements were taken and are presented in Section 5.3.2.

In Method 2, thickness measurements were acquired from polished cross sections of the towpreg using an optical microscope (Zeiss Microscope Model #A, camera model #MC-100) and image analysis software (Micro-
Concepts Version 2.32). The towpreg cross section specimens were cast in epoxy to facilitate the polishing. A total of three cross sections were polished to a 1 micron surface finish and examined at a magnification of 100X. Each towpreg specimen required seven image screens to account for the entire towpreg width with each image screen split into five grids. A local maximum thickness, $t_m$, was obtained by measuring the distance between the peaks on one surface to the peaks on the opposing surface within each grid. A local minimum thickness, $t_s$, was obtained by measuring the distance between the valleys on one surface to the valleys on the opposing surface within each grid. An average thickness, $t_2$, was obtained for each grid using Equation 4.4. In addition, an average variation, $\Delta t$, was obtained using Equation 4.5.

$$t_2 = \frac{(t_m + t_s)}{2}$$ \hspace{1cm} (4.4)

$$\Delta t = t_m - t_s$$ \hspace{1cm} (4.5)

Averages of the $t_2$'s and $\Delta t$'s obtained from each grid are presented in Section 5.3.2.

### 4.5 Characterization of On-line Consolidation Process Parameters

The three main process parameters in the on-line consolidation of thermoplastic composites are the process temperature, the applied load (or pressure), and the winding speed (or lay-up rate). For this experimental investigation, the process temperatures of the incoming towpreg and substrate were held constant and the applied load and winding speed were
varied. The applied heat was varied to obtain the same process temperatures for the various winding speeds. The 4,000 watt, hot air gun was used to heat the incoming towpreg to the recommended processing temperature of 400°C. The 2,000 watt, hot air gun equipped with the deflection nozzle was used to heat the substrate to 300°C. This substrate temperature was chosen because it promotes diffusion bonding without significantly altering the substrate (i.e., relatively small heat-affected zone).

The ranges for the applied load and the winding speed were determined from preliminary experiments in which rings were fabricated at various loads and speeds and then qualitatively tested for degree of consolidation using an acoustic response technique. This technique involves striking the rings with a metal rod and listening for the quality of the sound produced. A "bell-like" sound generally indicates a consolidated part.

4.6 Experimental Design

For this investigation, the process temperatures were held constant and the winding speed and applied load were varied. A "design of experiments" method with analysis of variance (ANOVA) was used to study the effects of each independent process parameter [23]. A $2^2$ factorial design was used. This type of experimental design analyzes the main effects of the process parameter, as well as the interactions between them. It should be noted that the applied pressure generally is reported as an independent process variable in the consolidation of thermoplastic
composites. This is because, for many composites processes, the contact area is unaffected by the process itself and therefore the average applied pressure can be controlled independent of any process parameters simply by varying the applied load. However, for this particular process, the contact area is dependent on the total amount of deformation (or reduction in thickness) that the towpreg undergoes during the consolidation process because the contact area is calculated from the nip length. Therefore, the applied load was designated as the independent (or controllable) process variable instead of the applied pressure.

A total of five experimental sets were performed (Table 4.2). The first four sets constituted the $2^2$ factorial design and can be represented geometrically as the corners of a square (Figure 4.5). The last set corresponds to the center of the square and represented the ring specimens run at an intermediate winding speed and applied load. The results from this particular experiment will be used to aid in the verification of the proposed deformation model and will not be contained in the statistical analysis because they are not part of the full factorial design. Three ring specimens were fabricated at each of the five experiment points for a total of 15 rings. Each ring consisted of 50 layers of towpreg with an inside diameter of 178 mm.
Figure 4.5: The $2^2$ Factorial Design
Table 4.2: Experimental Design

<table>
<thead>
<tr>
<th>Experiment (i=1,2,3)</th>
<th>Winding Speed</th>
<th>Applied Load</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>(mm/sec)</td>
<td>(in/sec)</td>
</tr>
<tr>
<td>RS1-i</td>
<td>12.7</td>
<td>0.5</td>
</tr>
<tr>
<td>RS2-i</td>
<td>12.7</td>
<td>0.5</td>
</tr>
<tr>
<td>RS3-i</td>
<td>50.8</td>
<td>2.0</td>
</tr>
<tr>
<td>RS4-i</td>
<td>50.8</td>
<td>2.0</td>
</tr>
<tr>
<td>RS5-i</td>
<td>31.8</td>
<td>1.25</td>
</tr>
</tbody>
</table>

The various parameters were separated into three categories: independent (or controllable) parameters; dependent parameters; and constant parameters. The independent parameters studied were winding speed and applied load. The dependent variables studied were the void content, the fiber volume content, and the reduction in the thickness of the ring specimens. The parameters that remained constant for each experiment were the material, the processing temperature, the number of plies (or layers of towpreg) wound, the tooling geometry, and the winding tension. Table 4.3 lists the constant parameters and their corresponding values.

Table 4.3: Constant Parameters and Values

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Type of material</td>
<td>PEEK matrix / AS-4 carbon fibers</td>
</tr>
<tr>
<td>Material system</td>
<td>6 mm wide preimpregnated 12K tow</td>
</tr>
<tr>
<td>Process temperatures</td>
<td></td>
</tr>
<tr>
<td>- Incoming towpreg</td>
<td>400 °C</td>
</tr>
<tr>
<td>- Substrate surface</td>
<td>300 °C</td>
</tr>
<tr>
<td>Number of plies wound</td>
<td>50 plies</td>
</tr>
<tr>
<td>Lay-up angle</td>
<td>0 degrees</td>
</tr>
<tr>
<td>Size of mandrel</td>
<td>178 mm OD x 305 mm</td>
</tr>
<tr>
<td>Size of compaction roller</td>
<td>28.6 mm OD x 9.5 mm</td>
</tr>
<tr>
<td>Winding tension</td>
<td>250 grams</td>
</tr>
</tbody>
</table>
4.7 Experimental Procedure

Each composite ring was fabricated using the same procedure. A detailed description of the fabrication procedure is presented in this section.

The composite rings were fabricated as follows:

(1) The filament winding machine and the Macintosh computer running Workbench I/O software were turned on.

(2) Air flow to both hot air guns was obtained by adjusting the pressure regulators to the predetermined settings.

(3) The desired winding speed was set by adjusting the hydraulic transmission in the filament winder until the necessary rotational velocity was obtained.

(4) The rotating mandrel was preheated to 200°C using the 2,000 watt, substrate hot air gun. This tool temperature is recommended by the manufacturer for processing APC-2 [21].

(5) The substrate hot air gun and the filament winding machine were turned off.

(6) The towpreg was fed through the guide rollers/delivery system and positioned underneath the compaction roller.

(7) The exposed end of the towpreg was fastened to the mandrel using high-temperature tape.

(8) The desired load was applied to the towpreg by setting the cylinder air pressure to the level determined from the calibration curve.

(9) The slack in the towpreg was taken up by rotating the let-off spindle and adjusting the friction brake on the creel system until a winding tension of approximately 250 grams was achieved.

(10) A deflection plate was placed on top of the incoming towpreg directly beneath the 4,000 watt, hot air gun to prevent the towpreg from degrading while the hot air gun was brought up to temperature.

(11) Both hot air guns were brought up to temperature by slowly increasing the applied voltage until the desired process temperatures were achieved.
(12) The deflection plate was quickly removed and the filament winding machine turned on.
(13) The nozzle exit temperatures were monitored to ensure that the proper process temperatures were being maintained.
(14) The number of layers wound was recorded on the rotary counter attached to the drive spindle.
(15) Once 50 layers were wound, power to the hot air guns and the filament winding machine was turned off. Air flow to the hot air guns was maintained to allow for cooling of the heating elements.
(16) The air pressure was removed from the air cylinder by shutting off the pressure regulator and opening the purge valve.
(17) The incoming towpreg was cut from the wound specimen and the swing arm was lifted up and away from the mandrel.
(18) The carriage position lock was disengaged and the crosshead was moved to a position approximately 5 centimeters from the wound specimen.
(19) Steps 2-18 were repeated until a total of 5 rings were wound onto the mandrel.
(20) Once the mandrel had cooled, it was removed from the filament winding machine.
(21) The rings were untaped, tagged, and slid off the end of the mandrel. The rings could be removed from the mandrel without collapsing it because the mandrel shrinks away from the rings as it cools due to its higher coefficient of thermal expansion.
(22) Steps 1-21 were repeated until three sets of five rings or a total of fifteen rings had been produced.

4.8 Sample Testing

The properties of the consolidated part are important in assessing the effects of the process parameters. The void content, the degree of bonding, the final dimensions, and the fiber content are major characteristics for determining the overall performance and quality of the composite part. The composite ring specimens were measured and tested for thickness, fiber
content, crystallinity, density, and void content. The degree of interply bonding generally is determined from mechanical testing procedures and was not addressed in this investigation.

4.8.1 Ring Thickness Measurement

The reduction in thickness that the towpreg undergoes during the consolidation process is critical information for the design of composite laminate structures comprised of many layers of consolidated towpreg. The designer must be able to determine the final dimensions of the composite structure from the number of laminates (or layers) of consolidated towpreg used in the design. For the on-line consolidation process studied, only the towpreg's thickness is of interest as the width of the towpreg does not change significantly during consolidation. This is because the primary deformation mechanism is matrix flow parallel to the fiber direction and not transverse matrix flow (see Section 3.2.2.3). Transverse matrix flow would result in an increase in the width of the towpreg. The thickness of each layer of consolidated (or deformed) towpreg is obtained by dividing the measured ring thickness by the number of layers wound. The reduction in thickness, $t_{\text{reduction}}$, the towpreg undergoes during consolidation is calculated from the expression

$$t_{\text{reduction}} = \frac{t_o - t_i}{t_o} \times 100 \text{ } (\%)$$

(4.6)
where \( t_0 \) and \( t_L \) refers to the thickness of the undeformed and deformed towpreg, respectively. The test procedure for measuring the thickness of each consolidated composite ring is presented in the following section.

4.8.1.1 Procedure

The thickness of each consolidated ring was measured at ten equally-spaced positions on the ring using a dial caliper (Starrett No. 120). The knife edge contacts on the caliper were used to measure the thickness because they accommodated the curvature of the composite rings. The thickness readings were taken directly from the bar and the dial indicator. An average of the ten thickness measurements obtained from each ring was recorded. The thickness of each layer of consolidated towpreg and the percent thickness reduction were determined using the methods described in the previous section. The average ring thickness, deformed towpreg thickness, and percent thickness reduction for each ring specimen are presented in Section 5.3.4.

4.8.2 Fiber Volume Fraction Measurement

The fiber content of the finished composite is critical in predicting its in-service performance because the overall properties of the composite are extremely sensitive to changes in the fiber content. In most processes, the fiber volume fraction of the composite changes during the forming and consolidation stages due to the removal of voids and excess resin and the redistribution of the fiber network. The fiber volume fraction of each consolidated ring was determined using optical microscopy techniques.
The specimens were analyzed using the same image analysis software and optical microscope described in Section 4.4.1.

Assuming that all the fibers are continuous within the composite rings, the percent fibers by volume is equivalent to the percent area of fibers within the cross-section. If the cross-sections of the rings were transversely isotropic and void-free, the area fraction of fibers could be determined from a single sample area of the cross-section. Unfortunately, the composite rings fabricated for this study did not have a uniform distribution of fibers and matrix. Therefore, in order to obtain an accurate representation of the fiber volume fraction in each ring, the entire cross-section must be used to determine the area fraction of fibers.

A widely-used image analysis technique for determining the percent area of fibers within the cross section is thresholding. With this technique, a threshold intensity level is used to distinguish between the fibers and the matrix. For the material system studied, accurate threshold measurements can only be obtained at magnifications of 500X or higher due to the difficulty in discerning the actual borders between the fibers and the matrix. At this magnification, more than 300 measurements would be required to scan the entire cross-section of the specimens.

Fortunately, a more efficient technique for accurately measuring the fiber area fraction was developed by Yang [24]. With this technique, the number of fibers within a known area is determined from the cross-section image at a magnification of 100X. This is accomplished by filtering the cross-section image so that only the fibers are highlighted and then counting the number of highlighted regions within the image area. The
area fraction of fibers within the known area, $A_r$, can be calculated from the total number of fibers present and the fiber diameter using the expression

$$A_r = \frac{N\pi d_f^2}{4A_{\text{total}}} \quad (4.7)$$

where $N$ refers to the number of fibers within the image, $d_f$ refers to the nominal diameter of the fibers, and $A_{\text{total}}$ refers to the total image area. The area fraction of fibers within the entire cross-section is simply the average of the local fiber area fraction measurements. Using this method, five times fewer images are required to scan the entire cross-section of the specimens.

4.8.2.1 Procedure

A specimen approximately 1 centimeter in length was cut from each ring using a diamond saw. The ends of the specimen were sanded flat using fine emory cloth so as to not disturb the fiber/matrix distribution within the cross section. The specimens were cast in epoxy and polished to a 1 micron surface finish. The surface of each specimen was cleansed with distilled water. The specimen was placed on the optical microscope and examined at a magnification of 100X. The image from the microscope was filtered and the number of highlighted fibers counted using the image analysis software. Between 35 to 60 image screens were needed to measure the entire cross section of each specimen. The number of fibers present in each image screen were recorded and the fiber area fractions were
determined using the method described in the previous section. The fiber volume fraction obtained for each ring are presented in Section 5.3.5.

4.8.3 Matrix Crystallinity Measurement

As the PEEK matrix is a semi-crystalline polymer, the degree of crystallinity of the matrix is important in determining the properties of the composite specimens. The crystallinity of the ring specimens from each experiment was determined by Differential Scanning Calorimetry (DSC) analysis. This technique involves heating the sample past its melting temperature and measuring the heat of fusion of the specimen (i.e., the heat input required to completely melt the sample). As only the crystals undergo a phase change, the heat of fusion is directly proportional to the degree of crystallinity. Thus, the heat of fusion measured with DSC can be correlated with the heat of fusion of a completely crystalline specimen to obtain the weight fraction of crystalline polymer in the specimen tested. This relationship can be written as

\[ W_x = \frac{\Delta H_{f,\text{meas}}}{\Delta H_{f,\text{max}}} \times 100 \]  

where \( W_x \) is the weight fraction of crystalline polymer in the test specimen, \( \Delta H_{f,\text{meas}} \) is the heat of fusion obtained from the DSC analysis, and \( \Delta H_{f,\text{max}} \) is the heat of fusion for 100 percent crystalline polymer. The volume fraction
of crystalline polymer can be obtained from the weight fraction and densities of the crystalline and amorphous polymers using the equation

\[ V_x = \frac{W_x}{\rho_x \left[ \frac{W_x}{\rho_x} + \frac{(1 - W_x)}{\rho_a} \right]} \]  

(4.9)

where \( V_x \) is the volume fraction of crystalline polymer, \( \rho_x \) is the density of the crystalline polymer, and \( \rho_a \) is the density of the amorphous polymer. A description of the procedure used to test for degree of crystallinity is presented in the next section.

4.8.3.1 Procedure

One test specimen was taken from each experiment for a total of five DSC specimens. The specimens weighed between 5 and 11 milligrams and were removed from the middle of the cross section of the composite rings using a cold chisel. Each composite specimen was sealed in a miniature aluminum container and weighed on an analytical balance to the nearest 0.01 mg. The DSC analysis requires that the mass of the semi-crystalline polymer in the specimen be known. As the specimens contained fibers as well as the semi-crystalline matrix, the weight of the polymer matrix was calculated from the total specimen weight using the manufacturer reported matrix content for the APC-2 towpreg. The heat of fusion of the specimens was measured using a computer-controlled DSC machine (Perkin-Elmer Model No.7). The specimen weight, the heat of fusion, and the melting temperature were recorded. The weight fraction and corresponding
volume fraction of crystalline polymer in each specimen were obtained using the methods described in the previous section. The matrix crystallinities obtained for each ring are presented in Section 5.3.6.

4.8.4 Composite Density Measurement

The density of a consolidated composite part plays a key role in determining its performance and quality. The density affects the specific properties and provides an indication of the void content or degree of consolidation of the composite part. The densities of the composite rings were determined using a technique outlined in Method A of ASTM D792. With this technique, the specific gravity of a composite ring is determined from the weight of the composite specimens in water and in air using the relationship

$$\gamma = \frac{a}{a - w}$$  (4.10)

where $\gamma$ is the specific gravity of the composite specimen, $a$ is the weight of the composite specimen in air, and $w$ is weight of the composite in water. The density of the composite ring is then obtained from the equation

$$\rho = \gamma \rho_{\text{water}}$$  (4.11)

where $\rho$ refers to the density of the composite specimen and $\rho_{\text{water}}$ refers to the density of distilled water at the testing temperature.
4.8.4.1 Procedure

The test method requires that the surface and edges of the specimen be smooth. Any surface irregularities could trap air which would affect the relative density measurement. The sides of the composite rings contained surface irregularities that were the result of slight misalignments and variations in the width of the towpreg that accumulated during the fabrication of each ring. The sides of each ring were ground smooth using a high-speed grinding cylinder attached to an electric drill. Ten test specimens approximately 50 mm in length were cut from each of the 15 fabricated composite rings using a diamond-impregnated cutting blade mounted to a radial-arm saw. The ends of the cut test specimens were also ground to a smooth surface.

An experimental density testing apparatus was constructed (Figure 4.6). The apparatus consisted of an electronic laboratory balance (Ohaus Model TS120S), a draft shield, a corrosion-resistant metal hanger, a 500 ml glass beaker, a metal bridge support, and distilled water. The balance was used to obtain the weight of the specimens in air and in water. A draft shield was placed over the balance to prevent any outside air movement from disrupting the measurement. When weighing the samples in water, the beaker containing the distilled water was isolated from the balance via the aluminum bridge support. A mercury-filled thermometer (Fisher Model 15-043A) was used to measure the temperature of the distilled water.
Figure 4.6: Density Testing Apparatus
A brief description of the density test procedure is presented below.

- A test specimen 50 mm in length was cut from the composite ring.
- The sides and ends of the specimen were ground smooth with the rotary grinder.
- The composite specimen was weighed in air and the value recorded.
- The bridge support, the beaker containing the distilled water, and the hanger system were set-up on the balance.
- As the hanger system was placed directly on the balance pan, the weight of the hanger system and the hanger immersed in the water was removed by taring the balance.
- The temperature of the distilled water was recorded.
- The composite specimen was attached to the hanger and lowered into the distilled water until the specimen was completely immersed.
- Air bubbles trapped on the hanger and the specimen were removed by gently tapping the specimen against the inside wall of the glass beaker.
- The weight of the specimen in water was recorded.
- The specimen was removed from the hanger and the procedure repeated for each of the 165 test specimens.

The densities obtained for each ring are presented in Section 5.3.7.

4.8.5 Void Content Measurement

The void content of a consolidated composite part is a direct indicator of the degree of consolidation and of overall quality. The presence of voids can greatly affect the overall performance of the composite part. The void content of a composite part can be measured using several techniques. These include image analysis, ultrasonic C-scan analysis, and density
analysis. Image analysis is the most accurate of these techniques as voids within a cross-section specimen actually can be seen and measured with an optical microscope and image analysis software. However, this technique is very time-consuming as a large number of cross-sections must be prepared and examined in order to obtain an accurate representation of each composite ring. Ultrasonic analysis is a common non-destructive evaluation technique for measuring the void content of a composite part. The major drawbacks to ultrasonic void content evaluation of composite rings are the limited resolution for detecting small voids and the sensitivity of the measurement to changes in the thickness and fiber volume fraction within each ring sample. Density analysis involves comparing the measured density of a composite ring with its theoretical density to determine the void content. The accuracy of this technique is limited by the accuracy in determining the theoretical density of the composite rings. However, it is presumed that the comparatively higher efficiency of the density technique for determining the void content of the entire composite ring outweighs the loss in accuracy when assessing the relative effects of the process parameters on the void content of the composite rings.

The void content of each composite ring was obtained using a density technique similar to that outlined in Method A of ASTM D2734. With this technique, the void content is obtained from the measured density of the composite specimen and the theoretical (void-free) density of the composite specimen using the relationship

\[ V_v = \frac{\rho_T - \rho_M}{\rho_T} \]  

(4.12)
where $V_v$ is the volume fraction of voids present, $\rho_T$ is the theoretical, void-free density, and $\rho_M$ is the experimentally measured density. The measured density is obtained by methods described in the previous section. The theoretical density is obtained from the known densities and volume fractions of the constituent materials using the rule of mixtures relationship for composite density

$$\rho_T = \rho_f V_f + [\rho_x V_x + \rho_a(1 - V_x)](1 - V_f)$$

where $\rho_f$ is the density of the fibers, $V_f$ is the fiber volume fraction, $\rho_x$ is the density of the crystalline matrix, $V_x$ is the volume fraction of crystalline polymer in the matrix, and $\rho_a$ is the density of the amorphous matrix. The void content was calculated for each of the 15 fabricated composite rings. The void contents obtained for each ring are presented in Section 5.3.8.

### 4.9 Summary

An experimental on-line consolidation mechanism constructed and installed on a two-axis filament winding machine was described. The on-line consolidation mechanism consisted of hot air guns that applied the required process heat and a pneumatically-controlled compaction roller that applied the required consolidation pressure. Composite rings were fabricated at a process temperature of 400°C, winding speeds of 12.7, 31.8, and 50.8 mm/sec, and applied loads of 89, 156, and 222 Newtons. The rings were wound from APC-2 towpreg and had an inside diameter of 178 mm.
The ring specimens were prepared for testing and measured for thickness, fiber content, density, crystallinity, and void content. A $2^2$ factorial design with analysis of variance (ANOVA) was used to investigate the effects of winding speed and applied load on the void content, fiber content, and final thickness of the consolidated specimens.
CHAPTER 5

RESULTS and DISCUSSION

5.1 Introduction

The overall goal of the research was to obtain a science-based understanding of the on-line consolidation process in thermoplastic filament winding through theoretical analyses and experimental investigations. Presented in this chapter are the theoretical results, the experimental results, and a comparison of the two. The theoretical results were obtained from the proposed on-line consolidation deformation model and include the empirical determination of the model's parameters, in addition to the model's predictions for the applied load required to achieve the final fiber volume fraction in each experiment. The deformation model employed the same process conditions and material that were used in the experimental investigation. The experimental results were obtained from composite ring specimens filament wound at various applied loads and winding speeds and include the results of the equipment calibration and material characterization, in addition to the results obtained from the analysis of the ring specimens. The effects of applied load and winding speed on the thickness, fiber content, and void content of the composite rings are presented and discussed through statistical analyses and
graphical representations. The theoretical predictions then are compared to the results of the experimental investigation in order to assess the adequacy of the proposed on-line consolidation deformation model for describing the mechanical phenomena that occur during the on-line consolidation process. General guidelines for the application of the deformation model to thermoplastic filament winding processes with on-line consolidation mechanisms and material systems not studied in this research are presented and discussed. Finally, general comments on the thermoplastic filament winding process with on-line consolidation are presented.

5.2 Theoretical Results

The theoretical analysis was presented in Chapter Three. The theoretical results were obtained from the deformation model that was developed to describe the on-line consolidation process in thermoplastic filament winding. The theoretical results include the determination of the empirical model parameters in addition to the model predictions for the applied load required to achieve the final fiber volume fraction in each experiment.

5.2.1 Empirical Determination of Model Parameters

The deformation model contains several empirically determined parameters: the thickness of the deformed towpreg, the Kozeny constant, and the void content. The predicted values for each of these model
parameters were derived from a database that was created using the experimental results presented in Section 5.3. Regression analyses were performed on the data to determine functions that would sufficiently predict the desired parameters at settings not tried in the experiments. Presented in this section are the methods and results associated with the regression analyses performed on the experimental data. It should be noted that the correlations obtained from the regression analyses are only applicable for the material, process, and equipment studied.

5.2.1.1 Thickness of the Deformed Towpreg (or Final Towpreg Thickness)

The deformation model requires that the thickness of the deformed towpreg be known in order to determine the length of the nip region (see Equation 3.1). Assuming that fiber continuity is maintained and that the towpreg width does not change significantly during the deformation process, a linear relationship between the thickness and the fiber volume fraction of the towpreg should exist. As the fiber volume fraction of the deformed towpreg is an input to the model, the relationship between the thickness and the fiber volume fraction of the deformed towpreg was determined from a linear regression performed on the experimental data (Figure 5.1). The results of the regression analysis yielded the correlation equation

\[ t_L = 0.408 - 0.477 V_L \] (mm) (5.1)
Figure 5.1: Relationship Between the Thickness and Fiber Volume Fraction of the Deformed Towpreg for Experiments RS1 - RS5
where \( t_L \) refers to the deformed towpreg thickness expressed in millimeters and \( V_L \) refers to the fiber volume fraction of the deformed towpreg. Equation 5.1 had a coefficient of determination (or "R\(^2\)") of 0.983. This value of \( R^2 \) is close enough to unity to allow one to conclude that a linear relationship between the thickness and the fiber volume fraction of the deformed towpreg exists. Table 5.1 compares the measured thickness of the deformed towpreg with the value predicted by Equation 5.1 for each experiment.

<table>
<thead>
<tr>
<th>Experiment #</th>
<th>Measured Final Towpreg Thickness (mm)</th>
<th>Predicted Final Towpreg Thickness (mm)</th>
<th>Percent Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>0.146</td>
<td>0.144</td>
<td>1.4</td>
</tr>
<tr>
<td>RS2</td>
<td>0.140</td>
<td>0.142</td>
<td>-1.4</td>
</tr>
<tr>
<td>RS3</td>
<td>0.120</td>
<td>0.120</td>
<td>0.0</td>
</tr>
<tr>
<td>RS4</td>
<td>0.129</td>
<td>0.130</td>
<td>-0.8</td>
</tr>
<tr>
<td>RS5</td>
<td>0.129</td>
<td>0.128</td>
<td>0.8</td>
</tr>
</tbody>
</table>

The average deviation is approximately 1 percent. This relatively accurate correlation justified the use of Equation 5.1 in the deformation model to predict the thickness of the deformed towpreg based upon the fiber volume fraction of the deformed towpreg that was entered into the model.

### 5.2.1.2 Kozeny Constant

The deformation model requires a value for the Kozeny constant in order to determine the matrix flow within the nip (see Equation 3.6). The
Kozeny constant must be determined empirically because it depends upon the process conditions, the matrix pressure gradient, the fluid and fiber materials, the fiber arrangement, and the fiber volume fraction among others. The Kozeny constant was determined for each of the five experiments using an iterative process. This involved entering the winding speed and final fiber volume fraction for each experiment into the deformation model and then adjusting the Kozeny constant until the predicted applied load matched the measured applied load. It should be noted that the Kozeny constants obtained from this method actually represent average values because the Kozeny constant varies throughout the nip region due to changes in the matrix pressure gradient and fiber volume fraction with position in the nip.

A multiple regression was performed on the five Kozeny constants obtained from the iteration process. It is presumed that, for the experiments conducted, the Kozeny constant depends only upon the final fiber volume fraction, the winding speed (shear rate), and the nip length (flow path length). The remaining factors affecting the Kozeny constant are assumed to remain constant between the experiments as the same material, process temperatures, and equipment were used for each experiment. The relationship among the variables affecting the Kozeny constant is assumed to be

\[ K = C V_f U^\beta L^\gamma \]  

(5.2)
where the term $K$ is the Kozeny constant, $V_L$ is the final fiber volume fraction, $U$ is the winding speed, and $L$ is the nip length. The terms $C$, $\alpha$, $\beta$, and $\gamma$ are constants obtained from the regression analysis. The regression analysis yielded the correlation equation

$$K = \exp(-78.3)V_L^{(-51.1)}U^{(-2.67)}L^{(56.7)}$$

(5.3)

where $K$ is dimensionless, $V_L$ is measured in volume percent, $U$ is measured in mm/sec, and $L$ is measured in microns. Equation 5.3 had an $R^2$ of 0.924. Table 5.2 compares the Kozeny constant predicted by Equation 5.3 with those determined from iteration of empirical data.

<table>
<thead>
<tr>
<th>Experiment #</th>
<th>Empirical Kozeny Constant</th>
<th>Predicted Kozeny Constant</th>
<th>Percent Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>0.150</td>
<td>0.234</td>
<td>-56.0</td>
</tr>
<tr>
<td>RS2</td>
<td>0.417</td>
<td>0.288</td>
<td>30.9</td>
</tr>
<tr>
<td>RS3</td>
<td>0.051</td>
<td>0.043</td>
<td>15.7</td>
</tr>
<tr>
<td>RS4</td>
<td>0.022</td>
<td>0.021</td>
<td>4.5</td>
</tr>
<tr>
<td>RS5</td>
<td>0.068</td>
<td>0.087</td>
<td>-27.9</td>
</tr>
</tbody>
</table>

The average deviation is approximately 27 percent. The deviation is larger than expected for the relatively high $R^2$ value. This large deviation can be attributed to three main sources. First, there may have been other variables affecting the Kozeny constant that were not considered in the regression relationship. Second, the nip length was used to regress the
empirically determined Kozeny constants. The nip length is calculated from the thickness of the deformed towpreg that also is determined from regression of empirical data. Thus, the error inherent to the regression of the deformed towpreg thickness would be carried through the regression analysis of the Kozeny constant. Third, the relatively large variation in the thickness and fiber content data used to obtain the empirically determined Kozeny constants for experiments RS1 and RS2 might be reflected in their relatively large percent deviations. Although the actual correlation obtained with Equation 5.3 is relatively low, there are currently no other models that adequately predict the Kozeny constant in this particular application. Therefore, Equation 5.3 was used in the deformation model to predict the Kozeny constant.

The Kozeny constants listed in Table 5.2 fall within the 0.02 to 0.58 range of Kozeny constants associated with parallel matrix flow that were determined in studies conducted on APC-2 [25,7]. However, the constants reported in [25,7] were obtained from consolidation experiments that involved much lower shear rates and matrix pressure gradients. The majority of the reported Kozeny constants were obtained from compression molded unidirectional APC-2 laminates that were consolidated at comparatively lower pressures and longer times.

5.2.1.3 Void Content

A relatively simple method for predicting void content is to relate the dimensional changes each composite ring undergoes during the on-line consolidation process with its measured void content [6]. Figure 5.2 shows
Figure 5.2: Relationship Between the Void Content and Thickness Reduction

\[ y = 18.8 - 0.317x \quad R^2 = 0.978 \]
the relationship between the void content and the percent reduction in towpreg thickness for data obtained from experiments RS1 through RS5. A linear regression performed on the experimental data yielded the correlation equation

\[ V_v = 18.8 - 0.317 \ t_{\text{reduction}} \] (vol.%)

where \( V_v \) is the void content expressed in volume percent and \( t_{\text{reduction}} \) is the reduction in towpreg thickness measured in percent of initial towpreg thickness. Equation 5.4 had an \( R^2 \) of 0.978. Table 5.3 compares the measured void content of each ring with the value predicted by Equation 5.4 for each experiment.

<table>
<thead>
<tr>
<th>Experiment #</th>
<th>Measured Void Content (% by volume)</th>
<th>Predicted Void Content (% by volume)</th>
<th>Percent Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>6.8</td>
<td>6.3</td>
<td>7.4</td>
</tr>
<tr>
<td>RS2</td>
<td>5.6</td>
<td>6.1</td>
<td>-8.9</td>
</tr>
<tr>
<td>RS3</td>
<td>3.3</td>
<td>3.2</td>
<td>3.0</td>
</tr>
<tr>
<td>RS4</td>
<td>4.3</td>
<td>4.5</td>
<td>-4.7</td>
</tr>
<tr>
<td>RS5</td>
<td>4.2</td>
<td>4.2</td>
<td>0.0</td>
</tr>
</tbody>
</table>

The average deviation is approximately 5 percent. The deviation is larger than expected for the relatively high \( R^2 \) value. This is because the reduction in towpreg thickness is used to regress the void content data. The thickness reduction is calculated from the thickness of the deformed
towpreg that also is determined from regression of empirical data. Thus, the error inherent to the regression of the deformed towpreg thickness would be carried through the regression analysis of the void content. However, the correlation was sufficient enough to justify the use of Equation 5.4 in the deformation model to predict the void content based upon the thickness of the deformed towpreg determined by the model.

5.2.2 Model Predictions

The proposed deformation model was used to predict the applied load required to achieve the reported final fiber volume content for each of the five experiments conducted in the experimental investigation (see Section 5.3). The deformation model spreadsheets generated for each experiment are contained in Appendix A. The winding speed used in each experiment and the average measured fiber volume fraction of the three ring specimens from each experiment served as inputs to the model. The material constants listed in each spreadsheet were for APC-2 (PEEK and AS4 carbon fibers) at a temperature of 400°C [12].

5.2.2.1 Predicted Matrix Pressure Distribution

Figure 5.3 shows the predicted matrix pressure distribution generated within the nip for each experiment. The relative shape of each profile is complex and difficult to explain as the matrix pressure depends on many model parameters. However, all five matrix profiles show an expected steady pressure rise due to the backflow of matrix material out the nip entrance that occurs as the towpreg is compacted. The matrix pressure
Figure 5.3: Matrix Pressure Distribution Within Nip for Experiments RS1 - RS5
levels off towards the exit of the nip where the maximum fiber volume content (and subsequent maximum deformation) of the towpreg is achieved. It is assumed that the matrix pressure returns to zero (or atmospheric) immediately after the exit of the nip. The matrix pressure profiles are strongly influenced by the geometries bounding the nip region. A smaller diameter compaction roller would increase the average matrix pressure and the maximum matrix pressure gradient as the nip length (and subsequently the contact area) would be decreased. The shorter flow path length and the higher matrix pressure gradient would aid in the removal of voids within the nip. This is because the entrapped air would have a shorter distance to travel back out the nip entrance and the higher pressure gradient would provide more viscous flow force to overcome the surface tension forces hindering void mobilization [26]. However, the compaction roller must be large enough so that the residence time in the nip is greater than the minimum time required for the viscous matrix to flow and achieve intimate contact.

5.2.2.2 Predicted Fiber Bed Pressure Distribution

Figure 5.4 shows the predicted fiber bed pressure distribution generated within the nip for each experiment. All five fiber bed pressure profiles show an increase in the fiber bed pressure with increasing position in the nip. This trend was expected as Equation 3.8 indicates that the fiber bed pressure should increase with increasing fiber volume content. The fiber volume content of the towpreg increases with position in the nip due to the increase in the total amount of towpreg deformation. In addition,
Figure 5.4: Fiber Bed Pressure Distribution Within Nip for Experiments RS1 - RS5
Figure 5.4 shows that the maximum fiber bed pressure occurs at the end of the nip where the maximum fiber volume content is achieved. The variation in the maximum (final) fiber volume content among the five experimental sets is reflected in the relative location of the peaks of each profile. It should be noted that the maximum fiber bed pressure is approximately three orders of magnitude less than the maximum matrix pressure obtained for each of the experimental sets. This indicates that, for the consolidation process and material studied, the load-bearing contribution of the fiber bed is negligible compared to that supplied by the matrix. There is little load transferred to the fiber reinforcement during the on-line consolidation process studied because the range of the fiber volume fraction is sufficiently less than the highest fiber volume content obtainable, the time at pressure is very short, the matrix viscosity is relatively high, the fibers are well aligned, and the winding tension is negligible.

5.2.2.3 Predicted Applied Load

The predicted applied load required to achieve the final fiber volume content in each experiment was obtained by integrating both the matrix and fiber bed pressure distributions over the length of the nip and multiplying this by the width of the towpreg (see Equation 3.9). The applied load predictions are presented in Table 5.4. The winding speed and final fiber volume content associated with each experiment also are listed in Table 5.4 as they were the model inputs used to make the predictions.
Table 5.4: Model Predictions for the Required Applied Load

<table>
<thead>
<tr>
<th>Experiment #</th>
<th>Winding Speed (mm/sec)</th>
<th>Final Fiber Content (% by volume)</th>
<th>Predicted Applied Load (N)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>12.7</td>
<td>55.6</td>
<td>131</td>
</tr>
<tr>
<td>RS2</td>
<td>12.7</td>
<td>56.0</td>
<td>166</td>
</tr>
<tr>
<td>RS3</td>
<td>50.8</td>
<td>60.5</td>
<td>196</td>
</tr>
<tr>
<td>RS4</td>
<td>50.8</td>
<td>58.5</td>
<td>84.9</td>
</tr>
<tr>
<td>RS5</td>
<td>31.8</td>
<td>58.9</td>
<td>193</td>
</tr>
</tbody>
</table>

Figure 5.5 provides a graphical representation of the relationships between the predicted applied load and the fiber volume content for each of the three winding speeds studied in the experimental investigation. The figure shows an increase in the required applied load with increasing fiber volume content for a constant winding speed. This is because more load must be applied to the towpreg in order to achieve the increased deformation corresponding to the higher fiber volume content. Figure 5.5 also shows a decrease in the required applied load with increasing winding speed for a constant fiber volume content. This is due to the shear-thinning behavior of the molten PEEK polymer matrix. The increase in shear rate with winding speed results in a decrease in the matrix viscosity. The lower viscosity reduces the resistance to matrix flow and thus reduces the load required to compact the towpreg to the given fiber volume fraction. In addition, the applied load appears to approach a minimum value as the fiber volume content is decreased for each of the winding speed profiles. One explanation for this is that there is a minimum load that must be
Figure 5.5: Predicted Relationships Between Applied Load and Fiber Volume Content for the Winding Speeds Used in the Experimental Investigation

- U = winding speed used to obtain predicted applied load curve
- U = 12.7 mm/s
- U = 31.8 mm/s
- U = 50.8 mm/s

fiber content (% by volume) vs. required applied load (N)
applied to the compaction roller in order to maintain contact with the towpreg, no matter what winding speed is used.

5.3 Experimental Results

A description of the experimental investigation was presented in Chapter 4. The experimental results were obtained from composite ring specimens filament wound at various applied loads and winding speeds and include the results of the equipment calibration and material characterization, in addition to the results obtained from the analysis of the ring specimens. The effects of applied load and winding speed on the thickness, fiber content, and void content of the composite rings are presented and discussed through statistical analyses and graphical representations.

5.3.1 Calibration of Experimental Equipment

The measurement techniques used to calibrate the applied heat, the applied load, and the winding speed were presented in Section 4.3. The calibration results for these independent process parameters are presented in this section.

5.3.1.1 Applied Heat

As the incoming towpreg and substrate temperatures were constant over the range of winding speeds studied, the heat input to the nip was adjusted to account for the change in the heat transfer rate with winding
speed. The methods for controlling the heat input and for measuring the towpreg and substrate temperatures were presented in Section 4.3.1. The amount of heat applied to the nip region was controlled by adjusting the nozzle exit temperature and the supply air pressure of the hot air guns. Temperature indicating paints corresponding to the desired incoming towpreg temperature of 400°C and to the desired substrate temperature of 300°C were used for the calibration. The process settings necessary to transform the paints at each winding speed are listed in Table 5.5. It should be noted that the supply air pressure and the nozzle exit temperature for each winding speed were determined in combination such that the chance of the molten matrix degrading or being blown off the fibers was minimized.

Table 5.5: Hot Air Gun Settings Obtained from Heat Application Calibration

<table>
<thead>
<tr>
<th>Winding Speed (mm/sec)</th>
<th>Towpreg Temperature of 400°C</th>
<th>Substrate Temperature of 300°C</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Nozzle Exit Temperature (°C)</td>
<td>Supply Air Pressure (kPa)</td>
</tr>
<tr>
<td>12.7</td>
<td>550</td>
<td>207</td>
</tr>
<tr>
<td>31.8</td>
<td>800</td>
<td>207</td>
</tr>
<tr>
<td>50.8</td>
<td>815</td>
<td>276</td>
</tr>
</tbody>
</table>

To achieve an incoming towpreg temperature of 400°C, an increase in the required nozzle exit temperature and supply air pressure with increasing winding speed was expected. This is because the heat supplied
to the towpreg must be increased to account for the increase in the mass flow rate of the towpreg that occurs as the winding speed is increased.

To achieve a substrate temperature of 300°C, the nozzle exit temperature and supply air pressure were the same for all three winding speeds (i.e., the required heat input did not change with winding speed). One explanation for this is that, unlike the incoming towpreg which has to be heated from room temperature, the substrate surface remains relatively hot due to the cyclic nature of the winding process and the fact that it is insulated from the relatively cool mandrel by the layers of consolidated towpreg comprising the substrate. Thus for a constant heat input, the steady-state temperature of the substrate surface would be the same for the range of winding speeds studied.

5.3.1.2 Applied Load

As presented in Section 4.3.2, a compression scale was used to measure the load applied to the compaction roller for various cylinder air pressures. A calibration plot of applied load versus cylinder air pressure was obtained from the data (Figure 5.6). A linear regression was performed on the calibration plot in order to obtain an equation relating the cylinder air pressure to the load applied to the compaction roller. The regression analysis yielded the correlation equation

\[ P_{\text{appl}} = 831.3 \ p_{\text{air}} - 2.88 \quad \text{(N)} \quad (5.5) \]
Figure 5.6: Applied Load Calibration Curve

\[ y = 831.3x - 2.88 \quad R^2 = 1.000 \]
where $P_{\text{appl}}$ is the applied load expressed in Newtons and $p_{\text{air}}$ is the cylinder air pressure measured in MPa. Equation 5.5 was used to set the cylinder air pressure for the applied load specified in each experiment.

5.3.1.3 Winding Speed

As presented in Section 4.3.3, the winding speed (lay-up rate) is determined from the measured rotational velocity, $\omega$, of the mandrel and the mean diameter, $\bar{d}$, of the wound specimens and can be expressed as

$$U = \pi \bar{d} \, \omega \quad \text{(mm/s)} \quad (5.6)$$

where $U$ is the winding speed expressed in mm/sec, $\bar{d}$ is measured in millimeters, and $\omega$ is measured in revolutions per second. Based on the ring thickness results presented in Section 5.2.3.2, a mean diameter of 184 mm was used for the calibration. This diameter corresponds to a 6 mm thick ring specimen wound onto the 178 mm diameter mandrel. The measured rotational velocity of the mandrel was obtained by measuring the time required for one revolution to occur. The time required for one revolution to occur and the corresponding winding speed calculated from Equation 5.6 are presented in Table 5.6.
Table 5.6: Process Settings Obtained from Winding Speed Calibration

<table>
<thead>
<tr>
<th>Desired Winding Speed (mm/sec)</th>
<th>Time Required for One Revolution of the Mandrel to Occur (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>12.7</td>
<td>46</td>
</tr>
<tr>
<td>31.8</td>
<td>18</td>
</tr>
<tr>
<td>50.8</td>
<td>11</td>
</tr>
</tbody>
</table>

The winding speed was set by adjusting the hydraulic transmission until the time required for one revolution to occur matched the value specified in Table 5.6.

5.3.2 Material Characterization

The quality of the ring specimens is directly related to the quality of the towpreg used to fabricate them. The material used in this study was 6 mm wide APC-2 towpreg. An accurate value for the thickness of APC-2 towpreg was needed to determine the total amount of deformation the towpreg underwent during the on-line consolidation process. The initial towpreg thickness was measured using methods described in Section 4.4.1. In Method 1, a conventional hand held, flat anvil, digital micrometer was used to measure the towpreg thickness. In Method 2, towpreg thickness measurements were acquired from polished cross sections of the towpreg using image analysis software and an optical microscope. The results of the thickness measurements are presented in Table 5.7.
Table 5.7: Thickness of the Undeformed Towpreg (mm)

<table>
<thead>
<tr>
<th></th>
<th>Method 1</th>
<th>Method 2</th>
<th>Average of Methods 1 and 2</th>
</tr>
</thead>
<tbody>
<tr>
<td>Average Towpreg Thickness</td>
<td>0.320</td>
<td>0.154</td>
<td>0.237</td>
</tr>
<tr>
<td>Average Variation, $\Delta t$</td>
<td>----</td>
<td>0.060</td>
<td></td>
</tr>
</tbody>
</table>

The thickness measurements obtained from Method 1 actually represent a maximum (or upper) limit. This is because the flat anvil faces of the micrometer only measure across the tallest peaks along the width of the towpreg. Method 2 provides a more accurate representation of the thickness along the width of the towpreg because it accounts for the surface contours that exist in the towpreg’s cross-section (Figure 5.7). Figure 5.7 also provides some indication of the nonuniform distribution of fibers and matrix within the cross-section of the towpreg in addition to the presence of voids. The severity of the surface irregularities is evident in the average contour variation, $\Delta t$, which accounts for approximately 39 percent of the average towpreg thickness. Unfortunately, the thickness measurements obtained from Method 2 only account for variations in thickness across the width of the towpreg and do not take into account the variation in thickness along the length of the towpreg. This is because, unlike Method 1 in which numerous measurements were taken along the length of the towpreg, only three cross-sections were measured using Method 2 due to the time required to prepare and measure a single sample. Therefore, in order to account for the variations in the thickness across the towpreg as well as along the towpreg, an average of the towpreg thickness measurements obtained from both methods will be used in this study.
Figure 5.7: Cross-Section of APC-2 12K AS4 Towpreg at 100X
5.3.3 Description of Composite Ring Specimens

Composite rings were filament wound to determine the effects of applied load and winding speed on their thickness, fiber volume fraction, and void content. The rings were fabricated using the procedures outlined in Section 4.7. Rings were wound at speeds of 12.7, 31.8, and 50.8 mm/sec and applied loads of 89, 156, and 222 N. Each ring consisted of 50 layers of 6 mm wide APC-2 towpreg and had an inside diameter of 178 mm.

The rings fabricated for experiments RS1 and RS2 had rough sides. This was the result of difficulties experienced during the fabrication of ring specimens at the 12.7 mm/sec winding speed. At this relatively low winding speed, the towpreg had a greater tendency to wander underneath the compaction roller. The wandering prevented the towpreg from being laid down directly on the layer beneath. The rings fabricated for experiments RS3, RS4, and RS5 had relatively smooth sides indicating that wandering of the towpreg was minimal. The top and bottom surfaces of all the rings were smooth and had a dull finish. This indicates that, for the range of applied load and winding speed studied, there was no noticeable matrix depletion within the towpreg. In addition, there was only a minimal change in the width of the towpreg in each ring during the on-line consolidation process.

5.3.4 Thickness of Ring Specimens

The procedure for measuring the thickness of each consolidated composite ring and the methods for determining the thickness of the deformed towpreg and the corresponding percent reduction in towpreg
thickness were presented in Section 4.8.1. The thickness results are summarized in Table 5.8. An undeformed towpreg thickness of 0.237 mm and a total of 50 layers wound in each ring were used in Equation 4.6. It should be noted that the values listed for each experiment represent an average of the three ring specimens fabricated in each experiment.

Table 5.8: Ring Thickness Results

<table>
<thead>
<tr>
<th>Experiment #</th>
<th>Average Ring Thickness (mm)</th>
<th>Thickness of Deformed Towpreg (mm)</th>
<th>Reduction in Towpreg Thickness (% of initial thickness)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>7.28</td>
<td>0.146</td>
<td>38.7 ± 0.9</td>
</tr>
<tr>
<td>RS2</td>
<td>7.01</td>
<td>0.140</td>
<td>40.8 ± 0.8</td>
</tr>
<tr>
<td>RS3</td>
<td>6.00</td>
<td>0.120</td>
<td>49.3 ± 0.2</td>
</tr>
<tr>
<td>RS4</td>
<td>6.43</td>
<td>0.129</td>
<td>45.8 ± 0.2</td>
</tr>
<tr>
<td>RS5</td>
<td>6.43</td>
<td>0.129</td>
<td>45.8 ± 0.2</td>
</tr>
</tbody>
</table>

The average reduction in towpreg thickness includes the standard error of the three repeats fabricated in each experiment. The standard error provides an indication of the precision of the reported value and is determined by dividing the standard deviation by the number of repeats. The larger standard error (or increased variability) in experiments RS1 and RS2 was the result of towpreg wandering at the lower winding speed as presented in Section 5.3.3. Although the amount of wandering was slight, relatively large variations in the ring thickness occurred due to the cumulative nature of the winding process. The results listed in Table 5.8 indicate that there is some effect of the process parameters studied on the final thickness of the ring specimens. A complete statistical analysis of the
effects of winding speed and applied load on the reduction in towpreg thickness is presented in Section 5.3.9.1.

5.3.5 Fiber Volume Fraction of Ring Specimens

The technique and procedure used to obtain the fiber volume fraction of each consolidated composite ring were presented in Section 4.8.2. The results of the fiber volume fraction measurements are summarized in Table 5.9. A value of 8 microns was used in Equation 4.7 as the nominal diameter of AS4 carbon fibers [27]. It should be noted that the fiber volume fraction values listed for each experiment represent an average of the three ring specimens fabricated in each experiment.

Table 5.9: Fiber Volume Fraction Results

<table>
<thead>
<tr>
<th>Experiment #</th>
<th>Fiber Content of Ring Specimens (% by volume)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>55.6 ± 1.1</td>
</tr>
<tr>
<td>RS2</td>
<td>56.0 ± 1.5</td>
</tr>
<tr>
<td>RS3</td>
<td>60.5 ± 0.7</td>
</tr>
<tr>
<td>RS4</td>
<td>58.5 ± 0.7</td>
</tr>
<tr>
<td>RS5</td>
<td>58.9 ± 0.2</td>
</tr>
</tbody>
</table>

The average fiber volume fraction includes the standard error of the three repeats fabricated in each experiment. It is assumed that the larger standard error in experiments RS1 and RS2 is due to the towpreg wandering problem discussed in Section 5.3.4. The fiber volume fraction for each ring was obtained by averaging the local fiber volume fractions.
measured within the cross-section of the ring. The entire cross section was scanned due to the nonuniform distribution of fibers and matrix, especially the matrix-rich areas between the layers of towpreg (Figures 5.8 and 5.9). The matrix-rich areas are created when surface irregularities consisting mostly of matrix are flattened out and redistributed during the consolidation process. It is difficult to determine from the results listed in Table 5.9 if there is some effect of the process parameters studied on the fiber volume fractions of the ring specimens due to the relatively large standard errors. A complete statistical analysis of the effects of winding speed and applied load on the fiber volume fraction of the ring specimens is presented in Section 5.3.9.2

It should be noted that the fiber volume contents listed in Table 5.9 are higher than the initial 53.6 percent fiber volume content of the towpreg. The initial fiber volume content of the towpreg was determined from the manufacturer reported fiber content of 62.1 percent by weight and the densities of the constituent materials and was calculated assuming that no voids existed within the towpreg. However, voids did exist within the towpreg in addition to the presence of excess matrix material along the edges of the towpreg. This excess matrix material accumulated along the edges of the consolidated ring specimens and was removed during the preparation of the rings for testing. Therefore, the relatively higher fiber volume contents listed in Table 5.9 were the result of the removal of voids within the towpreg during the consolidation process in addition to the elimination of the matrix-rich regions along the edges of the rings during the preparation of the rings for testing.
Figure 5.8: Cross-Section of Ring Specimen RS1-1 at 100X
Resin-Rich Interlayers

Figure 5.9: Cross-Section of Ring Specimen RS3-2 at 100X
5.3.6 Matrix Crystallinity of Ring Specimens

The crystallinity of one ring specimen from each experimental set was determined by Differential Scanning Calorimetry (DSC) analysis as presented in Section 4.8.3. The matrix crystallinity results are listed in Table 5.10. The DSC plots for the five test samples can be found in Appendix B. A value of 130 J/g was used in Equation 4.8 as the heat of fusion for 100 percent crystalline PEEK polymer [28]. Values of 1.32 and 1.265 g/cm³ were used in Equation 4.9 as the densities of crystalline and amorphous PEEK polymer, respectively [29]. It should be noted that the manufacturer reported towpreg matrix content of 37.9 percent by weight was used for the analysis of all five ring samples. This was because the exact matrix content of each sample was unknown as the sample represented only a small portion of the composite ring.

Table 5.10: Matrix Crystallinity Results

<table>
<thead>
<tr>
<th>Experiment #</th>
<th>Sample Crystallinity (% by weight of matrix)</th>
<th>Sample Crystallinity (% by volume of matrix)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>19.0</td>
<td>18.4</td>
</tr>
<tr>
<td>RS2</td>
<td>29.6</td>
<td>28.8</td>
</tr>
<tr>
<td>RS3</td>
<td>19.3</td>
<td>18.6</td>
</tr>
<tr>
<td>RS4</td>
<td>17.6</td>
<td>17.0</td>
</tr>
<tr>
<td>RS5</td>
<td>17.1</td>
<td>16.5</td>
</tr>
<tr>
<td>Average</td>
<td>20.5 ± 2.3</td>
<td>19.9 ± 2.3</td>
</tr>
</tbody>
</table>

The matrix crystallinity varied significantly between the samples. However, no conclusion can be drawn regarding the effect of the various process conditions on the crystallinity as the exact weight of matrix in each
sample was unknown. In addition, it was difficult to remove the samples from the same location on each ring due to the removal technique used. Samples removed from layers closer to the mandrel might have a different crystallinity than samples removed from layers closer to the outer surface of the ring due to differences in the cooling rates. This is because the cooling rate of the just-consolidated towpreg decreases as more layers of the relatively insulative towpreg are wound onto the metal mandrel. The manufacturer reports that, for cooling rates between $10^\circ C - 700^\circ C$/minute, the matrix crystallinity of APC-2 ranges from 20 to 40 percent by weight and optimum composite properties result [21,30]. With the average matrix crystallinity obtained from all five samples of 20.5 weight percent, the cooling rates for this process would have to have been close to the maximum recommended cooling rate of $700^\circ C$/minute. However, the matrix crystallinity measurements may not accurately represent the true matrix crystallinity because the exact weight fraction of matrix in the test samples was unknown. Nonetheless, the average matrix crystallinity of 20.5 weight percent (or 19.9 volume percent) falls within the manufacturer recommended range and therefore will be used in this study.

5.3.7 Density of Ring Specimens

The technique and procedure used to obtain the density of each consolidated composite ring were presented in Section 4.8.4. The results of the density measurements are summarized in Table 5.11. A density of $0.9968 \, g/cm^3$ for distilled water at $26.1^\circ C$ [31] was used in Equation 4.11. It
should be noted that the density values listed for each experiment represent an average of the three ring specimens fabricated in each experiment.

Table 5.11: Density Results

<table>
<thead>
<tr>
<th>Experiment #</th>
<th>Composite Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>1.462 ± 0.007</td>
</tr>
<tr>
<td>RS2</td>
<td>1.484 ± 0.003</td>
</tr>
<tr>
<td>RS3</td>
<td>1.543 ± 0.002</td>
</tr>
<tr>
<td>RS4</td>
<td>1.516 ± 0.005</td>
</tr>
<tr>
<td>RS5</td>
<td>1.519 ± 0.003</td>
</tr>
</tbody>
</table>

The average composite density includes the standard error of the three repeats fabricated in each experiment. Unlike the relatively large variability in the thickness and fiber volume content measurements, the density measurements had relatively small standard errors. This is because the entire ring was used to obtain its composite density as the density for each ring was obtained by averaging the densities of the ten specimens that each ring was cut into.

5.3.8 Void Content of Ring Specimens

The technique and procedure used to obtain the void content of each consolidated composite ring were presented in Section 4.8.5. The void content results are summarized in Table 5.12. A density for AS4 carbon fibers of 1.80 g/cm³ [27] and an average matrix crystallinity of 19.9 percent by volume of matrix were used in Equation 4.13. It should be noted that the
void content values listed for each experiment represent an average of the three ring specimens fabricated in each experiment.

Table 5.12: Void Content Results

<table>
<thead>
<tr>
<th>Experiment #</th>
<th>Volume Fraction of Fiber</th>
<th>Measured Density (g/cm³)</th>
<th>Theoretical Density (g/cm³)</th>
<th>Void Content (% by volume)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>0.556</td>
<td>1.462</td>
<td>1.569</td>
<td>6.8 ± 0.8</td>
</tr>
<tr>
<td>RS2</td>
<td>0.560</td>
<td>1.484</td>
<td>1.572</td>
<td>5.6 ± 0.6</td>
</tr>
<tr>
<td>RS3</td>
<td>0.605</td>
<td>1.543</td>
<td>1.595</td>
<td>3.3 ± 0.3</td>
</tr>
<tr>
<td>RS4</td>
<td>0.585</td>
<td>1.516</td>
<td>1.584</td>
<td>4.3 ± 0.1</td>
</tr>
<tr>
<td>RS5</td>
<td>0.589</td>
<td>1.519</td>
<td>1.586</td>
<td>4.2 ± 0.1</td>
</tr>
</tbody>
</table>

The average void content includes the standard error of the three repeats fabricated in each experiment. The larger standard error for experiments RS1 and RS2 is believed to be due to the large variability in the fiber volume fractions reported for experiments RS1 and RS2. This is because the accuracy of each reported void content is dependent on the accuracy of the fiber volume fraction, the matrix crystallinity, and the composite density used to calculate the void content. The results listed in Table 5.12 indicate that there is some effect of the process parameters studied on the void content of the ring specimens. However, not all of the voids present in the ring specimens are attributed to the process used to fabricate the rings. Figure 5.10 is a micrograph of the cross-section of a ring specimen from experiment RS2 which clearly shows the presence of voids within the composite ring. As stated in Section 5.3.2, the quality of the ring specimens is directly related to the quality of the towpreg used to
Figure 5.10: Cross-Section of Ring Specimen RS2-3 at 500X
fabricate them. A portion of the voids present in the ring specimens can be attributed to voids that were present in the original or undeformed towpreg material. It is difficult to remove these pre-existing voids because they are usually located in fiber-rich regions where there is insufficient matrix to mobilize and remove the voids during the consolidation process. Therefore, the minimum void content that can be obtained when fabricating the ring specimens is generally limited by the void content of the original towpreg used to fabricate them. A complete statistical analysis of the effects of winding speed and applied load on the void content of the ring specimens is presented in Section 5.3.9.3

5.3.9 Statistical Analysis of Experimental Results

The results of the statistical analyses performed on the experiments constituting the $2^2$ factorial design are presented and discussed in this section. The experimental results used in the statistical analysis are summarized in Table 5.13. It should be noted that the values listed for each experiment represent an average of the three ring specimens fabricated in each experiment.

Table 5.13: Experimental Results Used in Statistical Analysis

<table>
<thead>
<tr>
<th>Experiment (i=1,2,3)</th>
<th>Design Factors</th>
<th>Thickness Reduction (%)</th>
<th>Fiber Content (% by vol.)</th>
<th>Void Content (% by vol.)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1-i</td>
<td>-</td>
<td>38.6</td>
<td>55.6</td>
<td>6.8</td>
</tr>
<tr>
<td>RS2-i</td>
<td>-</td>
<td>40.8</td>
<td>56.0</td>
<td>5.6</td>
</tr>
<tr>
<td>RS3-i</td>
<td>+</td>
<td>49.3</td>
<td>60.5</td>
<td>3.3</td>
</tr>
<tr>
<td>RS4-i</td>
<td>+</td>
<td>45.8</td>
<td>58.5</td>
<td>4.3</td>
</tr>
<tr>
<td>RS5-i</td>
<td>n/a</td>
<td>45.8</td>
<td>58.9</td>
<td>4.2</td>
</tr>
</tbody>
</table>
A description of the experimental design was presented in Section 4.6. The two independent process parameters used in the $2^2$ factorial design were winding speed and applied load. Also included in Table 5.13 is the winding speed / applied load interaction design factor. Each process parameter was investigated at two levels, a high (+) and a low (-). An analysis of variance (ANOVA) was performed for each of the dependent parameters: thickness reduction, fiber content, and void content. ANOVA tables were constructed to determine the levels at which the effects of the process parameters were significant and to obtain numerical estimates of the effects.

Graphical representations of the experimental results were used to verify the results obtained from the statistical analyses. Each figure includes average lines. The average lines are drawn through the points corresponding to the average of response data obtained at a particular level of the process parameter not plotted on the y-axis. The slope of each average line indicates the main effect of the independent parameter studied. A positive slope represents a positive main effect. In addition, the relative orientation of the average lines is representative of the interaction between the two independent process parameters. If the lines are parallel, there is no interaction. In using average lines, it is assumed that a linear relationship exists between the independent and dependent parameters. Although the results of experiment RS5 were not used in the statistical analyses, they were used to assess the adequacy of the assumption of a linear relationship between the independent and dependent parameters. This is because the parameter settings used in experiment RS5 were at an
intermediate or "0" level between the low (-) and the high (+) parameter settings (see Section 4.6). If a linear relationship did exist, then the "0" level data points should fall within the average lines.

5.3.9.1 Thickness Reduction

The reduction in thickness that the towpreg undergoes during the consolidation process is important for determining the final dimensions of a composite part consisting of many layers of consolidated towpreg. An ANOVA was performed on the thickness reduction results listed in Table 5.13 to determine the main effects and interactions of winding speed and applied load. The results of the ANOVA are summarized in Table 5.14. It is assumed that the reduction in thickness the towpreg undergoes is representative of the reduction in thickness the composite ring undergoes.

Table 5.14: ANOVA Results for Reduction in Towpreg Thickness

<table>
<thead>
<tr>
<th>Source of Variation</th>
<th>Degrees of Freedom</th>
<th>Sum of Squares</th>
<th>Mean Square</th>
<th>F-ratio</th>
<th>Significance Level</th>
<th>Estimate of Effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>speed</td>
<td>1</td>
<td>181.95</td>
<td>181.95</td>
<td>162.75</td>
<td>99.99</td>
<td>7.79</td>
</tr>
<tr>
<td>load</td>
<td>1</td>
<td>24.50</td>
<td>24.50</td>
<td>21.92</td>
<td>99.84</td>
<td>2.86</td>
</tr>
<tr>
<td>speed/load</td>
<td>1</td>
<td>1.53</td>
<td>1.53</td>
<td>1.37</td>
<td>72.45</td>
<td>0.71</td>
</tr>
<tr>
<td>Error</td>
<td>8</td>
<td>8.94</td>
<td>1.12</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The ANOVA results indicate that both winding speed and applied load affect the thickness reduction at a 99 percent significance level for the parameter ranges studied. Numerical estimates of the effects of each source of variation also are listed in Table 5.14. The estimated effect of the
winding speed is relatively large and has a positive direction (i.e., increasing winding speed increases reduction in thickness), as changing the winding speed from low to high increased the reduction in thickness by 7.8 percent. The estimated effect of the applied load, while not as substantial as winding speed, also has a positive direction, as changing the applied load from low to high increased the reduction in thickness by 2.9 percent. The estimated effect of the speed/load interaction appears negligible as it is within the standard errors for thickness reduction in experiments RS1 and RS2. The small interaction allows the thickness reduction to be estimated from either process parameter. The results from the ANOVA were confirmed using graphical representations of the thickness reduction data. Figures 5.11 and 5.12 show the effect of winding speed and applied load, respectively, on the reduction in towpreg thickness. Both figures indicate a positive effect of winding speed and applied load on the thickness reduction as the slopes of the average lines are positive. The figures also verify that the speed/load interaction is small as the average lines are almost parallel. It should be noted that a linear relationship was assumed to exist between each process parameter and the thickness reduction. This assumption is questionable as the data points corresponding to experiment RS5 (i.e., the intermediate or "0" parameter levels) do not fall within the average lines in Figure 5.11. It appears from Figure 5.11 that a non-linear relationship exists between the thickness reduction and the winding speed. One explanation for this is that, as the molten PEEK polymer is a shear-thinning fluid, the viscosity of the matrix exhibits a non-linear relationship with respect to the winding speed and
Figure 5.11: Effect of Winding Speed on Thickness of Ring Specimens
Figure 5.12: Effect of Applied Load on Thickness of Ring Specimens
subsequent shear rate (see Equation 3.7). The increase in the thickness reduction with increasing winding speed is caused by the increased shear rates at the higher winding speed which result in a relative decrease in the viscous resistance of the matrix material. The lower viscous resistance causes an increase in the matrix flow thus reducing the thickness of the towpreg. One explanation for the increase in the thickness reduction with increasing applied load is that, at the higher applied load, a larger pressure gradient is established in the nip resulting in increased matrix flow and subsequent towpreg deformation.

5.3.9.2 Fiber Content

An ANOVA was performed on the fiber content results listed in Table 5.13 to determine the main effects and interactions of winding speed and applied load. The results of the ANOVA are summarized in Table 5.15.

Table 5.15: ANOVA Results for Fiber Content

<table>
<thead>
<tr>
<th>Source of Variation</th>
<th>Degrees of Freedom</th>
<th>Sum of Squares</th>
<th>Mean Square</th>
<th>F-ratio</th>
<th>Significance Level</th>
<th>Estimate of Effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>speed</td>
<td>1</td>
<td>41.07</td>
<td>41.07</td>
<td>11.97</td>
<td>99.1</td>
<td>3.70</td>
</tr>
<tr>
<td>load</td>
<td>1</td>
<td>4.32</td>
<td>4.32</td>
<td>1.26</td>
<td>70.6</td>
<td>1.20</td>
</tr>
<tr>
<td>speed/load</td>
<td>1</td>
<td>2.08</td>
<td>2.08</td>
<td>0.61</td>
<td>54.2</td>
<td>0.83</td>
</tr>
<tr>
<td>Error</td>
<td>8</td>
<td>27.45</td>
<td>3.43</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

Assuming that continuity can be applied to the fibers in the towpreg and that there is a negligible change in towpreg width during consolidation, the fiber volume fraction of a consolidated ring should be a
linear function of the reduction in towpreg thickness. If this is true, the effects of winding speed and applied load on the fiber content should be similar to those examined for the reduction in towpreg thickness. However, the ANOVA results listed in Table 5.15 indicate that the effect of applied load is only significant at a 70 percent level for the range of applied load studied. One explanation for the relatively low effect of applied load is that the large amount of variability in the fiber volume content data at the low winding speed may have overshadowed the true effect of applied load. The is because the towpreg wandering problem discussed in Section 5.3.4 may have affected the ability of the applied load to initiate towpreg deformation (and subsequent fiber volume content increase) as the towpreg was not placed directly on the layers beneath during the compaction process. The numerical estimates for each source of variation also are listed in Table 5.15. The estimated effect of winding speed is relatively large and has a positive direction, as increasing the winding speed from low to high increased the fiber content by almost 4 volume percent. Both the estimated effects of the applied load and the speed/load interaction are within the standard errors of the fiber content measurements for experiments RS1 and RS2. The results from the ANOVA were confirmed using graphical representations of the fiber content data. Figures 5.13 and 5.14 show the effect of winding speed and applied load, respectively, on the fiber volume content of the ring specimens. Figure 5.13 verifies the positive
Figure 5.13: Effect of Winding Speed on Fiber Content of Ring Specimens
Figure 5.14: Effect of Applied Load on Fiber Content of Ring Specimens
effect of winding speed on void content as the slopes of both average lines are positive. Figure 5.14 shows the small effect of applied load on the fiber volume content as one of the average lines is almost horizontal. In both figures, the speed/load interaction appears larger than that observed in the thickness reduction analysis as the difference in the slopes of the average lines is greater. However, the slopes of the average lines may be misleading as the true average fiber volume content associated with each level may be overshadowed by the large variability in the fiber content values at the low winding speed. It also should be noted that a linear relationship was assumed to exist between each process parameter and the fiber volume content. This assumption is questionable as the data points corresponding to the intermediate or "0" parameter levels do not fall within the average lines in Figure 5.13. It appears from Figure 5.13 that a non-linear relationship exists between the fiber volume content and the winding speed. One explanation for this is that, as the molten PEEK polymer is a shear-thinning fluid, the viscosity of the matrix exhibits a non-linear relationship with respect to the winding speed and subsequent shear rate (see Equation 3.7). The increase in fiber content with increasing winding speed is caused by the increased shear rates at the higher winding speed which result in a relative decrease in the viscous resistance of the matrix material. The lower viscous resistance causes an increase in the matrix flow thus enhancing fiber compaction and distribution.
5.3.9.3 Void Content

An ANOVA was performed on the void content results listed in Table 5.13 to determine the main effects and interactions of winding speed and applied load. The results of the ANOVA are summarized in Table 5.16.

Table 5.16: ANOVA Results for Void Content

<table>
<thead>
<tr>
<th>Source of Variation</th>
<th>Degrees of Freedom</th>
<th>Sum of Squares</th>
<th>Mean Square</th>
<th>F-ratio</th>
<th>Significance Level</th>
<th>Estimate of Effect</th>
</tr>
</thead>
<tbody>
<tr>
<td>speed</td>
<td>1</td>
<td>18.01</td>
<td>18.01</td>
<td>22.42</td>
<td>99.9</td>
<td>-2.45</td>
</tr>
<tr>
<td>load</td>
<td>1</td>
<td>3.97</td>
<td>3.97</td>
<td>4.94</td>
<td>94.3</td>
<td>-1.15</td>
</tr>
<tr>
<td>speed/load</td>
<td>1</td>
<td>0.04</td>
<td>0.04</td>
<td>0.05</td>
<td>17.3</td>
<td>0.12</td>
</tr>
<tr>
<td>Error</td>
<td>8</td>
<td>6.43</td>
<td>0.80</td>
<td></td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

The ANOVA results listed in Table 5.16 indicate that winding speed affects the void content of the composite rings at a 99 percent significance level and that applied load affects the void content at a 94 percent significance for the range of winding speed and applied load studied. Numerical estimates of the effects of each source of variation also are listed in Table 5.16. The estimated effect of winding speed is relatively large and has a negative direction (i.e., increasing winding speed decreases void content), as changing the winding speed from low to high decreased the void content by 2.45 volume percent. The estimated effect of applied load, while not as substantial as winding speed, also has a negative direction, as changing the applied load from low to high decreased the void content by 1.15 volume percent. The estimated effect of the speed/load interaction appears negligible as it is within the standard error for the void content in
all of the experiments. The results from the ANOVA were confirmed using graphical representations of the void content data. Figures 5.15 and 5.16 show the effect of winding speed and applied load, respectively, on the void content of the ring specimens. Both figures indicate a negative effect of winding speed and applied load on the void content as the slopes of the average lines are negative. The figures also verify that the speed/load interaction is small as the average lines are almost parallel. It should be noted that a linear relationship was assumed to exist between each process parameter and the void content. This assumption is questionable as the data points corresponding to the intermediate or "0" parameter levels do not fall within the average lines in Figure 5.15. It appears from Figure 5.15 that a non-linear relationship exists between the void content and the winding speed. One explanation for this is that, as the molten PEEK polymer is a shear-thinning fluid, the viscosity of the matrix exhibits a non-linear relationship with respect to the winding speed and subsequent shear rate (see Equation 3.7). The reduction in void content with increasing winding speed is caused by the increased shear rates at the higher winding speed which result in a relative decrease in the viscous resistance of the matrix material. The lower viscous resistance of the matrix material aids in the transportation of entrapped voids out of the nip region. One explanation for the reduction in void content with increasing applied load is that, at the higher applied load, a larger pressure gradient is established in the nip, thus providing more energy to force the entrapped voids out of the nip region.
Figure 5.15: Effect of Winding Speed on Void Content of Ring Specimens
Figure 5.16: Effect of Applied Load on Void Content of Ring Specimens
5.3.9.4 Discussion of Parameter Interactions

In all three ANOVA result summaries, the numerical estimates for the effect of the interaction between winding speed and applied load were relatively small compared to the main effects. If the interactions were negligible, then the desired thickness, fiber volume content, or void content could be determined from just one of the process parameters (winding speed or applied load) instead of both. However, the theoretical results presented in Section 5.2 predict that the winding speed and the applied load would have a coupled effect on the final fiber volume content of the ring specimens indicating that both parameters are needed to determine the desired effect. It is believed that the main reasons for the unexpectedly small interaction between winding speed and applied load were the limited parameter ranges studied and the large variation in the experimental data at the low winding speed.

5.4 Comparison of Theoretical and Experimental Results

The theoretical predictions were compared to the results of the experimental investigation in order to assess the adequacy of the proposed on-line consolidation deformation model for describing the mechanical phenomena that occur during the on-line consolidation process. The actual verification of the deformation model involved comparing the predicted applied load to the measured applied load for each experiment. Figure 5.17 provides a graphical representation of the correlation between the theoretical (predicted) applied load and the actual (measured) applied load.
Figure 5.17: Comparison of Predicted and Measured Applied Loads for Experiments RS1 - RS5
In general, the experimental results appear to follow the trends predicted by the deformation model: the required applied load increases with increasing fiber volume content and decreases with increasing winding speed. Table 5.17 provides a quantitative assessment of the adequacy of the deformation model for predicting the applied load necessary to achieve a given final fiber volume fraction at a given winding speed.

Table 5.17: Comparison of Measured and Predicted Applied Load

<table>
<thead>
<tr>
<th>Experiment #</th>
<th>Measured Applied Load (N)</th>
<th>Predicted Applied Load (N)</th>
<th>Percent Deviation</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>89</td>
<td>131</td>
<td>-47</td>
</tr>
<tr>
<td>RS2</td>
<td>222</td>
<td>166</td>
<td>25</td>
</tr>
<tr>
<td>RS3</td>
<td>222</td>
<td>196</td>
<td>12</td>
</tr>
<tr>
<td>RS4</td>
<td>89</td>
<td>84.9</td>
<td>5</td>
</tr>
<tr>
<td>RS5</td>
<td>156</td>
<td>193</td>
<td>-24</td>
</tr>
</tbody>
</table>

The average deviation is approximately 23 percent. It is believed that the main sources of error in the predicted applied load were error due to the regression analysis and error associated with the development of the deformation model. These are discussed in detail in the following section.

5.4.1 Description of Error in the Model Predictions for Applied Load

A major source of error in the predicted applied load can be attributed to the regression analyses performed on the Kozeny constant and the thickness of the deformed towpreg. As the predicted applied load was derived from these empirically-determined model parameters, the error
inherent to the regression analysis would be carried through in the calculation of the required applied load. This is evidenced by the similar variabilities for experiments RS1 and RS2 in both the Kozeny constant and the applied load regression analyses. The regression error associated with the Kozeny constant was discussed in Section 5.2.1.2 and was attributed to the error inherent to the regression of the deformed towpreg thickness that was carried through in the regression analysis of the Kozeny constant.

Another major source of error in the predicted applied load can be attributed to the preliminary assumptions used in developing the deformation model. First, the assumption of isothermal conditions within the nip was not realistic. The temperature of the towpreg decreases as it passes through the nip region due to the heat transferred to the compaction roller [3]. This decrease in temperature would cause a decrease in the matrix viscosity thus creating more resistance to flow and deformation as the towpreg passes through the nip. If the temperature decreased to the point where the matrix in the towpreg solidified before reaching the exit of the nip where the maximum consolidation pressure occurs, then the effect of the applied load on initiating matrix flow and void removal would be greatly reduced. However, the towpreg must be solidified at the exit of the nip and beyond to prevent the elastic deformation of the fiber bed from recovering and forming voids as there is no external pressure applied to the towpreg after it leaves the nip [6]. The actual temperature profile within the nip is difficult to predict, measure, and control. A more accurate thermal representation for this process might be to assume that the matrix temperature is above the melting point when it enters the nip and decreases
with position in the nip, dropping below the melting point upon exiting the nip. However, in order to employ any thermal model into the current deformation model, the material constants must be known as functions of temperature. In addition, the effects of viscous dissipation, thermal expansion, and of pressure on the matrix viscosity were not taken into consideration in the deformation model as they were beyond the scope of this research.

Second, the assumption of well-aligned and uniformly distributed fibers was not an accurate representation of the fiber arrangement within the towpreg. Figure 5.18 shows the poor distribution of the fibers within the cross-section of the towpreg. Misalignment and nonuniform distribution of the fibers can result in bridging of the fibers (Figure 5.19) which restricts matrix flow and hinders void removal. This is because the misalignment and bridging of the fibers increases the length of the flow path the matrix must travel through during the deformation process thus requiring more time (or applied load) to achieve the same amount of deformation. The longer flow path length also would hinder void removal as the viscous matrix must travel over a longer distance in order to transport the voids out the nip entrance.

Third, transverse matrix flow may have occurred during the consolidation process due to a lack of restraint along the edges of the towpreg. Pressure gradients established across these free surfaces may have resulted in a squeezing or barreling of the towpreg as it passed through the nip. However, no noticeable change in towpreg width occurred during the on-line consolidation process indicating that the contribution of
Figure 5.18: Cross-Section of APC-2 12K AS4 Towpreg at 100X
Showing Nonuniform Distribution of Fibers
Ideal Towpreg
no voids, transversely isotropic, uniform thickness, perfectly aligned fibers

Actual Towpreg
voids present, poor distribution of fibers and matrix, nonuniform thickness, misalignment and bridging of fibers

Figure 5.19: Graphical Representation of Composite Towpreg
transverse flow to the overall towpreg deformation was small compared to that supplied by flow parallel to the fibers.

5.4.2 General Guidelines for Application of the Deformation Model

In order for the deformation model to be robust, it must be applicable to material systems and on-line consolidation mechanisms other than those studied in this research. The proposed deformation model is capable of this, provided certain guidelines are followed. As the deformation model is semi-empirical in nature, the application of the model to other material systems and consolidation mechanisms requires that several experiments be conducted using the new material and consolidation mechanism in order to obtain the database required for the regression of the empirical model parameters. Summarized below are the steps that should be taken when applying the deformation model to material systems other than APC-2 12K towpreg and to compaction rollers with a diameter other than the diameter of the compaction roller used in the research.

1) Determine the required material parameters at the processing temperature of the material.

2) Conduct several experiments with the new material and/or roller diameter to obtain empirical data for the winding speed, applied load, final fiber volume content, final towpreg thickness, and the void content.

3) Use the regression analysis techniques described in Sections 5.2.1.1 and 5.2.1.3 to obtain the correlation equations for the deformed towpreg thickness and the void content.
4) Enter the material parameters and the correlation equations for the deformed towpreg thickness and void content into the spreadsheet.

5) Use the regression analysis technique described in Section 5.2.1.2 to obtain the correlation equation for the Kozeny constant.

6) Enter the correlation equation for the Kozeny constant into the spreadsheet.

7) Verify the adequacy of the deformation model spreadsheet by comparing the predicted applied load values to the actual (measured) applied load values.

It should be mentioned that if a pressure application device other than a single compaction roller is used, such as a track-laying band, the model only will describe the deformation and flow that occur at the nip or entrance to the band contact zone. In addition if an external compaction device is used that has an entrance or nip whose geometry is other than a circular arc or if off-axis winding is employed, the geometry-dependent equations (Equations 3.1 and 3.3) used to develop the deformation model must be altered accordingly.

5.5 General Comments on the Thermoplastic Filament Winding Process with On-Line Consolidation

Comments and suggestions on the process equipment used in this research, on the ramifications of the theoretical and experimental results, and on the potential extension of the thermoplastic filament winding process with on-line consolidation to the formation of the towpreg tape are presented and discussed in this section.
5.5.1 Comments on the Filament Winding / Consolidation Equipment

The filament winding machine and on-line consolidation mechanism used in this research proved to be adequate for fabricating consolidated rings out of APC-2 towpreg for the range of winding speeds studied. The towpreg wandering problem experienced at the low winding speed can be corrected by moving the towpreg placement guide roller closer to the compaction roller. However, this would require that the hot air guns be reconfigured. The geometry and configuration of the on-line consolidation mechanism may not have been the optimal design for thermoplastic filament winding. As presented in Sections 5.2.2.1 and 5.4.2, the diameter of the compaction roller plays a key role in determining such parameters as matrix pressure, matrix flow, nip length (contact area), and residence time in the nip. Decreasing the roller diameter causes a corresponding decrease in the nip length (contact length) and thus increases void mobilization and removal and decreases the applied load required to achieve a given fiber volume fraction (deformation) because of the larger pressure gradients established within the nip. However, a smaller diameter roller also decreases the time for flow to achieve intimate contact, the time required for bonding to occur, and the time the towpreg is under consolidation pressure. Therefore, the tradeoff between void elimination, the required applied load, the minimum matrix flow time, the minimum time-at-pressure, and the minimum bonding time must be taken into consideration when designing the geometry and dimensions of the pressure application and compaction device for the desired range of winding speeds. It should be noted that for winding speeds greater than
those studied in this research, the 4,000 watt, hot air gun used to melt the incoming towpreg may have to be modified to satisfy the process heat required at the higher winding speeds. Although the hot air gun is capable of higher heat outputs using a higher air flow rate, the increase in the exit velocity of the hot air stream could cause the molten matrix to be blown off the fibers in the towpreg. A focused infrared heat source would alleviate this problem provided that proper control equipment were used.

The heat application devices (hot air guns) only were used to heat the material to the proper process temperature before it encountered the consolidation pressure. If external pressure application devices with increased contact zones are employed to allow more time for bonding or flow to occur at higher winding speeds, then heat must somehow be applied within the consolidation pressure region to prevent the material from cooling or solidifying prematurely in the contact zone. It should be mentioned that increasing the length of the contact zone for the sole purpose of allowing more time for the voids to escape would not be useful. This is because the longer flow path length and reduced pressure gradient associated with the longer contact zone would hinder void mobilization and removal once the towpreg passed through the nip (entrance to the contact zone).

5.5.2 Comments on the Theoretical and Experimental Results

The predicted decrease in the required applied load with increasing winding speed indicates that the desired fiber volume content can be achieved at even higher winding speeds when fabricating with APC-2
towpreg. Thus, there exists the capability to continuously and efficiently produce quality APC-2 composite parts by filament winding at high winding speeds. However, the maximum winding speed is generally limited by the equipment and methods used to apply the process heat as the proper process temperatures must be maintained for complete consolidation to occur. In addition, as consolidation also involves the adhesion of the towpreg being laid down with the substrate beneath, the winding speed may also be limited by the time needed for complete adhesion (or diffusion bonding) to occur at the interface between the towpreg and the substrate. Muzzy [6] reports that the time necessary for complete adhesion to occur is directly proportional to the viscosity of the polymer matrix (Equation 5.7). Using an Arrhenius expression for the zero-shear rate viscosity of PEEK (Equation 5.8) [32], the contact time required for complete adhesion of APC-2 towpreg can be expressed as a function of the matrix temperature as

\[ t_c = 22.9 \eta_0(T) \]  \hspace{1cm} (5.7)

with

\[ \eta_0(T) = 1.13 \times 10^{-16} \exp\left[\frac{19100}{T}\right] \]  \hspace{1cm} (5.8)

where \( t_c \) refers to the contact time measured in seconds, \( \eta_0 \) refers to the zero-shear rate viscosity measured in MPa·sec, and \( T \) refers to the matrix temperature measured in degrees Kelvin. For an average matrix temperature between the towpreg and substrate surfaces of 350°C, the contact time is approximately 0.05 seconds. By comparing the contact time
to the residence time in the nip, one can predict whether complete adhesion will occur during the on-line consolidation process. Table 5.18 lists the residence times determined for experiments RS1 through RS5.

Table 5.18: Nip Residence Times for Experiments RS1 - RS5

<table>
<thead>
<tr>
<th>Experiment #</th>
<th>Winding Speed (mm/sec)</th>
<th>Residence Time in Nip (sec)</th>
</tr>
</thead>
<tbody>
<tr>
<td>RS1</td>
<td>12.7</td>
<td>0.129</td>
</tr>
<tr>
<td>RS2</td>
<td>12.7</td>
<td>0.130</td>
</tr>
<tr>
<td>RS3</td>
<td>50.8</td>
<td>0.036</td>
</tr>
<tr>
<td>RS4</td>
<td>50.8</td>
<td>0.034</td>
</tr>
<tr>
<td>RS5</td>
<td>31.8</td>
<td>0.056</td>
</tr>
</tbody>
</table>

Only the ring specimens fabricated at the low winding speed had a residence time in the nip that was greater than the calculated minimum contact time for complete adhesion to occur. This would indicate that the rings fabricated at the higher winding speeds did not have sufficient time to completely bond during the on-line consolidation process. However, the towpreg remains in contact with the substrate after it leaves the nip region and bonding can occur as long as the towpreg and substrate temperatures are above the glass transition temperature of the matrix. Therefore, the time for bonding to occur would actually be from the time the melted towpreg first comes into contact with the substrate to the time at which the towpreg and substrate temperatures drop below the glass transition temperature of the matrix. It should be noted that the residence time in the nip can be increased by increasing the diameter of the roller or by adding rollers in series provided that the temperature of the composite material is
maintained above the glass transition temperature of the matrix. This increased bonding time would allow for the use of higher winding speeds.

Although the lowest void content obtained with the single-roller, on-line consolidation mechanism in the experimental investigation was not within the range of void contents desired by industry, it is believed that the pre-existing voids within the towpreg limited the minimum void content that could be achieved in the consolidated, final part. This is because the pre-existing voids were generally located in fiber-rich regions where there was insufficient matrix to mobilize and remove all the voids. However, the lowest void content obtained with the experimental set-up theoretically can be improved by increasing the deformation (fiber volume content) of the towpreg. The deformation model indicates that more deformation and subsequently a higher fiber volume content can be achieved at winding speeds higher than those studied in the experimental investigation.

While it is difficult to quantify the optimum processing conditions for filament winding APC-2 towpreg based on the experimental and theoretical results, the results indicate that increasing either the applied load, the winding speed, or both will increase the degree of consolidation and overall quality of the finished part for the range of winding speed and applied load studied. In addition, the recommended processing temperature of 400°C for APC-2 proved adequate for achieving consolidation on-line during the filament winding process. The temperature was high enough above the melting point of PEEK to reduce sufficiently the matrix viscosity and was low enough to prevent degradation of the PEEK matrix.
5.5.3 Comments on the Extension of the On-Line Consolidation Process

There are many parallels between thermoplastic filament winding and consolidated tape formation and thus there exists the possibility of extending the on-line consolidation mechanism studied in this research to the process used to form the towpreg tape. The first step in making towpreg tape is to combine the matrix with the fiber tow. There are several techniques currently used to combine the fibers with the matrix. The powder fusion coating process developed by Muzzy, et al. [33], electrostatically coats the fiber tow with the polymer matrix in powder form and then uses heat to fuse the matrix to the fibers. This technique produces a flexible towpreg that can be formed into a tape using an on-line consolidation mechanism similar to the one studied in this research. However, instead of consolidating the towpreg with a substrate, the mechanism would be used to consolidate the towpreg with itself by gathering, compressing, and bonding the flexible towpreg into a consolidated, void-free, fully impregnated tape with a uniform thickness, a uniform width, a uniform fiber distribution, and completely wet-out fibers. In addition, there exists the potential to integrate the towpreg tape formation stage with the thermoplastic filament winding process using a single, on-line consolidation mechanism. This would be very desirable from a manufacturing standpoint because less material handling and consolidation process equipment would be required.
5.6 **Summary**

Theoretical results, experimental results, and a comparison and evaluation of the two were presented in this chapter. The theoretical results were obtained from the proposed on-line consolidation deformation model and included the determination of the empirical model parameters, in addition to the model predictions for the applied load required to achieve the final fiber volume fraction in each experiment. The empirical model parameters were obtained from correlation equations determined by performing regression analyses on the results of the experimental investigation. In addition, the deformation model also employed the same process conditions and material that were used in the experimental investigation.

The experimental results were obtained from composite ring specimens filament wound at speeds of 12.7, 31.8, and 50.8 mm/sec and applied loads of 89, 156, and 222 N. Each ring consisted of 50 layers of 6 mm wide APC-2 towpreg and had an inside diameter of 178 mm. The experimental results included the results of the equipment calibration and material characterization, in addition to the results obtained from the analysis of the ring specimens. The effects of applied load and winding speed on the thickness, fiber content, and void content of the composite rings were presented and discussed through statistical analyses and graphical representations. The applied load was found to affect the thickness and the void content of the ring specimens at a 90 percent significance level for the range of applied load studied. Increasing the
applied load resulted in an increase in the deformation (reduction in thickness) and a decrease in the void content of the rings. The winding speed was found to affect the thickness, fiber volume content, and void content of the ring specimens at a 99 percent significance level. Increasing the winding speed resulted in an increase in the thickness reduction and fiber volume content and a decrease in the void content of the rings. The interaction between winding speed and applied load was found to be insignificant in all three statistical analyses. The graphical representations confirmed the results obtained from the statistical analyses.

The theoretical predictions were compared to the results of the experimental investigation in order to assess the adequacy of the proposed on-line consolidation deformation model for describing the deformation and flow phenomena that occur during the on-line consolidation process. The actual verification of the deformation model involved comparing the predicted applied load to the measured applied load required to achieve the final fiber volume content for each experiment. Both the theoretical predictions and the experimental results followed the same general trend: the required applied load increases with increasing fiber volume content and decreases with increasing winding speed. The average deviation in the applied load predictions was 23 percent. General guidelines for the application of the proposed deformation model to material systems and on-line consolidation mechanisms other than those studied in this research were presented. As the deformation model is semi-empirical in nature, the application of the model to other material systems and consolidation
mechanisms requires that several experiments be conducted using the new material and consolidation mechanism in order to obtain the empirical database required for the regression of the empirical model parameters.

In addition, comments and suggestions were made regarding the filament winding and consolidation process equipment, the ramifications of the experimental and theoretical results, and the potential application of the on-line consolidation mechanism to the process used to form towpreg tape. The filament winding machine and the on-line consolidation mechanism used in this research proved to be adequate for fabricating rings from APC-2 towpreg for the range of winding speeds studied. The design of an optimal on-line consolidation mechanism involves a tradeoff between void elimination, the required applied load, the minimum matrix flow time, the minimum time-at-pressure, and the minimum bonding time. The theoretical results indicated that even higher winding speeds are possible when fabricating with APC-2. However, a limit to the maximum winding speed was presented as being a function of the time the towpreg material had to bond to the layers beneath. In addition, the theoretical results also indicated that lower void contents are possible with the on-line consolidation mechanism used in this research. However, a limit to the minimum void content was presented as being caused by the difficulty in removing pre-existing voids within the original towpreg material. Finally, suggestions were made for the possible extension of the on-line consolidation mechanism studied in this research to the process used to form the towpreg tape.
CHAPTER 6

CONCLUSIONS and RECOMMENDATIONS

6.1 Conclusions

The major obstacle to the wide-spread usage of continuous processes for thermoplastic composites is the lack of a science-based understanding of the phenomena associated with on-line consolidation. Consolidation is a processing step in which heat and pressure are applied to the thermoplastic composite in order to force out any entrapped air and thereby compress and bond the composite laminate into a void-free, finished part. Thermoplastic filament winding is a continuous manufacturing process in which continuous fiber-reinforced thermoplastic composite material is wound onto a mandrel and consolidated on-line thus building up the desired shape in a layer-by-layer fashion. A science-based understanding of the on-line consolidation process in thermoplastic filament winding was developed. This was accomplished through a theoretical analysis and an experimental investigation.

The theoretical analysis involved the development of a one-dimensional, semi-empirical, on-line consolidation process model that describes the deformation and flow phenomena that occur during the filament winding of thermoplastic composite towpregs using a single-
roller, on-line consolidation mechanism. The basic assumptions used in the development of the deformation model were the load applied to the consolidation region is borne by the fibers and matrix, the primary deformation mechanisms are matrix flow parallel to the fibers and fiber bed compaction, and the matrix and fiber distributions are functions of the fiber volume fraction. The deformation model was used to predict the applied load necessary to achieve a desired fiber volume content of rings consisting of 6 mm wide APC-2 AS4 12K towpreg consolidated at 400°C and at various winding speeds. The model also was used to provide an estimate of the process conditions necessary to achieve a given degree of consolidation (void content).

The experimental investigation involved the fabrication, testing, and analysis of filament wound thermoplastic composite rings consolidated at a temperature of 400°C, winding speeds of 12.7, 31.8, and 50.8 mm/sec, and applied loads of 89, 156, and 222 Newtons. Each ring consisted of 50 layers of 6 mm wide APC-2 AS4 12K towpreg and had an inside diameter of 178 mm. The rings were fabricated using an on-line consolidation mechanism that consisted of electric hot air guns for applying the required heat and a single, pneumatically-controlled, compaction roller for applying the consolidation pressure. A 2² factorial design with ANOVA was used to investigate the effects of winding speed and applied load on the void content, fiber content, and final thickness of the consolidated composite ring specimens. The results of the investigation were presented and discussed through statistical analyses and graphical representations. The winding speed was found to significantly affect all three of the dependent
parameters studied. The applied load's affect on the void content and the
thickness of the ring specimens was found to be statistically significant.
The effect of the speed/load interaction was found to be statistically
insignificant.

The results of the experimental investigation were compared to the
model's predictions in order to assess the adequacy of the proposed
deformation model for describing the deformation and flow phenomena
that occur during the on-line consolidation process. The average deviation
in the applied load predictions was 23 percent. General guidelines for the
application of the proposed deformation model to material systems and on-
line consolidation mechanisms other than those studied in this research
were presented. As the deformation model is semi-empirical in nature, the
application of the model to other material systems and consolidation
mechanisms requires that several experiments be conducted using the new
material and on-line consolidation mechanism in order to obtain the
empirical database required for the regression of the empirical model
parameters.

In addition, comments and suggestions were made regarding the
filament winding and consolidation process equipment, the ramifications
of the experimental and theoretical results, and the potential application of
the on-line consolidation mechanism to the process used to form the
towpreg tape. The filament winding machine and on-line consolidation
mechanism used in this research proved to be adequate for fabricating
rings from APC-2 towpreg for the range of winding speeds studied. The
design of an optimal on-line consolidation mechanism involves a tradeoff
between between void elimination, the required applied load, the minimum matrix flow time, the minimum time-at-pressure, and the minimum bonding time. The theoretical results indicated that even higher winding speeds are possible when fabricating with APC-2. However, a limit to the maximum winding speed was presented as being a function of the time the towpreg material had to bond to the layers beneath. In addition, the theoretical results also indicated that lower void contents are possible with the on-line consolidation mechanism used in this research. However, a limit to the minimum void content was presented as being caused by the difficulty in removing pre-existing voids within the original towpreg material. Finally, suggestions were made for the possible extension of the on-line consolidation mechanism studied in this research to the process used to form the towpreg tape.

6.2 Recommendations for Future Work

Perhaps the most important recommendation for future work is to remove as much empiricism as possible from the proposed on-line consolidation model so that it can be used in industry as a predictive tool for other material systems and on-line consolidation mechanisms without having to conduct costly experiments. This entails developing comprehensive and fundamental models for determining the Kozeny constant and for describing the phenomena associated with void mobilization, elimination, and prevention. In addition, a thermal model can be extended and integrated into the deformation model allowing for the
effects of temperature on the deformation and flow phenomena to be taken into account. This is assuming that the material parameters can be determined as functions of temperature. With an improved deformation model, further research can be conducted to determine the optimum on-line consolidation mechanism design for thermoplastic filament winding without having to conduct extensive experimentation.

In addition, the quality of the thermoplastic composite ring specimens is directly related to the quality of the towpreg used to fabricate them. The nonuniform distribution of fibers and matrix and the presence of voids within the towpreg must be corrected by the supplier if industry standards for part quality are to be met using a continuous thermoplastic composites manufacturing process such as filament winding with on-line consolidation. This is because it is difficult to redistribute the matrix and the fibers as well as remove the voids in the short process times associated with continuous processes involving on-line consolidation.
REFERENCES


21. APC-2 Product Data Sheets 1 and 5, ICI Fiberite.


27. Product Data from Hercules, Inc.


29. Product Data from ICI Americas, Inc.


APPENDIX A

DEFORMATION MODEL SPREADSHEETS
FOR EXPERIMENTS RS1 - RS5
<table>
<thead>
<tr>
<th></th>
<th></th>
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<th></th>
<th></th>
<th></th>
<th></th>
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</tr>
</thead>
<tbody>
<tr>
<td>Rollradius(m)=</td>
<td>Initial Vf</td>
<td>0.00E+00</td>
<td>0.336</td>
<td>1.79E+10</td>
<td>0.00E+00</td>
<td>0.00</td>
<td></td>
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<td>Initial Towpreg</td>
<td></td>
<td>5.45E-05</td>
<td>0.345</td>
<td>1.88E+10</td>
<td>9.99E-05</td>
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<td></td>
<td></td>
</tr>
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<td>Thickness(m)=</td>
<td>Final Towpreg</td>
<td>1.09E-04</td>
<td>0.354</td>
<td>1.97E+10</td>
<td>2.05E-06</td>
<td>52</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Desired</td>
<td>Thickness(m)=</td>
<td>1.63E-04</td>
<td>0.364</td>
<td>2.06E+10</td>
<td>3.16E-06</td>
<td>92</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Final Vf = 0.556</td>
<td>Thickness(m)= 0.000144</td>
<td>2.18E-04</td>
<td>0.373</td>
<td>2.16E+10</td>
<td>4.31E-06</td>
<td>144</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Winding = 0.0127</td>
<td>Constant = 0.234</td>
<td>3.27E-04</td>
<td>0.392</td>
<td>2.34E+10</td>
<td>6.76E-06</td>
<td>297</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Speed (m/s) = 0.0127</td>
<td>Void Content (% by volume) = 6.3</td>
<td>3.81E-04</td>
<td>0.402</td>
<td>2.43E+10</td>
<td>8.06E-06</td>
<td>406</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Zero-Shear Viscosity (Pa sec) = 280</td>
<td>Niplength(m)= 5.45E-04</td>
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<tr>
<td>Viscosity (Pa sec) = 280</td>
<td>Fiber Contact</td>
<td>7.06E-04</td>
<td>0.469</td>
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<td>1.67E-07</td>
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<tr>
<td>Fiber Radius (m) = 0.000004</td>
<td>Area (m^2) = 9.81E-06</td>
<td>8.17E-04</td>
<td>0.478</td>
<td>2.74E+10</td>
<td>1.97E-07</td>
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<tr>
<td>Constant (s) = 0.04</td>
<td>Residence</td>
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<td>2.69E+10</td>
<td>2.12E-07</td>
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<tr>
<td>Viscosity dial = 0.8</td>
<td>Time in Nip (s) = 0.129</td>
<td>9.26E-04</td>
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<tr>
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<tr>
<td>Fiber Spring Constant (Pa) = 159</td>
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<td>1.04E-03</td>
<td>0.511</td>
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<td>2.53E-07</td>
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<td>Constant(Avgpress)= 1.78E+10</td>
<td>MatrixLoad (N)= 131.27</td>
<td>1.09E-03</td>
<td>0.518</td>
<td>2.18E+10</td>
<td>2.66E-07</td>
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<tr>
<td>Highest Obtainable Vf= 0.829</td>
<td>FiberLoad (N)= 1.14E-03</td>
<td>1.20E-03</td>
<td>0.513</td>
<td>1.74E+10</td>
<td>2.87E-07</td>
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<td>Tow width(m)= 0.006</td>
<td>AppliedLoad (N)= 1.25E-03</td>
<td>1.25E-03</td>
<td>0.537</td>
<td>1.49E+10</td>
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<td>RegressConsts</td>
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<td>c0= 1.78E+10</td>
<td>AppliedLoad(db)= 1.31E-03</td>
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<td>AppliedLoad(db)= 1.36E-03</td>
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<td>9.49E+09</td>
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<td>c2= -2.44E+16</td>
<td>Avgpress(MPa)= 1.42E-03</td>
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<td>0.550</td>
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<td>c3= -6.91E+19</td>
<td>Avgpress(psi)= 1.53E-03</td>
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<td>0.554</td>
<td>4.25E+09</td>
<td>3.17E-07</td>
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<td>c4= -8.67E+22</td>
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<td>0.556</td>
<td>2.04E+09</td>
<td>3.18E-07</td>
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<tr>
<td>c5= 2.84E+25</td>
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<td>1.63E-03</td>
<td>0.556</td>
<td>5.29E+08</td>
<td>3.19E-07</td>
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Deformation Model Spreadsheet for Experiment RS1
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<tr>
<th>INPUTS:</th>
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</thead>
<tbody>
<tr>
<td>Rollradius(m) = 0.0143</td>
<td>Initial Vf</td>
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<tr>
<td>Initial Towpreg = 0.335</td>
<td>0.00E+00</td>
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<tr>
<td>Thickness(m) = 0.000237</td>
<td>0.343</td>
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<tr>
<td>Desired Parameters determined from empirical database</td>
<td>2.10E+10</td>
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<tr>
<td>Final Vf = 0.56</td>
<td>0.00E+00</td>
</tr>
<tr>
<td>Winding Constant = 3.08E-04</td>
<td>1.10E-04</td>
</tr>
<tr>
<td>Speed (m/s) = 0.0127</td>
<td>2.32E+10</td>
</tr>
<tr>
<td>Zero-Shear Viscosity (Pa sec) = 280</td>
<td>2.20E-04</td>
</tr>
<tr>
<td>Fiber Radius (m) = 0.000004</td>
<td>7.16E-04</td>
</tr>
<tr>
<td>Time Constant (s) = 0.04</td>
<td>7.19E-04</td>
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<tr>
<td>Viscosity Exponent = 0.8</td>
<td>8.38E-04</td>
</tr>
<tr>
<td>Fiber Spring Constant(Pa) = 159</td>
<td>4.10E-04</td>
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<tr>
<td>Highest Obtainable Vf = 0.829</td>
<td>2.07E+10</td>
</tr>
<tr>
<td>Tow width(m) = 0.006</td>
<td>1.10E-03</td>
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<table>
<thead>
<tr>
<th>RegressConsta</th>
<th>matrix</th>
<th>fiber</th>
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<tbody>
<tr>
<td>c0 = 2.08E+10</td>
<td>7.17E+01</td>
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<tr>
<td>c1 = 2.67E+13</td>
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<tr>
<td>c2 = -3.86E+16</td>
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<tr>
<td>c3 = 1.01E+20</td>
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<tr>
<td>c4 = -1.15E+23</td>
<td>5.31E+16</td>
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<tr>
<td>c5 = 3.59E+25</td>
<td>-1.69E+19</td>
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Deformation Model Spreadsheet for Experiment RS2
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</tr>
</thead>
<tbody>
<tr>
<td>Roll radius (m) = 0.0143</td>
<td>Nip position (m) = 0.037</td>
<td>Initial Vf = 0.00E+00</td>
<td>0.306</td>
<td>1.25E+10</td>
<td>0.00E+00</td>
<td>0</td>
<td>0.00</td>
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<tr>
<td>Initial Towpreg = 0.000237</td>
<td>Final Towpreg = 1.22E-04</td>
<td>0.327</td>
<td>1.44E+10</td>
<td>1.05E+06</td>
<td>42</td>
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<tr>
<td>Thickness (m) = 0.605</td>
<td>Desired Towpreg = 2.44E-04</td>
<td>0.340</td>
<td>1.66E+10</td>
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<tr>
<td>Winding = 0.0508</td>
<td>Constant = 3.05E-04</td>
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<tr>
<td>Speed (m/s) = 0.0508</td>
<td>Void Content = 3.66E-04</td>
<td>0.362</td>
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<tr>
<td>Zero-Shear Viscosity (Pa sec) = 280</td>
<td>Naplength (m) = 6.09E-04</td>
<td>0.373</td>
<td>2.34E+10</td>
<td>1.09E+07</td>
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<tr>
<td>Fiber Radius (m) = 0.000004</td>
<td>Area (m^2) = 7.13E-04</td>
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<td>12.44</td>
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<tr>
<td>Time = 1.10E-05</td>
<td>Contact = 7.92E-04</td>
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<td>Residence = 9.14E-04</td>
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<td>1.75E+07</td>
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<tr>
<td>Viscosity Exponent = 0.8</td>
<td>Time in Nip (s) = 1.04E-03</td>
<td>0.417</td>
<td>3.10E+10</td>
<td>2.13E+07</td>
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<td>23.11</td>
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<tr>
<td>Fiber Spring Constant (Pa) = 159</td>
<td>Matrix Load (N) = 1.22E-03</td>
<td>0.428</td>
<td>3.09E+10</td>
<td>2.50E+07</td>
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<tr>
<td>Highest</td>
<td>Fiber Load (N) = 1.28E-03</td>
<td>0.439</td>
<td>2.75E+10</td>
<td>3.04E+07</td>
<td>23220</td>
<td>30.41</td>
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<tr>
<td>Obtainable Vf = 0.829</td>
<td>Dow width (m) = 0.006</td>
<td>0.450</td>
<td>2.52E+10</td>
<td>3.20E+07</td>
<td>29074</td>
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<tr>
<td>Regress Consts matrix fiber</td>
<td>Applied Load (N) = 1.40E-03</td>
<td>0.461</td>
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<td>3.34E+07</td>
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<tr>
<td>c0= 1.92E+10</td>
<td>Applied Load (lb) = 1.52E-03</td>
<td>0.472</td>
<td>1.95E+10</td>
<td>3.47E+07</td>
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<tr>
<td>c1= 3.23E+13</td>
<td>Avg press (MPa) = 1.55E-03</td>
<td>0.483</td>
<td>1.51E+10</td>
<td>3.57E+07</td>
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<tr>
<td>c2= -3.47E+16</td>
<td>Avgpress (psi) = 1.57E-03</td>
<td>0.494</td>
<td>1.12E+10</td>
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<td>c3= 178.88</td>
<td>Avgpress (psi) = 1.59E-03</td>
<td>0.505</td>
<td>7.24E+09</td>
<td>3.71E+07</td>
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<tr>
<td>c4= 2.83E+20</td>
<td>Avgpress (psi) = 1.61E-03</td>
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<td>3.74E+07</td>
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<tr>
<td>c5= 2.73E+25</td>
<td>Avgpress (psi) = 1.63E-03</td>
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<td>2.52E+09</td>
<td>3.76E+07</td>
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Deformation Model Spreadsheet for Experiment RS3
<table>
<thead>
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<th>INPUTS:</th>
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<tbody>
<tr>
<td>Roll Radius (m) = 0.0143</td>
<td>Initial Vf = 0.00E+00</td>
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<tr>
<td>Initial Tow preg</td>
<td>Final Towpreg = 1.17E-04</td>
</tr>
<tr>
<td>Thickness (m) = 0.000237</td>
<td>Thickness (m) = 0.000130</td>
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<tr>
<td>Desired</td>
<td>Final Vf = 0.585</td>
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<tr>
<td>Final Vf = 0.320</td>
<td>Winding = 0.021</td>
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<tr>
<td>Speed (m/s) = 0.0508</td>
<td>Cozney = 2.34E-04</td>
</tr>
<tr>
<td>Zero-Shear</td>
<td>Viscosity = 280</td>
</tr>
<tr>
<td>Viscosity (Pa·s) = 280</td>
<td>Fiber = 7.12E+09</td>
</tr>
<tr>
<td>Fiber Contact</td>
<td>Area (m²) = 8.17E-04</td>
</tr>
<tr>
<td>Fiber Radius (m) = 0.000004</td>
<td>Time = 1.05E-05</td>
</tr>
<tr>
<td>Constant (s) = 0.04</td>
<td>Residence = 9.34E-04</td>
</tr>
<tr>
<td>Viscosity Exponent = 0.8</td>
<td>Time in Nip (s) = 0.004</td>
</tr>
<tr>
<td>Fiber Spring Constant (Pa) = 159</td>
<td>MatrixLoad (N) = 84.92</td>
</tr>
<tr>
<td>Highest FiberLoad (N) = 1.12E-03</td>
<td>FiberLoad (N) = 1.23E-03</td>
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<tr>
<td>Obtainable Vf = 0.829</td>
<td>AppliedLoad (N) = 1.34E-03</td>
</tr>
<tr>
<td>Tow width (m) = 0.006</td>
<td>RegressConsts = matrix fiber 84.92</td>
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<table>
<thead>
<tr>
<th>Nip Position x-position (m)</th>
<th>FiberVolFract Vt(x)</th>
<th>PressGradient dP/dx (Palm)</th>
<th>MatrixPress Pm(x) (Pa)</th>
<th>FiberBedPress Pf(x) (Pa)</th>
<th>MatrixPress Pm(x) (MPa)</th>
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<tbody>
<tr>
<td>0.00E+00</td>
<td>0.320</td>
<td>7.62E+09</td>
<td>0.00E+00</td>
<td>0.00E+00</td>
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Deformation Model Spreadsheet for Experiment RS4
### Deformation Model Spreadsheet for Experiment RS5

<table>
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<td>Rollradius (m)</td>
<td>Initial Vf</td>
</tr>
<tr>
<td>Initial Towpreg</td>
<td>0.0143</td>
</tr>
<tr>
<td>Thickness (m)</td>
<td>Final Towpreg</td>
</tr>
<tr>
<td>Desired</td>
<td>Parameters determined from database</td>
</tr>
<tr>
<td>Final Vf</td>
<td>Parameters determined from database</td>
</tr>
<tr>
<td>Speed (m/s)</td>
<td>0.0318</td>
</tr>
<tr>
<td>Zero-Shear Viscosity (Pa sec)</td>
<td>280</td>
</tr>
<tr>
<td>Fiber Contact</td>
<td>Contact</td>
</tr>
<tr>
<td>Area (m^2)</td>
<td>7.07E-04</td>
</tr>
<tr>
<td>Radius (m)</td>
<td>Contact</td>
</tr>
<tr>
<td>Time</td>
<td>1.06E-05</td>
</tr>
<tr>
<td>Constant (s)</td>
<td>Residence</td>
</tr>
<tr>
<td>Viscosity Exponent</td>
<td>0.8</td>
</tr>
<tr>
<td>Fiber Spring</td>
<td>Time in Nip (s)</td>
</tr>
<tr>
<td>Constant (Pa)</td>
<td>0.000004</td>
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<tr>
<td>Highest</td>
<td>Residence</td>
</tr>
<tr>
<td>Obtainable Vf</td>
<td>0.829</td>
</tr>
<tr>
<td>Tow width (m)</td>
<td>0.000020</td>
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<tr>
<td>RegressConsts c0</td>
<td>1.56E+10</td>
</tr>
<tr>
<td>c1</td>
<td>-1.55E+07</td>
</tr>
<tr>
<td>c2</td>
<td>9.16E+10</td>
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<tr>
<td>c3</td>
<td>1.85E+14</td>
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<tr>
<td>c4</td>
<td>1.58E+17</td>
</tr>
<tr>
<td>c5</td>
<td>4.21E+19</td>
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<tr>
<td>Nip Position x-position (m)</td>
<td>Initial Vf= 0.00E+00</td>
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<tr>
<td>FiberVolFrac Vf(x)</td>
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<tr>
<td>PressGradient dP/dx (Pa/m)</td>
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</tr>
<tr>
<td>MatrixPress Pm(x) (Pa)</td>
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<tr>
<td>FiberBedPress Pf(x) (Pa)</td>
<td>19.00E+00</td>
</tr>
<tr>
<td>MatrixPress Pm(x) (MPa)</td>
<td>0.00E+00</td>
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</table>

### Parameters

- Rollradius (m): 0.0143
- Initial Towpreg: 0.318
- Thickness (m): 0.000237
- Desired Final Vf: 0.589
- Speed (m/s): 0.0318
- Zero-Shear Viscosity (Pa sec): 280
- Fiber Contact Area (m^2): 7.07E-04
- Radius (m): 1.06E-05
- Constant (s): 0.04
- Viscosity Exponent: 0.8
- Fiber Spring Time in Nip (s): 1.00E-03
- Constant (Pa): 159
- Highest Fiber Spring Load (N): 12.4E-03
- Obtainable Vf: 0.829
- Tow width (m): 0.00020
- RegressConsts c0: 1.56E+10
- c1: -1.55E+07
- c2: 9.16E+10
- c3: 1.85E+14
- c4: 1.58E+17
- c5: 4.21E+19

### Deformation Parameters

- Initial Vf: 0.00E+00
- Final Vf: 1.16E-04
- Thickness (m): 0.000237
- Final Towpreg: 0.318
- Initial Towpreg: 0.317
- Nip Length (m): 6.48E-04
- Matrix Press (Pa): 1.12E-03
- Fiber Bed Press (Pa): 192.63
- Fiber Spring Press (Pa): 1.24E-03
- Fiber Load (N): 1.30E-03
- Matrix Load (N): 1.12E-03
- Applied Load (N): 1.35E-03
- Viscosity: 1.56E+10
- Avgpress (MPa): 1.59E-03
- Avgpress (psi): 1.71E-03
- Fiber Spring Constant (Pa): 159
- Fiber Spring Load (N): 12.4E-03
- Fiber Bed Press (Pa): 192.63
- Matrix Press (Pa): 192.63
- Fiber Contact Area (m^2): 7.07E-04
- Fiber Spring Time in Nip (s): 1.00E-03
- Fiber Spring Constant (s): 0.04
- Fiber Spring Constant (Pa): 159
- Fiber Spring Highest Fiber Load (N): 1.24E-03
- Fiber Spring Obtainable Vf: 0.829
- Fiber Spring Tow width (m): 0.00020
- Fiber Spring RegressConsts c0: 1.56E+10
- c1: -1.55E+07
- c2: 9.16E+10
- c3: 1.85E+14
- c4: 1.58E+17
- c5: 4.21E+19

### Deformation Model

- Deformation Model Spreadsheet for Experiment RS5
- Deformation Parameters
- Deformation Model Spreadsheet
- Deformation Input Parameters
- Deformation Output Parameters
- Deformation Variables
- Deformation Model Parameters
- Deformation Model Results
- Deformation Model Analysis
- Deformation Model Validation
- Deformation Model Comparison
- Deformation Model Conclusion
- Deformation Model Implications
- Deformation Model Applications
- Deformation Model Limitations
- Deformation Model Future Work
- Deformation Model Acknowledgments
- Deformation Model References
APPENDIX B

PLOTS OF THE DIFFERENTIAL SCANNING CALORIMETRY
RS1-2

WT: 5.54 mg
SCAN RATE: 20.00 deg/min

PEAK FROM: 301.44
TO: 351.46
ONSET: 323.01
CAL/GRAM: 6.08

ENDO

M/CAL/SEC

0.00  60.00  120.00  180.00  240.00  300.00  360.00

CARPENTER FILE: QSAVE.D4
TEMPERATURE (C)
DATE: 01/12/14  TIME: 12:36
DSC

MAX: 337.27
RS5-2

WT: 5.48 mg
SCAN RATE: 20.00 deg/min

PEAK FROM: 321.05
TD: 347.54
ONSET: 322.95
CAL/GRAM: 1.87

MAX: 335

CARPENTER	FILE: QSAVE.DI
DATE: 91/12/14	TIME: 14:28

TEMPERATURE (C)
DSC