RAPID LAYERED MANUFACTURING
OF SHORT-FIBRE-REINFORCED PARTS

by

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A thesis submitted in conformity with the requirements for the degree of Doctor of Philosophy
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Rapid Prototyping (RP) expedites the transition from the design concept to a physical model. With RP, objects are created from thin layers, i.e., by Layered Manufacturing (LM). The most common LM technique, stereolithography, uses a liquid photopolymer selectively solidified by an ultraviolet laser.

Mechanical properties of photopolymers can be improved by the addition of short-glass-fibres. In this thesis, a novel process for Rapid Layered Composites Manufacturing (RLCM) is developed. RLCM overcomes several fabrication problems caused by the introduction of fibres into the photopolymer liquid: (1) thin layer formation from a viscous mixture; (2) fibre settling; and (3) lack of inter-layer fibre penetration. The distinguishing features of the proposed process are: (1) external fibre-resin mixing supply source; (2) precisely controlled liquid deposition from above; and (3) selective solidification by a scanning pattern with layer-to-layer interconnecting features.

To arrive at the above process synthesis, first, the basic properties of the constituent materials were experimentally investigated; second, the RLCM process, originally synthesized using the Axiomatic Design Theory, was iteratively improved upon by analysis of the fabricated parts.

The constituent materials were studied to evaluate the fibre-photopolymer interaction. The high strength of the fibre-solid photopolymer interface was verified by single-fibre pull-out tests. Fibre-liquid photopolymer interaction was observed through rheological measurements which determined the relationship between the composite liquid's viscosity and (a) the fibre concentration and (b) the fibre aspect ratio.

Axiomatic Design Theory was employed for RLCM-process synthesis. The present design resulted from several cycles of evaluation and modification based upon the Independence and Information Axioms.

RLCM process output was analyzed in terms of parts' geometric quality and mechanical properties. The geometric quality was assessed by examining individual layers. Fluid-mechanical layer-formation models were employed to interpret layer-to-layer thickness variations.
Modelling mechanical properties of the short-fibre composites requires information about the fibre orientation and length. Thus, three-dimensional fibre-orientation distribution was measured with a novel methodology where two closely spaced consecutive cross-sections are examined. The method's novelty lies in accurately calculating the transformation between the cross-sections and additionally estimating the fibre length.

Mechanical properties of layered composites are of importance since such parts may directly serve as functional prototypes. These properties were modelled employing modified rule of mixtures. The models matched well the tensile test results, which demonstrated a significant improvement (80%) in modulus with reinforcements.
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Nomenclature

**Acronyms**

ADSA-CD  Axisymmetric Drop Shape Analysis-Contact Diameter
ADSA-D   Axisymmetric Drop Shape Analysis-Diameter
ADSA-MD  Axisymmetric Drop Shape Analysis-Maximum Diameter
ADSA-P   Axisymmetric Drop Shape Analysis-Profile
AS       Allied Signal 2202SF photopolymer resin
CG       Ciba Geigy SL5170 photopolymer resin
CLD      Composite-Liquid Delivery subsystem
DP       Design Parameter
FDM      Fused Deposition Modelling
FLD      Fibre-Length Distribution
FOD      Fibre-Orientation Distribution
FR       Functional Requirement
FRM      Fibre-Resin Mixing subsystem
HC_LIR   Horizontal Composite Large In-line Rivets specimen set
HC_LOR   Horizontal Composite Large Offset Rivets specimen set
HC_NR    Horizontal Composite Non-Riveted specimen set
HC_SOR   Horizontal Composite Small Offset Rivets specimen set
HP_LIR   Horizontal Pure-resin Large In-line Rivets specimen set
HP_NR    Horizontal Pure-resin Non-Riveted specimen set
LL Liquid-Levelling subsystem
LM Layered Manufacturing
LOM Laminated Object Manufacturing
LOR Large Offset Rivets specimen
LQ_C Layer-Quality study – Composite specimen
LQ_P Layer-Quality study – Pure-resin specimen
LQ_CE Layer-Quality study – Composite Early development-stage specimen
LQ_SL_P Layer-Quality study – SL-fabricated Pure-resin specimen
NR1 Non-Riveted specimen 1
NR2 Non-Riveted specimen 2
NURBS Non-Uniform Rational B-Spline
RLCM Rapid Layered Composites Manufacturing
RP Rapid Prototyping
SD Standard Deviation
SDM Shape Deposition Manufacturing
SL Stereolithography
SLS Selective Laser Sintering
SOR Small Offset Rivets specimen
SFRP Short-Fibre-Reinforced Polymers
VC_SOR Vertical Composite Small Offset Rivets specimen set
UV Ultraviolet
Latin alphabet symbols

A  major radius of an ellipse
B  minor radius of an ellipse
c_{\phi}  dimensionless parameter derived from the fibre azimuth and misalignment angle observations
C  6x6 stiffness matrix
C_d  cure depth of a photopolymer liquid
D_p  characteristic penetration depth of a photopolymer liquid
\Delta d  depth difference between the two sections in the Z direction
E_1  composite material tensile modulus in direction “1”
E_2  composite material tensile modulus in direction “2,” transverse to layer planes
E_c  characteristic critical exposure of a photopolymer liquid
E_{\text{max}}  maximum exposure at the photopolymer liquid surface
E_f  tensile modulus of the fibres
E_m  tensile modulus of the composite matrix
Fr  Froude number
G_{12}  composite shear modulus
G_f  fibre shear modulus
G_m  matrix shear modulus
h_0  gap between the wiper blade and the substrate
$h_{nom}$ nominal layer thickness equal to the incremental downward displacement of the platform for each layer

$H_0$ liquid height in a reservoir representing liquid bulge accumulated in front of the wiper

$l$ fibre length

$l_c$ critical fibre length

$L_0$ length of the under-the-blade channel (or blade-bottom thickness)

$n_{pix}$ number of image pixels representing an ellipse radius

$N_A$ number of particles per unit area

$N_V$ number of particles per unit volume

$N_T$ total number of fibres in a specimen

$N_{\phi_i}$ all fibres with the misalignment angle $\phi_i$ in a specimen

$\hat{N}_{\phi_i}$ number of fibres observed at a misalignment angle $\phi_i$ in a specimen plane section

$N_{\phi_i, s_j}$ number of fibres in the specimen with a misalignment angle $\phi_i$ and an aspect ratio $s_j$.

$\hat{N}_{\phi_i, s_j}$ number of fibres with $\phi_i$ and $s_j$ which would have been observed in a plane section

$N_{\phi_i, \theta_j}$ actual number of fibres with misalignment $\phi_i$ and azimuth $\theta_j$

$\hat{N}_{\phi_i, \theta_j}$ observed number of fibres with $\phi_i$ and $\theta_j$
<table>
<thead>
<tr>
<th>Symbol</th>
<th>Definition</th>
</tr>
</thead>
<tbody>
<tr>
<td>$N_{\phi,\theta,s}$</td>
<td>actual number of fibres with misalignment $\phi_i$, azimuth $\theta_j$, and aspect ratio $s_k$</td>
</tr>
<tr>
<td>$p(s_j)$</td>
<td>probability of a fibre having an aspect ratio of $s_j$</td>
</tr>
<tr>
<td>$p(\phi)$</td>
<td>probability of finding fibres oriented at an angle $\phi$</td>
</tr>
<tr>
<td>$p(\theta,\phi)$</td>
<td>probability of finding fibres oriented at an angle $\phi$ and $\theta$</td>
</tr>
<tr>
<td>$p_{obs}$</td>
<td>probability of observing a particular fibre type (length or orientation)</td>
</tr>
<tr>
<td>$P_L$</td>
<td>laser power</td>
</tr>
<tr>
<td>$P_{sk}$</td>
<td>fraction of the total fibre volume occupied by fibres with the aspect ratio $s_k$</td>
</tr>
<tr>
<td>$P_{slk}$</td>
<td>fraction of the total fibre volume occupied by <em>longer</em> fibres, having aspect ratio $(s_k &gt; s_c)$</td>
</tr>
<tr>
<td>$P_{ssk}$</td>
<td>fraction of the total fibre volume occupied by <em>shorter</em> fibres, having aspect ratio $(s_k \leq s_c)$</td>
</tr>
<tr>
<td>$\Delta P$</td>
<td>pressure difference between two points in a liquid flow</td>
</tr>
<tr>
<td>$Q_d$</td>
<td>drag-driven flow component</td>
</tr>
<tr>
<td>$Q_p$</td>
<td>pressure-driven flow component</td>
</tr>
<tr>
<td>$Re$</td>
<td>Reynolds number</td>
</tr>
<tr>
<td>$\Delta r$</td>
<td>distance between the matched ellipse centres when expressed in Section 1 coordinates</td>
</tr>
<tr>
<td>$s$</td>
<td>fibre aspect ratio</td>
</tr>
<tr>
<td>$s_c$</td>
<td>critical fibre aspect ratio</td>
</tr>
<tr>
<td>$s_{max}(\phi_i,\theta_j)$</td>
<td>maximum aspect ratio possible for a fibre with misalignment angle $\phi_i$ and azimuth angle $\theta_j$</td>
</tr>
</tbody>
</table>
\( S \) 6×6 compliance matrix

\( U_0 \) wiper blade translation velocity

\( V_S \) laser-beam scanning velocity

\( W \) wiper blade width transverse to the flow direction

\( W_0 \) laser beam's \( 1/e^2 \) Gaussian half-width or the beam radius

\( W_A \) reversible work of adhesion per unit area

\( W_{obs} \) observability-correction function

\( W_{\phi,\theta} \) weighting factor correcting the observed number of fibres at \((\phi_i, \theta_j)\)

\( x_0 \) X coordinate of the ellipse centre

\( x_i \) X translation shift required to align two section images

\( \Delta x \) actual X shift of the fibre ellipse centre from Section 1 to 2 when both are projected onto the \( X_1-Y_1 \) plane

\( \Delta \tilde{x} \) X difference between the ellipse centre positions observed in their respective frames

\( y_0 \) Y coordinate of the ellipse centre

\( y_i \) Y translation shift required to align two section images

\( \Delta y \) actual Y shift of the fibre ellipse centre from Section 1 to 2 when both are projected onto the \( X_1-Y_1 \) plane

\( \Delta \tilde{y} \) Y difference between the ellipse centre positions observed in their respective frames
Greek alphabet symbols

\( \dot{\gamma} \) shear rate

\( \gamma_{sv} \) surface tension at solid-vapour interface

\( \gamma_{lv} \) surface tension at liquid-vapour interface

\( \gamma_{sl} \) surface tension at solid-liquid interface

\( \gamma_c \) critical surface tension for a liquid

\( \varepsilon_1 \) composite material strain in direction "1"

\( \varepsilon \) 6×1 strain vector

\( \eta \) liquid viscosity

\( \theta \) azimuth angle, the angle of the ellipse major axis with respect to the coordinate X-axis

\( \lambda^{(1)} \) integration constant corresponding to the location of the maximum in the velocity profile curve in the normalized coordinates for a Case I flow

\( \lambda^{(II)} \) integration constant corresponding to the location of the maximum in the velocity profile curve in the normalized coordinates for a Case II flow

\( \nu_{12} \) Poisson ratio of the composite material

\( \nu_m \) Poisson ratio of the matrix material

\( \nu_f \) Poisson ratio of the fibre material

\( \sigma \) 6×1 stress vector

\( \sigma_{mu} \) ultimate tensile strength of the composite matrix

\( \sigma_{fu} \) ultimate tensile strength of the fibres
\( \sigma_c \)  ultimate tensile strength of the composite

\( \sigma_l \)  composite material stress in direction "1"

\( \sigma_{lu} \)  composite material strength in direction "1"

\( \sigma_f \)  stress in the fibre

\( \sigma_m \)  stress in the matrix

\( \sigma_{mu} \)  matrix material tensile strength

\( \tau_{iu} \)  interfacial shear strength for fibre-matrix system

\( \nu_m \)  volume fraction of matrix in a composite

\( \nu_f \)  volume fraction of fibres in a composite

\( \phi \)  misalignment angle, the angle between the fibre axis and the cross-section plane normal

\( \phi_{bias} \)  maximum misalignment angle that would still be affected by the bias in a single-section fibre orientation observation method

\( \chi_1 \)  orientation-efficiency factor for modulus

\( \chi_2 \)  fibre-length correction factor for modulus

\( \chi_3 \)  orientation-efficiency factor for strength

\( \chi_4 \)  fibre-length correction factor for strength

\( \psi \)  rotation angle required to align two section images

\( \Omega \)  ratio \( h/h_0 \) between the final layer height and the wiper gap height
Chapter 1. Introduction

1.1 Rapid Prototyping

Over the past 10-15 years, competition has forced manufacturers to sharply reduce their product development times and bring products to market at a lower cost. The consumers also have demanded more varied and customized products, and they wanted them for the same price as the mass-market products of the 60's and 70's (Pine, 1993). This situation has created a need to significantly speed up the product development process. Ability to go quickly and inexpensively from the design concept to a physical model is an important requirement for achieving this goal. In this context, several novel prototype-fabricating techniques have been developed and some commercially implemented. These Rapid Prototyping (RP) techniques allow free-form fabrication of complex-geometry parts directly from their CAD models (Schmidt, 1994).

With the use of the RP technology, the turnaround time for a typical part can be from a few hours to a few days. Conventional prototyping, on the other hand, may take weeks or even months, depending on the method used. Pratt & Whitney, for example, reported that they made 2000 castings from RP-made patterns in 1994 at a cost of US$600,000 to US$700,000. Using conventional prototyping methods, they estimated that the costs would have been about US$7 million. Also, time savings of 70 to 90 percent were realized (Ashley, 1995).

In general, all methods of object fabrication can be separated into three categories: subtractive, additive, and compressive (Pacheco, 1993). In a subtractive process, a block of material is carved out to produce the desired shape. An additive process builds an object by joining particles or layers of raw material. A compressive process forces a semi-solid or liquid material into the desired shape, in which it is then induced to harden or solidify.

All current RP technologies are additive processes. The objects are created by building them up out of thin layers, giving rise to the term Layered Manufacturing (LM). The LM processes can be further classified by the material used, such as photopolymer, thermoplastic, or adhesive. In photopolymer-based processes, the liquid resin is solidified selectively by
exposure to a light of specific wavelength. Thermoplastic-based processes start with the solid plastic, which is melted, and then fused upon cooling. Adhesive-based processes use a binder to link successive laminae of the primary construction material.

Objects fabricated by LM technologies can be used either as prototypes or, if their material properties are acceptable, as final products. As prototypes, the LM-fabricated products can be used either for form-and-fit testing or for functional testing, where they are subjected to the same environmental conditions and loads as the final product. In the latter case, the prototype must equal or at least approach the material properties of the final product. Currently, majority of RP technologies produce parts only suitable for form-and-fit testing due to their weak mechanical properties.

In either application (as functional prototypes or as final products), the usability of the LM-fabricated parts can be greatly extended if their mechanical properties are improved. Significant research effort has been devoted to improving the mechanical properties of LM-fabricated parts. These efforts can be categorized by the product material: plastic, metal, or ceramic. This thesis will focus on improving the mechanical properties of LM-fabricated plastic parts by the introduction of reinforcing fibres into the polymer matrix.

Reinforcement of plastics by fibres has been employed successfully for over fifty years as means of improving the mechanical properties of the manufactured products (Piggott, 1981). Glass-reinforced plastics are used in a wide variety of applications, which include automotive, aerospace, consumer goods, and many other industries. The key characteristics of these materials which have led to their wide-spread usage are ease of fabrication into complex shapes, high strength-to-weight ratio, good corrosion resistance, and thermal and electrical insulation (Kelly, 1994).

The reinforcing fibres can be introduced either in a continuous (long) or discontinuous (short) form. While continuous fibres provide greater relative improvement of the mechanical properties, they also significantly complicate composite-material processing. Short-fibre composites, on the other hand, can be easily manufactured by automated, and hence more economical, methods (Mallick, 1993).

The most commonly used LM technique for the production of plastic parts is Stereolithography (SL). As a building material, it uses a liquid photosensitive resin, which is
selectively solidified by an ultraviolet (UV) laser beam. By adding short reinforcing fibres to the liquid resin, plastic parts with improved mechanical properties can be manufactured. Thus, the research area of this thesis is the development of an LM process for the production of short-fibre-reinforced polymer-based parts.

1.2 Layered Manufacturing of functional parts

As mentioned above, significant research effort has been devoted to improving the mechanical properties of LM-fabricated parts. While this thesis will concentrate on polymeric materials, work on other materials will be mentioned here briefly for completeness.

1.2.1 Metallic materials

A number of approaches are being investigated for the direct fabrication of metallic LM objects. The methods include:

- Powder sintering
- Welding-based deposition
- Shape deposition

Powder sintering approach is being implemented via a Selective Laser Sintering (SLS) process. In SLS, powders of various materials are partially melted, and thus fused together, by the scanning of a high-power laser beam. For sintering of metallic powders, the most promising approach uses two-component powders – one component with a low melting point acting as a binding element and another with a high melting point acting as a structural element (Bourell et al., 1995). When such powder is melted by the laser, a liquid is formed containing solid particles. The low-melting-point component can be either another metal or a polymer. For example, bronze-nickel mixtures were studied in (Agarwala et al., 1995, König et al., 1994), copper-solder (Pb-Sn), copper-tin, and iron-copper mixture in (Van der Schueren and Kruth, 1994), and copper-PMMA (a polymer) in (Bourell et al., 1992).

Welding-based deposition approach uses a guided welding torch for building parts in a layered fashion (Dickens et al., 1992). The accuracy at the present stage of development is
about ±0.5 mm and surface roughness is unacceptable for many cases, but the researchers claim that better weld control and appropriate cooling can overcome these limitations.

In Shape Deposition Manufacturing (SDM), each layer consists of a primary material and a support-structure material (Hartmann et al., 1994). The layers are deposited by thermal spraying or by microcasting and subsequently shaped by 5-axis machining. Finally, the layer is shot-peened to control the residual stresses. The support structure is removed by melting when the building process is complete. Good surface quality and part integrity are achieved, but the process is very expensive in terms of time and material consumed.

1.2.2 Ceramic materials

Ceramics are used in many advanced engineering applications (e.g., ceramic tools, turbine blades, engine components). Most common fabrication methods use fine ceramic powders which are formed into the desired shape by methods such as die pressing, extrusion, slip casting or injection moulding. The green body produced by these methods is subjected to high-temperature oven treatment to sinter the particles into a void-free finished product.

Layered fabrication of ceramics not only eliminates the need for special tooling but also allows creation of complex shapes which may not be feasible via conventional methods. In one approach, photopolymer is combined with ceramic powder, and the mixture is selectively cured in an SL apparatus to produce the green body (Griffith and Halloran, 1995). An alternative method is an extension of the Fused Deposition Modelling (FDM) process (see Chapter 3). The FDM process creates parts by on-demand extrusion of the plastic filament through an X-Y translating nozzle. The ceramic particles are introduced into the thermoplastic filament which is then used as a feedstock to the existing FD hardware to manufacture the green body (Yardimci et al., 1995, Agarwala et al., 1996).

1.2.3 Polymeric materials

Recent research work has targeted improvement of the mechanical properties of polymer-based parts produced by SL methods. For example, in Barlage et al., 1992, long fibres were added to the polymer matrix by stacking rings with arrays of parallel horizontal fibres stretched across, and then curing the polymer via a standard SL procedure. In another
approach (Charan et al., 1994), continuous fibres were laid out by a dedicated apparatus before curing each layer of the part. Other approaches include using solid inserts within the polymer (Reiff, 1992) or building fibre-reinforced shells around solidified resin part (Thomas, 1994).

Feasibility of fabricating glass-reinforced composites by another LM process, Laminated Object Manufacturing (LOM), has been investigated in Klosterman, 1996. In LOM process, thin sheets of material (typically paper) are stacked and bonded, and then the desired layer contours are cut out by a CO$_2$ laser. In the reported work, sheets of continuous glass-fibre and epoxy resin prepreg were used instead of paper. However, to maintain a fully automated process required nearly complete curing of the resin, which resulted in a poor layer-to-layer bonding. On the other hand, reducing the degree of the resin pre-cure improved the inter-layer adhesion, but necessitated manual processing at every layer.

Feasibility studies were also reported regarding the use of discontinuous reinforcements in the form of either 10-15 mm chopped glass fibre bundles or 55 $\mu$m diameter glass microspheres (Ogale et al., 1991, Renault and Ogale, 1992). Composite samples several-layers thick were produced by manually spreading the glass fibres over the liquid resin on each layer. Fibres were not premixed due to the very high viscosity of the resulting liquid. Improvement of mechanical properties was reported for fibre-based reinforcements, while no improvement was attained by using microspheres.

The above methods of reinforcement (especially those using medium-length or continuous fibres, as in Barlage et al., 1992, Charan et al., 1994, Ogale et al., 1991, and Renault and Ogale, 1992) are suitable for the layered manufacturing of objects with relatively simple geometric shapes. However, difficulties may arise when applying these methods to the production of objects with small-scale features, thin walls, or intricate shapes.

Thus, herein, the reinforcement of LM-fabricated complex-geometry objects by short fibres introduced directly into the photopolymer matrix is proposed.

In order to provide significant reinforcement benefits, the fibre materials must have high strength and modulus values relative to the polymeric matrix. Given the requirements of transparency (for photo-curing) and the relative cost of reinforcing fibres, the primary candidate for the reinforcement of photosensitive resins is the glass fibre (Piggott, 1981).
Beneficial effects of the reinforcements on composite’s mechanical properties derive from the transfer of stresses from the matrix to the reinforcing fibres. The degree improvement depends mainly on the fibre concentration in the composite, the fibre aspect ratio (i.e., ratio of the fibre length to its diameter), and the strength of fibre-polymer interface. Adding more fibres to the composite and increasing their aspect ratio typically raises modulus and strength of a composite.

1.3 Stereolithography and fibre reinforcement

Stereolithography was the first commercial technique developed for the rapid manufacturing of complex 3-D plastic models (Figure 1.1) (3D Systems, 1994). In the SL method, a computer-controlled UV-laser traces the cross-section of each slice on a thin layer of a photosensitive resin to form a cured solid layer via a photo-polymerization reaction.

The SL technology was chosen in this thesis as a starting point for the process design of the LM of fibre-reinforced plastic parts. The fact that the resin remains liquid at room temperature simplifies the process of adding the fibres, storing and handling the mixture, and controlling the amount of fibres added.

Figure 1.1. Stereolithographic part building process.

SL-based prototype production of non-reinforced parts involves the following steps (Jacobs, 1992):

1. The solid model of an object is designed on a CAD system. It is subsequently prepared for the SL fabrication by selecting the optimum orientation, adding supporting structures where needed, and selecting the operating parameters for the SL process.
(2) The solid model is sliced to generate the cross-sectional data.

(3) For each layer, the laser-beam scan pattern is generated. The pattern choice affects the object's surface quality, shrinkage, shape distortion, and inter-layer adhesion.

(4) Each layer is created by moving a focused UV laser beam (e.g., a He-Cd or an Argon laser) over the surface of a photosensitive resin, according to the pattern determined in the previous step. Due to the adhesive properties of the resin, a firm bond is formed between successive layers.

(5) After a layer is completed, the platform supporting the object is lowered by several millimeters into the liquid to allow the resin to quickly flow over the solidified areas. The platform is then raised up until it is one-layer thickness below the previous layer height, and a horizontal wiper blade is passed over the layer to accelerate the liquid spreading.

(6) When the object is built, it is UV or heat cured to complete the polymerization.

It is important at this point to examine the SL part fabrication process for suitability to making fibre-reinforced parts. There are two main consequences of adding short glass fibres to a liquid photopolymer: the first is the significant increase of the liquid viscosity and the second is the fibre settling.

To build layered parts, the liquid has to be spread in a thin layer over the previously solidified surface. High liquid viscosity will make this step difficult to accomplish by dipping and wiping, as done in the SL process.

Since the fibre density is more than twice that of a typical resin (2.54 g/cm$^3$ for glass vs. 1.13-1.15 g/cm$^3$ for resin), the fibres tend to sink, if left undisturbed. In the SL process, after filling the processing vat, the liquid remains practically undisturbed during the part building, which can take many hours. For spheres in liquid, the terminal sinking velocity is given by (Batchelor, 1967):

$$V = (\rho_s - \rho_l) \frac{gd^2}{18\mu}$$

(1.1)

where $g$ is the gravitational acceleration, $d$ is the sphere diameter, $\rho_s$ is the density of the sphere, $\rho_l$ is the density of the liquid, and $\mu$ is the viscosity of the liquid. Using a typical resin
viscosity of 200 cP, a fibre length of 1.5 mm, and diameter of 15 μm, and by calculating the 
diameter of the sphere of the same volume, yields a sinking rate of about 85 mm/hr. Thus, 
after several hours of building, the fibre concentration would be very low near the liquid 
surface, which is where the liquid for the new layer is taken from in the SL process.

The foregoing considerations clearly demonstrate a need for the development of a new 
process suitable for the layered manufacturing of fibre-reinforced parts.

1.4 Problem formulation

The objective of this research is the development of a novel process for rapid layered 
composites manufacturing (RLCM) of fibre-reinforced plastic parts. This entails both 
conducting in-depth scientific investigations related to the process development and building 
of an experimental LM system (Figure 1.2).

The fundamental background information for the process design must be acquired 
through a thorough analysis of the raw-material characteristics. Subsequently, any proposed 
process should be analyzed through the formation of theoretical models, process simulation, 
and experiments. These provide the necessary feedback for process improvement. This 
iterative design procedure will be followed in this thesis until a satisfactory LM process is 
achieved.

![Figure 1.2. Proposed process-design steps.](image)

1.4.1 Constituent material properties

A number of important materials research issues can be identified. While there is ample 
information on the properties of the constituent materials individually, there exists little 
available data on their interaction. Therefore, the materials research part of this thesis will
concentrate on the interaction of the two constituent components involved: the glass fibres and the photopolymer. This research is categorized into two major areas: (a) interaction of fibres and liquid photopolymer and (b) interaction of fibres and solid photopolymer. The first area requires studies of the fibre-photopolymer mixture rheology and surface thermodynamics of the photopolymer liquids. The second area requires evaluations of the fibre-solid photopolymer interface strength and surface energy of the solid photopolymers.

Since the layers are formed from a liquid material, viscosity measurements of composite liquids are of primary importance. An increase in the volume fraction or in the aspect ratio of the fibres yields improved mechanical properties of the composite. However, prior studies have shown that the viscosity of the fibre-photopolymer composite liquids increases significantly with volume fraction and aspect ratio (Chan et al., 1978, Ganani and Powell, 1985). Thus, an investigation of the composite-liquid rheological properties is required in order to establish the dependence of the liquid viscosity on the fibre volume fraction and the fibre aspect ratio.

Surface thermodynamics and capillarity determine how the liquid behaves in phenomena such as formation of a meniscus, spreading over a solid, and surface levelling. To quantify these effects, measurements of the surface energy of liquid photopolymers must be conducted.

Since the stresses in the composite are transferred from the matrix to the fibres, the interface plays a major role in determining the overall performance of a composite (Piggott, 1981). Mechanical integrity of the fibre-photopolymer interface will be evaluated by measurements of the interface shear strength.

Thermodynamic compatibility between the fibre surfaces and the matrix will improve the adhesion and will assure long-term stability of the composite by preventing water from weakening the interface (Gutowski, 1987). As part of this research, surface energy of the solidified photopolymers will be measured, since such information is not currently available in the literature.

1.4.2 Process synthesis

Process synthesis will involve, first, identification of the overall design configuration, followed by the detailed design of the process components. The synthesis of the process will
be accomplished with the assistance of a formal design theory called Axiomatic Design (Suh, 1990). This approach provides a formal structure for the evaluation of any proposed design solution. Each solution is first verified to satisfy the Independence Axiom by maintaining the independence of the design’s functional requirements. Alternative solutions satisfying the Independence Axiom are then compared in terms of their information content, and the one with the minimum content is selected according to the Information Axiom.

The process synthesis results will be tested by building a prototype RLCM system. After system construction, correct operation of all components will be experimentally verified. Also, certain process parameters common to all photopolymer-based LM processes must be experimentally established. For example, a relationship between the cure depth and the laser scan speed needs to be known in order to build good-quality layers. The relationship is established by observing the cure depth for a range of scan speeds.

To fabricate parts, the RLCM system must receive their geometrical description, which consists of part contour definitions for every layer. The contours result from slicing a volumetric representation of the part with an infinite plane corresponding to the elevation of each layer. The most common source of part data is a CAD database. Thus, conversion of the CAD database information into the layered part description in the RLCM system format must be implemented.

1.4.3 Process analysis

Process analysis will provide feedback to the synthesis step in order to improve the process design. The process will be analyzed by examining its output (i.e., the fabricated parts) in terms of two broad quality metrics: part geometry and mechanical properties.

1.4.3.1 Examination of geometric quality

Geometric quality will be evaluated by examining individual part layers. Layer formation is at the heart of any LM process: the quality of individual layers determine the quality of the whole part. Ideally, layers of uniform, consistent, and predictable thickness are desired. For a photopolymer-based LM process, the layer formation can be separated into two steps: creation of a liquid layer and selective solidification of that layer. To aid with the analysis, a fluid-
mechanics model of the liquid-layer formation step will be created. The model will then be verified by building of test objects and characterizing the quality of solidified layers by microscopic examination.

1.4.3.2 Examination of mechanical properties

Examination of mechanical properties of the RLCM-fabricated parts will comprise three components: (1) experimental evaluation of the factors affecting the composite’s mechanical properties; (2) modelling of the mechanical properties; and (3) tensile tests of the RLCM-fabricated specimens.

Factors affecting composite properties

Factors that affect the composite properties, and that are themselves affected by the fabrication method, will be examined. These factors are the fibre volume fraction, the spatial orientation of the fibres, and their average length. The volume fraction must be examined to assure that the fibre content of the fabricated parts matches that of the raw material (i.e., to assure that the process is functioning correctly). The fibre alignment along any particular direction will significantly increase the modulus and strength along that direction. Processing of the composite may cause preferential alignment of the fibres. Therefore, the fibre orientations must be evaluated in order to fully characterize the process. Fibre length mainly affects the strength and to some degree the modulus of a composite. Fibre length may be affected by fibre breakage during processing, as the glass fibres are very fragile. Therefore, the fibre length also must be evaluated for process characterization.

Modelling composite's mechanical properties

A model will provide a better understanding of the composite’s mechanical properties. The model will use as input the data from the observations described in the preceding paragraph. Another factor which must be accounted by the model is the effect of the part building orientation on the material properties. It is expected that due to the layered fabrication process the parts will exhibit anisotropic material properties. Model predictions will be compared with the tensile-test results.
Tensile tests

Tensile-test specimens will be fabricated by the RLCM prototype and tested for modulus and strength. To test for anisotropy of material properties, the specimens will be built both parallel and transverse to the layer planes.

1.5 Thesis structure

Chapter 2 first provides background information on the properties of the constituent materials of composites considered in this thesis: the glass fibres and the photopolymers. The chapter then reports on the experimental observations of such material properties as surface energy of the photopolymers, strength of the interface between the glass fibres and the solidified photopolymer, and the rheology of the short-fibre-photopolymer mixtures. The chapter concludes by presenting the results of tensile tests of mould-fabricated fibre-reinforced photopolymers, which serve to confirm the potential benefits to be derived from reinforcing layered-fabricated parts.

Chapter 3 deals with the process design itself. The chapter shows how a formal design theory, Axiomatic Design, has been applied to the RLCM process. The design is described first in terms of its major components. Next, a detailed description of the experimental prototype is provided. Also reported is the experimental work conducted to verify correct operation of the prototype's subsystems.

The following three chapters concentrate on the process analysis. Chapter 4 deals with the liquid-layer-formation process. First, rheological models of the process are described; then, experimental observations of the layer profiles for parts fabricated on the RLCM prototype are provided. The observed quality of the layer profiles supplies the necessary feedback for the RLCM process improvements.

Chapter 5 addresses the theory and experiments required to characterize the geometrical arrangement of the fibres within the layered composites. The geometry of fibre reinforcements is described in terms of the fibre-orientation and fibre-length distributions. Both are required for predicting the mechanical properties of the short-fibre composites.
Chapter 6 focuses on the mechanical properties of the layered composite parts. The chapter first provides a theoretical overview for the prediction of the mechanical properties of short-fibre composites. The expected effect of the layered structure on the directional dependence of the mechanical properties is also dealt with. Finally, the chapter reports on the tensile tests conducted on the layered composites fabricated by the RLCM prototype system and compares the experimental observations with the previously derived models.

Chapter 7 provides conclusions to the present work and some recommendations for future study.
Chapter 2. Benefits of Fibre Reinforcement

2.1 Introduction

Short-fibre-reinforced plastics combine the advantages of polymers, such as the ease of fabrication into complex shapes, with the advantages of fibres, such as high stiffness and strength to produce strong and easily fabricated parts. These benefits are derived because the matrix material is able to transfer the load to the much stiffer fibres, while at the same time separating the brittle fibres to prevent crack propagation and surface damage due to fibre-to-fibre interference (Kelly, 1973).

Due to the presence of two phases, namely matrix and reinforcements, the mechanical properties of composite materials depend on (i) the material properties of the constituent components, (ii) their interface, and (iii) their geometrical arrangement. The first two items in the above list are determined by the material properties of the components, while the last item is the result of the processing parameters involved in the manufacturing of the composite.

In light of the above considerations, the work related to the mechanical properties of finished parts can be separated into two sub-tasks: one is to establish the relationship between the finished product properties and the materials, and the other is establishing the relationship between the finished product properties and the process parameters.

This chapter will describe the research conducted in support of the first sub-task referred to above, namely determination of the material properties of the constituent components and the interface. Once this information is obtained, theoretical predictions of the composite mechanical properties may be formulated. These models can be then verified through tensile testing of the composite specimens.

The chapter begins with an overview of the properties of constituent components employed in the layered fabrication process, namely fibres and photopolymer resins. Next, the interaction of the above components will be examined through characterization of the thermodynamic surface properties of the liquid and solid forms of the photopolymeric resin; empirical observation of the adhesion strength between the fibres and the resin; and investigation of the rheological properties of fibre-resin mixtures.
2.2 Properties of constituent materials

2.2.1 Photopolymers

Polymers are very-high-molecular-weight materials, mainly composed of carbon atoms. The atoms are connected to form long chains and networks. These chains form the backbone for the structure to which hydrogen atoms, organic and inorganic groups, and radicals may be attached. Polymers are characterized by low moduli and strengths, which is due to the deformability of the networks and the sliding which can occur between the long molecular chains. They have relatively low toughness compared to ductile metals.

Photopolymers were originally developed to be used as radiation-curable coatings in the late 1960's (Pang, 1996). Being composed mainly of photoinitiators and reactive monomers, they are liquids which undergo rapid polymerization when exposed to appropriate kind of type. Each photopolymer is designed to be activated by an appropriate type of radiation, which can vary over a wide spectrum, from gamma rays and x-rays, to ultraviolet and visible light. The specific photopolymers used in this thesis (Table 2.1) were formulated to polymerize in response to the ultraviolet (UV) light, with the best sensitivity at the wavelength of 325 nm.

Table 2.1. Material properties for photopolymers used in this thesis (manufacturers’ specifications).

<table>
<thead>
<tr>
<th>Photopolymer</th>
<th>Manufacturer</th>
<th>Composition</th>
<th>Density (g/cm³)</th>
<th>Strength (MPa)</th>
<th>Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>APS 950-806</td>
<td>Applied Polymer Systems</td>
<td>Acrylate-based</td>
<td>1.08</td>
<td>62.1</td>
<td>1.12</td>
</tr>
<tr>
<td>Cibatool SL5170</td>
<td>Ciba-Geigy</td>
<td>Epoxy-based</td>
<td>1.14</td>
<td>59-60</td>
<td>2.4-2.5</td>
</tr>
<tr>
<td>Exactomer 2202SF</td>
<td>Allied Signal</td>
<td>Vinyl-ether-based</td>
<td>1.13</td>
<td>69±7</td>
<td>1.45±0.14</td>
</tr>
<tr>
<td>Somos 6110</td>
<td>Dupont</td>
<td>Epoxy-based</td>
<td>1.15</td>
<td>54.4</td>
<td>2.69</td>
</tr>
</tbody>
</table>

The basic steps of any polymerization reaction are initiation, propagation, and termination (Table 2.2). In photopolymerization, the *initiation* occurs when photons ($h\nu$) are absorbed by
the photoinitiators (I) and yield free radicals (R•), which react with the monomer molecules (M), and thus begin the formation of polymer chains. During the chain propagation, additional monomers continue to react, forming (RMMM•) chains until termination occurs, mainly due to recombination and disproportionation.

**Table 2.2. Polymerization steps for photopolymers.**

| Initiation                      | $I + h\nu \rightarrow 2R\bullet$  
|                               | $R\bullet + M \rightarrow RM\bullet$ |
| Propagation                    | $RM\_n\bullet + M \rightarrow RM\_n+1\bullet$ |
| Termination                    | $RM\_n\bullet + RM\_p\bullet \rightarrow RM\_n+p\bullet R$ (combination)  
|                               | $RM\_n\bullet + RM\_p\bullet \rightarrow RM\_n + RM\_p$ (disproportionation)  
|                               | $RM\_n\bullet + Z \rightarrow RM\_nZ$ (inhibition by inhibitor Z) |

In addition to chain formation, cross-linking occurs when monomer molecules have three or more reactive chemical groups. Cross-linking forms an insoluble network of molecules, which is important for photopolymers because it prevents polymerized molecules from redissolving into the constituent liquid monomers and gives sufficient green strength to remain structurally sound.

Of particular significance to a laser-based fabrication process is that the photopolymerization reaction is highly localized to the site of initiation, which allows selective solidification of the liquid layer. As the concentration of the unreacted monomer decreases, the rate of polymerization is quickly reduced as well. With polymerization, the viscosity of the liquid increases, which restricts the mobility of free monomer to the site of the reaction and further reduces the reaction rate.

The first type of photopolymers developed were based on acrylates (Figure 2.1), which polymerize via a free-radical mechanism. Their one drawback is that free-radical polymerization reaction is inhibited by oxygen. Thus, incomplete cure of the surfaces results when UV irradiance is insufficient, which leads to "wet" feeling surfaces and poor layer-to-layer adhesion. Another drawback is their high viscosity (800-3000 cP at 30C).
Two other types of photopolymers, recently developed for stereolithography applications, are epoxy-based and vinylether-based formulations (Figure 2.1). Both of these rely on cationic photopolymerization (Pappas, 1985). Epoxy monomers form polymer chains by ring-opening reactions in the presence of cationic photoinitiators (Watt, 1979). Such reaction produces minimal volume change because the number and types of bonds remain essentially unchanged before and after the reaction. In acrylates and vinylethers, on the other hand, one double bond is converted into two single bonds.

One advantage of cationically polymerized formulations is that they are not inhibited by ambient oxygen, unlike the acrylate-based photopolymers. Lack of oxygen inhibition at the resin surface promotes better layer-to-layer adhesion. Another advantage is that cationically polymerized resins exhibit "thermal curing." Namely, the cationic reaction continues even after the activating light exposure has ended. In the context of layered manufacturing, this leads to stronger partially built parts, better able to withstand stresses imposed by the fabrication process (e.g., new layer formation by wiping). A third advantage is the much lower viscosity of the newest cationic photopolymers, with viscosity values in the range of 200-300 cP. One disadvantage of the cationic photopolymers is that their polymerization can be inhibited by bases and water, making them sensitive to the ambient humidity levels.

![Molecular structures of three types of photopolymers.](image)

**Figure 2.1. Molecular structures of three types of photopolymers.**

### 2.2.2 Fibres

Glass fibre was selected for the reinforcement of the photopolymers based on the requirements of high strength and modulus relative to the polymeric matrix, transparency (for photo-curing) (Renault et al., 1993), and considering glass fibre’s relatively low cost (Piggott, 1981). Glass is an amorphous substance comprising mostly silica (SiO₂) and several metal oxides. For example, E-glass, a commonly used material in reinforcement applications (Table 2.3), has a composition of SiO₂ 54.4%, CaO 17.5%, Al₂O₃ 14.4%, B₂O₃ 8%, and MgO 4.5%.
Chapter 2: Benefits of Fibre Reinforcement

Table 2.3. Material properties of E-glass fibres (Owen’s Corning, 1994)

<table>
<thead>
<tr>
<th>Density (g/cm³)</th>
<th>Strength (GPa)</th>
<th>Tensile Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>2.58</td>
<td>3.4</td>
<td>72</td>
</tr>
</tbody>
</table>

Glass fibres are fabricated by fast drawing of the molten ingredients through several hundred holes of 1-2 mm diameter in a heated platinum die. The drawing process reduces the filament diameters to 10-15 μm of the finished product. Immediately upon solidification, and before being brought into contact with any surface, the filaments must be covered by a special film called “sizing” to prevent surface damage and thus avoid significant reduction in the fibre’s strength.

The glass fibre’s high sensitivity to surface damage derives from its internal structure: most of glass fibre’s strength comes from its surface (Milewski, 1987). While the fibre core consists of a random network of amorphous glass, the surface has a semi-oriented structure, which is highly compressed in the longitudinal direction, and hence gives the fibres their high tensile strength. Damaging the surface reduces this stress as well as associated fibre strength.

A second function of the sizing is enhancing the adhesion of the fibres to the polymeric matrix. This function is performed by “coupling agents,” which are usually organofunctional silanes of structure $X_3\text{Si(CH}_2\text{)}_n\text{Y}$, comprising organofunctional groups (Y), compatible with the polymeric matrix, and hydrolyzable groups (X), compatible with the glass surface (Plueddemann, 1991).

Short glass fibres are produced in two different forms: chopped and milled (Milewski, 1987). Both types are produced from continuous fibre “rovings,” which are bundles of several hundred fibres held together by the sizing (or binder) applied during the glass filament manufacture. Chopped fibres are normally of lengths from 3 up to 100 mm. Chopped fibres remain in bundles even after the cutting operation. If the bundles remain intact after incorporation into the composite, the effective aspect ratio of the fibres is significantly reduced, as each fibre bundle acts effectively as a single thicker fibre. Processing is expected to separate fibres eventually from the bundles, but the degree of separation is difficult to
control. Since the chopped fibres are produced by cutting with equally spaced knives, their lengths can be accurately controlled.

In order to obtain milled fibres, chopped fibre bundles are milled into shorter lengths and then screened to a particular desired size, which range from 0.8 to 6 mm. Milling separates fibres from the chopped fibre bundles, so that the aspect ratio is not reduced by fibre bundling. Because of randomness of the milling process and subsequent screening, the fibre lengths vary relatively widely for the milled fibres. Another factor affecting the performance of milled fibres in composites is a significant amount of debris formed during the milling process. It can represent up to a quarter of the mass of milled fibre, particularly for shorter lengths (Milewski, 1987). This debris reduces the strength improvement provided by the fibres.

In this thesis, the two types of glass fibres used were 737BD milled glass-fibres and MFX milled recycled glass-fibres. The 737BD fibres, manufactured by Owens-Corning Corp., are made from virgin E-glass with 15.8 μm diameter and a specific gravity of 2.58. The fibre surface is coated with silane sizing, which is compatible with thermoplastics. MFX fibres from Pheonix Fibreglass Inc. are made of E-glass, recycled from a blend of re-manufactured Sheet-Moulded Compound (SMC) and virgin scrap glass-fibres. This product contains 85% glass, with 7.5% calcium carbonate and 7.5% cured polyester impurities. Its fibre diameter and specific gravity are 12 to 14 μm and 2.58, respectively. MFX milled fibres are coated with a generic surface sizing suitable for both thermoplastic and thermoset materials. Both types of milled fibres are manufactured in three fibre lengths, 0.8 mm, 1.6 mm, and 3.2 mm.

2.2.3 Composites

Composites typically have greater strength, higher modulus, and greater impact strength than the unreinforced matrix. In addition, the thermal expansion is reduced, and mechanical properties are less dependent on temperature. For example, a polyester moulding compound with fibre volume fraction of 15% has thermal expansion coefficient $\alpha=14 \, \mu K^{-1}$, compared with the unreinforced polymer $\alpha=50-100 \, \mu K^{-1}$ (cf. aluminum, $\alpha=23 \, \mu K^{-1}$) (Piggott, 1981). In another example, adding 30% by volume of 4.8 mm chopped glass fibres to a thermoplastic polyester (Polybutylene Terephthalate or PBT) brings a noticeable improvement of the unreinforced matrix mechanical properties: tensile strength increases from 57 to 133 MPa,
flexural modulus increases from 2.5 to 10 GPa, and notched Izod test results increase from 0.9 to 2.3 ft-lb/in. (Owen's Corning, 1994).

There is a limit however to the amount of reinforcements that can be added to a matrix. This limit depends on the uniformity of the fibre orientation and the fibre length. For uniformly oriented fibres, the concentration can reach 50-60% by volume; for randomly oriented fibres, the limit can be 10-40%, depending on the fibre length.

2.3 Interaction of constituent components

2.3.1 Characterization of surface properties

A composite's properties are affected by the interface between the matrix and the fibres. In terms of surface thermodynamics, the photopolymeric resin and the glass fibres can be regarded as an adhesive and a substrate system. Liquid adhesives bond to surfaces by physically conforming to the surface shape on a microscopic scale. Such close contact is necessary since the forces that act to hold the matter together are the same ones which to a large extent are responsible for the strength of adhesive joints, and they act over very short ranges (3 to 10 Å) (Zisman, 1971).

However, there is a large body of empirical and theoretical evidence indicating that good wetting in its own right does not guarantee a good adhesive bond (Mittal, 1975, Berg, 1986, Gutowski, 1987). Compatibility between the adhesive and substrate in terms of surface energies also plays a significant part in determining the bond's mechanical strength. Specifically, the best bond strength can be achieved by reducing the difference between the surface energy of the adhesive (as measured in its solidified form) and the substrate. Such compatibility will improve the adhesion between the resin and the matrix. These considerations demonstrate a need for obtaining surface tension values of the resin in its solid form.

In the following sub-sections, first, general concepts of adhesion are introduced. Subsequently, the experiments conducted to determine the surface energy of photopolymeric resins in their liquid and solid forms are described.
2.3.1.1 General adhesion concepts

When a liquid is placed on a solid surface, it will either wet the solid or form a drop with a definite contact angle at the solid-liquid-vapour interface. When a drop is formed, at equilibrium, the value of the contact angle is established by the magnitude of the surface tensions on the three interfaces involved (Figure 2.2). The relationship between these parameters has been stated qualitatively by Young and algebraically by Dupré and is commonly known as the Young's Equation (Neumann and Spelt, 1996):

$$\gamma_{SV} - \gamma_{SL} = \gamma_{LV} \cos \theta$$

(2.1)

where $\gamma$ designates the surface tension at the appropriate interface, as designated by the subscripts. These are $SV$ for solid-vapour, $LV$ for liquid-vapour, and $SL$ for solid-liquid interfaces.

Dupré also defined the reversible work of adhesion per unit area of the created surface as:

$$W_A = \gamma_{SV} + \gamma_{LV} - \gamma_{SL}$$

(2.2)

Equation (2.2) does not give an estimate of work involved in separating an actual adhesive joint, since it does not take into account various other mechanisms involved in separating physical surfaces, e.g., plastic work of deformation, friction etc. By substituting (2.1) into (2.2) one can also express $W_A$ as

$$W_A = \gamma_{LV} (1 + \cos \theta).$$

(2.3)

Figure 2.2. Definition of a contact angle.

For a liquid, wetting or spreading, occurs when the contact angle is zero. If this is the case, the Young's equation is no longer applicable. The surface energy balance is then given by a spreading coefficient:

$$S = \gamma_{SV} - \gamma_{LV} - \gamma_{SL}$$

(2.4)
Chapter 2: Benefits of Fibre Reinforcement

The coefficient is positive, if spreading is thermodynamically favourable (i.e., accompanied by the decrease in free energy).

In general, liquids have surface tension values under 100 mJ/m², hard solids have surface tensions from 500 to 5000 mJ/m², and soft organic solids (e.g., waxes and polymers) have values less than 100 mJ/m² (Zisman, 1971). Therefore, spreading is expected to occur for most liquids on hard solids with high-energy surfaces (such as glass), since this is favourable by lowering the free energy of the system, as expressed by Equation (2.4). Non-spreading occurs in many cases for liquids on organic solids since their surface energies are of similar order of magnitude.

Although theoretically any non-metallic liquid should spread on a high-energy surface, this is not always the case. In some cases, adsorption of water from the atmosphere converts such high-energy surfaces as copper, silver or gold to those with a much lower effective surface tension (Bernett and Zisman, 1968). In other cases, it was found that some organic liquids did not spread on high-energy surfaces because a film was adsorbed by the solid from the liquid, which created a low-energy surface on the substrate, preventing spreading of the bulk liquid itself (Fox et al., 1955).

For a liquid, the surface tension can be found by a number of experimental procedures. These procedures depend on the Laplace Equation of Capillarity, which relates the pressure difference across an interface, $\Delta P$, to the interface curvature and surface tension $\gamma$ (Neumann and Spelt, 1996):

$$\Delta P = \gamma \left( \frac{1}{R_1} + \frac{1}{R_2} \right),$$

(2.5)

where $R_1$ and $R_2$ are the two principal radii of curvature of the surface. Some of the ways to obtain the liquid surface tension include the ring method, Wilhelmy slide method, and sessile/pendant drop method (Adamson, 1976). A recent, highly accurate procedure based on the sessile drop method and computer image analysis is described in (Rotenberg et al., 1983).

Determining the surface tension of a solid is a much more difficult task. An indirect process whereby a value closely related to the surface tension of a solid, called the critical surface tension, $\gamma_c$, has been introduced by Zisman, 1964. Zisman and co-investigators have found that when the contact angles of a series of similar liquids with varying surface tensions
are measured on the same low-energy solid, a straight line relationship can be observed between the cosine of the contact angles and the surface tension of the liquids. By extrapolating the linear relationship up to the point where the line intersects the \( \cos \theta = 1 \) line, the value of the critical tension of the solid can be found. Since for a cosine equal one the contact angle is zero, the value corresponds to the maximum liquid surface tension such that the liquid will spread on the given solid. Figure 2.3 shows examples of such plots (Zisman, 1971).

![Figure 2.3. Contact angles formed by a series of liquid n-alkanes on various fluorinated low-energy solid surfaces. A. Polytetrafluoroethylene (Teflon). B. F.E.P. Teflon. C. Polyperfluoropropylene. D. Perfluorobutyric acid monolayer. E. Perfluorocaprylic acid monolayer. F. Perfluorolauric acid monolayer. G. Poly(1,1-dihydroperfluorooctyl methacrylate) (Zisman, 1971).](image)

The linear relationship with the slope \( b \) observed in Zisman plots can be written as:

\[
\cos \theta = 1 + b(\gamma_C - \gamma_{LV}).
\]  

(2.6)

If the critical surface tension value can be regarded as a measure of the surface tension of a solid, it is possible, by performing a sufficient number of contact angle experiments to produce a relationship:

\[
\gamma_{SL} = f(\gamma_S, \gamma_L)
\]  

(2.7)

Combining this relationship with the Young's equation, it is then possible to obtain all three surface tension values. Equation (2.7) can be considered as an equation of state for the
interfacial tensions. One possible formulation for the equation of state was suggested in
(Smith et al., 1986):

\[ \gamma_{SL} = \gamma_{LV} + \gamma_{SV} - 2\sqrt{\gamma_{LV}\gamma_{SV}} e^{-\beta(\gamma_{LV} - \gamma_{SV})^2}, \]  

(2.8)

where \( \beta \) is a constant equal to 0.0001247 \((m^2/mJ)^2\), according to Li and Neumann, 1992.

2.3.1.2 Surface energy of liquid photopolymers

The objective of this investigation was to determine the surface energy for the three
available liquid photosensitive resins.

Method

A method based on observing the shape of a suspended (or pendant) drop, called
Axisymmetric Drop Shape Analysis-Profile (Rotenberg et al., 1983) (ADSA-P), has been
selected to obtain the desired surface energy values. Measurements were conducted at the
Laboratory for Applied Surface Thermodynamics, Department of Mechanical and Industrial
Engineering, University of Toronto. ADSA-P allows accurate determination of the liquid’s
surface tension by fitting a curve to a digitized image of the drop’s profile. The curve is
generated by a numerical integration of the Laplace Equation of Capillarity (2.5). The
procedure involves optimization of the curve fit by the variation of the surface tension. As
input, the program requires the density difference across the liquid interface, and the
gravitational constant.

The experimental setup consists of a solid-state CCD camera, a microscope lens
attachment, a light, a light diffuser, a syringe holder, and a tube holder. A thin Teflon tube is
attached to the syringe. The camera points at the tip of the Teflon tube, where a drop of the
liquid under test is produced so that the drop remains suspended without falling. Behind the
drop is a glass diffuser plate, with a light behind it.

Surface Tension Measurements

After taking up a quantity of sample resin into the tubing using a screw-controlled
syringe, the fluid is expelled by turning of the syringe’s control valve until a pendant drop is
formed. The drop formation can be observed on the monitor. The image of the drop is
magnified and centred, so that it occupies most of the viewing area, with the tip of the tubing just visible at the top of the screen.

For each drop, five images, at one-second intervals, were taken. For each resin, 4 different drops were observed in this manner. After the images were stored in the computer, calibration images were also acquired. These consisted of an image of a 0.025-cm rectangular grid pattern, which allows the image-analysis program to identify the image-to-world transformation, and an image of a plumb line to identify the direction of the gravitational acceleration vector on the image.

The collected images are digitized to extract the drop boundary location and processed to extract the liquid surface tension values.

Results

The names and specific gravity values of the three available resins are given in Table 2.4. These values were provided to the algorithm for the calculation of the surface energies, which produced a surface energy value for each image taken. These values were averaged for each drop, and the results are presented in Table 2.5 and in Figure 2.4. The results are highly repeatable, with the 95% confidence interval on the order of ±0.1 mJ/m².

<table>
<thead>
<tr>
<th>Resin</th>
<th>Manufacturer</th>
<th>Specific Gravity</th>
</tr>
</thead>
<tbody>
<tr>
<td>APS 950-806</td>
<td>Applied Polymer Systems</td>
<td>1.08</td>
</tr>
<tr>
<td>SL5170</td>
<td>Ciba Geigy</td>
<td>1.15</td>
</tr>
<tr>
<td>Somos 6110</td>
<td>Dupont</td>
<td>1.15</td>
</tr>
</tbody>
</table>
2.3.1.3 Surface energy of solid (cured) photopolymers

As described in the introduction to Section 2.3.1 above, the solid surface energy of the resin affects the interfacial shear strength of the composite. As this data was not readily available in the literature, experiments were conducted to determine the surface energy of the three available photopolymers in their cured, solid state.

Procedure

The method employed in this thesis for measurements of solid surface energy relies on the assumption that a general functional relationship exists between the solid, liquid, and solid-liquid interfacial energies of any liquid-solid system (Section 2.3.1.1). This relationship is referred to as the Equation of State for the Interfacial Tensions, with one possible formulation given in (2.8). Combining this relationship with a measurement of contact angle $\theta$ and the Young's Equation (2.1), it is possible to obtain all three surface tension values.

Therefore, to determine the solid surface energy, a drop of liquid with a known surface tension is placed on the solid surface under examination and the liquid's contact angle is observed. With $\gamma_{LV}$ and $\theta$ known, and by solving Equations (2.8) and (2.1), the solid surface tension as well as the interfacial tension are determined.
Table 2.5. Experimental observations of the liquid surface energy of photopolymers.

<table>
<thead>
<tr>
<th>Resin Liquid</th>
<th>Drop</th>
<th>Num. of Samples</th>
<th>Liquid Surface Energy (mJ/m²)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Average</td>
</tr>
<tr>
<td>APS 950-806</td>
<td>A</td>
<td>5</td>
<td>34.085</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>5</td>
<td>34.102</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>5</td>
<td>34.125</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>5</td>
<td>33.961</td>
</tr>
<tr>
<td></td>
<td>Overall Average</td>
<td>20</td>
<td>34.068</td>
</tr>
<tr>
<td></td>
<td>Conf. Interval (95%)</td>
<td></td>
<td>0.030</td>
</tr>
<tr>
<td>Somos 6110</td>
<td>A</td>
<td>4</td>
<td>41.700</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>5</td>
<td>42.358</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>5</td>
<td>42.450</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>5</td>
<td>42.331</td>
</tr>
<tr>
<td></td>
<td>Overall Average</td>
<td>19</td>
<td>42.236</td>
</tr>
<tr>
<td></td>
<td>Conf. Interval (95%)</td>
<td></td>
<td>0.130</td>
</tr>
<tr>
<td>SL5170</td>
<td>A</td>
<td>4</td>
<td>44.523</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>5</td>
<td>44.368</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>5</td>
<td>44.583</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>5</td>
<td>44.538</td>
</tr>
<tr>
<td></td>
<td>Overall Average</td>
<td>19</td>
<td>44.502</td>
</tr>
<tr>
<td></td>
<td>Conf. Interval (95%)</td>
<td></td>
<td>0.039</td>
</tr>
</tbody>
</table>

The procedure used to determine the liquid's contact angle is called Axisymmetric Drop Shape Analysis-Diameter (ADSA-D) (Rotenberg et al., 1983). The contact angle is measured by viewing a drop from above and determining the contact diameter of the drop. There exist two variations of this procedure, depending on whether the contact angle is less than or greater than 90°. For angles over 90°, the contact line is obscured by the drop, and therefore ADSA-MD (Maximum Diameter) procedure is used. Otherwise, ADSA-CD (Contact Diameter) procedure is employed. It has been found that drop-shape analysis utilizing a top view is more suitable for the irregularly shaped drops which often occur on rough and heterogeneous surfaces. In these cases, an average contact diameter leads to an average contact angle.
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ADSA-CD requires the contact diameter, the volume and the liquid surface tension of the drop, the density difference across the liquid-fluid interface, and the gravitational constant as inputs to calculate the contact angle by means of a numerical integration of the Laplace Equation of Capillarity (2.5).

**Specimen Preparation:** To provide solid surfaces for the experiments, a small quantity of each of the three available resins was poured onto a microscope slide (37.5x76.0 mm). The liquid formed a thin, uniform film (~0.5 mm thick), on the glass surface. Due to liquid resin’s surface tension, it did not flow beyond the edges of the glass slides. The three slides with the liquid resin (one for each resin type) were placed under the UV lamp (Blak-Ray B-100A Long-Wave UV Lamp) for approximately two hours to completely cure the resin. The surfaces formed were relatively smooth, with only slight surface irregularity, possibly due to shrinkage of the resin during curing. After preparation, the samples were stored in covered transparent plastic Petri dishes to avoid any air-born surface contamination.

**Setup:** The experimental setup, located in the Laboratory for Applied Surface Thermodynamics, Department of Mechanical and Industrial Engineering, consists of a CCD camera, a macro-lens attachment, a micro-positioning table, with two controlled axes each for tilt and position control, another micro-positioner for liquid-dispensing syringe, a digital image acquisition system, a PC, and appropriate control software. The syringe has a long thin tube, which extends to the sample’s surface, and a micrometer-like dial control, which provides a reading of the amount of the dispensed liquid with the resolution of 0.01 microlitre.

**Contact Angle Measurement:** The solid surface tension is evaluated by placing a drop of test liquid on the solid’s surface and measuring the contact angle formed by the drop. The liquid must have surface energy higher than that of the solid, otherwise complete spreading (or a very low contact angle) will result. The Equation of State (2.8) does not apply in case of zero contact angle.

An initial attempt to use distilled water as a test liquid showed that apparently there was time-dependent interaction between the water and the polymer. In order to produce thermodynamically significant contact angle reading, a state of equilibrium (or near-equilibrium) must be reached. Therefore, water was replaced by glycerol as the test liquid, which lead to the desired stable system.
Drop Dispensing: To conduct the measurements, glycerol was placed in the syringe’s reservoir. The syringe’s volume indicator reading was taken before the liquid was dispensed onto the solid’s surface and then again after the needle was withdrawn. The dispensing procedure required that a small drop be first formed on the tip of the needle. Then, the drop was slowly brought into contact with the solid resin’s surface using the micro-manipulator holding the syringe. The drop was enlarged by adding more liquid to the drop. As more liquid was added, the needle’s tip was slowly raised, while maintaining contact with the drop. After the drop had grown to a sufficient size for taking the measurement of its diameter (3-6 mm), the needle was raised and moved out of the camera’s way.

Image acquisition and digitizing: For each resin sample, four drops were measured. For each drop, seven 640×480-pixel images were captured at ten-second intervals to verify there was no time dependence in the properties of the glycerol-resin system. There exists a possibility of moisture absorption from the atmosphere by glycerol, which would change its surface energy over time\(^1\). Also, a calibration image of a circle was taken in order to provide a reference for the drop-size calculations.

In order to determine the drop diameter, its image was opened within an image processing program and eight points were marked at equal intervals around the perimeter of the drop’s image – the edge location was determined through visual examination. An image of a reference circle (diameter 5.003 mm) was also digitized in the above manner to calculate drop’s dimensions in mm.

Calculating Contact Angles: To calculate the contact angle for each drop, the following information was provided to the program implementing the algorithm: glycerol surface tension (mJ/m\(^2\)) of 65.02 mJ/m\(^2\), glycerol specific gravity of 1.2583, and gravitational constant of 980.83 cm/s\(^2\). Also, for each drop, the boundary point coordinates in pixels and drop volume in millilitres were provided (Table 2.6).

\(^1\) Private communication with Z. Policova, who assisted with the experiments.
**Table 2.6. Observed drop volumes.**

<table>
<thead>
<tr>
<th>Resin</th>
<th>Drop</th>
<th>Drop Volume (µL)</th>
</tr>
</thead>
<tbody>
<tr>
<td>APS 950-806</td>
<td>A</td>
<td>19.00</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>13.00</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>15.00</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>14.49</td>
</tr>
<tr>
<td>Somos 6110</td>
<td>A</td>
<td>9.00</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>10.00</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>10.00</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>10.00</td>
</tr>
<tr>
<td>CibaTool 5170</td>
<td>A</td>
<td>11.00</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>11.00</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>11.00</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>11.00</td>
</tr>
</tbody>
</table>

**Results**

Contact angle values obtained over one-minute interval for each drop on the three resin surfaces showed no significant time dependence (Figure 2.5) and therefore the data were averaged. Then, using the Equation of State for Interfacial Tensions (2.8), the solid surface energy of each cured resin was calculated for each drop based on the average contact angle value. The results of these calculations are shown in Table 2.7.

For APS resin, the experiment showed considerable variation between contact angles of the four drops. The angles ranged from 72.5° to 116.4°. This is a range of almost 44° compared to ranges of 1.9° and 2.3° for the Somos 6110 and the SL5170 resins, respectively. The low contact angle value for drop A on the APS resin surface was obtained after the drop was observed to spread spontaneously within a few seconds of removing the tip of the syringe. The spreading occurred in a single jump, after which an apparently new equilibrium state was reached. One possible explanation of this phenomenon is the inhomogeneity of the solidified resin’s surface. This causes a meta-stable equilibrium state to be reached as the drop is grown (i.e., while the contact line is advancing). Then, due to small vibrations of the setup, for example, caused by moving the syringe out of the way, a jump to a new equilibrium state occurred.
Figure 2.5. Contact angle observations in (a) APS, (b) Somos 6110, and (c) SL5170 photopolymeric resins.

One can compare the surface energies of the resins in solid and liquid form (Figure 2.6). There is an apparent correlation between these quantities. The surface energies of the solids are about 7-10 mJ/m$^2$ lower than those of the liquids.

2.3.1.4 Conclusions

Surface tensions of the three solidified UV-sensitive photopolymers were obtained by measuring the contact angles formed by glycerol sessile drops on the cured polymer surfaces. The solid surface tension values were calculated based on the contact angles and the Equation of State for the Interfacial Tensions. The values were found to be consistently lower than the corresponding liquid surface tension. However, the overall trend of the observations is the same for solid and liquid surface tensions.
Figure 2.6. Surface energy of photopolymers in solid and liquid forms.

Table 2.7. Solid surface energy calculations\(^1\).

<table>
<thead>
<tr>
<th>Resin</th>
<th>Drop</th>
<th>Num. of Samples</th>
<th>Contact Angle (deg)</th>
<th>(\gamma_{sv} (\text{mJ/m}^2))</th>
<th>(\gamma_{sl} (\text{mJ/m}^2))</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td>Average</td>
<td>St. Dev.</td>
<td></td>
</tr>
<tr>
<td>APS</td>
<td>A</td>
<td>7</td>
<td>72.49</td>
<td>0.19</td>
<td>34.64</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>7</td>
<td>116.36</td>
<td>0.65</td>
<td>10.54</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>6</td>
<td>99.47</td>
<td>0.15</td>
<td>19.17</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>7</td>
<td>86.14</td>
<td>0.19</td>
<td>26.71</td>
</tr>
<tr>
<td></td>
<td>Overall Average</td>
<td>27</td>
<td>93.40</td>
<td>16.81</td>
<td>22.77</td>
</tr>
<tr>
<td></td>
<td>Conf. Interval (95%)</td>
<td></td>
<td>6.22</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Somos 6110</td>
<td>A</td>
<td>7</td>
<td>72.97</td>
<td>0.19</td>
<td>34.36</td>
</tr>
<tr>
<td></td>
<td>B</td>
<td>7</td>
<td>73.88</td>
<td>0.14</td>
<td>33.82</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>7</td>
<td>74.36</td>
<td>0.15</td>
<td>33.55</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>7</td>
<td>72.51</td>
<td>0.19</td>
<td>34.62</td>
</tr>
<tr>
<td></td>
<td>Overall Average</td>
<td>28</td>
<td>73.43</td>
<td>0.76</td>
<td>34.09</td>
</tr>
<tr>
<td></td>
<td>Conf. Interval (95%)</td>
<td></td>
<td>0.28</td>
<td></td>
<td></td>
</tr>
<tr>
<td>SL5170</td>
<td>B</td>
<td>7</td>
<td>67.87</td>
<td>0.15</td>
<td>37.31</td>
</tr>
<tr>
<td></td>
<td>C</td>
<td>7</td>
<td>65.60</td>
<td>0.19</td>
<td>38.62</td>
</tr>
<tr>
<td></td>
<td>D</td>
<td>7</td>
<td>66.63</td>
<td>0.31</td>
<td>38.03</td>
</tr>
<tr>
<td></td>
<td>Overall Average</td>
<td>21</td>
<td>66.70</td>
<td>0.97</td>
<td>37.99</td>
</tr>
<tr>
<td></td>
<td>Conf. Interval (95%)</td>
<td></td>
<td>0.42</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>

2.3.2 Characterization of the Interface Bond Strength

Literature refers to a number of techniques employed to evaluate the strength of the fibre-resin interface, which is most frequently expressed in terms of the interfacial shear strength, \(\tau_{iu}\), the shear stress required to break the fibre-resin bond. These techniques include fibre pull-

\(^{1}\) Drop A for SL5170 resin and observation at 40 sec. for drop C of APS resin produced invalid observations due to experimental error.
out, microtension, fragmentation, and microcompression (Figure 2.7). Fibre pull-out (Desarmot and Favre, 1991) and microtension (Miller et al., 1987) are similar in nature, as they both involve tensile loading applied directly to the fibre. Microcompression test (Mandell et al., 1986) requires finite element analysis to estimate $\tau_{\text{int}}$, and it involves compressive loading on the fibre. Fragmentation test (Wadsworth and Spilling, 1968) evaluates the interface shear strength by embedding a single fibre in the resin matrix of a tensile test specimen and loading the specimen until the fibre breaks into fragments. The average length of these fragments is used to evaluate the interface strength. However, the test may not be feasible where the matrix is not sufficiently compliant to achieve complete fibre fragmentation.

Except for the pull-out and microtension, which load the fibre similarly, the results of these different methods are not directly comparable (Piggott, 1991). Given that composite failure process involves fibre pull-out, Piggott, 1994, states that for estimation of composite strength, the pull-out test, and not fragmentation or microcompression tests, should be used to estimate the interface shear strength. Thus, the method used in this thesis to estimate the interface strength is the fibre pull-out, with the particular implementation of the test as described in Piggott and Dai, 1991.

The $\tau_{\text{int}}$ is, for example, required in the calculation of fibre’s critical aspect ratio for the particular fibre-resin system. The critical aspect ratio is used in the estimation of the composite’s tensile strength. If the fibre aspect ratios are below this value, then the composite cannot take full advantage of the fibre’s reinforcing properties (see Section 6.2.2 in Chapter 6). Direct empirical evidence for the significance of the fibre-resin interface strength is provided in Thomason and Schoolenberg, 1994, who demonstrate a linear relationship between $\tau_{\text{int}}$ and a flexural strength of glass-fibre laminates: the flexural strength increases by a factor of two as $\tau_{\text{int}}$ varies from 3 to 15 MPa.

The pull-out test is performed by embedding one end of a single fibre into the matrix material and then pulling out the fibre using a tensile test apparatus. By obtaining the maximum pull-out force required and measuring the surface area of the embedded fibre end, the interfacial shear strength can be calculated for a particular fibre-matrix combination. The
following sections will provide background information, the description of the test methodology, and its results.

![Diagram of Fibre Reinforcement Methods]

Figure 2.7. Methods for measuring fibre-resin interface strength.

2.3.2.1 Procedure

Introduction

An important consideration in fibre pull-out tests is the length of the embedded fibre end. If a fibre is embedded beyond a certain maximum length, the fibre will break instead of being pulled out. Assuming a cylindrical fibre, the condition for pull-out can be expressed as:

\[ 2\pi RL \tau_{iu} < \sigma_{fu} \pi R^2, \]

where \( R \) is the fibre radius, \( L \) is the embedded fibre length, \( \tau_{iu} \) is the interfacial shear strength, and \( \sigma_{fu} \) is the fibre tensile strength. Thus, to avoid fibre failure, the embedded fibre length must satisfy:
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\[ L < \sigma_{fu} R / 2 \tau_{iu} . \] (2.10)

Given typical values for the parameters in (2.10), \( \sigma_{fu} = 3.4 \text{ GPa}, R = 8 \mu\text{m}, \) and \( \tau_{iu} = 40 \text{ MPa}, \) the longest possible embedded fibre length for successful pull-out is only 0.34 mm. Thus, great care must be taken to control the embedded fibre length.

Upon achieving a successful pull-out, the average interfacial shear strength can be calculated by

\[ \tau_{iu} = \frac{F_{\text{max}}}{2 \pi RL} , \] (2.11)

where \( F_{\text{max}} \) is the peak load force during the pull-out.

A pull-out test consists of the following steps:

(1) Insert the fibre filament into the liquid resin,

(2) Solidify the resin by curing, and

(3) Pull out the fibre in a tensile testing apparatus.

Materials

Two of the available resins most frequently utilized for building parts in this thesis, Ciba Geigy SL5170 and Allied Signal 2202SF, were chosen to be tested. For ease of reference, the first resin will be referred to as “CG”, and the second one as “AS” resin. The fibres were the Owen’s Corning 737BD milled glass fibres.

Specimen Preparation

One difficulty with conducting the pull-out test is the short length of the fibres. Typically, continuous strands of fibres are tested, with the fibres being cut to the required length of about 35-40 mm. However, the milled fibres used for building the layered parts were not available in continuous form with the same sizing.

In general, the following procedure is used to insert the fibres into the resin (Piggott and Dai, 1991). First, the fibres are placed inside a glass tube with a capillary hole in it, Figure 2.8. The capillary tube holds the fibres while they are inserted into the liquid resin. The glass capillary itself is held inside a cylindrical metal sleeve. The fibre to be inserted into the resin protrudes from one end of the tube by about 10 mm.
Capillary tubes containing the fibres are placed in a specially designed carrousel (Figure 2.9). The carrousel consists of two parts: the top disk, where the capillary tubes are placed, and the bottom disk, where the capsules with resin are located. The top disk has cylindrical holes around its perimeter to hold the capillary tubes, which are retained within the holes by spring action of a wire placed around the carrousel’s perimeter. The locations of holes in the bottom disk align with the holes of the top disk.

Since the capillary tubes are approximately 30 mm long while the fibres are not longer than 3-5 mm, the short fibres to be tested were glued to “carrier” filaments. Several different carriers were tested for suitability during preliminary trials. These included (a) pieces of steel wire, approximately 70 μm in diameter, (b) bundles of long glass fibres, and (c) single-strand long glass fibres. The carrier glass fibres were 22 μm in diameter. The single-strand long glass fibres were found to be most suitable for this purpose. Steel wires would frequently bend, making it impossible to insert the fibres perpendicularly to the resin surface; glass-fibre bundles made aligning the short fibre to be tested with the axis of the bundle difficult and were prone to curving as well.

To attach the short fibres to the carriers,

(1) the carrier fibre was threaded through the capillary,

(2) some cyano-acrylate (“Krazy”) glue was spread on the end of the carrier,

(3) short glass fibres were laid out on a flat surface, and

(4) the carrier fibre was brought in contact with one end of the short fibre, where the fibre was attached to the end of the carrier by the wetting action of the glue.
Figure 2.8. Glass capillary tube and metal sleeve used to hold the glass fibres.

During the specimen preparation, it was noted that a sufficient overlapping length between the carrier and the short fibre must be used to assure successful attachment of the short fibre: using overlapping lengths of less than 1-2 mm leads to short fibres falling off the carrier. To verify that the short fibres were properly attached, they were examined under an optical microscope.

The next step requires placing the resin inside the capsules. The capsules are cylindrical containers made of plastic, 12.8 mm high, 7.1 mm in diameter, and with the wall thickness of 0.9 mm (Figure 2.10). Before inserting the fibres, to reduce the amount of resin shrinkage during the curing step and to improve the curing speed, the capsules were pre-filled with the resin up to about 8 mm level and that resin was cured under an ultraviolet lamp (Blak-Ray B-100A Long-Wave UV Lamp) for about 2 hours. Next, the capsules with pre-cured resin were placed into the bottom disk of the carrousel, and the resin was again dispensed by the pipette. About 4-5 drops were placed into each capsule, until the resin formed a meniscus above the capsule’s rim. The top disk of the carrousel was attached to the bottom disk, so that the capillary tubes lined up vertically with the capsules.
To insert the fibres into the liquid resin, the carrousel is placed within the setup shown in Figure 2.9. It consists of a metal base-plate to which are attached a shaft for mounting the carrousel, a micrometer for inserting the fibres, and a microscope (not shown on the figure) for monitoring the insertion process. The shaft allows rotation of the carrousel to align the capillary tubes with an arm connected to the micrometer. This arm is used to push down on each capillary tube to insert the attached fibre into the liquid resin. By carefully observing the end of the fibre and the resin’s surface, it is possible to detect the moment when the fibre just
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touches the liquid. Then, the reading on the micrometer is noted and the dial is rotated until the desired insertion depth is reached. Based on the results of preliminary tests, embedded lengths between 0.05 and 0.15 mm were used.

The resin was cured with the same UV lamp used for pre-curing by maintaining the lamp in close proximity to resin-filled capsules for about 35-40 min. The lamp position was rotated with respect to the specimens in steps of 30-45 degrees, and the curing cycle repeated. After curing was completed, the fibres were cut so that a length of about 5-10 mm protruded from the resin surface. The capsules were then removed from the carrousel and taken to the tensile testing setup.

Tensile Test Setup

The setup consisted of a tensile testing machine, a chart recorder, and specimen-mounting hardware. The tensile testing machine was Instron table-top model equipped with a 0.5-kg loadcell. Cross-head speed was set to 0.5 mm/min. The test procedure consisted of mounting the specimen in the lower and upper grips, activating the cross-head and the chart recorder, and stopping them when the fibre pulled out of the resin or broke.

Mounting of the specimen required special hardware, which was already available at the testing facility. The mounting hardware consisted of a cylindrical capsule holder, a flat square copper plate (25×75×0.6 mm), a copper-plate holder, and a lower grip (Figure 2.11). The capsule holder is open at one end, where the capsule can be inserted and held by three set screws. The opposite end of the holder has a hook by which the holder can be suspended from the upper grip. The copper-plate holder is a square metal block with a thin slit along its upper face. The copper plate sits in the slit held by two set screws. The lower grip is mounted on wooden block, which is free to move on the horizontal table-top surface.

Tensile Tests

To test each specimen, the following procedure was followed:

1. The capsule with the resin and the fibre was inserted into the capsule-holder, the set screws on the holder were tightened, and the capsule holder was suspended on the upper grip.
(2) The longer edge of the copper plate was inserted into the slit on the plate holder and the two set screws were tightened.

(3) The holder with the copper plate was placed between the faces of the lower grip and the screw of the grip was tightened.

(4) The cross-head of the testing machine was lowered until the distance between the upper edge of the copper plate and the surface of the resin within the capsule was less than 1 mm.

(5) The lower grip was then shifted on the table until the surface of the copper plate facing the experimenter touched the fibre. Subsequent fibres tested were attached to the edge of the copper plate at intervals of 3-4 mm.

Figure 2.11. Specimen-mounting hardware.

(6) The fibre was then glued to the copper plate’s surface by the application of a cyanoacrylate (“Krazy”) glue. The glue was applied by placing a bead of the liquid adhesive onto the curved end of a metal paper clip and spreading it on the plate’s surface with one or two up-and-down strokes. A thin layer of the glue is desirable to minimize the drying time. Typically, about one minute was required for the glue to dry.

(7) After the glue had dried, the test was started by activating the strip chart recorder and the cross-head.
(8) The cross-head and the chart recorder were stopped when the sharp reduction of the force reading indicated that the fibre either pulled out or broke.

After the pull-out, the maximum pull-out force was obtained by examining the chart recorder output, Figure 2.12. The figure shows an example of a force-displacement plot from a successful pull-out test. The curve features an elastic extension region followed by quick drop in load as the interfacial bond is broken. The pull-out region is due to the frictional forces exerted by the matrix as the fibre is sliding out. The force is proportional to the area, causing an approximately linearly decreasing load reading.

The diameter of each pulled out fibre and its embedded length were measured with a calibrated viewfinder on an optical microscope (measurement error ±0.3 μm). The embedded length could be determined by measuring the distance between the pulled out end of the fibre attached to the copper plate and a characteristic “ring” left by the meniscus, which surrounded the fibre prior to resin curing (Figure 2.13). Based on these two measurements, the embedded fibre area is obtained as the surface area of a cylinder (without the ends). Note that the embedded length could not be measured for fibres broken during the pull-out attempt since the embedded fibre segment could not be extracted from the resin.

![Figure 2.12. Characteristic force-displacement curve in a fibre pull-out test.](image-url)
2.3.2.2 Results

Four batches of specimens were prepared for the CG resin and two batches for AS resin. The test results for the successfully pulled out fibres are given in Table 2.8 for CG and in Table 2.9 for AS. The results are listed in order of increasing embedded area, with the batch indicated by the first number in the specimen ID. Out of 38 fibres tested with CG resin, about 40% pulled out successfully. The corresponding number for the AS resin is 60% out of 17 fibres tested.

Table 2.8. Pull-out test results for CG resin (SL5170) and 737BD fibres.

<table>
<thead>
<tr>
<th>Used?</th>
<th>Sp. ID</th>
<th>Fibre Diameter (μm)</th>
<th>Embedded Length (μm)</th>
<th>Embedded Area (μm²)</th>
<th>Peak Load (g)</th>
<th>Peak Force (mN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>X</td>
<td>4-5w</td>
<td>18.1</td>
<td>18.1</td>
<td>1024</td>
<td>3.75</td>
<td>37</td>
</tr>
<tr>
<td>X</td>
<td>3-20</td>
<td>15.2</td>
<td>25.4</td>
<td>1210</td>
<td>3.1</td>
<td>30</td>
</tr>
<tr>
<td>X</td>
<td>3-19</td>
<td>17.8</td>
<td>24.9</td>
<td>1389</td>
<td>14.3</td>
<td>141</td>
</tr>
<tr>
<td>X</td>
<td>3-14</td>
<td>14.1</td>
<td>33.0</td>
<td>1463</td>
<td>11.1</td>
<td>109</td>
</tr>
<tr>
<td>√</td>
<td>1-1</td>
<td>17.3</td>
<td>35.1</td>
<td>1902</td>
<td>6.0</td>
<td>59</td>
</tr>
<tr>
<td>√</td>
<td>1-9</td>
<td>16.2</td>
<td>42.9</td>
<td>2186</td>
<td>5.5</td>
<td>54</td>
</tr>
<tr>
<td>√</td>
<td>4-6w</td>
<td>17.3</td>
<td>41.1</td>
<td>2228</td>
<td>11.25</td>
<td>110</td>
</tr>
<tr>
<td>√</td>
<td>2-11w</td>
<td>14.9</td>
<td>51.5</td>
<td>2414</td>
<td>9.0</td>
<td>88</td>
</tr>
<tr>
<td>√</td>
<td>1-5</td>
<td>13.1</td>
<td>79.0</td>
<td>3247</td>
<td>11.0</td>
<td>108</td>
</tr>
<tr>
<td>√</td>
<td>3-21</td>
<td>15.4</td>
<td>99.4</td>
<td>4820</td>
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<td>2-4w</td>
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<td>5138</td>
<td>20.5</td>
<td>201</td>
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<tr>
<td>√</td>
<td>3-1</td>
<td>17.0</td>
<td>136.8</td>
<td>7309</td>
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<td>285</td>
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<tr>
<td>√</td>
<td>1-6</td>
<td>17.8</td>
<td>137.6</td>
<td>7690</td>
<td>29.5</td>
<td>289</td>
</tr>
<tr>
<td>√</td>
<td>1-3</td>
<td>17.0</td>
<td>144.1</td>
<td>7701</td>
<td>30.0</td>
<td>294</td>
</tr>
</tbody>
</table>
Table 2.9. Pull-out test results for AS resin (2202SF) and 737BD fibres.

<table>
<thead>
<tr>
<th>Used?</th>
<th>Sp. ID</th>
<th>Fibre Diameter (µm)</th>
<th>Embedded Length (µm)</th>
<th>Embedded Area (µm²)</th>
<th>Peak Load (g)</th>
<th>Peak Force (mN)</th>
</tr>
</thead>
<tbody>
<tr>
<td>✓</td>
<td>4-15b</td>
<td>17.8</td>
<td>30.3</td>
<td>1696</td>
<td>5.0</td>
<td>49</td>
</tr>
<tr>
<td>✓</td>
<td>2-2b</td>
<td>12.8</td>
<td>85.0</td>
<td>3424</td>
<td>10.2</td>
<td>101</td>
</tr>
<tr>
<td>✓</td>
<td>2-3b</td>
<td>17.3</td>
<td>68.5</td>
<td>3718</td>
<td>10.2</td>
<td>101</td>
</tr>
<tr>
<td>✓</td>
<td>4-12b</td>
<td>17.5</td>
<td>68.3</td>
<td>3760</td>
<td>14.4</td>
<td>141</td>
</tr>
<tr>
<td>✓</td>
<td>2-5b</td>
<td>15.2</td>
<td>91.0</td>
<td>4340</td>
<td>13.1</td>
<td>129</td>
</tr>
<tr>
<td>✗</td>
<td>4-11b</td>
<td>16.0</td>
<td>116.7</td>
<td>5850</td>
<td>26.9</td>
<td>264</td>
</tr>
<tr>
<td>✓</td>
<td>2-10b</td>
<td>18.8</td>
<td>103.3</td>
<td>6115</td>
<td>18.9</td>
<td>185</td>
</tr>
<tr>
<td>✓</td>
<td>4-18b</td>
<td>16.0</td>
<td>166.1</td>
<td>8328</td>
<td>27.5</td>
<td>270</td>
</tr>
<tr>
<td>✓</td>
<td>2-6b</td>
<td>17.3</td>
<td>158.8</td>
<td>8614</td>
<td>27.5</td>
<td>269</td>
</tr>
</tbody>
</table>

Instead of using Equation (2.11), a more accurate way of obtaining an estimate of interfacial shear strength is by plotting the peak pull-out force against the embedded surface area (Chua and Piggott, 1985). Theoretically, a straight-line plot should result, with a slope equal to the shear strength of the interface and with the line passing through the origin. The data is plotted in Figure 2.14. An interfacial shear strength value of 40±7 MPa was obtained for glass fibres embedded in CG resin, compared with shear strength of 32±5 MPa for AS resin. In order to obtain these values, an outlier point (4-11b) (determined per Lipson and Sheth, 1973) and observations for fibres with embedded lengths approaching one fibre diameter were discarded (denoted by “✗” marks in first column of both tables). The short embedded-length points were eliminated since the stress concentrations near the resin surface and at the embedded end of the fibre are likely to affect the results (Chua and Piggott, 1985). Unreliability of these results was confirmed in these tests by very wide data scatter observed. Statistical comparison test showed the interfacial shear strength for CG resin is greater than that for AS resin at 5% level of significance.

To estimate the critical aspect ratio based on the interfacial shear strength observations, the following relationship is used (Piggott, 1981):

\[
s_c = \sigma_{fu} / 2\tau_{iu}.
\]  

(2.12)
This expression gives critical aspect ratios of 42.5 for the CG resin and 52.3 for the AS resin. Given a fibre diameter of 16 μm, these ratios translate to critical fibre lengths of 0.68 and 0.84 mm for CG and AS resins, respectively. These calculations were made based on the fibre strength $\sigma_{fu}$ of 3.4 GPa (Table 2.3).

![Figure 2.14. Fibre pull-out test results.](image)

2.3.2.3 Conclusions

Comparing the interfacial shear strength observations for glass fibres and photopolymeric resins with those for other thermoset plastics (Desarmot and Favre, 1991) indicates existence of a good bond. CG resin demonstrated bond strength about 25% greater than the AS resin. For the fibres used in the study, critical fibre lengths of 0.68 mm for CG resin and 0.84 mm AS resin were obtained.
2.3.3 Characterization of the rheological properties of composite liquid

2.3.3.1 Introduction

In a composite, increasing the fibre volume fraction or fibre aspect ratio yields improved mechanical properties. However, prior studies have shown that the viscosity of the fibre-photopolymer composite liquids increases significantly with volume fraction and aspect ratio (Chan et al., 1978, Ganami and Powell, 1985). Increased viscosity will affect the layer-formation and the post-processing steps in any layered-manufacturing process utilizing polymer-based composite liquids as raw materials (Ogale et al., 1992).

As has been pointed out in (Renap and Kruth, 1995), in any SL-based process, the depth of the liquid layer formed by the wiper is dependent on such factors as blade thickness, presence of the entrapped liquid volumes, blade traversal speed and also on the viscosity of the resin. In addition, the time required for the surface of the composite liquid layer to settle depends on its viscosity.

Thus, the objective of this investigation was to assess the effects of aspect ratio, volume fraction, and surface coating on the viscosity of the glass fibre and photopolymer composite liquids. The results of the experimental viscosity analyses provided the fundamental knowledge needed for the development of any composite-polymer-based layered manufacturing processes. Specifically, the experimental results helped with the selection of suitable operating parameters for the experimental system described in Chapter 3.

2.3.3.2 Viscosity measurement techniques for fibre suspensions

Rheological science background

Fibre-photopolymer composite liquids, which are the subject of this study, can be classified as suspensions. Suspension is a mixture of a liquid phase and solid particles, where the particles are dispersed within the liquid. Fibre suspensions can be further classified according to their concentration as dilute, semi-concentrated, and concentrated (Doi and

---

1 This research was conducted by A. Y. F. Chan under the author's direction. See Chan, 1995, for more details.
Edwards, 1986). The concentration of fibre suspensions is described by a "number" density of the dispersed phase, \( n \), as \#/mm\(^3\):

\[
n = \frac{4v_f}{\pi d^2},
\]

(2.13)

where \( l \), \( d \), and \( v_f \) are the length, the diameter, and the volume fraction of the dispersed fibres, respectively. A fibre suspension is considered dilute when \( n < 1/l^2 \), semi-concentrated when \( (1/l^2 < n < 1/d^2) \), and concentrated when \( n > 1/d^2 \).

Study of the rheological behaviour of suspensions is complicated by the presence of two phases. Numerous uncertainties arise from the effects of the distribution and shape of the dispersed phase as well as the interface between the two phases. When the dispersed particles are fibres, the rheological properties are even less well understood than for the low-aspect-ratio particle suspensions. The main reason for this is that the rheological behaviour of the fibre-suspension system is affected by the particle orientation, in addition to other factors. The complex particle interactions make the theoretical predictions for such suspensions extremely difficult.

Due to the aforementioned fibre-orientation effect, dilute suspensions with freely tumbling particles usually have to be treated separately from semi-concentrated and concentrated ones. In the past, a number of well-known formulations (Ziegel, 1970, Dinh and Armstrong, 1984, Shaqfeh and Fredrickson, 1990) have been proposed for dilute and semi-concentrated systems. However, only empirical formulations have been developed for estimating the rheological properties of concentrated suspensions (Utracki, 1988).

Using Equation (2.13) and the typical dimensions of the short reinforcing fibres (0.8–3.2 mm), the suspension can be noted to be in the concentrated regime for any fibre volume fraction above 0.6%. In the present case, fibre suspensions are expected to have volume fractions of at least 5%, in order to achieve worthwhile improvements in the composite's properties.

During past experimental studies (Chan et al., 1978, Maschmeyer and Hill, 1974, Han and Lem, 1983, Crowson and Folkes, 1980), certain common phenomena were observed regarding the effects of the volume fraction and the aspect ratio of fibres on viscosity. Firstly, shear thinning (i.e., decreasing viscosity with increasing shear rate) was observed in all
concentrated fibre suspensions. Secondly, it was noted that the effects of volume fraction and aspect ratio on viscosity of fibres are more pronounced at low shear rates.

**Selection of experimental equipment**

Viscosity measurement techniques include capillary, slit, rotational, and oscillatory rheometries (Collyer and Clegg, 1988). In the present case, a number of factors were considered when selecting the appropriate viscosity measurement equipment. First, the effect of the "slip layer" formed at the boundary of the measuring instrument and the fluid must be minimized. The slip layer occurs when the fibres in the suspension migrate away from the wall effectively lowering the observed viscosity. Second, the equipment must be able to provide measurements for a wide range of shear rates, as it is desired to quantify the shear-thinning effect in the composite liquids under study.

Both above-mentioned requirements can be satisfied by the capillary and concentric-cylinder rheometers. However, capillary rheometers require relatively large sample sizes to establish constant flow conditions. The photopolymers under study were not available in sufficient quantities for these types of measurements. As a result, concentric-cylinder rheometer was chosen to conduct viscosity measurements of the fibre-photopolymer composite liquids.

**Operation of the concentric-cylinder rheometer**

The primary geometric parameters in a concentric-cylinder rheometer are the radii of the two cylinders (cup and rotor, \( R_1 \) and \( R_0 \), respectively), the gap between the two cylinders, \( h \), and the length of the cylinders, \( L \), as depicted in Figure 2.15. The measured quantities are: the torque on the rotor and its rotational speed. It is assumed that (a) the fluid elements move in circles about the common axis of the cylinders with an angular velocity, \( \Omega \), which is a function of radius \( r \) only; (b) the flow is associated with a shear stress, \( \sigma_r \), at radius \( r \); and (c) the flow is independent of time.

The constitutive equation for the concentric-cylinder rheometer is derived by starting with the definition of viscosity as the ratio of the shear stress \( \sigma \) and the shear rate \( \dot{\gamma} \)
\[ \eta = \frac{\sigma}{\dot{\gamma}}. \]  
(2.14)

By using a simple force-balance analysis, the shear stress on the surface of the inner cylinder, \( \sigma_1 \), is related to the torque per unit length of cylinder, \( G \), as:

\[ G = 2\pi R_1^2 \sigma_1. \]  
(2.15)

From a simple shear-flow analysis, the shear rate is defined as the inner-cylinder velocity, \( V \), divided by the gap width, \( h \), where the two parameters can be expressed in terms of the cylinder radii and the rotor angular velocity,

\[ \dot{\gamma} = \frac{V}{h} = \frac{R_i \Omega}{R_o - R_i}, \]  
(2.16)

since \( V = \Omega R_i \) and \( h = R_o - R_i \).

![Concentric cylinder rheometer](image)

Figure 2.15. Concentric cylinder rheometer.

From the above three relationships, an expression is obtained for the viscosity as a function of the measured torque, \( G \), the cylinder radii, \( R_o \) and \( R_i \), and the angular velocity, \( \Omega \), as

\[ \eta = \frac{G/2\pi R_i^2}{R_i \Omega/ R_o - R_i}. \]  
(2.17)

Some limitations of the concentric-cylinder rheometer are as follows: (a) high shear-rate measurements cannot be achieved in viscous materials due to the centrifugal expulsion; (b)
the reproducibility of measurements is not as good as that in a capillary rheometer; and (c) the fluid below the inner cylinder exerts an additional torque at the base of the inner cylinder, potentially leading to measurement inaccuracies.

2.3.3.3 Experimental setup and procedure

**Equipment**

The Haake Rotovisco RV12 (Haake Buchler, 1980) concentric-cylinder rheometer was used for the viscosity measurements. A cup with an inner diameter of 42 mm and a depth of 90 mm was utilized. Rotors of two different sizes were used: the smaller rotor has an outer diameter of 30.4 mm and a height of 60 mm, while the larger rotor has an outer diameter of 36.8 mm and a height of 60 mm. The smaller rotor was used in most of the experiments to provide a wide gap between the rotor and the cup for the concentrated suspensions with comparatively long fibres. The resulting gap, in which the concentrated suspensions are sheared, is 5.8 mm. The required volume of sample is 70 cm$^3$. The larger rotor, on the other hand, was used when either the viscosity of the concentrated suspensions was expected to be lower than 400 cP or the length of the dispersed fibres was 0.8 mm (the shortest fibre length used in this study). In this case, the resulting gap is 2.6 mm and the required volume of sample is 55 cm$^3$.

**Calibration experiments**

Prior to conducting viscosity measurements, the rheometer was calibrated by three pure fluids of known viscosity, ranging from about 100 to 4600 cP. The fluids were Brookfield 100, Brookfield 1000, and Fluid 5000 (Brookfield, 1985). The results obtained were within the error bounds specified for the fluid viscosities.

**Assessing possible error sources**

The material nature of the fibre suspensions can cause a variety of difficulties and errors in measurement, including: fibre migration from the equipment wall (i.e., slip layer), sample non-homogeneity due to fibre sedimentation, fibre alignment, and liquid evaporation. Consequently, preliminary experiments were performed to assess the significance of these effects on the measured viscosity.
Evaporation was studied by observing the change in sample weight over time. No significant weight change was found. Sedimentation was observed by recording the sinking rate of the fibres as well as by the increase in volume of the top clear photopolymer region over time. It was found that it took up to 30 minutes for visible sedimentation to occur, which is much longer than the time frame of the viscosity measurements.

Slip-layer effect was assessed by measuring the viscosity of the same fibre suspension with two different rotor sizes. If the measured viscosity remains unchanged for both experiments, no "slip layer" is formed near the wall of the cylinders (Yilmazer and Kalyon, 1989). No significant difference was found between the two measurements.

The effect of fibre alignment on the measured viscosity was assessed by observing the change of viscosity of the suspension with time. The experimental results showed that the change in the measured viscosity was less than 4.5% during the 6-minute interval when the applied shear rate was 29 s\(^{-1}\), and less than 1.4% when the applied shear rate was 115 s\(^{-1}\).

**Viscosity measurements of glass-fibre and photopolymer composite liquids**

**Materials**

In this study, three different photopolymers were employed as the dispersing phase (Table 2.10), and two types of milled glass-fibres with three fibre lengths were used as the dispersed phase. The two types of glass fibres were 737BD milled glass-fibres and MFX milled recycled glass-fibres. The 737BD fibre surface is coated with a silane sizing, which is compatible with thermoplastics, whereas MFX fibres are coated with a generic surface sizing suitable for both thermoplastic and thermoset materials. Both types of milled fibres are manufactured in three fibre lengths, 0.8 mm, 1.6 mm, and 3.2 mm.

**Table 2.10. Properties of the photopolymers used in the study.**

<table>
<thead>
<tr>
<th>Photopolymer</th>
<th>Manufacturer</th>
<th>Composition</th>
<th>Viscosity (cP)</th>
<th>Density (g/cm(^3))</th>
</tr>
</thead>
<tbody>
<tr>
<td>Cibatool 5170</td>
<td>Ciba-Geigy</td>
<td>Epoxy-based</td>
<td>165-195 (@30°C)</td>
<td>1.14</td>
</tr>
<tr>
<td>Somos 6110</td>
<td>DuPont</td>
<td>Epoxy-based</td>
<td>391 (@25°C)</td>
<td>1.15</td>
</tr>
<tr>
<td>Exactomer 2202</td>
<td>Allied Signal</td>
<td>Vinyl-ether-based</td>
<td>250 (@30°C)</td>
<td>1.13</td>
</tr>
</tbody>
</table>
Sample preparation: For each combination of fibres and photopolymer, 90 ml of mixture was prepared. First, the desired volumes of the photopolymer and the glass fibres were converted to weights based on the specified fibre and photopolymer densities. The desired weights of the photopolymer followed by the glass fibres were deposited into a beaker placed on a digital balance (Mettler Toledo AB204). The sample was stirred gently until all the fibres were wetted by the photopolymer. To remove the entrapped air bubbles, the sample was then placed in a vacuum chamber for half an hour. Lastly, the sample was drained from the beaker into the rheometer cup and stirred before testing.

Viscosity measurement: Measurements for each fibre suspension were started at the lowest rotor speed, which was subsequently raised in predetermined increments. The actual range of rotor speeds used in an experiment was determined by the viscosity of the sample and the equipment capabilities. Measurements at each shear rate were repeated three times successively in order to verify the repeatability of the tests. Between successive measurements, the cup was removed from the instrument and the sample was stirred to assure a random fibre orientation. All measurements were made at room temperature.

As the present study focused exclusively on the viscosity of composite liquids with random fibre orientation, the initial, stabilized reading at each measurement was recorded. The torque value was obtained by averaging the torque over one revolution of rotor. The measured quantities (the torque and the rotor speed) were converted to viscosities and shear rates by Equations (2.16) and (2.17).

2.3.3.4 Results and discussion

Effect of photopolymer type

The viscosity measurements of the three pure photopolymers used in the study are shown in Figure 2.16. Without the fibres, the pure photopolymers behaved largely as Newtonian fluids, showing only a minor increase in viscosity with shear rate.
The measurements were repeated for three composite liquids with 20% volume fraction of 1.6-mm 737 fibres, Figure 2.17. The results reveal similar shear-thinning behaviour regardless of the photopolymer type.

**Effect of fibre volume fraction**

Results displaying the dependence of suspension viscosity on the fibre volume fraction are shown in Figure 2.18. The measurements were made over a range of shear rates from 0.1 to 100 s⁻¹, for SL5170 photopolymer mixed with 1.6-mm 737 fibres. The shape of the curves confirms the findings reported previously in (Chan et al., 1978, Ganani and Powell, 1985, Utracki, 1988, Collyer and Clegg, 1988).

The viscosity is seen to increase with the increase of the fibre volume fraction. However, the degree of increase depends on the shear rate. At the lowest measured shear rate, the increase is from about 200 cP for pure liquid to almost 20000 cP for composite liquid with 20% fibre volume fraction. At the highest measured shear rate, the increase is only from 250 cP for pure liquid to 950 cP for the composite liquid. This is a manifestation of the shear-thinning behaviour typically noted in fibre suspensions. Within the range of the shear rates observed, the shear-thinning effect is not apparent until a fibre volume fraction of 15% is reached.

**Effect of fibre aspect ratio**

The effect of fibre aspect ratio on viscosity for the composite liquids made from Cibatool 5170 photopolymer and MFX fibre is shown in Figure 2.19. For the range of fibre lengths tested, experimental results show that the fibre length has only a minor effect on the viscosity at high shear rates (100 s⁻¹), but it does have a considerable effect at low and medium shear rates. For example, at a shear rate of 0.4 s⁻¹, viscosity increases by a factor of 10 as the fibre length doubles, from 0.8 mm to 1.6 mm.

Examining Figure 2.19, one can note that increasing the fibre length, while maintaining constant fibre volume fraction, shifts the viscosity-shear-rate curves in the direction of increasing shear rates, with the same maximum and minimum viscosity values being approached asymptotically by all three curves.
Figure 2.16. Viscosity vs. shear rate for pure photopolymer liquids.

Figure 2.17. Viscosity vs. shear-rate for composite liquids made from 20% 1.6-mm 737 fibre.
Figure 2.18. Viscosity vs. shear-rate for composite liquids made from CibaTool SL5170 photopolymer and 1.6-mm 737 fibres.

Figure 2.19. Viscosity vs. shear-rate composite liquids made from CibaTool SL5170 photopolymer and MFX fibres.
Effect of fibre surface coating

The effect of fibre surface coating is displayed in Figure 2.20 by comparing the viscosity measured for composite liquids with 737 and MFX fibres. The comparison is shown for 8% and 24% volume fractions.

In general, the viscosities of composite liquids with 737 fibres are lower than those of liquids with the MFX fibres. The difference increases with increasing fibre volume fraction, but decreases with increasing shear rates. It is conjectured that the MFX fibres’ surface sizing and impurities might have caused formation of agglomerates of fibres, consequently yielding higher viscosities at low shear rates. However, the agglomerates break down at high shear rates, leading to comparable viscosity levels with composite liquids containing 737 fibres.

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Figure 2.20. Viscosity vs. shear-rate for composite liquids made from CibaTool SL5170 photopolymer and 0.8-mm fibres.

2.3.3.5 Conclusions

This study provided important information about the rheological properties of fibre-photopolymer composite liquids. These properties helped to establish the upper limits for the processability of composite liquids in terms of the fibre length and the volume fraction. The
pronounced shear-thinning behaviour of the samples is also of importance when addressing the design of the composite liquid’s deposition system.

The high viscosity values, especially at low shear rates, exhibited by the composite liquids with significant (at least 15%) volume fractions of fibres, confirmed the qualitative observations of the difficulties encountered in formation of the new part layers by submerging the part as opposed to by depositing the composite liquid from the top.

These experiments showed an apparently strong effect of the fibre surface coating on the mixture viscosity. Future work may exploit this effect by selecting a suitable fibre coating in order to reduce the viscosity and thus allow processing of the liquids with higher fibre concentrations.

2.4 Experimental verification of fibre-reinforcement benefits

While the benefits of short-fibre reinforcement for regular plastics are well documented (Gibson, 1994), this is not the case for photopolymers. Thus, during the early phase of this research, mechanical tests were conducted on mould-fabricated fibre-reinforced photopolymers. The objectives of these tests were first to quantify the benefits to be derived from short-fibre reinforcements and second to establish a benchmark for comparison of mechanical properties of the mould- and layered-fabricated composites.

Tests were conducted on dogbone-shaped specimens, 66 mm long with 3x3 mm cross-sections (ASTM Standard Test Method D638-91a, Type M-III), which were produced by pouring the liquid (either a pure resin or a resin-fibre mixture) into an open Teflon mould (Figure 2.21) and UV-curing. The specimens were produced two-at-a-time. Four specimens were made for most fibre concentration settings; except, only two specimens were made with 16% fibre content. The constituent materials were CibaTool SL5170 resin and 737BD 1.6-mm short milled glass fibres.
Chapter 2: Benefits of Fibre Reinforcement

Figure 2.21. Dimensions of the mould used to fabricate the tensile test specimens.

Figure 2.22 shows the tensile modulus and Figure 2.23 shows the tensile strength observations plotted against the fibre volume fraction. Error bars indicate a range of ± one standard deviation. Addition of only 15% by volume of short glass fibres can be seen to increase the modulus by 64%, from 1.4 GPa to 2.3 GPa, and the strength by 20%, from 53 MPa to 63 MPa.

The experimental measurements follow the expected linear relationship for both strength and modulus; however, the slope of the line fitted to the modulus data is less than theoretically expected (Hull, 1981). The discrepancy may exist either because the theoretical model is not accurate or because of the inaccurate information assumed about the fibre length or orientation within the specimen. For example, the difference between expected and actual fibre lengths may be due to fibre breakage during the composite preparation.

The strength model underestimates the improvement in strength due to fibre reinforcements. The theoretical plot was produced by assuming the observed tensile strength
values for the unreinforced matrix and employing a strain energy approach to account for the effect of fibre misalignment (Sanadi and Piggott, 1985a). This approach however predicts a very sharp drop-off of reinforcement efficiency with the deviation from direction of estimate (see Figure 6.5 in Chapter 6), which is claimed to underestimate the composite strength (Piggott, 1994), as confirmed by the test data.

![Figure 2.22. Tensile modulus of mould-fabricated short-fibre composites.](image)

![Figure 2.23. Tensile strength of mould-fabricated short-fibre composites.](image)

### 2.5 Summary

This chapter provided background information about photopolymers and glass fibres, which are constituent components of the composite liquid used as a raw material in the proposed manufacturing process. Liquid photopolymers undergo local solidification when exposed to sufficient level of radiation, such as the UV laser light. They possess relatively
weak mechanical properties (1-3 GPa modulus and 50-70 MPa strength). Glass fibres have high modulus and strength (70 GPa modulus and 3.4 GPa strength), but are brittle and sensitive to surface damage. To fabricate glass fibres in short lengths required for the proposed manufacturing process, chopped fibre bundles are hammer milled, which results in a distribution of fibre lengths. When polymers and fibres are combined in a composite material, there is a practical limit to the amount of randomly oriented short fibres that can be added to the matrix. This limit is 10-40%: the longer the fibre length, the lower the limit.

Next, the chapter examined the interaction of the constituent components. The examination involved measurement of the surface energy of the photopolymers in liquid and solid forms, measurement of the interfacial shear strength between the fibres and the solidified photopolymers, and measurement of the viscosity of the photopolymer-fibre composite liquids.

The observed low surface energy of the photopolymer liquids (34-45 mJ/m² for photopolymers vs. 73 mJ/m² for water (Weast, 1981), for example) will enhance their ability to spread when forming a thin liquid layer and when wetting the fibres. Both layer formation and fibre wetting are required in the proposed layered manufacturing process.

Strong bond between the fibres and the photopolymeric matrix will enhance the mechanical properties of the fabricated composites. The bond strength was evaluated by measuring the interfacial shear strength using a single-fibre pull-out test. Strength values of 32 MPa and 40 MPa obtained for the two photopolymers tested indicate existence of a good bond between the fibre and the matrix materials.

Viscosities of composite liquids were measured over a range of shear rates. Pure photopolymers were found to behave as Newtonian liquids over the observed shear-rate range. Composite liquids exhibited pronounced non-Newtonian behaviour: the viscosity decreased with increasing shear rate. Knowledge of the viscosity dependence on the shear rate is essential for modelling of the liquid-layer formation by wiping, as the choice of wiping speed, for example, will affect the shear rate. The viscosity is also important in designing effective fibre-resin mixing apparatus. Additionally, the experiments established practical limits for the fibre concentration. The viscosity was observed to rise sharply (increasing by a factor of 50-
100 at shear rates of 0.2-0.5 s⁻¹) when fibre concentration reached 20% by volume for 1.6-mm fibres.

Benefits of short-fibre reinforcement for photopolymer resins were ascertained via mechanical testing of mould-fabricated composites. Addition of 15% by volume of short fibres increased modulus by 64% and strength by 20%. Increasing fibre concentration was determined to cause proportionate rise in the composite modulus and strength.
Chapter 3. Process Design

3.1 Introduction

The high viscosity of a composite liquid, fibre settling, lack of interlayer fibre penetration, and other engineering issues necessitate the development of a new process for the layered fabrication of reinforced polymeric parts.

In regard to the first issue mentioned above, rheological studies reported in Chapter 2 have demonstrated that the addition of fibres to a photopolymer resin produces a highly viscous liquid. At low shear rates, such as those encountered in layer formation by liquid spreading, the viscosity of the composite liquid can be up to five times greater than that of the pure resin. In the commercial SL process, a wiper blade is used to spread the liquid photopolymer onto the newly solidified layer. With a significant rise in viscosity, the composite liquid would have difficulty spreading over the entire solidified surface, especially for larger surface areas.

Secondly, if the composite liquid were left undisturbed in a vat during the part building, the fibres would settle continuously due to having a density more than twice that of a typical resin (2.54 g/cm³ for glass vs. 1.13-1.15 g/cm³ for resin).

Thirdly, if the composite layered parts were produced via complete layer-by-layer solidification (as in the regular SL process), there would be no fibres extending across the layer boundaries; the fibres would be prevented from protruding above the surface by the liquid surface tension.

This chapter first presents a brief overview of three existing layered manufacturing processes, the proposed RLCM process is presented next, followed by a detailed discussion addressing the use of Axiomatic Design theory in arriving at the final process design solution.
3.2 Existing layered manufacturing processes

3.2.1 Stereolithography process

The stereolithography (SL) process was invented in 1982 by Charles Hull. In 1986 he founded 3D Systems (3D Systems, 1998) to manufacture the SL machines. The SL process builds objects by selective curing of a liquid photopolymer resin by an ultraviolet laser (Figure 3.1). It starts with a vat filled with the liquid photopolymer and an elevator platform set just below the liquid's surface. After loading the object data file and slicing, the optical scanning system moves the focused laser beam over the liquid surface to solidify the cross-section. The elevator is lowered then so that the next layer of liquid can be formed on the solidified object's surface. A leveling wiper is used to speed up the smoothing of the liquid. The process continues, as the part is built from bottom up until completion. The part is then raised, the excess liquid resin is drained and cleaned off, and the part is transferred to a post-curing apparatus where its cure is completed under an intense conventional UV lighting.

Figure 3.1. Stereolithography process.

3.2.2 Selective Laser Sintering process

Carl Deckard developed Selective Laser Sintering (SLS) at the University of Texas at Austin. After becoming aware of his work, Dr. Paul F. McClure founded DTM Corporation
(DTM Corp., 1998) in Austin in 1986. Before starting the SLS process, the working chamber has to be filled with nitrogen and heated to the operating temperature. After obtaining the cross-sectional part data, the powder-feed piston rises to deliver the new layer of material. At the same time, the part-building cylinder is lowered to the desired layer thickness (Figure 3.2). The second powder feed piston then also lowers to accommodate any surplus powder as it is pushed across by the levelling roller. The powder is heated to the temperature just below its melting point. A high-power (typically 50W) CO₂ laser draws the part’s cross-section in a raster scan pattern, sintering the powder particles together. After the part is completed, the part-building cylinder rises so that the part can be removed and cooled. Materials utilized by the process include polycarbonate, investment casting wax, and regular and glass-filled nylon powders.

![Figure 3.2. Selective Laser Sintering process.](image)

### 3.2.3 Fused Deposition Modelling process

Fused Deposition Modelling (FDM) was developed in 1988 by Scott Crump, who is now the president of Stratasys, Inc. (Stratasys, 1998) of Minneapolis, MN. In the FDM process, after model slicing, a spool of 0.050-inch diameter thermoplastic filament is fed to the heated extruding head (Figure 3.3). The liquefied thermoplastic is maintained at 1°F above solidification temperature prior to deposition. It takes 0.1 seconds for the material to solidify once it is deposited by the X-Y extruding head. For each layer, the Z-platform lowers the part.
Layer thickness ranges from 0.025 to 1.3 mm. The materials used include ABS, investment casting wax, and elastomers, similar to low/high density polyethylene and polypropylene.

**Figure 3.3. Fused Deposition Modelling process.**

### 3.3 Synthesis of the proposed process

The primary fabrication steps of the proposed RLCM process are as follows, Figure 3.4:

1. A precise volume of the composite liquid is withdrawn from an external source and deposited from above for each layer (Figure 3.4(a));
2. A wiper levels the liquid at the required height (Figure 3.4(b));
3. The layer is selectively cured by a UV laser in a pattern to be discussed in Section 3.3.3.2 (Figure 3.4(c)); and,
4. A platform supporting the part is lowered into the vat (Figure 3.4(d)).

The process steps are repeated until the entire part is built. Subsequently, the platform is raised, and the part is removed, cleaned, and post-cured. As the fabrication process continues, the composite liquid is continuously mixed in a separate container, i.e., an external raw-material source. This process solves all the primary problems addressed in the introduction of this chapter.

The following sub-sections will first provide a brief overview of an organized approach to design, Axiomatic Design theory, which has been employed in the design of the RLCM
process. This section will be followed by the description of the proposed process design at the highest level of the design hierarchy. Then, detailed description of each system component will be presented.

3.3.1 Axiomatic Design

There exists a number of "design strategies," such as robust design, adaptive design, and design for flexibility. However, there are very few formal theories to provide an organized framework for the design process (Mistree et al., 1990). One recently introduced approach is referred to as the Decision-Based Design (Mistree et al., 1990). With this approach the design process is viewed as a series of decisions. Risk and uncertainty are some of the issues that can be better analyzed and handled by focusing on the decision. Other advantages include the promise of reducing the number of design iterations, and the ability to integrate manufacturing analysis issues throughout the design process. However, research is still on-going to provide a formal framework for this approach.

Axiomatic Design theory, on the other hand, attempts to capture the essence of the design process by postulating that all good designs must satisfy two axiomatic statements (Suh, 1990). Based on these fundamental statements, the theory proceeds to develop a set of theorems and corollaries to guide the designer. The theory does not claim to replace the creative element of the design process, but instead aims to enhance the design process by placing the designer’s creativity within a well-organized framework.

Axiomatic Design Theory defines a design solution as a mapping between the Functional Requirements (FRs) of the functional domain and the Design Parameters (DPs) of the physical
domain. FRs comprise a minimum set of independent requirements that completely characterize the design objective.

The Independence Axiom provides guidance for finding the appropriate DPs by stating that a good design must maintain independence of the Functional Requirements. In other words, DPs must be chosen in such a way that each FR can be satisfied by the adjustment of the corresponding DP without affecting other FRs.

The Information Axiom further specifies that among the designs satisfying the Independence Axiom, the best design choice is the one which minimizes the information content of the design. The information content of a design increases with greater complexity and tighter tolerances. The Axiomatic Theory defines numerical measures of the information content which can be derived from the design specifications.

The design process is hierarchical in nature (Figure 3.5). The FRs and DPs are determined first at the highest level where the basic design configuration is defined. Then, for each DP of one level, a set of FRs must be stated at the next lower level, and subsequently the design solution in terms of corresponding DPs is sought at that level.

![Functional Domain (FRs) and Physical Domain (DPs)](image)

**Figure 3.5. Hierarchical nature of design process.**

The relationship between the FRs and DPs is expressed via a design matrix:

\[
\begin{bmatrix}
FR_1 \\
FR_2
\end{bmatrix} = \begin{bmatrix} X & 0 \\ 0 & X \end{bmatrix} \begin{bmatrix} DP_1 \\
DP_2
\end{bmatrix},
\]

(3.1)
where X at \((i,j)\) position indicates that DP\(_j\) affects FR\(_i\), and 0 indicates that it does not. The matrix in (3.1) represents an uncoupled design with two FRs and DPs. Uncoupled design matrices are characterized by a diagonal shape, since each DP only affects the corresponding FR.

However, complete uncoupling is not the only way to maintain independence of FRs. If the design can be represented by a triangular matrix, such as:

\[
\begin{bmatrix}
  \text{FR}_1 \\
  \text{FR}_2
\end{bmatrix} =
\begin{bmatrix}
  X & 0 \\
  X & X
\end{bmatrix}
\begin{bmatrix}
  \text{DP}_1 \\
  \text{DP}_2
\end{bmatrix},
\]

then, the design is referred to as decoupled. A decoupled design requires that the DPs be adjusted in a particular sequence in order to assure independence of FRs. In the case of (3.2), DP\(_1\) must be adjusted first, as it affects both FR\(_1\) and FR\(_2\). Once the desired setting of FR\(_1\) has been achieved, DP\(_2\) is adjusted to achieve the desired setting of FR\(_2\). Since DP\(_2\) only affects FR\(_2\), this last adjustment can be made without affecting FR\(_1\).

### 3.3.2 The top level of the design hierarchy – Conceptual design

The design objective for the RLCM process has been formulated as:

*Design a process which produces fibre-reinforced plastic parts by selectively solidifying thin layers of liquid photopolymer containing short glass fibres.*

Examining the design objective, three Functional Requirements are identified, Table 3.1. By fulfilling these requirements, the design will achieve the above-stated objective. The top-level design parameters for the current RLCM embodiment have been selected based on the Axiomatic Theory guidelines, specifically the Independence Axiom. The following design matrix expresses the inter-relationship of the top-level FRs and DPs:

\[
\begin{bmatrix}
  \text{FR}_1 \\
  \text{FR}_2 \\
  \text{FR}_3
\end{bmatrix} =
\begin{bmatrix}
  X & 0 & 0 \\
  0 & X & 0 \\
  X & 0 & X
\end{bmatrix}
\begin{bmatrix}
  \text{DP}_1 \\
  \text{DP}_2 \\
  \text{DP}_3
\end{bmatrix}.
\]
Table 3.1. Top-level FRs and DPs.

<table>
<thead>
<tr>
<th>FR</th>
<th>Description</th>
<th>DP</th>
</tr>
</thead>
<tbody>
<tr>
<td>FR₁</td>
<td>Build part layers of accurate height</td>
<td>DP₁ = Liquid-Layer Formation subsystem</td>
</tr>
<tr>
<td>FR₂</td>
<td>Build part layers with accurate horizontal dimensions</td>
<td>DP₂ = Laser-Light Delivery subsystem</td>
</tr>
<tr>
<td>FR₃</td>
<td>Build part layers with a specified fibre content</td>
<td>DP₃ = Fibre-Resin Mixing subsystem</td>
</tr>
</tbody>
</table>

The liquid-layer formation subsystem, DP₁, not only affects the layer height, FR₁, but also the fibre content within the layer, FR₃, (both denoted by an X at elements (1,1) and (3,1)). The laser-light delivery subsystem, DP₂, through the X-Y translation of the laser beam, will affect only the horizontal dimensions of a layer, FR₂, (represented by an X at (2,2)). Finally, the fibre-resin mixing subsystem, DP₃, is responsible for providing the desired fibre content in the raw material, FR₃, (represented by an X at (3,3)).

The design matrix (3.3) is triangular and therefore characterizes a decoupled design. Since the liquid-layer formation (DP₁) affects both FR₁ and FR₂, it must be adjusted first, while the DP₂ and DP₃ can be adjusted afterwards in any sequence.

The following sections will describe in detail the components of the proposed RLCM process.

### 3.3.3 The lower levels of the design hierarchy - Detailed design

Equation (3.3) defines the RLCM design at the highest level of the design hierarchy. To be completed, the design must be “decomposed” down to the lowest level. For each DP on one level, a corresponding set of FRs must be identified on the next lower level. The lower level design then continues with the selection of DPs to satisfy the lower-level FRs. Figure 3.6 illustrates the design hierarchy in terms of FRs, and Figure 3.7 in terms of corresponding DPs.
Chapter 3. Process Design

3.3.3.1 Liquid-layer formation (DP₁)

Formation of high-quality liquid layers is a necessary condition for the fabrication of the high-quality parts. The design objective for the Liquid-Layer-Formation subsystem can be formulated as:

*Design a subsystem which can form thin layers of composite liquid characterized by prescribed thickness, minimum height variability, and fibre content equal to that in the externally supplied liquid raw material.*

The FRs and DPs for this subsystem are shown in Table 3.2, and their inter-relationship is described by the following equation:
Table 3.2. FRs and DPs for the Liquid-Layer-Formation subsystem.

<table>
<thead>
<tr>
<th>FR</th>
<th>Description</th>
<th>DP</th>
</tr>
</thead>
<tbody>
<tr>
<td>FR$_{11}$</td>
<td>Distribute liquid equally over the vat surface</td>
<td>DP$_{11}$</td>
</tr>
<tr>
<td>FR$_{12}$</td>
<td>Deliver liquid onto previously solidified surfaces</td>
<td>DP$_{12}$</td>
</tr>
<tr>
<td>FR$_{13}$</td>
<td>Create layers with minimum height variation</td>
<td>DP$_{13}$</td>
</tr>
<tr>
<td>FR$_{14}$</td>
<td>Create layers with specified average thickness</td>
<td>DP$_{14}$</td>
</tr>
</tbody>
</table>

(3.4)

![RLCM Process Diagram](image)

Figure 3.7. Design parameter hierarchy of RLCM.
Chapter 3. Process Design

The composite liquid must be distributed uniformly over the vat’s top surface to prevent unequal local accumulation of solid material. The composite liquid also must be delivered directly onto the previously solidified parts of the layer to assure adequate fibre content. Finally, liquid layers of prescribed thickness and minimum height variation must be created to assure solid layers of correct shape.

Area-wide deposition (DP₁₁) must occur before the layers can be formed, and, therefore, affects both FR₁₃ and FR₁₄ in addition to FR₁₁. Liquid levelling (DP₁₃) not only smoothes the layer (FR₁₃) but also levels it to the specified thickness (FR₁₄). Lowering of the Z-platform (DP₁₄) only affects the layer thickness (FR₁₄).

The triangular matrix in (3.4) indicates that this is a decoupled design, which implies that the DPs must be adjusted in the sequence (DP₁₁, DP₁₂, DP₁₃, DP₁₄) in order to assure independence of the FRs. This establishes the correct sequence for process parameter adjustments: (1) area deposition, (2) direct deposition, (3) liquid leveling, and (3) Z-platform.

The design parameters of area and direct deposition (DP₁₁ and DP₁₂) are a part of the Composite-Liquid-Delivery subsystem, which will be described in detail in Subsection 3.3.3.4.

3.3.3.2 Laser-light delivery (DP₂)

The Laser-Light Delivery subsystem must perform the following function:

*Translate a focused beam of UV-laser light over the liquid’s surface in the X-Y (horizontal) plane.*

The FRs and DPs for the laser-light-delivery subsystem are shown in Table 3.3, and they are related to each other by:

\[
\begin{bmatrix}
FR_{21} \\
FR_{22} \\
FR_{23} \\
FR_{24}
\end{bmatrix}
= \begin{bmatrix}
X & 0 & 0 & 0 \\
0 & X & 0 & 0 \\
0 & 0 & X & 0 \\
0 & 0 & 0 & X
\end{bmatrix}
\begin{bmatrix}
DP_{21} \\
DP_{22} \\
DP_{23} \\
DP_{24}
\end{bmatrix}.
\]  

(3.5)
Table 3.3. FRs and DPs for the Laser-Light-Delivery subsystem.

| FR1 = Deliver light to the focusing lens | DP1 = Fibre-optic cable |
| FR2 = Focus the beam on the liquid's surface | DP2 = Lens attached to X-Y carrier |
| FR3 = Move beam along the X axis | DP3 = X axis of X-Y table |
| FR4 = Move beam along the Y axis | DP4 = Y axis of X-Y table |

The design equation (3.5) represents an uncoupled design (a diagonal design matrix). Each design parameter can be independently adjusted to satisfy the corresponding functional requirement.

As laser scanning converts the liquid resin into a solid part, it determines the part's structural integrity, correctness of its final shape, and presence of residual stresses within the part. In the scanning method adopted for the RLCM process, a border scan is followed by parallel hatch lines along the X and Y axes. The hatch lines are offset by half of line separation from layer to layer to improve the part integrity. The scanning sequence is also varied from layer to layer, so that the lines are drawn either in ascending or descending order (in terms of their position along the coordinate axis), which equalizes the build up of residual stresses within the part due to resin shrinkage.

A novel aspect incorporated into the proposed X-Y scanning method is the creation of interlayer rivets. The motivation was to improve the layer-to-layer bonding. Since the liquid surface tension keeps the fibres from protruding above the surface, with the normally performed complete-surface solidification, there would be no fibres crossing the layer boundaries. The proposed rivets are formed by intentionally leaving small volumes of resin uncured within each layer. Fibres protrude into these volumes from the surrounding solidified resin. When the subsequent layer is spread and cured, it will extend into the uncured pockets left in the preceding layer, and will form interlayer rivets, with the fibres extending into the rivets.

Figure 3.8 attempts to illustrate this concept graphically. It shows the sequential formation of three consecutive layers. The figure shows, from bottom to top, one-layer, two-
layer, and three-layer cross-sections of the same rectangular part as the layers are added. (The darker shading indicates solidified resin, while the lighter semi-transparent volumes denote uncured liquid.) For the first layer, there are three small volumes left with the uncured liquid. The uncured volumes are shifted by half of the inter-rivet distance in the next layer. When this second layer is cured, the uncured volumes of the previous layer are partially solidified; the solidification is completed during post-curing.

![Image of interlayer rivets](image)

**Figure 3.8.** Schematic illustration of the interlayer rivets (half of a cross-section is shown).

### 3.3.3.3 Fibre-resin mixing (DP₃)

The design objective of the fibre-resin mixing subsystem can be stated as:

*Supply a composite liquid with the desired fibre concentration to the rest of the system throughout the building process.*

The basic design configuration of the subsystem is a container with a rotary agitator, which is a commonly used approach for achieving solid suspensions in industrial processing (McDonough, 1992).
The mixing mechanism must be capable of suspending glass fibres within a liquid photopolymer. At least one area of uniform suspension should be created and sustained where draw-off can occur. Uniform suspension refers to a constant percentage of fibres per unit volume. The mixer should deliver a fibre concentration at draw-off point which matches as closely as possible the fibre concentration in the container as a whole. The mixer must also continue to supply the liquid with the constant fibre concentration throughout the building process and as the liquid level decreases. In addition, the agitation of the liquid must not be accompanied by aeration.

The functional requirement of this subsystem (FR31) is to achieve good circulation of the composite liquid throughout the container (Table 3.4). This will lead to the desired fibre concentration at the draw-off point. Once the basic configuration of the apparatus is established, to achieve good circulation, the design parameter of agitator rotation speed (DP31) must be adjusted to an appropriate value.

Table 3.4. FR and DP for the Fibre-Resin Mixing subsystem.

<table>
<thead>
<tr>
<th>FR31</th>
<th>DP31</th>
</tr>
</thead>
<tbody>
<tr>
<td>Circulate liquid throughout container</td>
<td>Agitator rotation speed</td>
</tr>
</tbody>
</table>

3.3.3.4 Composite-liquid delivery (DP11 and DP12)

Two design parameters, area deposition and direct deposition, are embodied in the composite-liquid-delivery subsystem.

For the area deposition (DP11), the composite liquid must be spread as uniformly as possible. This is achieved by translating the nozzle in a raster pattern over the vat, while delivering the liquid through a narrow nozzle in a tightly controlled manner. The FRs and DPs for the area deposition are shown in Table 3.5, and they are related to each other by:

\[
\begin{bmatrix}
FR_{111} \\
FR_{112}
\end{bmatrix} = \begin{bmatrix}
X & X \\
0 & X
\end{bmatrix}
\begin{bmatrix}
DP_{111} \\
DP_{112}
\end{bmatrix}
\] (3.6)

\[
FR_{111} = \begin{bmatrix}
X & X \\
0 & X
\end{bmatrix} DP_{111}
\]

\[
FR_{112} = \begin{bmatrix}
X & X \\
0 & X
\end{bmatrix} DP_{112}
\]
Table 3.5. FRs and DPs for the Area Deposition.

| FR111 = Deliver required volume per layer | DP111 = Flow rate per unit distance travelled |
| FR112 = Spread the composite liquid uniformly | DP112 = Number of raster lines |

The number of raster lines (DP112) must be established first to assure even spreading of the composite liquid (FR112). Then, the flow rate (DP111) is adjusted to deliver the required volume of the composite liquid per layer (FR111).

Direct deposition (DP12) is the second design parameter embodied in the composite-liquid delivery subsystem. Experience has shown that the method of composite-liquid delivery has significant impact on achieving the desired fibre content in the finished parts. The composite liquid has to be delivered directly onto the solidified areas of the previous layer in order to achieve the desired fibre content. The fibres within the liquid surrounding the solidified part settle out relatively quickly; after the surrounding liquid reaches the depth of several millimetres, a layer of mostly pure resin forms near its surface. Thus, if the composite liquid were deposited only onto the liquid areas of the vat's surface, the fibres would start sinking, leading to a wiping operation that covers the part with a liquid of lower fibre concentration.

To quantify the performance of the delivery system, one can use the ratio between the fibre content of the liquid delivered into the vat ($v_{f1}$) and the fibre content of the finished parts ($v_{f2}$); ideally, this ratio should be 1.0. By implementing the proposed liquid delivery process, the ratio ($v_{f2} / v_{f1}$) was improved from about 0.78 with no direct deposition to 0.98±0.06 with the current process.

The FRs and DPs for the direct deposition are shown in Table 3.6, and they are related to each other by:

$$\begin{bmatrix}
    FR_{121} \\
    FR_{122}
\end{bmatrix} = \begin{bmatrix}
    X & X \\
    0 & X
\end{bmatrix} \begin{bmatrix}
    DP_{121} \\
    DP_{122}
\end{bmatrix}. \quad (3.7)$$
Table 3.6. FRs and DPs for the Direct Deposition.

| FR$_{121}$ = Deliver required volume per layer | DP$_{121}$ = Flow rate per unit distance travelled |
| FR$_{122}$ = Cover previously solidified surfaces | DP$_{122}$ = Deposition travel path |

The deposition travel path (DP$_{112}$) must be established first to assure coverage of the previously solidified surfaces by the composite liquid (FR$_{122}$). Then, the flow rate (DP$_{121}$) is adjusted to deliver the required volume of the composite liquid per layer (FR$_{121}$).

3.3.3.5 Liquid leveling (DP$_{13}$)

The liquid-leveling operation must achieve two objectives: create a liquid layer of specified thickness and of minimum height variability. The FRs and DPs for the liquid leveling are shown in Table 3.7, and they are related to each other by:

$$\begin{bmatrix}
    \text{FR}_{131} \\
    \text{FR}_{132}
\end{bmatrix} =
\begin{bmatrix}
    X & 0 \\
    0 & X
\end{bmatrix}
\begin{bmatrix}
    \text{DP}_{131} \\
    \text{DP}_{132}
\end{bmatrix}$$

Table 3.7. FRs and DPs for the Liquid-Levelling subsystem.

| FR$_{131}$ = Correct layer thickness | DP$_{131}$ = Wiper traversal speed |
| FR$_{132}$ = Minimize thickness variability within layer | DP$_{132}$ = Pre-wiping stroke |

Formation of a thin liquid layer by a straight-edge blade is a complex fluid-mechanics problem affected by a number of parameters. These include the shape of the blade edge, the wiping speed, the amount of material accumulated at the leading edge, and the liquid viscosity (see Chapter 4).

The model in Chapter 4 points to the significance of the liquid bulge formed at the wiper's leading edge. As the wiper moves, the bulge height changes. This variation can affect the layer height locally, leading to variation in the layer thickness. The proposed wiping process aims to minimize this effect through performing two wiping stroke sequences to form each layer: the first stroke sequence is carried out after the platform has been lowered by a depth of several layers, and the second stroke sequence when the platform has been raised.
back to the height one-layer lower than the last layer built. Pre-wiping prior to the final
levelling helps to spread the liquid more uniformly by removing most of the excess liquid and
roughly spreading the liquid during the first stroke. Pre-wiping also reduces the leading edge
liquid accumulation during the final wiping stroke. Part cross-section studies have confirmed
the effectiveness of this approach.

3.3.3.6 Z-platform subsystem (DP14)

The Z-platform moves up and down the platform which supports the part and acts as the
part bottom. The FRs and DPs for the Z-platform subsystem are shown in Table 3.8, and they
are related to each other by:

\[
\begin{bmatrix}
FR_{141} \\
FR_{142}
\end{bmatrix} =
\begin{bmatrix}
X & 0 \\
0 & X
\end{bmatrix}
\begin{bmatrix}
DP_{141} \\
DP_{142}
\end{bmatrix}
\]

Table 3.8. FRs and DPs for the Z-platform subsystem.

<table>
<thead>
<tr>
<th>FR141 = Move platform vertically</th>
<th>DP141 = Vertical translation stage guides</th>
</tr>
</thead>
<tbody>
<tr>
<td>FR142 = Move platform by a required distance</td>
<td>DP142 = Z-motor rotation</td>
</tr>
</tbody>
</table>

The precise movement in a vertical direction (FR141) is assured by the linear guides of the
translation stage (DP141), and the translation distance (FR142) is determined by the rotation of
the driving motor (DP142).

3.4 Design of the experimental prototype

An experimental prototype system was built to serve as a test-bed for process
development. Since the emphasis was on the process analysis, and not on designing a
commercial product, the choice of some components was dictated not by the greatest
suitability to the task but by the equipment availability and simplicity of implementation. For
example, to achieve fast building speeds, majority of commercial systems utilizes
galvanometrically actuated mirrors for the X-Y laser beam scanning. However, since the
building time was not a significant issue, the RLCM prototype system uses a precision X-Y
translation system for the same purpose due to the equipment availability.
The current design presented in this thesis was a result of several iterations. For each of these, Axiomatic Design principles were used to help with the design analysis and to supply ideas for the new designs, leading to a significantly improved system performance. The subsystems of the current RLCM prototype (Figure 3.9) are individually discussed below.

![Prototype RLCM system for the fabrication of fibre-reinforced composites.](image)

**3.4.1 Fibre-resin mixing subsystem**

The *fibre-resin mixing subsystem* keeps the fibres in suspension throughout the building process. It consists of an open-top cylindrical container, an impeller, and a flexible plastic draw-off tubing fixed to container walls by magnetic clamping. The fibres are maintained in suspension by inducing constant circulation within the container. This is achieved by the following design features (McDonough, 1992):

- An impeller in the form of a three-blade marine propeller. The variable pitch angle of the blades produces large axial flow component and reduces radial swirling.
- A container with a near-one height-to-width ratio. Given a single impeller, excessive container height hinders liquid circulation throughout the container volume.
- Cylindrical container shape and radiused transition between container bottom and side walls to avoid stagnation zones due to sharp corners.
• Draw-off tubing and magnetic clamp serving as a baffle to reduce vortexing.

3.4.1.1 Performance evaluation experiments

Experiments were conducted to determine:

(a) The impeller rotation speed required to establish good circulation, and

(b) The effect of decreasing batch volume on the fibre concentration.

Equipment

A 400-mL beaker served as a container, and a 1-inch diameter three-blade marine-type propeller served as an impeller. The impeller was attached to a shaft which was immersed vertically into the liquid and driven directly by a DC electric motor. To draw off the liquid, a flexible plastic tube was held within the beaker by magnetic clamping.

Method

The impeller rotation speed was varied by the adjustment of the motor voltage and measured by an optical digital tachometer. The liquid samples were drawn off with the plastic tubing by operating a single peristaltic pump. The samples taken were solidified by UV curing and their density was measured to evaluate the fibre content.

To find the suitable impeller speed, three different values were tried: 589, 1254, and 1915 rpm. To determine the effect of the decreasing batch volume, samples were drawn from batches of progressively decreasing volumes from 320 to 130 mL. The impeller speed was set at 1254 rpm.

Results

Figure 3.10 shows the fibre volume fractions observed at three different motor speeds. The solid horizontal line represents the expected volume fraction (18.9%) based on the relative volumes of fibres and resin in the original mixture preparation. The results (Table 3.9) indicate that the middle speed setting produces the highest average sample fibre content and the smallest variability.

---

1 This research was conducted by H. Scandalis under the author’s direct supervision. See Scandalis, 1998, for more details.
Figure 3.11 shows the variation in fibre content in withdrawn samples as the liquid level in the container is varied over a wide range. The solid horizontal line at 13.7% indicates the expected sample fibre content based on the relative volumes of fibres and resin in the original mixture preparation. The observed average fibre content and the standard deviation were 13.7% and 0.4%, respectively. The results indicate no significant effect of the container liquid level on the fibre content of the withdrawn samples. The samples also match very closely the expected fibre content values.

The above experimental results indicate that the proposed design of the mixing subsystem is capable of achieving the stated design objectives.

![Graph](image)

**Figure 3.10. Fibre-resin mixing subsystem performance at three impeller speeds.**

**Table 3.9. Fibre-resin mixing subsystem performance evaluation at three speeds.**

<table>
<thead>
<tr>
<th>Speed (rpm)</th>
<th>Average Fibre Content (%)</th>
<th>St. Dev. of Fibre Content (%)</th>
<th>Number of Samples</th>
</tr>
</thead>
<tbody>
<tr>
<td>589</td>
<td>16.8</td>
<td>2.1</td>
<td>9</td>
</tr>
<tr>
<td>1254</td>
<td>18.3</td>
<td>0.9</td>
<td>9</td>
</tr>
<tr>
<td>1915</td>
<td>17.2</td>
<td>1.4</td>
<td>12</td>
</tr>
</tbody>
</table>
Figure 3.11. Fibre-resin mixing subsystem performance with varying batch volume.

3.4.2 Composite-liquid delivery subsystem

The composite-liquid delivery subsystem transfers a desired amount of resin into the vat and deposits it in a predetermined pattern. The material is transferred by two paired peristaltic pumps, which use rollers to squeeze the liquid through a flexible plastic tube. One end of the tubing is inserted into the container of the mixing subsystem; the other end is attached to the platform of the X-Y translator, Figure 3.12. To deposit the material in a desired pattern, the pumping action is combined with simultaneous translation of the dispensing nozzle.

Figure 3.12. Composite-Liquid Delivery and Fibre-Resin Mixing subsystems.

The advantages of delivery via a peristaltic pump are: (i) accurate metering of the liquid volume delivered, since the pump is of a positive displacement type, and (ii) simplified system maintenance, since there is no contact between the pump mechanism and the liquid, and since the tubing is easily replaceable. In the RLCM prototype, the liquid was delivered into a vat
with inside dimensions of 93x93 mm. Deposition from above did not cause any observable air bubble entrapment.

Two pumps are used to reduce the flow rate pulsation caused by the squeezing action of three rollers of each pump. With a single pump, the pulsation was observed to occur at a rate of three pulses per pump revolution. By adding a second pump on the same drive shaft, and by placing it with rollers rotated by 180° relative to the first pump, the pulsation was significantly reduced.

The repeatability of a single pump in delivering a volume of liquid over one pump revolution was verified experimentally. Using flexible tubing with inner diameter of 4.8 mm, for pure resin (2202SF), the volume delivered was 1.72±0.05 mL and, for a composite liquid (2202SF with 15% by volume of 1.6-mm fibres), the volume delivered was 1.69±0.05 mL. Indicated ranges (±σ) correspond to one standard deviation.

To improve the consistency of fibre concentration in the composite liquid delivered into the vat, the contents of the plastic tubing are pumped back into the mixing container after each deposition sequence. This step prevents fibre settling within the tubing system between the depositions. By adding the above reverse pumping to the process, the experimentally observed ratio between the fibre content of the liquid delivered into the vat (υ_1) and the fibre content of the parts built (υ_2), υ_2 / υ_1, was improved from about 0.76±0.08 to 1.03±0.09.

The deposition sequence (as currently implemented in the prototype RLCM) consists of two main steps: the area deposition and the direct deposition (as explained in Section 3.3.3.4).

The area deposition is carried out by eight deposition lines parallel to the wiper (i.e., along the Y direction) (Figure 3.13(a)). The solid straight lines in the figure indicate the pump is on during translation and the arrowheads indicate the direction of X-Y carrier translation. The curved lines indicate the X-Y carrier point-to-point traversal with the pump off. To reduce the effect of continuous repetitions of the same deposition sequence, the process is varied in two ways: either the direction of traversal is reversed or the sequence of deposition lines is reversed (i.e., from right to left as opposed to from left to right, as drawn in the figure). The above two settings are varied through four possible combinations every four layers.
Additionally, the deposit lines are shifted by half of the between-line separation at every second layer.

The direct deposition has been implemented at this point only for relatively simple shapes. To determine the deposition path, first a bounding box is found for each curve among the layer contours. Then, the deposit path is set as the line bisecting the bounding box along its long dimension. Figure 3.1(b) shows an example for the two tensile-test specimens.

![Wiper](image)

**Figure 3.13. Traversal path definitions for the (a) area and (b) direct deposition sequences.**

### 3.4.3 Liquid-levelling subsystem

The *liquid-levelling subsystem* assures uniform spreading of the liquid over the vat’s top surface to create a layer of consistent thickness. This is achieved by translating a wiper with a triangular edge profile (Figure 3.14). The wiper movement is actuated by a pneumatic cylinder.

The wiping speed is controlled by adjusting the opening of an air outlet valve. The valve controls the rate at which the air is allowed to escape from the air cylinder. Closing the valve reduces and opening the valve increases the traversal speed. Since the contact with the liquid occurs only during the forward stroke, the traversal speed is adjusted to be slower in the forward stroke (about 1.5 cm/s) than in the reverse (about 7 cm/s).
Figure 3.14. Wiper-blade edge profile (all dimensions in mm).

3.4.4 Laser-light delivery

The laser-light delivery subsystem delivers a focused beam of UV light to the surface of the composite liquid and translates this beam in the X-Y plane. The light is carried by a fibre-optic cable attached to a focusing lens. The lens is translated over the vat by an X-Y table with a 1-micrometer resolution.

3.4.4.1 Cure depth dependence on scan speed

For any process based on laser curing of photopolymers, a relationship between the exposure and the cure depth must be established. Analytically, the dependence of the cure depth, $C_d$, on the laser exposure is expressed by (Jacobs, 1992):

$$C_d = D_p \ln\left(\frac{E_{\text{max}}}{E_c}\right),$$  \hspace{1cm} (4.10)

where $E_{\text{max}}$ is the maximum exposure occurring at the beam centre for the Gaussian beam profile, $D_p$ is the penetration depth, and $E_c$ is the critical exposure. The last two are characteristic parameters of a particular photopolymer used (Table 3.10). A plot of the cure depth versus the log of the exposure is expected to produce a straight line with a slope $D_p$ and an X-axis intercept of $E_c$. This plot is referred to as the working curve.
Table 3.10. Manufacturer-specified photosensitivity parameters of the photopolymers.

<table>
<thead>
<tr>
<th>Photopolymer</th>
<th>Penetration Depth, Dp (mm)</th>
<th>Critical Exposure, Ec (mJ/cm²)</th>
</tr>
</thead>
<tbody>
<tr>
<td>CibaTool SL5170</td>
<td>0.12</td>
<td>13.5</td>
</tr>
<tr>
<td>Exactomer 2202SF</td>
<td>0.17</td>
<td>8.5</td>
</tr>
</tbody>
</table>

When the surface is scanned by a linear translation of a laser beam with a Gaussian intensity profile, the maximum exposure is given by (Jacobs, 1992):

\[ E_{\text{max}} = \left( \frac{2}{\pi} \right)^{\frac{1}{2}} \frac{P_L}{W_0 V_S}, \]  

(4.11)

where \( P_L \) is the laser power delivered to the photopolymer surface, \( W_0 \) is the \( 1/e^2 \) Gaussian half-width (or the beam radius), and \( V_S \) is the scanning velocity.

Thus, for a laser beam of a particular power, radius, and intensity profile, the cure depth will be a function of the scanning velocity. The working curve is then experimentally established by drawing either separate or partially overlapping series of lines on the resin surface and observing the variation of the cure depth with the scanning velocity.

Figure 3.15 shows the cure depth of separate scan lines plotted against the scan speed for SL5170 and 2202SF photopolymers (for the laser power of 14 mW). The observations confirm the lower photosensitivity of the SL5170 resin (its \( E_c \) is 59% higher than that of 2202SF, and thus SL5170 requires greater exposure to begin curing). Based on these observations, one can set an appropriate scan speed for the desired layer thickness. For example, to get a layer thickness of 0.3 mm requires a scan speed of 25 mm/s for SL5170 and 68 mm/s for 2202SF.
3.4.4.2 Effect of finite acceleration of the X-Y translator

Since the cure depth is a function of the scan speed, it is apparent that the depth will vary as the X-Y translator accelerates to the commanded speed. When the galvanometric mirrors are used for beam translation, high accelerations are feasible, and so the effect of finite acceleration is negligible. The X-Y table used for beam translation in RLCM, on the other hand, cannot accelerate as fast.

Therefore, tests were conducted to evaluate the effect of the finite acceleration for the X-Y table. A series of parallel lines separated by 0.3 mm were scanned for five different acceleration settings. For each line, the table motion is started, stopped, resumed, and stopped again. The laser shutter is opened in the beginning of the sequence and closed at the end. The intention was to observe the effect of point-to-point motion executed without closing the shutter.

The X-Y table motion controller performs the movement using a trapezoidal velocity profile. The acceleration is set by specifying the acceleration/deceleration ramp times. The commanded velocity was 40 mm/s and the translation consisted of two segments of 8 mm each.

The cured line thickness profiles were sampled every 0.25 mm. Representative results for two acceleration values (27 cm/s² and 67 cm/s²) are plotted in Figure 3.16. Theoretical cure-
depth predictions were fitted to the data based on Equations (4.10) and (4.11), and these can be seen to follow the observed data closely. The peaks observed at 4 and 12.5 mm are due to two single strands drawn perpendicular to the scan lines for support and to keep them together.

The acceleration and deceleration segments require 3 mm each for 27 cm/s$^2$ and 1.2 mm for 67 cm/s$^2$ acceleration settings. However, the effect of acceleration is relatively insignificant beyond about 0.5 mm from the start and stop peak depth points.

![Graphs](image)

**Figure 3.16.** Scan line profiles for acceleration values of (a) 27 cm/s$^2$ and (b) 67 cm/s$^2$ and scan velocity of 4 cm/s.

### 3.4.4.3 Scanning patterns – rivets

As described in Section 3.3.3.2, a special laser-scanning pattern, herein referred to as "rivets," is used in the RLCM process to enhance the part’s mechanical properties. This
pattern leaves small uncured volumes within each layer which are cured when the next layer above is solidified, thus joining the two layers.

To build a part with rivets, a new scanning pattern had to be implemented to meet the following three requirements:

1) The rivet horizontal locations cannot overlap from layer to layer since the uncured areas must be cured when the next layer is solidified.

2) Increased exposure should be applied over the rivet areas of the previous layer since the liquid depth is twice the layer thickness at these locations.

3) Scanning should involve continuous lines extending between the boundaries of each part contour within the layer. Since the layer may contain a large number of rivets, it would be inefficient to interrupt the scan lines to leave the rivet areas uncured (Figure 3.17).

A scan pattern satisfying the above requirements is shown in Figure 3.18. The figure shows patterns for alternating layers, with six rivets in pattern (a) and two in pattern (b). (The scan-line spacing has been exaggerated for clarity.) Note that the new layer surface above each rivet will be scanned twice—once in X and once in Y direction, so as to double the exposure locally. Also, all scan lines extend continuously between the layer contour boundaries.

Figure 3.17. Undesirable effect of scan lines interrupted by rivets (interrupted line segments are highlighted by thicker lines).
3.4.5 Z-platform

The Z-platform subsystem moves the supporting platform vertically. Since the height of the platform directly affects the layer thickness, its vertical displacement must be accurately controlled. To achieve the required accuracy, the Z platform is actuated by a stepper motor driving a micrometer attached to a vertical translation stage with a 70 mm range of motion.

Evaluation of the translation accuracy of the Z-platform has shown a backlash of about 0.5 mm. To compensate, the platform is always positioned while travelling in an upward direction. Thus, when downward displacement is required, the platform is moved about 1 mm further down and then up to the required height.

The above approach was tested by commanding the platform to move repeatedly 1.5 mm down and 1.2 mm up, for a net downward displacement of 0.3 mm (a typical layer height). A gauge was placed in the middle of the Z-platform for these measurements, and movement commands were issued through the system controller. Gauge measurement reading resolution was 0.003 mm. The incremental displacements measured gave an average of 0.299 mm and a standard deviation of 0.01 mm. The results indicate a satisfactory accuracy and repeatability of the Z-platform translation system.

3.4.6 System control

The RLCM prototype is operated by an Intel-486-based PC through a dedicated controller (Unidex 12 by Aerotech). Figure 3.19 shows the inter-relationship between various system components. The prototype system is run by software written in Matlab® script language,
which runs in the Matlab® program environment. Low-level serial communication between the Unidex 12 controller and the PC is handled by a dedicated code written in C language.

3.4.6.1 Unidex controller tasks

Tasks executed by the Unidex controller are:

(8) Motion control of four axes:
   - X and Y translation axes
   - Z-platform translation
   - Rotation of the peristaltic pump drive shaft

(9) On/off switching to:
   - Activate the solenoids which direct the air driving the pneumatic cylinder of the wiper
   - Activate the solenoid of the laser-beam shutter

(10) Receiving feedback from microswitches sensing completion of the wiper's forward and return strokes.

3.4.6.2 PC-controller communication

The Unidex controller is operated via a built-in programming language. To operate the controller remotely, the Unidex language commands are transmitted from the PC to the controller as ASCII codes via an RS-232 serial communication link. The Unidex sends back acknowledgement of command completions (e.g., end-of-move condition) via a single digital I/O line connected to one of the pins on a parallel port of the PC.

3.4.6.3 Generation of the controller commands

The low-level motion commands required for system operation are automatically generated by the main control program based on the geometrical description of the object to be created, the specified laser-scanning pattern, and the process parameter settings.
Figure 3.19. RLCM process components.

1 Drawing prepared with the assistance of M. Haberer.
The object's geometrical description is conveyed by the layer contour data, which is generated from a CAD solid model of the part to be built (Figure 3.19). The layer contour data defines the part's X-Y boundaries at the Z elevation of each layer.

The laser-scanning pattern is defined by the layer hatch data, which is generated off-line for each layer from the layer contours and the scanning pattern specifications. It consists of start- and end-point coordinates of the scan line segments required to solidify each layer.

The process parameters are entered into the main program interactively via a dialog box of a graphical user interface. Table 3.1 lists the parameters required by the program, their descriptions, and possible values.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Possible Values</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td><strong>Main Process Parameters</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>ScanSpeed</td>
<td>&lt;150 mm/s</td>
<td>Laser scanning speed</td>
</tr>
<tr>
<td>DVolume</td>
<td>0 &gt; (mL)</td>
<td>Area deposit volume per layer (5-6 mL typical)</td>
</tr>
<tr>
<td>DvolDirPerCm</td>
<td>0 &gt; (mL/cm)</td>
<td>Direct deposit volume per cm travel of deposit nozzle (0.5-0.6 mL/cm typical)</td>
</tr>
<tr>
<td><strong>Auxiliary Process Parameters (used for debugging)</strong></td>
<td></td>
<td></td>
</tr>
<tr>
<td>DoXYMove</td>
<td>ON/OFF</td>
<td>Turns on/off movement of the axes</td>
</tr>
<tr>
<td>DoGraphics</td>
<td>ON/OFF</td>
<td>Turns on/off graphical display of layer hatching</td>
</tr>
<tr>
<td>Light_On</td>
<td>ON/OFF</td>
<td>Enables or disables laser shutter</td>
</tr>
<tr>
<td>DoPure</td>
<td>ON/OFF</td>
<td>Sets system up for pure-resin or composite parts</td>
</tr>
<tr>
<td>DoDeposit</td>
<td>ON/OFF</td>
<td>Turns on/off deposition function</td>
</tr>
<tr>
<td>DoWiper</td>
<td>ON/OFF</td>
<td>Enables or disables the wiping operation</td>
</tr>
<tr>
<td>DoBorder</td>
<td>ON/OFF</td>
<td>Enables or disables scanning of the layer borders</td>
</tr>
<tr>
<td>nStartLayer</td>
<td>0 &gt;</td>
<td>Number of first layer to be built (1 typically, used to resume failed build)</td>
</tr>
</tbody>
</table>

**3.4.7 Generation of the layer data**

**3.4.7.1 Layer contour data generation**

In order to build parts, their geometric information must be provided to an RP system. The most common way of obtaining this information is from a solid model of the part created
within a CAD program. Since the part is fabricated through the layer-by-layer accretion, the part geometry is described with a set of contours for every part layer.

**Tessellated representation – STL format**

To generate the required layer data, the object's model needs to be "sliced" at the layer's height. As a result, the information defining boundaries of the solid material at that layer is obtained. While this information can be in principle extracted directly from any solid model of an object, an indirect approach was introduced by 3D Systems in 1987 in the form of an "STL" file. Since 3D Systems were the originators of the RP industry, this file format has become a universally accepted standard.

The STL file format works by representing object's surfaces with a mesh of connected triangles called "tessellations" (Figure 3.20). Each triangle must be connected to other triangles at all its vertices. The STL file data consist of the coordinates of all the triangle vertices as well as the surface normal vectors corresponding to each triangle. By convention, the vectors are assumed to point away from the solid material. Thus, determining the object's boundary contours at each layer entails finding the intersection between a horizontal plane and the object's surface represented by the triangles.

All the major CAD vendors supply translators from their CAD representation to STL, and all RP systems accept the STL file format for object definition. When the object's solid model is translated to a tessellated format, an "accuracy," or offset parameter, is input by the user. This is the acceptable chordal error between the plane of a triangle and the surface it is approximating. This value has no effect on the shapes made of planar faces such as a cube. In these cases, tessellating an object for file transfer to RP is very efficient. However, with highly curved objects, requirement for high accuracy will result in a very large data file.
Advantages of tessellation are as follows:

1. It provides a simple method of representing 3D CAD data.

2. This is already a de facto standard, which most CAD and RP systems support.

3. For certain shapes, it can provide small and accurate files for data transfer.

Disadvantages of tessellation include the following:

1. It creates files many times larger than the original CAD data file for an acceptable accuracy parameter.

2. The implementation of STL translators within CAD systems varies and consistency of quality is a problem.

3. The subsequent slicing of large STL files can take a very long time.

4. Sometimes, the designer will be unable to get the CAD model through the STL interface successfully, requiring changes to the model.

STL model slicing program has been implemented in Matlab® language to produce the layer contour data in the format required by the RLCM.

**Direct slicing**

An alternative to using an intermediary tessellation file is to slice the CAD model directly and transfer the resultant contours to the RP process. There are a number of reasons for using direct slicing, mainly related to the disadvantages of using tessellation:

1. Reduced file size (compared with the faceted models).

2. Greater model accuracy.
(3) Reduced RP machine pre-processing time.

(4) Greater reliability of the model data due to bypassing of an intermediary step.

Such a direct slicing method has been implemented within the SDRC I-DEAS CAD environment\(^1\). The algorithm finds intersections between the part's solid boundaries and a stack of infinite slicing planes. The intersections, mathematically represented by NURBS (Non-Uniform Rational B-Spline) curves, are output from I-DEAS in an IGES (Initial Graphics Exchange Specification) format. The IGES-format contours are then optimally converted to the point-wise layer contour definition required by the RLCM\(^2\). The optimization involves finding the minimum number of points required to represent a curve at a specified accuracy level.

While a greater number of contour curve points will improve the accuracy, it will also result in an increased data file size and longer layer scan times. The RLCM controller is only capable of motion in discrete linear or circular segments. Thus, when tracing the layer boundaries, increasing the number of points will require more discrete motion segments: the controller then must decelerate at every intermediate curve point before beginning the motion towards the next point.

3.4.7.2 Layer hatch data generation

The layer hatch data specifies for the RLCM prototype how all the part layers will be solidified. The solidification is accomplished by tracing with a laser beam individual line segments on the liquid surface. For ease of implementation, all line segments are aligned with either X or Y axis. Thus, the hatch data consists of end-points of the scan line segments to be drawn on each layer.

The hatch data generation is implemented via a stand-alone Matlab\(^\circ\) program. The program requires two types of input data: the layer contours and the scanning pattern specification. The line-segment end-points are determined by finding the intersections of the scanning pattern lines with the layer-contour curves.

---

1 Program written by Frank Truillet, a visiting graduate student from France.

2 This research was conducted by O. Kornienko and S. Orzechovsky under the author's supervision. See Kornienko, 1998.
The layer contours are obtained as described in the preceding subsection. The scanning pattern (described in Sections 3.3.3.2 and 3.4.4.3) is determined by three factors:

1. A set of parameters listed in Table 3.12 (mainly the scan line spacing).
2. Layer-to-layer *alternation matrix*, which specifies variation in the scan pattern with layer.
3. Presence and specifications of the rivet features.

**Table 3.12. Scan pattern parameters accepted by the hatch data generation program.**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Possible Values</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>DoDrawX</td>
<td>ON/OFF</td>
<td>Activate scan lines perpendicular to X axis (typically ON)</td>
</tr>
<tr>
<td>DoDrawY</td>
<td>ON/OFF</td>
<td>Activate scan lines perpendicular to Y axis (typically ON)</td>
</tr>
<tr>
<td>DoStagger</td>
<td>ON/OFF</td>
<td>Turns on/off scan line staggering (typically ON)</td>
</tr>
<tr>
<td>HSpaceX</td>
<td>0 &gt; (mm)</td>
<td>Scan line spacing along X direction (typically 0.3 mm)</td>
</tr>
<tr>
<td>HSpaceY</td>
<td>0 &gt; (mm)</td>
<td>Scan line spacing along Y direction (typically 0.3 mm)</td>
</tr>
</tbody>
</table>

The alternation matrix specifies values of three on/off parameters, which determine in what sequence the layer scan pattern will be drawn for each layer (Table 3.13). The parameter settings are cycled through eight different combinations, starting with the layer 1 (Table 3.14). The sequence is repeated every eight layers.

**Table 3.13. Alternation matrix parameters.**

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Possible Values</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>XFirst</td>
<td>TRUE/FALSE</td>
<td>If true, do scan lines perpendicular to X axis first (before Y axis)</td>
</tr>
<tr>
<td>XAscend</td>
<td>TRUE/FALSE</td>
<td>If true, do scan lines perpendicular to X in order of ascending X coordinate</td>
</tr>
<tr>
<td>YAscend</td>
<td>TRUE/FALSE</td>
<td>If true, do scan lines perpendicular to Y in order of ascending Y coordinate</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Layer Sequence</th>
<th>1</th>
<th>2</th>
<th>3</th>
<th>4</th>
<th>5</th>
<th>6</th>
<th>7</th>
<th>8</th>
</tr>
</thead>
<tbody>
<tr>
<td>XFirst</td>
<td>1</td>
<td>0</td>
<td>0</td>
<td>1</td>
<td>0</td>
<td>1</td>
<td>1</td>
<td>0</td>
</tr>
<tr>
<td>XAscend</td>
<td>1</td>
<td>1</td>
<td>0</td>
<td>0</td>
<td>1</td>
<td>1</td>
<td>0</td>
<td>0</td>
</tr>
<tr>
<td>YAscend</td>
<td>1</td>
<td>0</td>
<td>0</td>
<td>1</td>
<td>1</td>
<td>0</td>
<td>0</td>
<td>1</td>
</tr>
</tbody>
</table>

The third factor which determines the scanning pattern is the presence of the rivet features. If the rivets are present, their specifications are included as part of the data file containing the layer-contour information. The rivets are specified by four parameters listed in Table 3.15. Figure 3.21 further clarifies the parameter definitions. The figure shows rivets from two consecutive layers, with rivets from one layer drawn with solid and from the other with dashed lines.

Table 3.15. Rivet specification parameters.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>rvtSize</td>
<td>[X, Y] size of the rivets</td>
</tr>
<tr>
<td>rvtSpacing</td>
<td>[X, Y] spacing between rivet centres</td>
</tr>
<tr>
<td>rvtShift</td>
<td>[X, Y] shift in layer positions between alternate layers</td>
</tr>
<tr>
<td>rvtOrigin</td>
<td>[X, Y] point from which rivet positions are calculated</td>
</tr>
</tbody>
</table>

Figure 3.21. Definition of the rivet parameters (rivets from two consecutive layers are distinguished by the solid and dashed lines).

The rivet parameters are specified via an interactive graphical interface. The program accepts the rivet definition parameters listed in Table 3.15 and then overlays the rivet outlines.
over the layer contours (Figure 3.22). The graphical display permits confirmation of correct rivet positioning. The program also allows verification of the hatch line locations on each layer. Figure 3.23 shows a zoomed-in view of the mid-section of a dogbone-shaped tensile-test specimen with the hatch lines and the layer contour outline visible.

![Graphical interface for definition and visualization of rivets.](image)

**Figure 3.22.** Graphical interface for definition and visualization of rivets.

### 3.5 A brief discussion of the process design history

Prior to arriving at the current version of the RLCM process several design iterations have been explored. Most of the redesigns were applied to the Layer-Formation and Fibre-Resin Mixing (FRM) subsystems. The Layer-Formation subsystem consists of the Composite-Liquid Delivery (CLD), the Liquid-Leveling (LL), and the Z-platform subsystems.
3.5.1 Version 1 of the Fibre-Resin Mixing (FRM) and the Composite-Liquid Delivery (CLD) subsystems

The initial design of the FRM and the CLD subsystems (hereafter referred to as Version 1) utilized a rotating cylindrical container placed on two parallel tracks (Figure 3.24). Mixing function was achieved by keeping the container in a constantly oscillating motion. The deposition function was achieved by rotating the cylinder further forward, until the opening was over the vat. At this point the liquid was gravity forced into the vat. After a short pause, the container was retracted.
Chapter 3. Process Design

Examining the Version 1 embodiment, the following design equation with four design parameters (DPs) can be written:

\[
\begin{array}{c|c|c|c|c}
\text{Deposit Mixture} & \text{FR1} & X & 0 & 0 & X \\
\text{Meter Mixture} & \text{FR2} & X & X & 0 & 0 \\
\text{Constant Fiber Content} & \text{FR3} & 0 & 0 & 0 & X \\
\end{array}
\begin{array}{c|c|c|c|c}
\text{DP1} & \text{Rolling of Cylinder} \\
\text{DP2} & \text{Time in Overturned Position} \\
\text{DP3} & \text{Size of the Cylinder Slot} \\
\text{DP4} & \text{Cylinder Oscillation} \\
\end{array}
\]

(3.12)

Let us consider the design parameters listed in the equation and their effect on the functional requirements. Rolling of the cylinder (DP1) is required in order to deposit the mixture. However, the rolling will also affect the metering function since the cylinder has to be rolled before metering out of the mixture. Also, if the rolling is too slow, the mixture will start pouring too early, which will affect the total amount deposited. The time in the overturned position and the size of the slot directly control the amount of mixture deposited. The rate and the amplitude of cylinder oscillation will affect the degree of mixing.

Since there are more DPs than FRs, this design must be either redundant or coupled by Theorem 3 in (Suh, 1990). This design can be decoupled by adjusting the DPs in a particular sequence. In this case, the sequence is DP1 followed DP2 and DP3 in any sequence, then DP4. However, by carefully considering the operation of this system, one can observe that its performance will be highly dependent on the level of mixture present in the cylinder, i.e., this is a non-linear system. As the level decreases, as it will during normal operation, nearly all the elements in the above matrix will be affected. Below certain mixture level, some of the elements in the design matrix will change from zero to a non-zero value, coupling the design. For example, mixture deposition may not occur at all for some slot size and mixture level combinations.

After extensive testing and modifications, Version 1 of FRM and CLD was found to be incapable of delivering repeatable amounts of composite liquid into the vat: a complete redesign was required. In Version 2 of FRM and CLD subsystems, the mixing and deposition functions were physically separated as described in Section 3.3.3. Evaluation of the Version 2 has shown significantly improved consistency of the liquid volume delivered.
3.5.2 Improvements to Version 2 of the Composite-Liquid Delivery subsystem

In the first implementation of Version 2, the composite liquid was delivered to the vat by passing through two channels within the wiper. This design however resulted in non-uniform spreading of the composite liquid. The liquid tended to form mounds under the outlet of each channel. Also, occasional blockages of one of the channels diverted majority of the liquid to the other channel outlet. Layer profile studies have shown that even after wiping, layers of inconsistent and non-uniform thickness were produced.

The above problems were addressed by attaching the liquid-delivery tubing to the platform of the X-Y translator. This yielded two benefits:

- The liquid could be deposited in any desired pattern.
- The liquid travelled a much shorter and more direct path from the tubing, through a single short connector nozzle, and into the vat, thus minimizing the possibility of blockage.

3.6 Summary

The chapter described the design of the proposed RLCM process. The synthesis of the design was founded on the Axiomatic Design theory. This design theory rests upon two axioms, Axiom 1, the Independence Axiom, and Axiom 2, the Information Axiom.

Axiom 1 was particularly useful during the initial stages of design. It forces us to clarify the design objectives by unambiguously stating the functional requirements of the design. To satisfy the requirement of minimizing the information content (Axiom 2), whenever possible, the design avoided the use of tight tolerances and utilized off-the-shelf components.

The chapter then dealt with the design of the experimental prototype of the RLCM process. Here, the individual subsystems and their design details were described. Also given were the description of the experiments conducted to verify the correct operation of each subsystem.
Chapter 4. Layer Quality Studies

4.1 Introduction

Since the parts produced by a layered manufacturing process are constructed from individual layers, good surface quality and dimensional accuracy can be achieved only by building high-quality layers. As presented in Chapter 3, layer creation in the RLCM process consists of the following steps:

1. Deposit composite liquid material onto the previously solidified substrate;
2. Use a wiper blade to smooth the liquid surface and remove the excess material to form a thin and uniform liquid layer; and,
3. Solidify the liquid layer by selective curing with a UV laser.

The quality of the solid layers thus formed can be evaluated by three metrics:

1. Average layer thickness,
2. Within-layer variability of layer thickness, and
3. Between-layer variability of layer thickness.

Since the solid layers are formed from liquid precursors, formation of a liquid layer with desirable attributes is required to produce a good quality solid layer. In a pure-resin-based layered fabrication process, achievement of a smooth liquid layer relies to a large extent on the liquid’s ability to flow and thus level itself. To allow for this, a time delay from 10 to 90 sec, referred to as “Z-wait” is typically added after the wiping step (Jacobs, 1992). However, with a composite liquid, self-levelling would require excessively long delay time due to the liquid’s high viscosity (particularly at low shear rates associated with the gravity-induced self-levelling process). Thus, for composite layered fabrication, the liquid layers formed by the wiping action must be free from gross shape irregularities, as self-levelling will not produce any substantial correction.

However, exact matching of the average layer thickness with the nominal layer thickness is not required for every layer. Though, it is important is that the cumulative part height meets the specifications. As the total distance between the platform supporting the part and the
wiper bottom surface is precisely controlled by the vertical positioning sub-system, the errors in the layer height will tend to correct themselves, with thinner layers followed by thicker layers and vice versa. This self-correction occurs since a thinner-than-specified layer leaves a greater gap between the previously solidified part surface and the wiper, and a thicker-than-specified layer will leave a correspondingly thinner gap. In this manner, the layer-to-layer thickness variations tend to average out.

However, consistently thick layers present a danger of exceeding the clearance between the wiper and the previously solidified substrate, causing a part-damaging collision. Likewise, exceedingly thin layers may lead to delamination due to undercuring.

Between-layer thickness variability would indicate an unstable process. However, some variability in the first several layers is to be expected, while the process stabilizes itself.

Regarding the within-layer irregularities, because of the liquid state of the raw material, they tend to be compensated to some degree in the next layer. Thus, a thinner area within one layer will have a thicker area directly above it within the next layer. This however can lead to structural problems for the parts. If the layer thickening exceeds the cure depth, the material will remain partially liquid until post-curing stage. If localized to a small area of a layer, this may not pose a problem. However, larger uncured areas can allow air to reach interlayer boundaries, causing air bubbles to be entrapped within parts, or complete delamination in extreme cases.

4.2 Layer formation in manufacturing processes

Forming liquid into thin sheets with the aid of a straight-edge blade is a technique encountered in a number of industrial applications. These include forming adhesive layers, protective polymeric films, photographic emulsions, and magnetic dispersions. In studying these processes, the most important parameter examined is the final thickness of the formed sheet.

For example, Sullivan et al., 1987, model a blade-over-roll coating system, where a fluid contained in a bath is entrained by a rotating roll partially submerged into the fluid. The fluid is carried by the roll towards a blade resting above it, where it passes under the blade to form a thin film. FEM is used to represent the behaviour of non-Newtonian fluids in this process.
The model is divided into three regions: free surface liquid upstream of the blade, under-the-blade channel flow, and free surface liquid downstream of the blade. Setting the boundary conditions for the upstream region presents the greatest difficulty. Here, the fluid forms a "bulge" as it encounters the blade. The model is simplified by treating this region as an infinite bath of fluid of fixed elevation. The model also accounts for the surface tension effect at the downstream free surface. The authors also perform measurements of the final coating thickness with a micrometer-driven needle. Good agreement with the model (on the order of 1-3%) is reported.

Tape casting is another process employing blade-coating technique. The process forms liquid ceramic slurry into thin sheets (0.03-1 mm), which are subsequently cut into the desired shapes and fired to produce electronic ceramics such as capacitors, piezoeactive devices, and catalyst substrates (Wang, 1976). The tape-casting process is similar to the layer formation process employed in this thesis since in both cases thin sheets must be formed out of viscous liquid with suspended solid particles. The processes differ, however, in terms of particle dimensions: the ceramic slurries typically contain roughly spherical particles, 0.5-2 μm in size, compared with 15 μm diameter and 500-1500 μm long fibres employed in this thesis.

Tape-casting equipment consists of a moving sheet-like carrier above which sits a slurry-filled reservoir with a narrow rectangular outlet on one side. As the carrier passes under the reservoir, the fluid is "pulled" through the outlet to form a thin sheet on the carrier surface. The outlet height is controlled by raising or lowering of a blade.

To analyze the tape-casting process, one can either employ analytical fluid mechanics formulations with some simplifying assumptions, or resort to numerical modelling. The latter approach is taken in Loest et al., 1993. The paper utilizes an FE model to represent only the under-the-blade channel and the downstream free surface liquid. Also reported are measurements of the free surface shape obtained by projecting a line shadow onto the liquid surface and using digital image processing to analyze it. Numerical results assuming a Newtonian and non-Newtonian (shear-thinning) liquid models were presented. The non-Newtonian model was found to give smaller maximum error (7% vs. 12%) for the predicted surface height.
Loest et al., 1994, employ FEM to represent the fluid reservoir in addition to the under-the-channel and the downstream regions. Based on the model, the reservoir/blade design changes are proposed. The model predicted that by tapering the reservoir cross-section towards the liquid outlet one could reduce the size of the recirculating regions within the reservoir. Such regions are undesirable since they lead to material stagnation.

Chou et al., 1987, developed an analytical model by assuming Newtonian fluid behaviour and using linear superposition of drag- (Couette) and pressure-driven flows. Experiments were conducted with 50% by weight ceramic slurries (viscosity 1500 cP, density 2.03 g/cm³), and operating parameters consisting of blade gap of 0.4 mm, reservoir height of 1 cm, and a range of tape speeds from 0.44 to 4.4 cm/s. Measuring tape thickness after the firing and adjusting for tape shrinkage, good agreement (within 1-2%) was obtained for tape speeds over 2 cm/s, while for lower speeds the prediction error increased to about 10%.

Pitchumani and Karbhari, 1995, recognizing the non-Newtonian nature of most ceramic slurries, use the Ostwald-de Waele fluid model, with its viscosity described by a power-law dependence on the shear rate. The model yields an analytical expression for the final tape thickness as a function of various slurry and process parameters. The analysis could not employ the linear superposition of pressure and drag flows due to the non-Newtonian fluid assumption. Instead, the flow continuity and momentum equations, which take into account the pressure gradient effects and the moving substrate boundary conditions, were solved.

Layer formation in a stereolithography process (based on pure photopolymers) has been empirically investigated by Renap and Kruth, 1995. The factors studied include the gap width, the wiper speed, wiper thickness, and resin viscosity. A special setup was created which allowed accurate measurements of the liquid height after wiping and the gap between the substrate and the wiper blade. The liquid height was measured with a laser distance sensor. On a completely solid substrate, a layer height of 0.25 mm was obtained for wiping speeds ranging from 2 to 12 cm/s, blade width of 10 mm, and gap set at 0.5 mm. The height increased slightly (to 0.27 mm) at 0.5 cm/s. The result is explained in the paper with a rheological model similar to that of Chou et al., 1987.

Renap and Kruth, 1995, also investigate the wiping of a solid substrate containing a trapped liquid volume. Trapped volume refers to a volume of liquid resin completely
enclosed by a solid substrate and unconnected with the rest of the liquid in the vat. For the wiping process, this means that the wiper-to-substrate gap becomes effectively greater when the blade is traversing above the trapped volume. The experiments found that the layer height above the trapped volume was a strong function of the wiping speed. As the speed was increased, the blade entrained greater amount of liquid from the trapped volume, leading to locally thin layers. With slower speeds, an opposite effect was observed: due to the wide opening, the liquid was able to flow back under the wiper blade, and an excessive local layer thickness resulted. The authors then find the optimum velocity, which results in layers of correct thickness. This velocity is found to be a function of the trapped volume depth and geometry.

In the following section, two models of layer creation in the RLCM process are developed. First, a simple rheological model based on Newtonian fluid assumption is presented (similar to Chou et al., 1987). Second, a model taking into account the shear-thinning viscosity of the composite liquids is given (Pitchumani and Karbhari, 1995). After presenting the models, the subsequent section describes experimental observations of the layer heights obtained from the cross-sections of layered specimens.

4.3 Rheology of layer formation

Formation of a thin liquid layer by a straight-edge blade is a complex fluid mechanics problem affected by a number of parameters. These include the shape of the blade edge, the wiping speed, the amount of material accumulated at the leading edge, and the liquid viscosity, Figure 4.1. The height of the resulting liquid layer, \( h \), is normally not equal to the gap between the underlying solid surface and the blade bottom, \( h_0 \).
4.3.1 Layer formation model based on Newtonian fluid assumption

The interrelationship of the configuration parameters can be approximated by a simplified fluid-flow model (Figure 4.2) based on a combination of drag- and pressure-driven flows (also known as Couette and Poiseille flows, respectively), Figure 4.3 (Fay, 1994). The model is derived by assuming (a) laminar viscous flow, (b) constant viscosity, and (c) a channel length $L_0$ which is much greater than the gap $h_0$. The last assumption is only a rough approximation, as the actual gap $h_0$ in this thesis is 0.3-0.6 mm, while the flat bottom surface of the RLCM wiper is only 1-mm wide ($L_0$).

\[ Q_d = \frac{U_0 h_0 W}{2} \quad \text{and} \quad Q_p = \frac{h_0^3 \Delta P W}{12\eta}, \]  

(4.1)
where $W$ is the channel width (transverse to the flow direction), $\eta$ is the viscosity of the composite liquid, and $\Delta P$ is the pressure difference between the entry and exit points of the under-the-blade channel. $\Delta P$ is given by:

$$\Delta P = \frac{\rho g H_0}{L_0},$$  \hspace{1cm} (4.2)$$

where $\rho$ is the density of the composite liquid and $g$ is the gravitational acceleration.

**Figure 4.3. Velocity profiles in the under-the-blade channel due to (a) drag- and (b) pressure-driven flows.**

Combining the two flow types by linear superposition, the total flow is given by:

$$Q_1 = Q_d + Q_p = \left( \frac{U_0 h_0}{2} + \frac{h_0^3 \rho g H_0}{12 \eta L_0} \right) W.$$  \hspace{1cm} (4.3)$$

At some distance downstream from the blade, the fluid velocity must equal the substrate velocity. Then, this flow rate is:

$$Q_2 = U_0 h W.$$  \hspace{1cm} (4.4)$$

Conservation of mass dictates that the flow rates $Q_1$ and $Q_2$ must be equal. Therefore, equating (4.3) and (4.4), the following expression is obtained for the final layer thickness after wiping:

$$h = \frac{h_0}{2} + \frac{h_0^3 \rho g H_0}{12 \eta U_0 L_0}.$$  \hspace{1cm} (4.5)$$
4.3.2 Layer formation model based on non-Newtonian fluid assumption

As the rheological measurements of the fibre-photopolymer mixtures have shown, the mixtures behave as non-Newtonian fluids with a strong shear-thinning characteristic: the viscosity decreases with the increase of shear rate (Chapter 2). The linear region in the log-log plot of the viscosity versus the shear-rate can be described by a power-law equation (Fay, 1994):

\[ \eta = m |\dot{\gamma}|^{n-1} \]  \hspace{1cm} (4.6)

where \( m \) and \( n \) are empirically determined constants and \( |\dot{\gamma}| \) is the shear rate, \( \dot{\gamma} = U_0 / h_0 \). For a Newtonian fluid, \( n = 1 \), and thus the viscosity does not depend on the shear rate.

When a fluid's viscosity is shear-rate dependent, one cannot derive the fluid flow expression by linearly superimposing the drag- and pressure-driven flows (Pitchumani and Karbhari, 1995). To arrive at the desired flow expression, one must integrate the flow velocity over the channel vertical dimension. To perform the integration task, two possible flow cases must be treated separately (Figure 4.4): Case I is characterized by the monotonically decreasing velocity profile with height, and Case II is characterized by the presence of a maximum in the velocity profile. In Case II, the integration is performed separately for the monotonically increasing and decreasing regions.

![Figure 4.4. Velocity profiles in the under-the-blade channel for (a) Case I and (b) Case II flow regimes.](image)

The following dimensionless parameters need to be defined before proceeding with the derivation of the layer-height expressions:
Chapter 4. Layer Quality Studies

\[ \text{Re} = \frac{\rho U_0 h_0}{\eta}, \quad \text{Fr} = \frac{U_0^2}{gH_0}, \quad \text{and} \quad \alpha = \frac{\text{Re} h_0}{\text{Fr} L_0}, \quad (4.7) \]

where Re is the Reynolds number, denoting the ratio of the inertial and viscous effects; Fr is the Froude number, representing the hydrostatic head effects due to the height of the liquid accumulated in front of the wiper; and \( \alpha \) is a combined design parameter.

After performing the required integration, the layer height for Case I is given by

\[ h^{(I)} = h_0 \frac{\alpha^N}{1 + N} \left[ (1 - \lambda^{(I)})^{1+N} - \frac{(1 - \lambda^{(I)})^{2+N}}{2 + N} + \frac{(-\lambda^{(I)})^{2+N}}{2 + N} \right], \quad (4.8) \]

where \( N = 1/n \) (for notational convenience), and \( \lambda^{(I)} \) is an integration constant corresponding to the location of the maximum in the velocity profile curve in the normalized coordinates (i.e., \( \lambda = y_{\max}/h_0 \), where \( y_{\max} \) is the location of the maximum). The constant \( \lambda^{(I)} \) is defined by the transcendental equation:

\[ (1 - \lambda^{(I)})^{1+N} - (-\lambda^{(I)})^{1+N} = (1 + N)\alpha^{-N}. \quad (4.9) \]

Note that by definition, \( \lambda^{(I)} \) must be negative, since the maximum in Case I occurs at negative \( \lambda \).

For Case II, the results are:

\[ h^{(II)} = h_0 \left( \lambda^{(II)} + \frac{\alpha^N}{2 + N} \left[ (1 - \lambda^{(II)})^{2+N} + (\lambda^{(II)})^{2+N} \right] \right), \quad (4.10) \]

where \( \lambda^{(II)} \) is obtained by solving

\[ (1 - \lambda^{(II)})^{1+N} - (\lambda^{(II)})^{1+N} = (1 + N)\alpha^{-N}, \quad 0 < \lambda^{(II)} < 1. \quad (4.11) \]

In order to choose which of the two cases is appropriate, a critical value of \( \alpha, \alpha^* \), is used:

\[ \text{if} \quad \alpha \leq \alpha^*, \quad \text{use Case I} \quad \text{and}, \]

\[ \text{if} \quad \alpha > \alpha^*, \quad \text{use Case II} \quad (4.12) \]

where \( \alpha^* \) is given by

\[ \alpha^* = \left(1 + N\right)^{\frac{1}{2}} \quad (4.13) \]
4.3.3 Layer-thickness prediction based on the layer-formation models

The layer height will be predicted herein for the composite liquid formulation most commonly used in this thesis, namely SL5170 resin with 15% by volume of 1.6-mm 737BD short-glass fibres. First, the power-law relationship for the liquid's viscosity must be established from the experimental data (Chapter 2). Figure 4.5 shows the linear portion of the log-log plot for the 15% fibre-resin mixture (shear rate range from 1.4 to 225) (see **Figure 2.18** for the original data). By fitting the following linear equation to the data, the coefficients \( m \) and \( n \) in Equation (4.6) are obtained:

\[
\log(\eta) = \log(m) + (n - 1)\log(\dot{\gamma})
\]  

(4.14)

The power-law expression for this composite liquid (expressed in the units of cP) is now obtained as:

\[
\eta = 857.01 \dot{\gamma}^{0.0622}
\]

(4.15)

Also, the critical value of \( \alpha \), from Equation (4.13), is \( \alpha^* = 1.975 \).

![Figure 4.5. Power-law dependence of the viscosity on the shear rate for the mixture of SL5170 resin and 15% by volume 737BD short-glass fibres.](image)

Table 4.1 shows the parameters used in modelling the layer-formation process. The table lists the default parameter values used whenever the parameter value is held constant. In the
following figures, the layer thickness predictions are normalized by the wiper gap height, $h_0$. Each figure shows two plots: the results of the model based on the Newtonian fluid assumption are shown by a dashed line (Model 1) and the corresponding results using the model based on the non-Newtonian fluid assumption are shown by a solid line (Model 2). Also indicated on each figure is a boundary between the Case I and Case II operating regimes.

Table 4.1. Layer-formation model parameters.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Density, $\rho$</td>
<td>1.42 g/cm$^3$</td>
</tr>
<tr>
<td>Fluid bulge height, $H_0$</td>
<td>5.0 mm</td>
</tr>
<tr>
<td>Wiper gap height, $h_0$</td>
<td>0.6 mm</td>
</tr>
<tr>
<td>Wiper blade thickness, $L_0$</td>
<td>1.0 mm</td>
</tr>
<tr>
<td>Wiping speed, $U_0$</td>
<td>1.5 cm/s</td>
</tr>
</tbody>
</table>

Figure 4.6 shows the relationship between the layer thickness and the wiping speed. The layer thickness is predicted to decrease with the increasing wiping speed. As the wiper moves faster, the fluid has less time to flow back under the blade, making the remaining fluid layer thinner. Figure 4.7 shows that an increase of the wiper gap leads to an increase in the layer height expressed as a fraction of the wiper gap. The increased gap significantly decreases the resistance to the reverse flow of the fluid due to the bulge accumulating in front of the wiper, which in turn leads to a thicker fluid layer. Figure 4.8 demonstrates that an increase of the bulge height leads to a proportionate increase in the layer height. Thus, if the height of the accumulated liquid in front of the blade is not constant throughout the wiping stroke, variability in the layer height may result.

In all cases, the predictions of the Newtonian model are lower than the non-Newtonian model. Sullivan et al., 1987, noted similar results when using FEM to model the blade coating process: when the shear-thinning (as opposed to constant-viscosity) fluid model was used, thicker layers resulted.
Figure 4.6. Normalized layer thickness as a function of the wiping speed.

Figure 4.7. Normalized layer thickness as a function of the wiper gap.
4.4 Experiments

The objective of the experiments in this thesis was to evaluate the geometrical quality of the layers fabricated by the RLCM prototype system. Specifically, the layers were to be evaluated in terms of:

1. Average layer thickness,
2. Within-layer variability of layer thickness, and
3. Between-layer variability of layer thickness.

4.4.1 Procedure

To evaluate the layer quality, the experimental system was used to build pure and composite rectangular parts (25×30×4.8 mm) whose cross-sectional profiles were examined microscopically (Figure 4.9). The parts were fabricated with one corner removed to identify orientation during building. Results from four test parts will be presented below (Table 4.2). These parts comprise a composite part (LQ_C) and a pure-resin part (LQ_P) built on the RLCM prototype; a benchmark pure-resin part (LQ_SL_P) fabricated on a commercial SL machine (Sony JSC-2000 Solid Creator); and a composite part (LQ_CE) fabricated during the early stages of the RLCM process development.
Table 4.2. Layer quality test parts.

<table>
<thead>
<tr>
<th>Specimen Name</th>
<th>Description</th>
</tr>
</thead>
<tbody>
<tr>
<td>LQ_SL_P</td>
<td>Reference pure-resin part built on a commercial SL equipment</td>
</tr>
<tr>
<td>LQ_P</td>
<td>Pure-resin part built on RLCM after process improvements</td>
</tr>
<tr>
<td>LQ_C</td>
<td>Composite part built on RLCM after process improvements</td>
</tr>
<tr>
<td>LQ_CE</td>
<td>Composite part built on RLCM during the early process development stage</td>
</tr>
</tbody>
</table>

The composite parts (LQ_C and LQ_CE) were built from Allied Signal 2202SF photopolymer and Owens Corning 737BD 1.6 mm milled glass fibres (15% by volume). The RLCM-fabricated pure-resin part (LQ_P) was built from 2202SF photopolymer and the benchmark part (LQ_SL_P) was built from DeSolite SCR310 photopolymer. All RLCM-fabricated parts were built at a location within the machine vat as specified in Figure 4.10. Additionally, the specimens LQ_P and LQ_C were built on acrylic support blocks. The intention was to elevate the specimens from the platform surface and to provide a flat reference surface.

Layer quality was evaluated by examining a number of vertical sections for each specimen (Figure 4.11). Only the LQ_CE specimen was sectioned 9 times. For the remaining specimens, either 5 or 3 sections were made. The greater number of sections was examined for the early LQ_CE specimen in order to verify the procedure for extracting three-dimensional layer shape. Once the feasibility of such procedure was established, the number of sections was reduced to expedite data collection.
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Figure 4.9. Specimen built for layer-quality study.

Figure 4.10. Location of the layer-quality-study specimens in the vat.

Figure 4.11. Location of the cross-sections within a specimen.
The following method was used to examine the sections. First, to facilitate handling, each specimen was encased in a cylindrical mould (37 mm in diameter and 25.5 mm in height) made from a polyester-based potting resin cured by a hardener (methyl ethyl ketone peroxide), and the mould was mounted onto an aluminum plate (Figure 4.12). To section the mounted specimen, it was milled to the desired height and then wet-polished with SiC abrasive papers of progressively decreasing grit size.

![Diagram of mounting process]

**Figure 4.12. Mounting of the layer-quality specimens.**

Location of the layer boundaries was measured through a microscope equipped with a precision X-Y translation stage, with a measurement resolution of 0.0001 inch. However, the X-Y table micrometers’ range was only 25 mm, which is less than the 30 mm required. To overcome this, another micrometer and a translation stage with a 50 mm travel were mounted on top of the microscope’s table. Thus, the Z-axis measurements were made by the microscope’s stage and the Y-axis measurements were made with the external stage (Figure 4.13). The external micrometer’s resolution is 0.001 mm.

The boundary locations were sampled by first setting the Y coordinate and then measuring the boundary position in the Z coordinate. The process was repeated at equal intervals along the Y direction (intervals ranging from 1.5 to 3 mm, depending on the specimen).
Figure 4.13. Orientation of specimen in the measuring setup.

The layer boundaries were numbered starting from the bottom of the plate, with the boundary 1 corresponding to the top surface of the bottom-most layer. The location of part's bottom surface was not measured for LQ_CE specimen since it was difficult to locate the surface precisely due to the presence of supports and significant overcure. The last boundary measured is the top surface of the specimen. The sections were numbered according to the cutting sequence, with Section 1 being at the point furthest away from the wiper in its resting position (Figure 4.11).

Since cutting and polishing each section requires removing the specimen from the microscope stage, unless special measures are taken, the positions of layer boundaries measured in each section will be referenced to a different coordinate origin due to a small shift occurring when the specimen is removed from, sectioned, and remounted onto the microscope stage. Thus, to evaluate the layer shape along the X direction, the following steps were taken. Four mounting holes were drilled in the aluminum plate holding the specimen. These holes aligned precisely with the corresponding threaded holes on the microscope stage. The aluminum plate could then be remounted repeatedly onto the same location on the microscope stage. To adjust for minor positioning errors, registration markers were inscribed on the aluminum plate, and their position was measured for each section. The location of each section in the X direction was measured using a height gauge.
4.4.2 Results

4.4.2.1 Layer profiles

Figure 4.14 shows the layer profiles for one cross-section of a pure-resin part built on the commercial machine (LQ_SL_P) and for an equivalent pure-resin part built on the RLCM prototype system (LQ_P). Figure 4.15, on the other hand, shows layer profiles for two composite parts built on the RLCM prototype. Ideally, all the plots should consist of straight horizontal lines, representing layer boundaries, separated by the nominal layer thickness of 0.3 mm.

Statistical analysis of the measurements from several sections of the above four parts was carried out after discarding the data for the first 8-10 layers in order to allow for process stabilization. (Normally, production parts would be built on supports of at least a few millimetres high, i.e., separated by several layers from the supporting platform.) Three parameters are shown in Table 4.3 for each part: the average layer thickness, which is the mean of layer boundary separation values measured across all the part sections; the standard deviation within the layers, which is a measure of the uniformity of the individual layers; and the standard deviation between the layers, which is a measure of the layer-to-layer variability of the average layer thicknesses. The results verify that the prototype RLCM system is able to build layers with a mean thickness very close to the nominal value. Also, the variabilities in the building process of the layers are quite close to those obtained on the commercial machine. The composite part layer variability is only slightly higher than that for the pure-resin part.
Figure 4.14. Layer profiles for pure-resin parts made (a) on a commercial SL machine (LQ_SL_P) and (b) on the RLCM prototype (LQ_P).

Figure 4.15. Layer profiles for composite test parts made on the RLCM prototype: (a) early in the process design (LQ_CE) and (b) after process improvements (LQ_C).

The improvements in layer quality brought about by process modifications are demonstrated by comparing the part fabricated early in the process development (Figure 4.15(a)) and another made after some key process adjustments (Figure 4.15(b)). A number of layers in the LQ_CE specimen are seen to be extremely distorted, with wide variations in layer thickness. Also, the layers tend to be excessively thin on average (mean layer thickness of 0.266 mm was observed). The main process change between the two specimens is a switch from the delivery of mixture through the nozzles within the wiper to a mixture delivery via a simultaneous deposit-and-translate method. The new delivery method resulted in a more uniform spreading of the raw material, which led to a more consistent layer shape.
4.4.2.2 Layer thickness variations

Layer-to-layer thickness variations can be observed by plotting the average layer thickness against the layer number. Figure 4.16 shows such plots for the pure-resin parts (LQ_SL_P and LQ_P), and Figure 4.17 shows the corresponding plots for the composite parts (LQ_C and LQ_CE). First, one notes the very consistent layer thickness achieved in the commercial process after some initial minor instability in the layers 1-7. A notable feature of the plot for the pure-resin part built on RLCM prototype (LQ_P) is that, starting from 0.5 mm for the first built layer, the layer thickness values can be seen to decline steadily, reaching the nominal value after 8-9 layers. For the composite specimen LQ_C, the layer thickness averages also show a similar, but faster, convergence to the nominal value (Figure 4.17(a)). (The sharp drop of layer thickness at layer 15 was caused by equipment malfunction and the data for that layer are not included in the layer statistics calculations.) Finally, Figure 4.17(b) shows the consistently thin layers and large thickness variability observed in the LQ_CE specimen.

As has been noted above, the average layer thickness plots for LQ_P and LQ_C specimens both display a gradual asymptotic change in layer thickness as it approaches the nominal value. The layer formation models developed earlier in this chapter provide an explanation for this phenomenon. As shown by these models, the actual thickness of each layer formed is equal to a fraction of the gap between the wiping blade and the solid substrate.

<table>
<thead>
<tr>
<th>Specimen Name</th>
<th>Number of Sections</th>
<th>Total Number of Points</th>
<th>Layers Used for Statistics</th>
<th>Average Layer Thickness (mm)</th>
<th>St. Dev. Within Layers (mm)</th>
<th>St. Dev. Between Layers (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LQ_CE</td>
<td>9</td>
<td>2835</td>
<td>10-16</td>
<td>0.266</td>
<td>0.026</td>
<td>0.013</td>
</tr>
<tr>
<td>LQ_C</td>
<td>3</td>
<td>528</td>
<td>8-14</td>
<td>0.315</td>
<td>0.021</td>
<td>0.010</td>
</tr>
<tr>
<td>LQ_P</td>
<td>5</td>
<td>1280</td>
<td>10-16</td>
<td>0.306</td>
<td>0.016</td>
<td>0.007</td>
</tr>
<tr>
<td>LQ_SL_P</td>
<td>3</td>
<td>816</td>
<td>11-17</td>
<td>0.313</td>
<td>0.008</td>
<td>0.002</td>
</tr>
</tbody>
</table>
Layer Quality Studies

of the previous layer, i.e. \( h/h_0 = \Omega \), where \( \Omega \) is itself a function of the gap height. Then, given a value for the first surface-to-blade gap, \( h_0^{(1)} \), and \( \Omega \), the thickness of layer \((i+1)\), \( h^{(i+1)} \), can be calculated iteratively using the following relationships:

\[
h^{(1)} = \Omega h_0^{(1)} \quad \text{and} \quad h^{(i+1)} = \Omega \left( h_0^{(1)} + ih_{\text{nom}} - \sum_{k=1}^{i} h^{(k)} \right)
\]

(4.16)

where \( h_{\text{nom}} \) is the nominal layer thickness, which equals the incremental downward displacement of the platform for each layer.

The model shown by a dashed line in Figure 4.16(b) was obtained by assuming the first surface-to-blade gap \( h_0^{(1)} = 1.7 \) mm and a constant fraction value \( \Omega = 0.3 \). Note that these numbers were only used as parameters for the best fit of the model to the layer thickness data: actual initial gap value was not measured in this test. One may observe that the layer formation models predict that \( \Omega \) cannot be below 0.5. The discrepancy may be due to the divergence between the model assumptions and the actual experimental environment.

A similar model was fitted to the plot of the composite part layer thickness averages (Figure 4.17(a)), with the only difference being that the value of \( \Omega \) was now a variable calculated from Equation (4.5), which is the layer formation model based on the Newtonian fluid assumption. This model fits well, and correctly predicts the much quicker approach to the nominal layer thickness than the pure-resin specimen.

To confirm that \( \Omega \) is indeed a fraction of the wiper-to-substrate gap, a pure-resin test part was built where the gap height was measured between the wiper blade and the platform surface prior to building the first layer. The cross-section examination showed that, although the gap was 0.60 mm, the first part layer built was on average 0.40 mm, i.e., \( \Omega = 0.67 \).
Figure 4.16. Average layer thickness observations for pure-resin parts made (a) on a commercial SL machine (LQ\_SL\_P) and (b) on the RLCM prototype (LQ\_P).
Figure 4.17. Average layer thickness observations for composite test parts made on the RLCM prototype: (a) after process improvements (LQ_C) and (b) early in the process design (LQ_CE).

4.4.2.3 Microscopic observations of rivets

The interlayer rivets’ shapes within the dogbone tensile test specimens were examined microscopically. Figure 4.18 shows a vertical section through the central plane of the specimen. All the layer boundaries are visible on the right half of the photograph. The interruptions of the boundary seen on every second layer in the middle correspond to interlayer rivets. The rivet boundaries can be more clearly discerned in a photo taken at a higher magnification (Figure 4.19). Layer boundaries are indicated by triangular markers on both photographs.
4.5 Summary

This chapter emphasized the importance of forming good quality liquid layers to building of good quality layered composite parts. Similarity between the layer formation in the RLCM process and in several well-researched industrial processes was pointed out. The layer formation by a blade is employed to create thin layers of ceramic slurries in a tape-casting process. Two fluid mechanics models of this process available in the literature were employed to model the layer formation in the RLCM process. One model is developed based on an assumption of a Newtonian fluid and thus allows linear superposition of the two types of flows occurring between the wiper and the substrate: the drag- and pressure-induced flows. The second model assumes a non-Newtonian fluid and thus must integrate the flow velocity field over channel height.
By employing the above models, the layer thickness was predicted as a function of such factors as the wiper translation speed, wiper-to-substrate gap, and the height of the liquid bulge accumulating ahead of the wiper. The predictions show that all of the above factors indeed influence the final layer height.

The chapter also reports on a number of experimental studies designed to evaluate the layer quality in terms of (a) average thickness, (b) variation of thickness within layers, and (c) variation of thickness between layers. Evaluation of a benchmark part built on a commercial machine utilizing a pure photopolymer, showed that layers of comparably good quality can be obtained for the parts built on the RLCM prototype.

Also explained in the chapter is the gradual change in the average layer height observed in some RLCM-fabricated specimens. The variation manifests itself as a steady decline from an initial excessive layer thickness to the nominal value. This variation is explained by assuming that the layer height is equal to a fraction of the wiper-to-substrate gap (as predicted by the layer formation models).

The experiments presented in the chapter also established the procedure for creating a three-dimensional representation of the layer shape via a contour plot. Such results were obtained by precise recording of absolute coordinates of the individual specimen sections and subsequently expressing the layer boundary positions in a common coordinate frame.
Chapter 5. Fibre-Orientation Distribution Studies

5.1 Introduction

5.1.1 Motivation

Properties of fibre-reinforced composites are largely determined by three primary factors: fibre content, fibre aspect ratio, and fibre orientation. In particular, fibre orientation plays an important role. For example, in the equation below, the prediction of composite tensile modulus using a modified rule-of-mixtures model (Piggott, 1981) is dependent on the orientation-efficiency factor $\chi_1$:

$$E_c = \chi_1 \chi_2 v_f E_f + v_m E_m$$ (5.1)

where $E_f$ and $E_m$ are, respectively, fibre and matrix tensile moduli, and $v_f$ and $v_m$ are, respectively, fibre and matrix volume fractions. The $\chi_1$ varies from 0 to 1 and depends on the degree of alignment of the fibres with the direction in which the modulus is estimated: greater alignment leads to values closer to 1. The fibre-length correction factor $\chi_2$ depends on the fibre aspect ratio: its value approaches 1 for aspect ratios over 100.

Using Equation (5.1), one can note that, given typical parameters of a layered short-fibre composite ($v_f = 0.15$, $E_f = 70$ GPa and $E_m = 1.5$ Gpa), as $\chi_1$ varies from 0.3 to 0.8, i.e., from uniformly distributed to nearly aligned orientation state, the predicted composite modulus more than doubles, from 4.4 GPa to 9.9 GPa.

This chapter presents a novel method for statistical characterization of the fibre-orientation state within short-fibre layered composites. Since fibre orientation plays such an important role in determining the composite material properties, its characterization is essential for providing a complete analysis of any process producing composite parts. Certain aspects of the layered manufacturing process presented in this thesis, such as dimensional restrictions imposed on fibres by the layers and broad distribution in fibre alignment, necessitated development of new methodologies for determining the fibre orientation.

The proposed method is based on two principles: (1) intersection of a plane and a cylindrical fibre is an ellipse, and (2) this ellipse shape is a function of fibre orientation.
relative to the intersecting plane. Therefore, by sectioning a specimen, acquiring images of
the polished cross-section surfaces, and collecting the fibre ellipse data from these images, the
orientation of every fibre intersecting the sectioning plane can be determined.

The set of fibres intersected by the plane is a sample of the total set of fibres existing
within a region of interest in the specimen. Based on this sample, it is desired to find the
probability of encountering fibres with a particular orientation within this region of interest.
This probability is determined by first obtaining the probability distribution for fibre
orientations within the sectioning plane, and then relating this distribution to that within the
volume of the region of interest.

5.1.2 Previous work

The orientation of an individual fibre in a composite can be described by a single angle in
a two-dimensional case and by two angles, e.g., $\theta$ and $\phi$ (Figure 5.1), in a three-dimensional
case. The overall fibre orientation in a composite can be statistically summarized by a fibre
orientation distribution (FOD), $p(\theta, \phi)$, which is a joint probability density function
describing the probability of finding fibres with orientation $(\theta, \phi)$ in the specimen.
Frequently, it is desired to find the variation of probability as a function of either one of the
two angles. This leads to calculation of marginal probability density functions\(^1\), $p(\phi)$ and
$p(\theta)$, Figure 5.2. In the most general case, for fibres with variable length, $l$, to completely
characterize their state in a composite, a joint orientation/length distribution function $p(\theta, \phi, l)$
has to be used.

Fibre orientation studies can be subdivided into two major areas: those dealing with
continuous and those dealing with short fibres. Continuous fibres tend to be highly aligned in
their typical applications. Subsequently, the corresponding FOD measurement methods are
mainly focused on small angle misalignments from the nominal fibre direction (Yurgatis,
1987, Stecenko and Piggott, 1997). Majority of short-fibre orientation studies, on the other
hand, concentrates on injection-moulded thermoplastic composites. These studies characterize

---

\(^1\) If $p(x_1, x_2)$ is a joint probability density function, then $p(x_1) = \int p(x_1, x_2) \, dx_2$ and $p(x_2) = \int p(x_1, x_2) \, dx_1$ are
the marginal probability density functions of $x_1$ and $x_2$, respectively (Guttmann et al., 1982).
local preferred fibre orientation due to the mould flow patterns. Short-fibre composites typically have wide distributions of fibre orientations. As will be discussed shortly, this variability creates a number of difficulties.

Figure 5.1. Fibre intersected by a section plane.

Figure 5.2. Examples of possible FOD for (a) misalignment angle and (b) azimuth angle.

Short-fibre orientation studies can be further categorized by whether two- or three-dimensional fibre orientations are assumed. Experimental results in the former category are reported in Sirkis et al., 1994, Kamal, 1986, Vincent and Agassant, 1986, Ranganathan and Advani, 1990. The two-dimensional nature of the problem considerably simplifies the data extraction and makes it amenable to automation. Data can be derived either from polished
sections by reflective microscopy, or from microtomed samples by contact microradiography or transmission optical microscopy.

For the layered composites considered in this thesis, while there exists a degree of preferential fibre alignment within the layers, there also exists a significant out-of-plane component. Therefore, it is necessary to obtain a complete three-dimensional FOD.

Lian et al., 1994, used an "optical sectioning" technique to obtain three-dimensional FOD, whereby transparent samples with opaque tracer fibres were observed with an optical microscope. By focusing on a sequence of depths up to 1.5 mm into the sample, a series of sample "slices" were obtained. These 2-D slices were "stacked" to derive 3-D fibre orientation. Although reliable results were obtained, the requirements for transparent matrix, opaque fibres, and depth restriction significantly limit the utility of this method, especially as applied to the layered composites with glass fibres.

Fischer and Eyerer, 1988, Bay and Tucker, 1992, and Hine et al., 1993, measured short-fibre orientations by examining polished cross-sections with an optical microscope. A similar method is suggested by Zhu et al., 1997a, who however did not report on any experimental results. The method considers the intersection of a cylinder (i.e., a fibre) with a plane (Figure 5.1), to calculate the misalignment angle $\phi$ between the plane's normal and the cylinder's longitudinal axis:

$$\phi = \arccos(B/A)$$  \hspace{1cm} (5.2)

where $A$ is the major radius and $B$ is the minor radius of the ellipse, respectively. The rotation about the section-plane normal, or azimuth angle, $\theta$, is defined by the direction of the major axis. The assumptions implicit in applying this method are: (a) fibres are circular, and (b) fibres are straight on the scale of the ellipse dimensions.

Several problems arise when applying the above method directly to obtain an accurate 3-D FOD. First, the ellipse-based calculation of fibre orientations is biased for the near-zero misalignment angles. Second, the method always produces two alternative solutions for individual fibre orientation, both with the same $\phi$, but with $\theta$ separated by 180 degrees (Figure 5.3). The ambiguity of the orientation data makes it impossible to predict composite properties in any other direction except that normal to the section plane, as the orientation data
cannot be transformed. Third, as will be explained shortly, the raw FOD data obtained from specimen sectioning has to be adjusted to correctly represent the distribution of fibre orientations within the specimen.

![Diagram of fibre orientations](image)

**Figure 5.3. Two possible orientations derived from the same fibre cross-section.**

In this chapter, a novel method is proposed for obtaining a three-dimensional FOD by combining data from two consecutive closely spaced cross-sections of a specimen. This method produces unbiased distribution data for the near-zero misalignment angles and resolves the orientation duality problem. The chapter will first present the supporting theory for the proposed methodology, and subsequently will deal with the practical issues of image acquisition and processing (in particular, ellipse matching between cross-sections) in Section 5.3. Experimental results for layered composites built via two alternative building methods are reported in Section 5.4.

### 5.2 Proposed methodology for obtaining 3-D fibre-orientation distribution

This section first explains the shortcomings of the currently employed single-section method of finding fibre orientations and then derives the necessary theoretical foundation for the two-section-based method. The two-section method provides additional important information when used on short-fibre composites: it can be used to estimate the average fibre length in the specimen. Section 5.2.4 derives the relationship needed to obtain this estimate.
Chapter 5. Fibre-Orientation Distribution Studies

The last subsection addresses the issue of relating the FOD observed within a plane to the FOD within the specimen volume. The two distributions are different because the probability of fibre intersection by a plane is orientation-dependent: it decreases with the increase of the misalignment angle. Alternative methods for adjusting for this effect are shown, with a novel approach developed for the case of variable-length fibres within layered composites.

5.2.1 Orientation-duality problem and estimation bias in a single-section method

In a single-section method, fibre orientations are obtained from only one planar section of a specimen. This approach, however, has two significant shortcomings. The first shortcoming is the significant bias and noise sensitivity in the estimation of the near-zero misalignment angles. This bias causes consistent errors in the misalignment angle estimates for fibres nearly aligned with the section normal, since noisy data makes it impossible to select correctly the major and minor radii of the resulting nearly circular ellipses. Simply choosing the smaller measured radius as the minor one leads to undercounting of fibres in the determination of FOD for very small angles, as the noisy data is effectively “reflected” back on itself as opposed to being averaged around zero. The maximum fibre misalignment angle that would still be affected by the bias, $\phi_{bias}$, is thus related to the measurement error of the ellipse radii. It can be roughly estimated as:

$$\phi_{bias} = \arccos\left(\frac{n_{pix} - 1}{n_{pix}}\right)$$

where $n_{pix}$ is an integer equal to the fibre radius in image pixels. In our experiments, $n_{pix} = 10$, which results in $\phi_{bias} = 25.8^\circ = 30^\circ$. In other words, one can conclude that $\phi$ angles cannot be measured accurately below this $\phi_{bias}$.

Yurgatis, 1987, addressed the above problem, while dealing with continuous fibres, by sectioning the specimen so that the average misalignment angle is about 85 degrees, and thus avoiding the biased estimation range. Hine et al., 1993, also noted the existence of the above problem when dealing with aligned short fibres, and recommended sectioning at a non-zero angle to the preferential fibre direction in order to improve the measurement accuracy. Such an approach is suitable for continuous or short fibres which are well aligned (e.g., Yurgatis
reported orientation variability of no more than ±2° and Hine et al. reported ±10° variability); however it is not suited for short fibres with widely distributed orientations, as in this thesis. Bay and Tucker, 1992, correctly noted the true cause of the estimation bias for nearly aligned fibres. The authors' proposed solution, however, appears to rely on an operator's discretion during image digitization: the operator decides in advance which axis is to be measured, and, if the impossible case results (i.e., \( B > A \)), the imaginary values obtained in parameter estimation are replaced by zeroes.

Appendix A provides a formal proof for the existence of the misalignment-angle-estimation bias and supplies supporting data derived by simulating the random measurement noise and assessing the resulting error in the misalignment angle estimates.

A second shortcoming of ellipse-based fibre orientation estimates is the existence of two equally possible alternative orientations for each elliptical cross-section (Figure 5.3), i.e., the orientation duality problem. Both alternatives have the same misalignment angle \( \phi \), but the azimuth angles \( \theta \) differ by 180°. This does not pose a problem only in a special case when (a) the direction of interest is along the section plane normal and (b) the property of interest only depends on the misalignment angle.

For example, let the property of interest be the tensile modulus in a rectangular specimen in Figure 5.4. Then, consider two alternative directions of interest: (i) along the section-plane normal and (ii) along the Z-axis. According to some models, modulus is not affected by the fibre rotation about the direction of interest (see Chapter 6). Then, one would be able to calculate this property in the former case above, but not in the latter. In the latter case, each alternative azimuth angle about the plane normal results in a different azimuth angle about the Z-axis.

Orientation duality is not a significant problem for aligned fibres since the correct orientation can be selected by comparing it to the preferred fibre orientation. However, such a solution is not feasible for short fibres due to the large variability of their orientations. Zhu et al., 1997a, proposed a method which derives a complete three-dimensional FOD by combining data from two orthogonal plane sections, while Bay and Tucker, 1992, claim that data from three orthogonal section planes is required. The experimental implementation of such approaches may be practically difficult even though they are shown to be theoretically
possible by such investigations. Additionally, the results would be valid only if the FOD remains unchanged between the two section locations. However, in short-fibre composites, the fibre orientation is frequently a strong function of location.

Figure 5.4. Two possible fibre orientations based on an inclined section plane.

5.2.2 Overview of the two-section method

The proposed method consists of the following steps:

1. Obtain a planar cross-section of the specimen, hereafter referred to as Section 1.

2. Fit ellipses to the fibre cross-sections observed within this Section 1, obtaining for each fibre five ellipse parameters: ellipse centre coordinates, \((x_0, y_0)\); Z-rotation angle, \(\theta\); and major and minor radii, \(A\) and \(B\), respectively.

3. Obtain a second planar cross-section of the same specimen, hereafter referred to as Section 2, parallel to Section 1 and separated from it by a depth roughly equal to one fibre diameter (about 10-15 \(\mu\)m).

4. Fit ellipses to the fibre cross-sections of this Section 2, obtaining a second set of the ellipse parameters.

5. Using the above two sets, (i) identify the ellipses belonging to the same fibres in the two data sets, i.e., identify the matching ellipse pairs, and (ii) estimate the section-to-section alignment angle \(\psi\).
(6) Using the matched ellipse data outside of the biased region (\(\phi > 30^\circ\) in our case), obtain accurate estimates of transformation parameters between Section 1 and Section 2 data: \(x_n, y_n,\) and \(\Delta d\) (i.e., the X-Y plane shift and the depth distance).

(7) Using the section-to-section transformation parameters, calculate (i) the estimates of \(\phi\) which are unbiased and (ii) estimates of \(\theta\) which span the full \(360^\circ\) range for each fibre with a matching ellipse pair.

(8) Obtain a raw FOD from matched fibre data.

(9) Correct the above distribution for the orientation-dependent bias to obtain the desired FOD.

5.2.3 Solving the orientation-duality problem

This section describes the proposed methodology that simultaneously resolves the orientation duality problem and yields an unbiased and accurate estimate of the misalignment angles.

By processing two-section data, a set of Section 1 parameters \((x_o^{(1)}, y_o^{(1)}, \theta^{(1)}, \phi^{(1)})\) and a set of Section 2 parameters \((x_o^{(2)}, y_o^{(2)}, \theta^{(2)}, \phi^{(2)})\) are obtained for each fibre. The primary difficulty lies in finding the section-to-section transformation parameters that would allow us to take advantage of the extra information.

(i) Defining the transformation parameters

For parallel section planes, four transformation parameters are required: \(x_n, y_n, \psi_r,\) and \(\Delta d\) (Figure 5.5). The first three are the translation and rotation parameters for the alignment of the two section frames and \(\Delta d\) is the depth difference between the two sections in the \(Z\) direction.

The rotation alignment angle \(\psi_r\) can be readily determined, for example, by matching the orientation of linear features (such as the layer edges in our case). Thus, only three of the four parameters need to be determined through the following derivation.
Figure 5.5. Section-to-section transformation parameters.

(ii) Estimating the parameters $\Delta d$ and $x_t$

For each matched fibre cross-section, information available from the examination of two specimen sections comprises ellipse centre coordinates from Section 1, $\left( x_o^{(1)}, y_o^{(1)} \right)$, and those from Section 2, $\left( x_o^{(2)}, y_o^{(2)} \right)$, both expressed in their own respective frames; and angles $\theta$ and $\phi$ from both sections. (Note that these $\theta$ values only span a range of $180^\circ$, not $360^\circ$ actually spanned by the fibres, since an assumption had to be made about these values due to the orientation duality problem. Also, $\phi$ estimates suffer from the bias and variability problems for $\phi < \phi_{bias}$, as explained above.)

Section 2 ellipse centre coordinates can be expressed in Section 1 frame as:

$$ x_o^{(21)} = x_o^{(2)} + x_t \quad \text{and} \quad y_o^{(21)} = y_o^{(2)} + y_t. \quad (5.4) $$

Let us also define the following variables:

$$ \Delta x = x_o^{(21)} - x_o^{(1)} \quad \text{and} \quad \Delta y = y_o^{(21)} - y_o^{(1)}, \quad (5.5) $$

$$ \Delta \bar{x} = x_o^{(2)} - x_o^{(1)} \quad \text{and} \quad \Delta \bar{y} = y_o^{(2)} - y_o^{(1)}, \quad (5.6) $$

where $(\Delta x, \Delta y)$ is the actual shift of the fibre ellipse centre from Section 1 to 2 when both are projected onto the $X_1-Y_1$ plane, and $(\Delta \bar{x}, \Delta \bar{y})$ are the $X$ and $Y$ differences between the ellipse centre positions observed in their respective frames. The $(\Delta \bar{x}, \Delta \bar{y})$ values would be available directly, but $(x_t, y_t)$ are required to obtain the $(\Delta x, \Delta y)$ values.
Next, from Figure 5.6:

\[ \tan \theta = \frac{\Delta y}{\Delta x}, \]  
\[ \tan \phi = \frac{\sqrt{\Delta x^2 + \Delta y^2}}{\Delta d}. \]

Substituting \( \Delta y \) from (5.7) into (5.8) and rearranging:

\[ \frac{\tan^2 \phi}{1 + \tan^2 \theta} = \left( \frac{\Delta x}{\Delta d} \right)^2. \]  

Let us define a variable \( c_{\theta \phi} \), such that

\[ c_{\theta \phi} = \pm \sqrt{\frac{\tan^2 \phi}{1 + \tan^2 \theta}}, \]

Also, from (5.4), (5.5), and (5.6),

\[ \Delta x = \Delta \bar{x} + x_i, \quad \Delta y = \Delta \bar{y} + y_i. \]  

Substituting \( \Delta x \) from (5.11) into (5.9) and rearranging:

\[ c_{\theta \phi} = \frac{\Delta \bar{x} + x_i}{\Delta d}. \]  

Equation (5.12) can be rewritten as:

\[ \Delta \bar{x} = \Delta d \ c_{\theta \phi} - x_i. \]  

Equation (5.13) describes a linear relationship between \( c_{\theta \phi} \) and \( \Delta \bar{x} \), defined by a slope \( \Delta d \), and a Y-intercept \(-x_i\). For each matched ellipse pair, \( \Delta \bar{x} \) can be obtained from the ellipse centre coordinates and \( c_{\theta \phi} \) can be obtained from the single-section-based angle estimates. Subsequently, \( \Delta d \) and \( x_i \) can be accurately estimated via linear regression for the above linear relationship.

(iii) Estimating the parameter \( y_i \)

The following relationship from (5.11) is used to determine the last of the three section-to-section transformation parameters, \( y_i \):

\[ y_i = \Delta y - \Delta \bar{y}. \]
Figure 5.6. Nomenclature for the two-section method.

Examining Figure 5.6,

\[ \Delta y = \Delta r \sin \theta \quad \text{and} \quad \Delta r = \Delta d \tan \phi,\]

(5.15)

where \( \Delta r \) is the distance between the matched ellipse centres when expressed in Section 1 coordinates. Substituting for \( \Delta y \), in (5.14):

\[ y_i = \Delta d \tan \phi \tan \theta - \Delta \bar{y}.\]

(5.16)

The \( \phi \) and \( \theta \) are known from the single-section ellipse observations, and \( \Delta \bar{y} \) is known from matched ellipse pairs. Thus, if there are \( n \) fibres found with matched ellipse pairs, \( y_i \) can be estimated using (5.16) by:

\[ \hat{y}_i = \frac{1}{n} \sum_{i=1}^{n} (\Delta d \tan \phi_i \tan \theta_i - \Delta \bar{y}_i).\]

(5.17)

To estimate the three section-to-section transformation parameters, one should only use data for fibres with \( \phi > \phi_{bias} \), where the ellipse-based \( \phi \) estimates are reliable.
(iv) Obtaining the fibre orientation

Since the correct signs of $\Delta x$ and $\Delta y$ are now known, the ellipse-based orientation estimation duality problem is eliminated, yielding $\theta$ values over the full $360^\circ$ range from Equation (5.7). The $\phi$ angle is also obtained from Equation (5.8).

5.2.4 Determining the average fibre length from two-section fibre data

As pointed out in the introduction to this chapter, fibre length is an important characteristic of a composite, with a direct impact on its properties (e.g., see Equation (5.1)). The layered manufacturing process proposed in this thesis, however, can significantly reduce the fibre length through breakage. Similarly, Karnal et al., 1986, reported a decrease from 0.71 to 0.27 mm in injection moulding of short-fibre thermoplastics.

It is possible to characterize the fibre length distribution in a specimen by burning off the resin and directly measuring the fibre lengths. However, estimating the average fibre length indirectly from the two-section data collected for the fibre orientation measurements can eliminate this additional time-consuming step. Moreover, if the characteristic shape of the fibre length distribution is known a priori, average fibre length measurement may be sufficient to completely define the distribution.

Zhu et al., 1997a, proposed to derive the average fibre length of a short-fibre composite by calculating the fraction of fibres whose ends have been intersected by the sectioning plane out of a total number of intersected fibres. However, as pointed out by the authors, this method is likely to produce very inaccurate results. In numerous cases, it is difficult to be certain whether one intersected a fibre end (as characterized by an incomplete ellipse boundary). For example, it is possible that the incomplete ellipse boundary is caused by a piece of fibre chipped away by polishing.

Herein, an alternative, more robust, method is proposed for estimation of average fibre length based on data derived from two consecutive closely spaced sections of a specimen. The method relies on the fibre matching procedure described in Section 5.2.3. The average fibre length is estimated based on the ratio of the number of matched fibres to the total number observed in a single cross-section.
A well-known relationship in quantitative microscopy (DeHoff & Rhines, 1968) gives the probability of a randomly positioned sectioning plane intersecting a particle located in a cubical \( L \times L \times L \) sample space as:

\[
\text{Prob(planes intersect a particle)} = \frac{H}{L},
\]

where \( H \) is the extent of the particle along the section plane normal (or height for a horizontal plane).

Similar considerations lead to the conclusion that the probability of two sectioning planes, separated by a distance \( \Delta d \), intersecting the same particle of height \( H \) is:

\[
\text{Prob(two planes intersect a particle)} = \frac{(H-\Delta d)}{L}.
\]

In case of non-spherical particles, such as fibres, \( H \) will depend on the fibre orientation:

\[
H = l \cos \phi + d \sin \phi,
\]

where \( l \) is the average fibre length, \( d \) the average fibre diameter, and \( \phi \) the fibre misalignment angle.

Then, if the number of fibres oriented at angle \( \phi \) and intersected by a single section is \( n_1(\phi) \) and that intersected by two sections is \( n_2(\phi) \),

\[
n_1(\phi) = K_\phi N_T p(\phi) \frac{H}{L},
\]

\[
n_2(\phi) = K_\phi N_T p(\phi) \frac{(H-\Delta d)}{L},
\]

where \( N_T \) is the total number of fibres in the specimen, \( K_\phi \) is a constant, and \( p(\phi) \) is the probability of finding fibres oriented at an angle \( \phi \).

For a range of angles from \( \phi_1 \) to \( \phi_2 \), the number of fibres intersected by one cross-section, \( N_1 \), and by both cross-sections, \( N_2 \), will be given by integrals:

\[
N_1 = \int_{\phi_1}^{\phi_2} n_1(\phi) \, d\phi \quad \text{and} \quad N_2 = \int_{\phi_1}^{\phi_2} n_2(\phi) \, d\phi.
\]

Substituting from (5.21) into (5.22) and rearranging:

\[
N_1 = \frac{K_\phi N_T}{L} \int_{\phi_1}^{\phi_2} p(\phi) H \, d\phi,
\]

\[
N_2 = \frac{K_\phi N_T}{L} \left[ \int_{\phi_1}^{\phi_2} p(\phi) H \, d\phi - \Delta d \int_{\phi_1}^{\phi_2} p(\phi) \, d\phi \right].
\]
Taking the ratio of the two fibre counts and substituting for $H$ from (5.20):

\[
\frac{N_2}{N_1} = 1 - \frac{\Delta d \int_{\phi_1}^{\phi_2} p(\phi) \, d\phi}{\int_{\phi_1}^{\phi_2} p(\phi) \, d\phi} = 1 - \frac{\Delta d \int_{\phi_1}^{\phi_2} p(\phi) \, d\phi}{l \int_{\phi_1}^{\phi_2} p(\phi) \cos(\phi) \, d\phi + d \int_{\phi_1}^{\phi_2} p(\phi) \cos(\phi) \, d\phi} = 1 - \frac{\Delta d \, P}{l \, Q + d \, R}, \quad (5.24)
\]

where $P = \int_{\phi_1}^{\phi_2} p(\phi) \, d\phi$, $Q = \int_{\phi_1}^{\phi_2} p(\phi) \cos(\phi) \, d\phi$ and $R = \int_{\phi_1}^{\phi_2} p(\phi) \sin(\phi) \, d\phi$.

Finally, rearranging (5.24) one obtains the average fibre length as:

\[
l = \frac{1}{Q} \left( \frac{\Delta d \, P}{1 - \frac{N_2}{N_1} - d \, R} \right).
\]

(5.25)

It would be beneficial to evaluate the error sensitivity of the above estimate. It is assumed that the most significant error sources are the ratio $N_2/N_1$ and the section separation distance $\Delta d$. In (5.25), the second term, $(d \, R)$, can be ignored, as its contribution to the $l$ estimate is much smaller than the first term's. Evaluating partial derivatives with respect to $N_2/N_1$ ratio and $\Delta d$, the following error estimate is obtained:

\[
\Delta l = \left[ \left( \frac{-\Delta d \, P}{Q(1-w)^2} \right)^2 + \left( \frac{P}{Q(1-w)} \Delta (\Delta d) \right)^2 \right]^{1/2},
\]

(5.26)

where $w = N_2/N_1$ and $\Delta w$ and $\Delta (\Delta d)$ are errors in $w$ and $\Delta d$, respectively.

The contributions of individual terms and the total error estimate are displayed in Figure 5.7 for exemplary data provided in Appendix F. The plot shows that the fibre length estimate is highly sensitive to the error in the ratio $w$: a potential error of about 2-3% in $w$ would result in 10-15% variability in the length estimate. On the other hand, contribution of the $\Delta d$ error is relatively insignificant.
5.2.5 Adjusting for variation in fibre observability with misalignment angle

When characterizing a specimen's FOD, it is the distribution of fibre orientations within the specimen volume which is sought (volume-based FOD). However, the data collected is the FOD within a planar section (area-based FOD). Thus, the desired volume-based data must be derived from area-based observations. The relationship between the two types of observations depends on the fibre geometry and orientation: the probability of fibre intersection decreases with the increased misalignment angle $\phi$ and reduced fibre length, leading to progressive undercounting of fibres as the misalignment angle increases (DeHoff and Rhines, 1968).

To correct for this bias, a weighting function, $W_{obs}$, which will be referred to as the observability-correction function, must be applied to the area-based distribution data. Alternative forms of this function were presented by Zhu et al., 1997a, Bay and Tucker, 1992, and Möginger and Eyerer, 1991 (Table 5.1). The first two are similar in nature and base the correction on a well-known stereological relationship that expresses the number of particles per unit volume, $N_v$, in terms of the number of particles of the same type counted per unit area, $N_A$ (DeHoff and Rhines, 1968).

$$N_v = N_A / H$$ (5.27)
where $H$ is the length of a particle (or fibre, in our case) projected onto the section normal axis. Figure 5.8 illustrates this for a fibre of length $l$ and diameter $d$, and cross-section parallel to the $X$-$Y$ plane (i.e., a section-plane normal vector along the $Z$-axis). Zhu et al. use $H = l \cos \phi + d \sin \phi$, while Bay and Tucker assume $H = l \cos \phi$, where $l$ is the fibre length and $d$ is the fibre diameter. Dividing by fibre diameter expresses both in terms of the fibre aspect ratio: $s = l/d$.

Möginger and Eyerer add a dependence on the fibre volume fraction, $\nu_f$. The dependence on $\nu_f$ is accounted for through $\delta$, which the authors define as the average fibre-to-fibre distance divided by the fibre diameter. Figure 5.9 displays these alternative weighting functions plotted versus $\phi$. The plots are for the aspect ratio of 10, with the Möginger and Eyerer function plotted for 15% and 60% volume fractions.

![Figure 5.8. Nomenclature for fibre observability correction.](image)

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Table 5.1. Alternative forms of the observability-correction function.

<table>
<thead>
<tr>
<th>Author(s)</th>
<th>Formula</th>
</tr>
</thead>
<tbody>
<tr>
<td>Zhu et al., 1997a</td>
<td>( W_{\text{obs}} = \frac{1}{s \cos \phi + \sin \phi} )</td>
</tr>
<tr>
<td>Bay and Tucker, 1992</td>
<td>( W_{\text{obs}} = \begin{cases} \frac{1}{s \cos \phi} &amp; \text{for } \phi \leq \phi_c = \acos \left( \frac{1}{s} \right) \ 1 &amp; \text{for } \phi &gt; \phi_c \end{cases} )</td>
</tr>
<tr>
<td>Möginter and Eyerer, 1991</td>
<td>( W_{\text{obs}} = \frac{1}{\left[ s^4 \cos^4 \phi + \left( \frac{s + \delta}{1 + \delta} \right)^2 + \left( \frac{s(1 + \delta)}{s + \delta} \right)^2 \right] \sin^2 \phi \cos^2 \phi + \sin^4 \phi} )</td>
</tr>
<tr>
<td></td>
<td>where ( \delta ) is found by solving ( s = \frac{\delta(1 + 2\delta + \delta^2)}{\frac{\pi}{4V_f} - 1 - 2\delta - \delta^2} )</td>
</tr>
</tbody>
</table>

Figure 5.9. Alternative observability-correction functions (aspect ratio = 10).

Three cases of aspect ratios are considered below:

1. constant aspect ratio for all fibres,
2. variable aspect ratio, and
3. variable aspect ratio constrained by layer height.
The first case applies to all composites with relatively narrow fibre aspect ratio distributions; the second applies when the aspect ratio is widely distributed and the distribution function is known; and the third applies to layered composites with widely distributed fibre aspect ratio where the layer height restricts certain combinations of fibre length and orientation (as is the case in this thesis).

In the following derivations, a function \( p_{\text{obs}} \), which is the probability of observing a particular fibre type (i.e., particular orientation, length, or both) when sectioning a sample, is used. It is related to the above-mentioned weighting functions as:

\[
p_{\text{obs}} = (K_{\text{obs}} W_{\text{obs}})^{-1},
\]

where \( K_{\text{obs}} \) is an unknown proportionality constant. Any one of the above forms of \( W_{\text{obs}} \), Table 5.1, can be used in the following derivations.

5.2.5.1 Case 1: Constant aspect ratio

In order to obtain the desired marginal FOD for \( \phi \), the probability of finding fibres with the misalignment angle centred on \( \phi_i \), for \( 0^\circ < \phi_i < 90^\circ \) needs to be determined. Ideally, this probability could be estimated by counting all the fibres with the misalignment angle \( \phi_i \), \( N_{\phi_i} \), and dividing it by the total number of fibres in the specimen, \( N_T = \sum_i N_{\phi_i} \):

\[
p(\phi) = \frac{N_{\phi_i}}{N_T}.
\]

However, only \( \hat{N}_{\phi_i} \), the number of fibres observed at a misalignment angle \( \phi_i \) in a specimen plane section, and not in a complete specimen, is available. From the definition of \( p_{\text{obs}} \) as the probability of observing a particular fibre type,

\[
\hat{N}_{\phi_i} = p_{\text{obs}}(\phi_i) N_{\phi_i}.
\]

Substituting \( N_{\phi_i} \) from (5.30) into (5.29),
5.2.5.2 Case 2: Variable aspect ratio

All observability-correction functions depend on fibre aspect ratio as well as on orientation. Therefore, if the fibre aspect ratio distribution function is available, it must be taken into account in deriving a corrected fibre-orientation-distribution function.

Let $N_{\phi,s}$ be the number of fibres in the specimen with misalignment angle $\phi_i$ and aspect ratio $s_j$. Also, let $\hat{N}_{\phi,s}$ be the number of fibres with $\phi_i$ and $s_j$ which would have been observed in a plane section. Assuming that the fibre aspect ratio distribution is independent of $\phi_i$,

$$N_{\phi,s} = p(s_j)N_{\phi},$$

where $p(s_j)$ is the probability of a fibre having an aspect ratio of $s_j$. As in (5.30), from the definition of $p_{obs}$:

$$\hat{N}_{\phi,s} = p_{obs}(\phi_i, s_j)N_{\phi,s}.$$ (5.33)

However, only the total number of fibres of all lengths at $\phi_i$ is known. Thus, using Equations (5.28), (5.32), and (5.33):

$$\hat{N}_{\phi} = \sum_j \hat{N}_{\phi,s} = \sum_j p_{obs}(\phi_i, s_j)N_{\phi,s} = \sum_j p_{obs}(\phi_i, s_j)p(s_j)N_{\phi} = \frac{N_{\phi}}{K_{obs}}\sum_j \frac{p(s_j)}{W_{obs}(\phi_i, s_j)}.$$ (5.34)

Rearranging the above equation,

$$N_{\phi} = \frac{K_{obs}\hat{N}_{\phi}}{\sum_j \frac{p(s_j)}{W_{obs}(\phi_i, s_j)}}.$$ (5.35)
Normalizing (5.35) by $N_T = \sum_i N_{a_i}$ and cancelling the constant $K_{obs}$, the desired expression for the corrected fibre orientation distribution is obtained:

$$p(\phi_i) = \frac{\tilde{N}_{\phi_i}}{N_T \sum_j W_{obs}(\phi_j, s_j)}.$$

(5.36)

5.2.5.3 Case 3: Variable aspect ratio constrained by layer height

In layered manufacturing, if the layer thickness is less than the length of some fibres, the fibre orientation distribution will be affected by the layer thickness. As the fibres are unlikely to protrude from the liquid's surface due to the surface tension, it will be assumed that all the fibres are within the layer boundaries. A possible exception occurs if any part of the preceding layer remains uncured (e.g., the "rivet" features used in this thesis); in such cases, the fibres can extend over two-layer depth. However, this effect would be localized, as the majority of the layer must be hardened before the next layer of liquid material is added.

Consider a fibre of a length exceeding the layer thickness $\Delta y_L$ (Figure 5.10). It is required to find a relationship between the fibre orientation and its maximum length, given the above layer height restriction. From the figure,

$$L_{sv} = \frac{\Delta y_L}{\sin \theta} \text{ and }$$

$$L_{\max} = \frac{L_{sv}}{\sin \phi} = \frac{\Delta y_L}{\sin \phi \sin \theta} \quad (5.38)$$

Dividing (5.38) by the fibre diameter, the maximum aspect ratio for a given orientation is defined as:

$$\frac{L_{\max}}{d} = s_{\max} = \frac{\Delta y_L / d}{\sin \phi \sin \theta}. \quad (5.39)$$

The above expression implies that, depending on the fibre's orientation, the aspect ratio must be limited to $s_{\max}$. This constraint must be incorporated into the observability-correction function.
Figure 5.10. Calculating maximum fibre length for a given fibre orientation.

Let $N_{a,\theta,i}$ be the actual number of fibres with misalignment $\phi_i$, azimuth $\theta_j$, and aspect ratio $s_k$; $N_{a,\theta}$ be the actual number of fibres with misalignment $\phi_i$ and azimuth $\theta_j$; $\hat{N}_{a,\theta,i}$ be the observed number of fibres with $\phi_i$ and $\theta_j$; and finally, the maximum aspect ratio be a function of fibre orientation, given by:

$$s_{k_{\text{max}}} = s_{\text{max}}(\phi_i, \theta_j) = \frac{\Delta y_k / d}{\sin \phi_i \sin \theta_j}.$$  \hspace{1cm} (5.40)

Then, the conditional probability of a fibre being of aspect ratio $s_k$ is:

$$\text{Prob}[s_k < s_{\text{max}}(\phi_i, \theta_j)] = \frac{p(s_k)}{\text{Prob}[s_k < s_{\text{max}}(\phi_i, \theta_j)]}. \hspace{1cm} (5.41)$$

The above expression is used as:

$$N_{a,\theta,i} = \frac{p(s_k)}{\text{Prob}[s_k < s_{\text{max}}(\phi_i, \theta_j)]} \cdot N_{a,\theta}. \hspace{1cm} (5.42)$$

Next, relating the observed and the actual data with the weighting function yields:
Chapter 5. Fibre-Orientation Distribution Studies

Note that the term $\text{Prob} [s_k < s_{\text{max}} (\phi, \theta)]$ can be taken out of the summation, as it is a function of the two angles only. Rearranging (5.43):

$$\hat{N}_{\phi, \theta} = \frac{N_{\phi, \theta}}{K \text{Prob} [s_k < s_{\text{max}} (\phi, \theta)]} \sum_{k=1}^{\text{max}} \frac{p(s_k)}{W_{\phi, \theta} (\phi, \theta)}.$$

where $W_{\phi, \theta}$ are the weighting factors correcting the observed number of fibres with $(\phi, \theta)$. Then, the desired corrected fibre-orientation distribution is defined as:

$$p(\phi) = \frac{N_{\phi}}{N_T} = \sum_{j} \sum_{i} N_{\phi, \theta} (\phi, \theta).$$

5.2.6 Calculating fibre-orientation distribution with respect to a different coordinate frame

When the fibre orientations are measured, the misalignment angle $\phi$ is obtained with respect to the section plane normal. However, in order to estimate mechanical properties for any direction other than the section plane normal, the fibre misalignment angle distribution needs to be calculated with respect to the new direction.

The transformation itself is a straightforward procedure:

1. Given the two fibre orientation angles in the original frame, $(\phi, \theta)$, represent the fibre orientation as a vector $v$ as (Paul, 1982):

$$v = \begin{pmatrix} \cos \theta \sin \phi \\ \sin \theta \sin \phi \\ \cos \phi \end{pmatrix}.$$

2. Transform the vector to a new frame using a $3 \times 3$ rotation matrix $R$:

$$v' = Rv.$$

3. Calculate the two angles defining fibre orientation with respect to the new frame, $(\phi', \theta')$: 

$$\hat{N}_{\phi', \theta'} = \frac{N_{\phi', \theta'}}{K \text{Prob} [s_k < s_{\text{max}} (\phi', \theta')]} \sum_{k=1}^{\text{max}} \frac{p(s_k)}{W_{\phi', \theta'} (\phi', \theta')}.$$
\( \phi' = \cos(\mathbf{v}' \cdot \mathbf{v}) \) and \( \theta' = \arctan(2\mathbf{v}' \cdot \mathbf{v}) \)

(5.48)

where \( \mathbf{v}' = [v'_x, v'_y, v'_z]^T \) and \( \arctan2 \) is a four-quadrant inverse tangent function.

However, in deriving the fibre angle distributions with respect to the new frame, one must still correct for the variation in fibre observability in the original data. This correction can be accomplished as follows.

Based on the Equation (5.44), the probability for existence in the specimen of fibres with angles \((\phi, \theta)\) can be expressed as:

\[
p(\phi, \theta) = \frac{N_{\phi, \theta}}{N_T} = \frac{\hat{N}_{\phi, \theta} W_{\phi, \theta}}{N_T}
\]

(5.49)

Note that all \( \hat{N}_{\phi, \theta} \) fibres in the \((\phi, \theta)\) group have the same associated weighting factor \( W_{\phi, \theta} \).

After expressing each fibre’s orientation in terms of the new misalignment and azimuth angles, the probability of finding fibres with \((\phi'_i, \theta'_i)\) in the specimen will be given by:

\[
p(\phi'_i, \theta'_i) = \frac{\sum_{p=1}^{N_{\phi, \theta}} W_{\phi, \theta} p}{N_T}
\]

(5.50)

where \( \hat{N}_{\phi, \theta} \) is the number of fibres observed with \((\phi'_i, \theta'_i)\) and \( W_{\phi, \theta} p \) is the weighting factor associated with the \( p \)th fibre within the \((\phi'_i, \theta'_i)\) group. The \( W_{\phi, \theta} p \) factor is equal to the \( W_{\phi, \theta} \) factor of the \((\phi, \theta)\) group to which the \( p \)th fibre belonged prior to coordinate transformation. Note also that, unlike for the \((\phi, \theta)\) group, within the \((\phi'_i, \theta'_i)\) group, each fibre has a different associated weighting factor, depending on the fibre’s orientation with respect to the original frame.

Then, the desired distributions of the misalignment and azimuth angles with respect to the transformed frame can be calculated by:

\[
p(\phi'_i) = \sum_{i} p(\phi'_i, \theta'_i),
\]

\[
p(\theta'_i) = \sum_{i} p(\phi'_i, \theta'_i).
\]

(5.51)
5.3 Fibre-orientation data acquisition from cross-sectional images

In order to acquire geometric data about the elliptical fibre cross-sections, digital images of polished specimen sections can be taken via an optical microscope. The information sought from these images is the location of the fibre ellipse boundaries. By processing this data, the desired information about the fibre cross-sections can be extracted.

The two-section methodology described above would not be affected by whether the boundary data were to be obtained manually or automatically. However, to expedite data acquisition and to make it more consistent, it would be preferable to obtain the boundary data automatically using available digital image-processing techniques (Castleman, 1996). However, automatic image processing is a complicated task that requires careful sample preparation, use of special techniques that assure acquisition of good quality images, and use of robust image processing algorithms.

For short fibres, Bay and Tucker, 1992, resort to manual image processing that requires location of four end-points of the ellipse's two diameters. Fisher and Eyerer, 1988, perform automatic image processing on thresholded grey-scale images, with some interactive manual correction in "critical regions." For continuous fibres, Yurgatis, 1987, also uses manual digitization, but only identifies the end-points of the ellipse major axis; the minor axis is equal to the fibre diameter, which the author assumes to be constant and thus not requiring measurement. Sirkis et al., 1994, provide a good description of image processing techniques suitable for fibre imaging, which they apply, however, only to the acquisition of two-dimensional FOD, and not to ellipse-based 3-D fibre orientations.

In the present work, images with 640×480 256-gray-level pixels were acquired. Given the expediency of automatic processing, several attempts were made to extract the ellipse boundary data automatically. However, even by manually isolating the region of interest containing the ellipse, reliable edge detection could not be achieved. Causes included variations in image contrast, surface impurities obscuring the ellipse boundary, incomplete ellipse boundaries, and large variability of the analyzed shapes (from nearly circular cross-sections of aligned fibres to square cross-sections of perpendicular fibres). Examples of good and poor quality ellipse images are provided in Figure 5.11. Since image processing per se is
not an objective of this work, it was decided to resort to manual identification of the ellipse boundary points.

![Figure 5.11. Examples of (a) good and (b) poor images of elliptical fibre cross-sections](image)

To extract the five ellipse parameters \((x_0, y_0, \theta, A, B)\), the ellipse-curve equation was fitted to the manually selected boundary points using the least-squares fit for all but a few special cases (e.g., incomplete elongated ellipses of nearly perpendicular fibres). The following sections will describe how the images were calibrated to obtain actual dimensions of the fibres and how the ellipse parameters were extracted by fitting the curves to the boundary points. Also described is merging of the data from individual images of small sub-areas into a combined data set representing a larger area of the specimen (e.g., a complete layer cross-section in the case of layered specimens).

The next important step for implementation of the two-section method is matching of the ellipses between consecutive sections. Described below is the algorithm enabling this process. Finally, estimates of the expected measurement errors are given for the two-section-based orientation estimates.

A detailed procedure for collecting and processing the image data, including the names of programs to be executed at each step, is provided in Appendix C.

### 5.3.1 Image-to-world transformation

The image-to-world scaling is necessary for two reasons: first, to obtain the fibre dimensions and, second, to correct for any disparity in the image X and Y scaling. The latter is particularly important, as accurate determination of the ellipse axes ratio is required to derive the orientation data.
The following procedure was used for image scaling:

1. Measure several distances between identifiable features in X and Y directions on the imaged area of the sample using an optical microscope with a calibrated viewfinder, while making sure that all the features are contained within the same image;
2. Measure the distance in pixels between the same features on the image; and,
3. Determine the X and Y image-to-world scaling by calculating the average ratio between the corresponding world and image measurements in both directions.

5.3.2 Fitting ellipses to fibre cross-sections

Fibre cross-sections must be located in the digital images of specimen cross-sections in order to obtain the ellipse parameters. While some researchers propose to identify the end-points of the ellipse's two diameters manually (Bay and Tucker, 1992), such an approach is imprecise and even impossible to apply in some cases. For example, many fibre cross-sections result in incomplete ellipses, where visual identification of the diameters' end-points is impossible. In addition, the accuracy of the diameter estimation would depend directly on operator's ability to accurately locate each point.

On the other hand, by selecting a number of points along the ellipse boundary and calculating the best-fitting set of ellipse parameters, one can average out the uncertainties in the individual point selection. In implementing the algorithm for ellipse-parameter estimation, steps were taken to assure high accuracy through good numerical conditioning of the linear equations: first, the boundary points are shifted so that their average (X, Y) position is near the origin. Second, the point coordinates are rescaled so that all the ellipse parameters are of similar magnitude. Appendix D describes in detail the ellipse-parameter estimation algorithm.

5.3.2.1 Ellipse-data collection procedure

Ellipses are identified by visual examination of the images. A custom MATLAB® image processing software was created to assist in the process. It allows close-up view of each ellipse to enable accurate boundary identification. Data collection procedure consists of the following:
(1) Visually identify the fibre's cross-section;

(2) Select appropriate processing method for the ellipse (see Section 5.3.2.2 below);

(3) Identify several points along the fibre cross-section boundary by moving the cursor over the boundary and issuing a selection command. (For a regular ellipse, a minimum of five is required and typically 9-12 are used. For special cases described in the following section, specific points must be selected, depending on the case);

(4) Fit an ellipse to these points and verify the correctness of the fitted curve overlaid on the image by visually comparing it to the actual boundary; and,

(5) Store the five ellipse parameters \((x_0, y_0, \theta, A, B)\) for each identified fibre cross-section, together with a unique identification number. (The same number is also overprinted on the image itself for reference purposes.)

5.3.2.2 Special cases of fibre-plane intersections

In general, any fibre cross-section that is not a complete ellipse can be considered a special case. Both Zhu et al., 1997a, and Fischer and Eyerer, 1988, identify the conditions when such incomplete ellipses result: a partial ellipse is observed when a plane intersecting a finite-length cylinder passes through one or more of its ends. There are five distinct possibilities, as illustrated in Figure 5.12. Figure 5.13 also gives examples of special cases observed in our specimen cross-section images.

Zhu et al., 1997a, propose handling of the above cases through the selection of specific points on these cross-sections, with appropriate relationships used to derive the ellipse parameters from the distances between these special points. This approach, however, either results in no improvement compared to the regular curve fitting method or altogether fails to estimate the parameters. In this thesis, it was determined that the standard curve-fitting procedure works well for all special cases where the cross-section includes the ellipse centre (cases B and D, Figure 5.12). Problems do occur when only a "corner" of an elongated ellipse is to be fitted (cases A and C). In the latter case, curve-fitting results in very poorly conditioned linear systems, frequently failing to calculate the ellipse parameters.
In this thesis, only two situations are treated as special cases, with the others handled by the standard curve fitting. The first case is the nearly in-plane fibre ($\phi \to 90^\circ$), with the ellipse that does not extend past its mid-point (cases A and C), and the second case is the exactly in-plane fibre ($\phi = 90^\circ$, case E). The two special cases are identified visually and the appropriate processing method is selected. For the first case, a special estimating algorithm has been developed that does not suffer from the ill conditioning of the regular curve fitting procedure (Appendix D). For the second case, only the four corner points are selected in order to calculate the fibre’s azimuth ($\theta$), while the misalignment angle is set by definition to 90 degrees.
5.3.3 Merging images

In order to provide sufficient resolution, each image covers only a part of the total sampled cross-section area. Thus, to record fibre locations within this area, individual data from several images must be merged into a combined set referenced to a common coordinate system. In order to produce such a combined set, transformations between images must be obtained. This is accomplished by identifying cross-sections of the same fibres found at the boundaries of adjacent images. Since the locations of these fibres are known in both image frames, the image-to-image transformation is easily derived. Normally, several such common fibres are identified, and the frame translation is obtained by averaging the relative shift for the chosen fibres. It is assumed that there is no rotation between the images, as the specimen is only translated in the X-Y plane during image acquisition. When the image data is combined into a single set, fibres common to two images are removed from one of them to avoid duplication. As part of the data merging, the ellipse dimensions and location coordinates are also rescaled, as described in Section 5.3.1 above.
5.3.4 Matching fibres between consecutive sections

Once the combined data for both consecutive specimen sections is obtained, ellipses belonging to the same fibres must be identified. Initial attempts to match the fibres by unassisted visual examination of the section images showed that the process is difficult or even impossible in regions of high fibre density; the process is also prone to misidentification. Further compounding the difficulty of the task is the significant shifting in the fibre cross-section locations from one section to the other for the more inclined fibres, which changes the fibre arrangement patterns between the two section images. Thus, to facilitate the fibre-matching task, custom software was written in MATLAB®.

The developed software overlays ellipse outlines from both sections onto the same display. Figure 5.14 shows an example of a Section 2 image acquired from the “Large Offset Rivets” (LOR) specimen (see Section 5.4.1.2 for the specimen description); Figure 5.15 displays the ellipse boundaries extracted from Section 1 and 2 images of the same area; and finally, Figure 5.16 provides an overlaid display of both section ellipses after necessary adjustments have been made.

The overlaid display is adjusted interactively in the following manner until a good visual match is attained:

1. Section 2 ellipses are rotated in the X-Y plane by the angle $\psi_i$. An appropriate value for the rotation angle $\psi_i$ is calculated by performing a linear fit to the layer edge points in both sections and then taking the difference between the inclination angles of the fitted lines;

2. Section 2 ellipses are shifted in the X-Y plane by $(x_i, y_i)$;

3. Section 1 ellipses are individually shifted to reflect the shift in the fibre cross-section caused by the fibre inclination with respect to the section plane. The shift magnitude and direction are determined by the depth parameter $\Delta d$ and by the fibre orientation; and,

4. Each fibre’s orientation is switched as needed between two possible alternatives of the ellipse-based orientation estimate, with the consequent difference in the shift direction.
After identifying a matching pair of ellipses, the pair is selected, and the software automatically records the corresponding fibre ID numbers and disables their future selection. Once all the possible matches have been made and the satisfactory rotation angle $\psi_r$ has been found, the section-to-section parameters ($x_i$, $y_i$, and $\Delta x$) are calculated separately for each image of Section 1 data using the methodology described in Section 5.2.1.

Figure 5.14. An example image of a specimen cross-section. Figure shows leftmost image of the upper layer in LOR specimen (see Section 5.4.1.2 for specimen description) that served as the source for ellipses displayed in Figure 5.15(b). Processed fibres are labelled with ID numbers.
Chapter 5. Fibre-Orientation Distribution Studies

5.3.5 Evaluating measurement errors

The sensitivity of the two-section-based misalignment angle estimate is evaluated in this section. This estimate is made using Equation (5.8), which is restated below for convenience:

$$\tan \phi = \frac{\sqrt{\Delta x^2 + \Delta y^2}}{\Delta d}. \quad (5.8)$$
To simplify the notation, let \( L^2 = \Delta x^2 + \Delta y^2 \) and \( D = \Delta d \). Then,

\[
\phi = \tan^{-1} \frac{L}{D} = f(L, D) .
\]

(5.52)

The error in \( \phi, \Delta \phi \), can be estimated as:

\[
\Delta \phi = \left( \frac{\partial f}{\partial L} \Delta L \right)^2 + \left( \frac{\partial f}{\partial D} \Delta D \right)^2 \right)^{1/2} .
\]

(5.53)

Evaluating the derivatives,

\[
\Delta \phi = \frac{\left( D \Delta L \right)^2 + \left( L \Delta D \right)^2}{D^2 + L^2} .
\]

(5.54)

For example, given a typical value of \( D=15 \, \mu m \) and \( L \) calculated from Equation (5.52) for \( 0^\circ < \phi < 30^\circ \), a region where one would like to improve the accuracy of the \( \phi \) estimate, and approximate expected errors of \( \Delta L = \Delta D = 0.5 \, \mu m \), \( \Delta \phi \) is estimated as \( 2^\circ \). This value is significantly lower than the expected error in the single-section method used by other researchers, approximately 15-20\%, as shown in Appendix A.

A numerical simulation of the section-to-section parameter estimation was also performed. Its aim was to assess the method's accuracy, given the noisy nature of the fibre-orientation data. A detailed account of the methodology and results is provided in Appendix F. The results indicate that the method can provide accurate estimates of the transformation parameters: given a typical standard deviation of the boundary-point position uncertainty of \( 0.4 \, \mu m \), the standard deviation of the three parameter estimates over ten simulation runs was approximately \( 0.2 \, \mu m \).

### 5.4 Experiments

As mentioned earlier, the three principal factors affecting the composite mechanical properties are fibre content (or volume fraction), fibre aspect ratio, and fibre orientation. In this section, the third significant factor influencing the composite's mechanical properties, namely fibre orientation, is experimentally examined.

Fibre-orientation analysis tools are also utilized to explain empirically observed differences between the mechanical properties of layered parts fabricated by two different
building methods, Chapter 6. As explained in Chapter 3, layered parts are fabricated by curing thin layers of composite liquid consisting of photopolymer resin and short glass fibres. In the first method, each layer is cured completely by the UV laser beam scanning. In the second method, small rectangular areas are left uncured within each layer. The locations of the uncured areas alternate, from layer to layer, in a checkerboard pattern. The exposure is doubled above the uncured area on the next layer to achieve curing through two layers. The uncured features form interlayer “rivets” to further join the consecutive layers. The first approach is referred to as the “no-rivet” and the second as the “rivet” building method, and the specimens produced are called “non-riveted” and “riveted,” accordingly.

5.4.1 Experimental procedure

5.4.1.1 Overview

Collection of fibre-orientation data is a very time-consuming task, with the bulk of the time spent on the manual acquisition of the fibre cross-section boundary data. Given below are the steps required to collect this data and to derive the FOD:

1. Set specimens in a resin mould to facilitate handling during the polishing step;
2. Mill the top surface of the mould to obtain an even plane surface to be polished;
3. Polish the surface of the mould encasing the specimen using a sequence of abrasives with progressively decreasing grit sizes;
4. Acquire digitized images of the desired cross-section region. Typically, a sequence of images is taken to cover one (non-riveted) or two (riveted) complete layer cross-sections;
5. Determine image-to-world scaling using a microscope to measure distances between identifiable features on the cross-sections;
6. Measure same feature dimensions on a sample image (in pixels);
7. Determine scaling factors based on the above measurements;
8. Collect fibre ellipse cross-section data from the acquired images by manually tracing each ellipse border. A data file is generated corresponding to each image that
records ID numbers for each fibre together with the collected ellipse data. The same ID numbers are added to each processed image for cross-referencing;

(9) Collect layer boundary curve data from each image by locating several points along each curve. This information is to be used for display purposes and for orientation adjustment when matching fibre cross-sections (see Step (13) below). Individual data files are generated for each image;

(10) Visually identify identical fibres within adjacent images and record their ID numbers in a data file to enable calculation of image-to-image transformations;

(11) Combine fibre ellipse cross-section data from individual images into a single data file. Three tasks are performed to combine the fibre data: the fibre positions are expressed with respect to the same coordinate frame; identical fibres identified in adjacent images are removed from one of the image data sets to avoid duplication; and the data is rescaled from image to world measurement units. The first two steps are accomplished based on data collected in Step (10) and the third step employs scaling data collected in Step (7);

(12) Combine layer boundary curve data from individual images into a single file. This step also involves expressing curve positions in the same frame and rescaling the data;

(13) Identify matching fibres in Sections 1 and 2. Use one combined fibre ellipse data file from each cross-section as input. As a result of matching, the rotation angle between the two cross-section, $\psi$, is identified and ID numbers of matching fibres are stored in a data file;

(14) Estimate parameters for section-to-section transformation $(x_i, y_i, \Delta d)$. A separate parameter set is calculated and recorded for each sub-area covered by a single image;

(15) Calculate the fibre orientations for the matched fibres based on the parameters identified in the preceding step; and,

(16) Obtain fibre-orientation distribution using individual fibre orientations calculated above.
5.4.1.2 Specimen description

Fibre orientation was examined for a small sample selected from a larger set of dog-bone-shaped specimens originally prepared for the examination of tensile properties (Figure 5.17). The sample comprised two riveted specimens, each with a different rivet style, and two non-riveted specimens. Due to the time-consuming nature of the fibre-orientation measurements, only several layers within each of the four specimens were examined. Table 5.2 lists the fibre content observed for the specimens selected for fibre-orientation measurements. The four specimens will be henceforth identified as “LOR,” for Large Offset Rivets, “SOR,” for Small Offset Rivets, and “NR1” and “NR2,” for No Rivets 1 and 2.

The arrangement of the rivet features and the approximate location of the sectioning plane are depicted in Figure 5.18. Figure 5.19 shows the expected view of the specimen cross-section for several layers. (A specimen consists of thirteen layers, each nominally 0.3 mm thick.)

Figure 5.17. Dog-bone-shaped tensile test specimen (all dimensions are in mm).

Figure 5.18. Close-up view of the gauge section (dashed square outlines designate rivets in the alternate layer).
5.4.1.3 Specimen preparation

From each dogbone, a small part about 5 mm in length was cut away from the narrow waist section. The four parts, one from each specimen, were mounted with paper glue onto the sides of an acrylic cube (17×17×17 mm) to assure their alignment with respect to each other and with respect to the cutting plane normal. The four specimens were then placed at the bottom of a cylindrical open-top plastic mould of 37-mm diameter, with a metallic weight placed on top of the cube to prevent it from floating in the resin (Figure 5.20). Upon filling the mould with a polyester-based potting resin and adding a prescribed small amount of hardener (methyl ethyl ketone peroxide), the mixture was left to cure for several hours at room temperature.

After de-moulding, the specimens embedded in the resin were milled to the desired height and metallographically wet-polished first with SiC papers (particle mesh sizes progressively decreasing from grade 80 to 2400), and then, with 1-μm alumina powder. When repolishing to create the second section for two-section fibre-orientation measurements, only 2400-grade
SiC paper and 1-μm alumina powder were used. The approximate depth change during repolishing was monitored with a micrometer.

![Diagram of sample arrangement](image)

**Figure 5.20. Arrangement of specimens in a mould.**

### 5.4.1.4 Image acquisition

Specimens were observed via a microscope (Olympus Vanox) under a yellow-filtered reflected light. Images were digitized using a CCD (Sony XC-77) video camera passing its signal to a frame grabber (Scion LG3) at a resolution of 640×480 pixels, 256 grey levels per pixel. Each image captured an area of 0.5×0.4 mm², translating to a spatial resolution of 0.83 μm per pixel.

Images were acquired in sets of eight for the non-riveted specimens and in sets of sixteen for the riveted ones. Eight images are required to completely represent a cross-section of one layer, which is approximately 3.4 mm wide and 0.3 mm thick. Two consecutive layer cross-sections were examined for the riveted specimens to observe the interlayer rivets, as these features extend from one layer to another.

### 5.4.2 Results

A total of three cross-sections were made by successive grinding and polishing of the same surface for the four specimens under test. The first cross-section, identified henceforth as Section 0, was examined using the single-section fibre-orientation-measurement technique; the subsequent two cross-sections, which will be referred to as Section 1 and 2, were examined using the two-section fibre-orientation-measurement technique. Section 1 is
separated from Section 0 by a distance of about 120 μm. Sections 1 and 2 are closely spaced (10-15 μm), as required by the two-section method.

Table 5.3 presents a summary of the experiments. The layer numbers are referenced according to the fabrication sequence, from the bottom up. Complete layer cross-sections were examined for all specimens except NR2, where only the left half of a layer was processed. Partial sampling was done for expediency, since visual observation of the images did not indicate significant variation in the fibre orientations between the left and right half of the layer cross-section. Each image required about 25-30 minutes of processing for collection of ellipse data and a similar time for each image-pair in a two-section method for fibre matching. Including section preparation, image acquisition, and other steps, the experiments took about 120-150 hours.

Table 5.4 shows calculated parameters of the section-to-section transformation. All section-to-section transformation parameters, except ψ_t, are the averages over all images examined. The rotation parameter ψ_t has the same value for all images. Error ranges given are equal to one standard deviation.

Table 5.3. Experimental observation summary for FOD measurements.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Cross-section Number</th>
<th>Layers Examined</th>
<th>Number of Images Acquired</th>
<th>Number of Images Processed</th>
<th>Number of FibresMeasured</th>
</tr>
</thead>
<tbody>
<tr>
<td>LOR</td>
<td>0</td>
<td>7,8</td>
<td>16</td>
<td>16</td>
<td>853</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>11,12</td>
<td>16</td>
<td>16</td>
<td>832</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>11,12</td>
<td>16</td>
<td>16</td>
<td>814</td>
</tr>
<tr>
<td>SOR</td>
<td>0</td>
<td>9,10</td>
<td>16</td>
<td>16</td>
<td>1113</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>9,10</td>
<td>16</td>
<td>16</td>
<td>1061</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>9,10</td>
<td>16</td>
<td>16</td>
<td>1096</td>
</tr>
<tr>
<td>NR1</td>
<td>0</td>
<td>7</td>
<td>8</td>
<td>8</td>
<td>670</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>10</td>
<td>8</td>
<td>8</td>
<td>591</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>10</td>
<td>8</td>
<td>8</td>
<td>597</td>
</tr>
<tr>
<td>NR2</td>
<td>0</td>
<td>8</td>
<td>8</td>
<td>4</td>
<td>217</td>
</tr>
<tr>
<td></td>
<td>1</td>
<td>7</td>
<td>8</td>
<td>4</td>
<td>153</td>
</tr>
<tr>
<td></td>
<td>2</td>
<td>7</td>
<td>8</td>
<td>4</td>
<td>148</td>
</tr>
</tbody>
</table>
5.4.2.1 Distribution of fibres within a layer

Table 5.4. Results of section-to-section matching.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Number of Matched Fibres</th>
<th>Number of Fibres Used to Estimate Parameters</th>
<th>$x_r$ (μm)</th>
<th>$y_r$ (μm)</th>
<th>$Δd$ (μm)</th>
<th>$ψ_r$ (deg)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LOR</td>
<td>647</td>
<td>303</td>
<td>-6.4±1.4</td>
<td>-8.4±0.9</td>
<td>13.9±0.5</td>
<td>1.45</td>
</tr>
<tr>
<td>SOR</td>
<td>928</td>
<td>300</td>
<td>25.9±0.8</td>
<td>8.7±1.6</td>
<td>10.8±0.9</td>
<td>0.45</td>
</tr>
<tr>
<td>NR1</td>
<td>504</td>
<td>195</td>
<td>3.1±0.6</td>
<td>-10.9±0.5</td>
<td>10.0±0.8</td>
<td>0.93</td>
</tr>
<tr>
<td>NR2</td>
<td>120</td>
<td>80</td>
<td>41.7±1.3</td>
<td>7.3±1.8</td>
<td>11.4±0.7</td>
<td>2.1</td>
</tr>
</tbody>
</table>

5.4.2.1 Distribution of fibres within a layer

Figure 5.22 contains photographs of the specimen cross-sections. Layer numbers are indicated on the figures for reference. Figure 5.22 defines the orientation of the coordinate system with respect to the specimens. Figure 5.23 indicates locations of fibre cross-section centres observed in the LOR specimen. The plot combines observations from 16 separate images of Section 1. It shows two layers, with an interlayer rivet located on the right side. The rivet is distinguished by the interruption of the boundary between upper and lower layers. Settling of the fibres can be seen as the fibre density decreases near the top for both layers. One can also note the effectiveness of the rivet in maintaining a continuity of fibre distribution between the two layers. Figure 5.24 displays the fibre cross-section locations for the NR1 specimen. Similar fibre settling can be also seen in this diagram.
Figure 5.21. Photographs of (a) LOR, (b) SOR, (c) NR1, and (d) NR2 specimen cross-sections.

Figure 5.22. Orientation of the coordinate system.
5.4.2.2 Average fibre length

Fibre length is required to adjust FOD observations for variable fibre observability with misalignment angle (Section 5.2.5). Average fibre lengths were estimated for all the specimens using the methodology described in Section 5.2.4. To simplify calculations, the length estimates were made using only fibres with the misalignment angle in the range $0^\circ < \phi < 30^\circ$. Since at low misalignment angles the orientation-dependent bias is insignificant, the above restriction allowed us to use the raw FOD data for these calculations. A second simplifying assumption was that the angles are distributed uniformly in this range. Thus, $\rho(\phi) = K$ ($K$ is a constant), $\phi_1 = 0$, and $\phi_2 = \pi/6$, which yields factors $P$, $Q$, and $R$ in Equation (5.25) as $P = \frac{\pi}{6}K$, $Q = \frac{1}{2}K$, and $R = \left(1 - \frac{\sqrt{3}}{2}\right)K$. For example, using the data for
the specimen LOR (fibre diameter $d = 16 \, \mu m$, section separation depth $\Delta d = 13.8 \, \mu m$, $N_2 = 344$, and $N_1 = 414$) yields an average fibre length estimate of $84 \, \mu m$. All estimation results are given in Table 5.5.

<table>
<thead>
<tr>
<th>Specimen</th>
<th>Number of Matched Fibres, $N_2$</th>
<th>Total Number of Fibres with $\phi &lt; 30^\circ$, $N_1$</th>
<th>Ratio $(N_2/N_1)$</th>
<th>Average Fibre Length ($\mu m$)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LOR</td>
<td>344</td>
<td>414</td>
<td>0.831</td>
<td>84</td>
</tr>
<tr>
<td>SOR</td>
<td>621</td>
<td>683</td>
<td>0.909</td>
<td>138</td>
</tr>
<tr>
<td>NR1</td>
<td>313</td>
<td>350</td>
<td>0.894</td>
<td>110</td>
</tr>
<tr>
<td>NR2</td>
<td>39</td>
<td>48</td>
<td>0.813</td>
<td>54</td>
</tr>
</tbody>
</table>

All length values in Table 5.5 are significantly lower than the average fibre length of 900 $\mu m$ observed prior to mixing of the fibre-resin composite liquid. A decrease in the average fibre length is expected due to fibre breakage in processing. However, the extent of the decrease is still considered to be significant, and thus, before drawing further conclusions, the above results should be verified in a future study by direct measurement of fibre length. This can be accomplished by burning out the polymer and examining the fibres under a microscope equipped with an X-Y measurement table (as was done for measuring fibre length prior to mixture compounding).

Another notable point about the length observations is that the average length shows a decreasing trend according to the building sequence of the specimens. The four specimens were built in a sequence NR1, SOR, LOR, NR2, corresponding to the observed lengths of 110, 138, 84, and 54 $\mu m$. This provides further evidence for fibre breakage during processing, as the same composite liquid was recycled for building of all of the specimens.

When the fibre length falls below a certain critical value, its contribution to the composite's strength falls off sharply (Piggott, 1981). Through the fibre pullout experiments, it was roughly estimated that the critical length is 680 $\mu m$ (Chapter 2). If the average fibre...
length is indeed significantly shorter, as noticed from fibre observations, then the fabrication process needs to be modified for reducing fibre breakage.

5.4.2.3 Fibre length distribution

Fibre-length distribution (FLD) is needed in the calculation of the orientation-dependent-bias correction function (Section 5.2.5). Figure 5.25 shows a fibre-length histogram obtained prior to mixture preparation. A two-parameter Weibull distribution fits this data well:

\[ p(L) = \frac{b}{a} \left( \frac{L}{a} \right)^{b-1} e^{-\left( \frac{L}{a} \right)^b}, \quad L > 0, \]  

with \( a = 0.9 \) and \( b = 1.2 \). Other researchers also noted that Weibull distribution represents well fibre-length histograms (Fu and Lauke, 1996, Chin et al., 1988).

Given the average fibre length estimate and the distribution shape defined by the shape parameter \( b \) of the Weibull distribution, the FLD can be approximately represented by adjusting the so-called scale parameter, \( a \), of the Weibull distribution. The expected mean value of the Weibull distribution is given by:

\[ \mu = a \Gamma(1 + 1/b). \]  

Equating \( \mu \) to 100 \( \mu m \), based on our fibre length observations, and setting \( b = 1.2 \), \( a = 0.106 = 0.1 \) results for the examined specimens. Figure 5.26 presents for comparison the Weibull distribution fitted to the fibre length histogram observed prior to mixture preparation with the Weibull distribution adjusted to correspond to the average fibre length observed in the sample. The latter distribution was used in calculation of the orientation-dependent-bias correction function. (Note that the vertical scale of the two plots was equalized to a maximum of one for display purposes.)
5.4.2.4 Comparing fibre-orientation distributions

In this section the FODs are presented for the four specimens examined. Figure 5.27 shows the FODs for the riveted and non-riveted specimens. Dashed lines show the raw histograms; solid lines indicate the same histograms adjusted by the observability-correction function (Section 5.2.5.3). Both riveted specimens (especially SOR) exhibit tendency for
greater alignment with the cross-section normal, i.e., they show greater probability of finding fibres with lower misalignment angles (\( \phi \)). The non-riveted specimens show notable difference between them: NR1 shows alignment tendency similar to LOR specimen, while NR2 shows preferential orientation at \( \phi=90 \) degrees.

In order to verify the consistency of our fibre orientation observations, FODs obtained for different sections or different layers of the same specimen were examined. Due to the time-consuming nature of the data collection process, the two-section method was applied to two layers in each riveted specimen and to one layer in each non-riveted specimen. Since the two layers in riveted specimens are interconnected, they are considered as the smallest repeatable unit.

One can also use the single-section-based orientation measurements from Section 0 to compare with those from Sections 1 or 2. Even though these orientation distributions will be not correct in an absolute sense due to the estimation bias for near-zero \( \phi \), they can still be used for comparison purposes. Figure 5.28 shows the single-section-based distributions. For each specimen, one distribution based on Section 0 and another based on Section 2 are presented. Section 2 is the furthest from 0 and thus most likely to have a different distribution. The distributions show similar trends for all the sections.
Figure 5.27. Fibre-orientation distributions for riveted specimens (a) LOR and (b) SOR, and for non-riveted specimens (c) NR1 and (d) NR2. (Dashed lines – raw data; solid lines – data adjusted by observability-correction function.)
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(a) Weighted FOD (407a-0)

(b) Weighted FOD (407a-2)

(c) Weighted FOD (403b-0)

(d) Weighted FOD (403b-2)

(e) Weighted FOD (401b-0)

(f) Weighted FOD (401b-2)
5.4.2.5 Variation of fibre orientation within layers

In this section, the variation of the fibre-orientation distribution within layers of the riveted specimens is examined. This is accomplished by calculating the FODs of different sub-areas of the same cross-section. The sub-areas examined span two layers (Figure 5.29). They include: upper layer – left half (U-L), upper layer – right half (U-R), lower layer – left half (L-L), and lower layer – right half (L-R).

![Figure 5.29. Sub-areas where fibre orientation was evaluated locally for two-layer cross-sections of riveted specimens.](image)

It would be desirable to isolate the effect of the interlayer rivets on fibre orientation: if rivets cause fibre alignment, then it would be reasonable to assume that this alignment occurs somewhere within the rivet volume. The FODs obtained for the sub-areas are shown in Figure 5.30 for LOR and in Figure 5.31 for SOR. In the upper-right corner of each distribution plot is a small diagram indicating by crosshatching the sub-area that the plot represents.

Figure 5.30(d) (area L-R), for LOR specimen, clearly shows the FOD pattern having a greater alignment in the rivet area. The least alignment is shown in Figure 5.30(a), which
diagonally opposes the bottom of the rivet area. Figure 5.31 for SOR specimen shows more evenly distributed orientation pattern for all sub-areas. One can, thus, speculate that due to the smaller size of the SOR rivets, their effect on FOD is not as pronounced as in the LOR specimens.

![Weighted FOD graphs](image)

Figure 5.30. FOD for four sub-areas in the riveted specimen LOR: (a) upper-left (b) upper-right; (c) lower-left; and (d) lower-right.
5.4.2.6 Fibre-orientation distributions with respect to three orthogonal directions

The misalignment and azimuth angle distributions were calculated with respect to X, Y, and Z axes of the original frame, shown in Figure 5.32(a). To align the new Z axis with the X axis of the original frame, the original frame is rotated by 90° about its Y axis, Figure 5.32(b). To align the new Z axis with the Y axis of the original frame, the original frame is rotated about X by -90°, Figure 5.32(c). The FODs for the original and the two rotated frames are shown in Figure 5.33.
The methodology used to obtain these plots is described in Section 5.2.6. The distributions for X and Y axes were obtained using the rotation matrices $R_x$ and $R_y$, respectively:

$$
R_x = \begin{bmatrix} 0 & 0 & -1 \\ 0 & 1 & 0 \\ 1 & 0 & 0 \end{bmatrix}, \quad \text{and} \quad R_y = \begin{bmatrix} 1 & 0 & 0 \\ 0 & 0 & -1 \\ 0 & 1 & 0 \end{bmatrix}
$$

Figure 5.32. Definition of coordinate frames for calculation of FODs with respect to three orthogonal axes.

The exemplary plots shown in Figure 5.33 are for the SOR specimen. All plots indicate a strong preference for in-the-layer-plane alignment of the fibres. This is expected both due to the presence of the layers, which restrict transverse-to-layer-plane fibre orientation, and due to fibre settling, which causes a significant fraction of fibres to lie in a horizontal plane.

The alignment is especially clear in Figure 5.33(a). The two peaks at 180 and -180 degrees correspond with the alignment along the new X axis (Figure 5.33(b)), which is the direction along the specimen long axis. Distribution of azimuth angles in Figure 5.33(c) corresponds to the in-the-layer-plane rotation of the fibres. The peak at 90° clearly shows the preferential fibre alignment along the specimen long axis.
Figure 5.33. Misalignment and azimuth angle histograms for SOR specimen calculated with respect to the (a-b) X, (c-d) Y, and (e-f) Z axes of the original coordinates.
5.5 Summary

In this chapter, a novel method is proposed for obtaining three-dimensional FOD. The two-section method (i) produces unbiased distribution data for the near-zero misalignment angle range, (ii) resolves the orientation duality problem, and (iii) uses an observability-correction function taking into account the fibre length distribution and the effect of finite layer thickness.

In addition, a new technique is proposed to accurately estimate the average fibre length in the specimen by calculating the ratio between the number of fibres observed in one section and the number observed in both sections. Such information can be directly used in the prediction of the material's mechanical properties, as well as in the estimation of the observability-correction function for FOD calculations.

The above methodology has been applied to investigate the fibre-orientation distribution within a set of layered short-glass-fibre composites. Four dogbone-shaped tensile test specimens have been examined: two fabricated using a standard building procedure and two using a “riveted” building procedure. Further examination of the local variation of the FOD within one riveted specimen has shown significantly increased alignment of fibres in the neighbourhood of the “rivet” feature. This indicates that the increased alignment of fibres is a likely contributing factor to the improved mechanical properties.
Chapter 6. Mechanical Properties of Layered Composites

6.1 Introduction

Since composite layered parts are expected to be employed as functional prototypes, their mechanical properties acquire a much greater significance. In fibre-reinforced composite materials, the inherent mechanical properties of the matrix material are modified by the introduction of the reinforcing fibres. The property improvement derives from the transfer of stresses from the matrix to the fibres through the fibre-matrix interface. Thus, the mechanical properties of composite materials depend on the material properties of the two constituent components and their interface, as well as the amount of reinforcing material and its geometrical arrangement within the matrix.

The amount of reinforcements and their arrangement are determined by the composite fabrication process. To understand how the manufacturing process affects the finished product properties, one needs a model which relates the above-mentioned characteristics of composites to their mechanical properties.

There exist a number of models for predicting composite-material properties. A good overview of the subject is given in Hashin, 1983. For example, the Mori-Tanaka theory (Mori & Tanaka, 1973) evaluates the average internal stress in the matrix of a material with ellipsoidal inclusions. While the theory requires knowledge of the shape and orientation of every particle, in practice, statistical descriptions in the form of the fibre-orientation and fibre-length distributions are used.

Both Pettermann et al., 1997, and Biolzi et al., 1994, use the generalized Mori-Tanaka mean-field approach to take into account the fibre-orientation distribution (FOD) and thus predict modulus and strength of short-fibre composites. Biolzi et al., 1994, obtain both fibre-length distribution (FLD) and 2-D FOD and apply these to predict modulus and Poisson’s ratio of short-fibre-reinforced polymers (SFRP) with volume fractions up to 48%. In their work, the fibres were preferentially aligned in the injection-moulded specimens used. Good agreement (within 5%) between predicted and experimental moduli was obtained. Authors
report on the importance of having correct microstructural data (i.e., FLD and FOD), and, as an example, point out that modulus predictions based only on the mean fibre length overestimated the effective elastic moduli.

Zhu et al., 1997b, develop a strength-prediction theory for short-fibre composites, taking into account the three-dimensional FOD. Fu and Lauke, 1996, predict the tensile strength of SFRP based on both fibre-length and fibre-orientation distributions. They use modified rule-of-mixtures expression and calculate the product of the fibre-length and orientation-correction factors as a function of the FOD and FLD. They represent fibre length by a two-parameter Weibull distribution function, and use two-parameter exponential function to describe FOD as a function of the misalignment angle only.

In this thesis, the most commonly used and comparatively straightforward model based on the modified rule-of-mixtures expression is employed for mechanical property prediction. In this chapter, after presenting the model, it is used to predict mechanical properties of layered parts based on the previously obtained information on the fibre-matrix interface (Chapter 2) and the fibre geometrical arrangement (Chapter 5). Finally the results of the mechanical tests on the layered composites fabricated on the experimental system described in Chapter 3 are presented.

### 6.2 Estimation of the mechanical properties of composites

Models used to predict mechanical properties of a composite material differ depending on whether the material contains long- or short-fibre reinforcements. While in this thesis only short-fibre reinforcements have been employed, it is instructive to consider the simpler case of long fibres to introduce the subject of composite mechanics. For either case, the main quantities of interest are the composite’s tensile modulus and strength. Both are frequently direction dependent. Namely, the modulus and strength estimates are sought along a particular direction. Such a direction will be referred to as the direction of interest.

---

1 A fibre can be considered “long” when its aspect ratio exceeds approximately 1000. For example, for a fibre with diameter 15 μm, the corresponding fibre length would be 15 mm.
6.2.1 Mechanics of long-fibre composites

The simplest possible case is that of a long-fibre composite with all its fibres aligned along the direction of interest (Figure 6.1). It is assumed that the average strain in the fibres and in the matrix are the same, i.e., when stressed along the fibre alignment direction by $\sigma_f$, the matrix and the fibres will extend by the same distance.

![Figure 6.1. Stress applied to a long-fibre composite.](image)

Since the fibre, matrix, and the composite have the same strain, it can be stated that

$$\sigma_i = E_i \varepsilon_i \quad \sigma_f = E_f \varepsilon_i \quad \sigma_m = E_m \varepsilon_i,$$

(6.1)

where $\sigma_i$, $E_i$, and $\varepsilon_i$ are the composite stress, tensile modulus, and strain in direction “1,” respectively; and $\sigma_f$ and $E_f$ are the fibre, and $\sigma_m$ and $E_m$ are the matrix stress and tensile modulus, respectively. The load carried by the composite is

$$\sigma_i A = \sigma_f A \nu_f + \sigma_m A \nu_m,$$

(6.2)

where $A$ is the cross-section area, $\nu_f$ is the fibre and $\nu_m$ is the matrix volume fractions. Fractions of the cross-section area occupied by the fibres and the matrix equal to their volume fractions. Using the definitions of $\sigma_f$ and $\sigma_m$ in Equation (6.1), and cancelling $A$ in Equation (6.2),

$$\sigma_i = (\nu_f E_f + \nu_m E_m) \varepsilon_i = E_i \varepsilon_i,$$

(6.3)

which yields the desired expression for the tensile modulus of long-fibre composite in the fibre direction as:

$$E_i = \nu_f E_f + \nu_m E_m.$$

(6.4)
This type of expression, where the composite material property depends on the corresponding constituent component properties, is referred to as the Rule of Mixtures; therefore, the composite is said to obey the Rule of Mixtures for modulus.

Similar arguments produce an expression for tensile strength in the direction "1" as:

$$\sigma_{tu} = v_f \sigma_{fu} + v_m \sigma_{mu},$$

(6.5)

where $\sigma_{fu}$ and $\sigma_{mu}$ are the fibre and matrix ultimate tensile strengths, respectively.

### 6.2.2 Mechanics of short-fibre composites

#### 6.2.2.1 Modulus prediction for aligned fibres

For short fibres, the assumption of equal strain in the matrix and fibres is no longer valid. When the short-fibre composite is stressed, the stresses are transferred to the fibres by shear at the fibre surfaces, which causes the fibres to deform. Since the fibres are much stiffer than the matrix, the fibre strain is smaller than the overall strain in the composite.

To estimate the tensile modulus of short-fibre composite, Cox, 1952, proposed a theory (later referred to as "shear-lag" theory) that assumes a completely elastic transfer of stresses from the matrix to the fibre. Cox analysis considers an ideal case of short fibres aligned with the stress direction and arranged in a particular pattern (e.g., a square or a hexagonal array). The theory derives the distribution of tensile stress within the fibre, along its axial direction. Thus, the stress at distance $x$ from the fibre centre is given by

$$\sigma_f(x) = E_f \varepsilon_i \left(1 - \frac{\cosh(2n_c x/d)}{\cosh(n_c s)}\right),$$

(6.6)

where

$$n_c = \sqrt{\frac{2G_m}{E_f \ln(2R/d)}}, \quad G_m = \frac{E_m}{2(1 + v_m)}, \quad \frac{2R}{d} = \frac{\pi}{4v_f}, \quad \text{and} \quad s = \frac{l}{d}. \quad (6.7)$$

Above, $s$ is the aspect ratio for fibres of length $l$ and diameter $d$, $G_m$ is the matrix shear modulus, and $v_m$ is the matrix Poisson ratio.

The fibre stress calculated from Equation (6.6) and the shear stress at the fibre surface predicted by the same theory are plotted in Figure 6.2 for a 1-mm long fibre at 1% strain. One
can note how the stress in the fibre is zero at the fibre ends and how it rises quickly to its maximum value. Also, the shear stress at the fibre-matrix interface is at its maximum at the fibre ends. When the composite is stressed, the theory indicates that the initial debonding should occur near the fibre ends. Piggott and Xiong, 1994, confirm this by direct observation of glass fibres in epoxy resin. Complete debonding of fibre ends is observed to occur at only 0.5% strain.

By averaging the stress distribution within the fibres, the theory predicts the tensile modulus for aligned short-fibre composites, in the direction of alignment, to be equal to:

$$E_1 = \chi_2 \nu_f E_f + \nu_m E_m,$$  \hspace{1cm} (6.8)

where *fibre-length correction factor*, $\chi_2$, is equal to:

$$\chi_2 = 1 - \frac{\tanh(n_c s)}{n_c s}.$$  \hspace{1cm} (6.9)

The fibre-length correction factor is plotted in Figure 6.3 for the same data used for Figure 6.2. The relationship shows that the effectiveness of reinforcements quickly drops for fibres with aspect ratios below 30 (i.e., fibre length of 0.5 mm).

![Figure 6.2](image)

*Figure 6.2. Variation of (a) stress within fibre and (b) shear stress at fibre surface predicted by the shear-lag theory (for $E_f=70$ GPa, $E_m=1.5$ GPa, $\nu_f=0.15$, $\nu_m=0.3$, $d=16$ $\mu$m, $t=1$ mm, $\varepsilon=0.01$).*
6.2.2.2 Modulus prediction based on fibre-orientation distribution

When fibres are not perfectly aligned with the direction of modulus estimate, an orientation-efficiency factor, $\chi_l$, is added to Equation (6.8) (Hull, 1981):

$$ E_c = \chi_l \chi_s v_f E_f + v_m E_m, \quad 0 < \chi_l < 1. \quad (6.10) $$

For in-plane uniformly distributed fibre orientations, the factor is $3/8$ when estimating the in-plane composite modulus. The factor is $1/5$ for three-dimensional uniform fibre-orientation distribution.

The orientation-efficiency factor can be calculated more precisely if the fibre-orientation distribution is known. While in general FOD is a joint probability density function of two angles, $p(\theta, \phi)$, for the approach described below, a marginal distribution with respect to angle $\phi$ only is required, $p(\phi)$. Angle $\phi$ is defined as the angle between the direction of interest and the fibre axis (see Figure 5.1 in Chapter 5 for definition of $\theta$ and $\phi$).

To estimate the orientation-efficiency factor, $\chi_l$, the composite is modelled as a laminate consisting of laminae with different fibre orientations (Sanadi and Piggott, 1985a). Each lamina $k$ is assigned a thickness, $t_k$, proportional to the fraction of fibres at this particular angle.
Laminate theory predicts the modulus along the Z direction for fibres aligned at angle $\phi$ to Z as (Jones, 1975):

$$\frac{1}{E_\phi} = \frac{\cos^4 \phi}{E_1} + \left(\frac{1}{G_{12}} - \frac{2v_{12}}{E_1}\right)\sin^2 \phi \cos^2 \phi + \frac{\sin^4 \phi}{E_2}.$$  \hspace{1cm} (6.12)

This relationship is plotted in Figure 6.4 for a typical composite used in these studies ($E_f=70$ GPa, $v_f=0.15$, $v_m=0.3$, $E_m=1.5$ GPa, $v_m=0.3$). In the above, $E_1$ is the tensile modulus for perfectly aligned long fibres given by Equation (6.4). Also, $E_2$, the composite tensile modulus in the transverse direction, $G_{12}$, the composite shear modulus, and $v_{12}$, the composite Poisson's ratio, are given by:

$$\frac{1}{E_2} = \frac{v_f}{E_f} + \frac{v_m}{E_m}, \quad \frac{1}{G_{12}} = \frac{v_f}{G_f} + \frac{v_m}{G_m}, \quad \text{and} \quad v_{12} = v_f v_f + v_m v_m.$$  \hspace{1cm} (6.13)

![Modulus vs. Fiber Orientation](image)

Figure 6.4. Effect of fibre orientation on tensile modulus for aligned fibre within a lamina.

Given the relative thickness of each lamina $k$, $t_k$, from Equation (6.11), the value of $\chi_1$ is estimated by:

$$\chi_1 = \frac{\sum_{k=1}^{n} E_{ek} t_k}{E_1 \sum_{k=1}^{n} t_k}.$$  \hspace{1cm} (6.14)
6.2.2.3 Strength prediction based on fibre-orientation distribution

Prediction of the tensile strength for short-fibre composites is a difficult task, and no universally accepted theory exists on this subject (Piggott et al., 1993). The difficulty arises because the material's ultimate strength in the case of composites is determined by the onset of fracture, and not via a yielding mechanism. Composite strength models most frequently take the form of:

\[ \sigma_c = \chi_3 \chi_4 v_f \sigma_{fu} + \nu_m \sigma^*_m, \]  
(6.15)

where \( \sigma_{fu} \) is the fibre tensile strength, \( \sigma^*_m \) is the tensile stress in the matrix at composite failure strain, and \( \chi_3 \) and \( \chi_4 \) are the fibre-length correction and orientation-efficiency factors, respectively, for composite strength. The difficulty lies in the estimation of the correction factors.

For example, a commonly used "Kelly-Tyson" model based on slip theory (Kelly, 1973), proposes a fibre-length correction factor

\[ \chi_4 = \begin{cases} 
1 - s_c / 2s & \text{for } s > s_c, \\
\frac{s_c}{2s} & \text{for } s \leq s_c,
\end{cases} \]  
(6.16)

where \( s_c = \sigma_{fu} / 2\tau_{iu} \) is the critical aspect ratio for the composite, \( \sigma_{fu} \) is the fibre's ultimate tensile strength and \( \tau_{iu} \) is the interfacial shear strength.

Piggott, 1994, proposes that the short-fibre composite strength model should be derived from a fracture-based, instead of slip-based, theory that gives the fibre-length correction factor as:

\[ \chi_4 = \begin{cases} 
1 - s_s / 2s - 12.5 / s \frac{ss}{s_c} + 5\sigma_{mu} / s\sigma_{fu} & \text{for } s > s_c, \\
(s / s_c)(1 - 25 / s^2) + 5\sigma_{mu} / s\sigma_{fu} & \text{for } s \leq s_c,
\end{cases} \]  
(6.17)

where \( \sigma_{mu} \) is the matrix ultimate tensile strength.

To account for fibre misalignment in the strength model, an approach based on the strain-energy concept can be used (Sanadi and Piggott, 1985a). This approach is similar to the method used for modulus calculation in Equation (6.14). The orientation-efficiency factor is calculated by summing contributions from \( n \) "laminae" of thickness \( t_k \), with fibres in each
lamina oriented at angle $\phi_k$ and thickness proportionate to the number of fibres oriented at an angle $\phi_k$:

$$\chi_3 = \frac{\sum_{k=1}^{n} \sigma_{u\theta_k} t_k}{\sigma_{1u} \sum_{k=1}^{n} t_k},$$

with $\sigma_{u\theta_k}$ given by

$$\frac{1}{\sigma_{u\theta_k}^2} = \frac{\cos^4 \phi}{\sigma_{1u}^2} + \left( \frac{1}{\tau_{12u}^2} - \frac{1}{\sigma_{1u}^2} \right) \sin^2 \phi \cos^2 \phi + \frac{\sin^4 \phi}{\sigma_{2u}^2},$$

where

$$\sigma_{1u} = \nu_j \sigma_{fu} + \nu_m \sigma_{m}, \quad \tau_{12u} = \frac{\sigma_{mu}}{2}, \quad \text{and} \quad \sigma_{2u} = \sigma_{mu}. \quad (6.20)$$

Figure 6.5 shows the dependence of strength reinforcement efficiency on the misalignment angle $\phi$, as predicted by Equation (6.19). The curve is plotted for the volume fraction of 15%, with the remaining data the same as for Figure 6.6. Figure 6.6 displays an exemplary plot of composite tensile strength as a function of fibre volume fraction based on Equation (6.15). The plot uses material data for the 737BD 1.6-mm fibres and SL5170 resin (see Table 6.2 in Section 6.3.2). To obtain the orientation-efficiency factor, $\chi_3$, Equation (6.18) was applied for the case of in-plane uniformly distributed fibre orientations. For the fibre-length correction factor, $\chi_4$, Equation (6.16) according to Kelly, 1973, was used to give 0.788. Using Equation (6.17) according to Piggott, 1994, to estimate $\chi_4$ resulted in a small reduction of this parameter (0.785). The model predicts a modest strength improvement with the addition of fibre reinforcements. For example, addition of 20% fibres by volume increases the strength by 6.5%.
6.2.2.4 Accounting for fibre-length distribution

As shown by Equation (6.10) for modulus and Equation (6.15) for strength, both material properties are affected by the fibre length in short-fibre composites. The effect of finite fibre length is accounted for by the fibre-length correction factors: \( \chi_2 \) for modulus and \( \chi_4 \) for strength. When the fibre length is not uniform, the factors can be obtained as weighted sums, with the weights determined by the fibre-length distribution (Piggott et al., 1993). To calculate these weights, the fibre-length distribution has to be modified to represent the fraction of the total fibre volume, as opposed to the number of fibres, for each fibre length interval. This adjustment is needed since the mechanical properties are a function of fibre volume, and not the number of fibres.

Since the fibre diameter can be assumed to be the same for fibres of all lengths, the total volume occupied by fibres in any particular length range is proportional to the number of fibres in that range multiplied by the fibre length. If the probability of finding fibres of length \( l \) is \( p(l) \), then the weighting factor proportional to the total volume of all length \( l \) fibres is

\[
P_s = l p(l).
\]  

(6.21)

For example, the FLD represented by Weibull distribution with \( a=0.1 \) and \( b=1.2 \) is plotted in Figure 6.7. The plot shows the length distribution both in terms of a "number fraction," \( p(l) \), and in terms of a "volume fraction," \( P_s \). Since the longer fibres occupy relatively greater
volume than the shorter ones, the $P_x$ distribution is skewed towards the greater fibre lengths compared with original $p(l)$ distribution.

Figure 6.7. Fibre-length distribution plotted as "number fraction" and "volume fraction" of the fibres (Weibull distribution with $a=0.1$ and $b=1.2$).

Therefore, to account for fibre-length distribution, the fibre-length correction factor for modulus is calculated as (Piggott et al., 1993):

$$
\chi_2 = \sum_{k=1}^{m} P_{sk} \left\{ 1 - \tanh(ns_k) / ns_k \right\} ,
$$

(6.22)

where $P_{sk}$ is the fraction of the total fibre volume occupied by fibres with the aspect ratio $s_k$.

The fibre-length correction factor for strength is given by (Piggott et al., 1993):

$$
\chi_s = \sum_{k=1}^{m} P_{sk} \left\{ 1 - s_c / 2s_k \right\} + \sum_{k=1}^{m} P_{sk}s_c / 2s_k ,
$$

(6.23)

where $P_{sk}$ is the fraction of the total fibre volume occupied by longer fibres, having aspect ratio ($s_k > s_c$), and $P_{sk}$ is the corresponding fraction occupied by shorter fibres, having aspect ratio ($s_k \leq s_c$).
6.3 Effect of layered structure

6.3.1 Orthotropic materials

For elastic solids, the relationship between the applied stresses and the resulting strains is described by a constitutive equation known as the generalized Hooke's Law (Gibson, 1994). The law can be expressed in terms of a stiffness matrix as

$$\sigma = C \varepsilon,$$

(6.24)

where $C$ is a $6 \times 6$ stiffness matrix, $\sigma$ is a $6 \times 1$ stress vector, and $\varepsilon$ is a $6 \times 1$ strain vector. Alternately, the generalized Hooke's Law can be written in terms of a compliance matrix $S = C^{-1}$:

$$\varepsilon = S \sigma.$$

(6.25)

For an isotropic material, the coefficients of the compliance matrix can be expressed in terms of only two independent parameters, the elastic modulus $E$ and the Poisson's ratio, $\nu = -\varepsilon_3/\varepsilon_1$:

$$S = \begin{bmatrix}
\frac{1}{E} & -\nu/E & -\nu/E \\
1/E & -\nu/E & 0 \\
1/E & 0 & 0 \\
\end{bmatrix}
\begin{bmatrix}
0 & 0 & 0 \\
0 & 0 & 0 \\
0 & 0 & 0 \\
\end{bmatrix},$$  

(6.26)

where $G = E/2(1 + \nu)$ is the shear modulus.

However, the short-fibre layered composites in this thesis are not isotropic materials, i.e., their material properties are a function of direction. The anisotropy is mainly caused by non-uniformity in spatial distribution of fibre orientations. As shown in Chapter 5, the fibres have a tendency to align parallel to layer planes. The interlayer boundaries may also weaken the mechanical properties transverse to the layer plane. Therefore, the short-fibre layered composites can be described as either (a) orthotropic materials or (b) transversely isotropic materials. Orthotropic materials have three mutually orthogonal planes of symmetry for material properties (Figure 6.8). Transversely isotropic materials have mechanical properties independent of the direction within the layer plane (normal to $Z$ axis in Figure 6.8).
Chapter 6. Mechanical Properties of Layered Composites

For orthotropic materials, the number of independent parameters in a compliance matrix is 9, compared to 2 for isotropic materials.

\[
S = \begin{bmatrix}
S_{11} & S_{12} & S_{13} & 0 & 0 & 0 \\
S_{12} & S_{22} & S_{23} & 0 & 0 & 0 \\
S_{13} & S_{23} & S_{33} & 0 & 0 & 0 \\
0 & 0 & 0 & S_{44} & 0 & 0 \\
0 & 0 & 0 & 0 & S_{55} & 0 \\
0 & 0 & 0 & 0 & 0 & S_{66}
\end{bmatrix}
\] (6.27)

Expressing the elastic constants in terms of engineering constants, the compliance matrix can be written as:

\[
S = \begin{bmatrix}
1/E_1 & -v_{12}/E_1 & -v_{13}/E_1 & 0 & 0 & 0 \\
-1/v_{12}/E_2 & 1/E_2 & -v_{23}/E_2 & 0 & 0 & 0 \\
-1/v_{13}/E_3 & -1/v_{23}/E_2 & 1/E_3 & 0 & 0 & 0 \\
0 & 0 & 0 & 1/G_{23} & 0 & 0 \\
0 & 0 & 0 & 0 & 1/G_{31} & 0 \\
0 & 0 & 0 & 0 & 0 & 1/G_{12}
\end{bmatrix}
\] (6.28)

where \(E_1, E_2, \text{ and } E_3\) are the Young's moduli, \(G_{ij}\) are the shear moduli, and \(v_{ij}\) are the Poisson's ratios

\[
v_{ij} = -e_{j}/e_{i}, \quad i, j = 1, 2, 3, \ i \neq j,
\] (6.29)

where the \(j\) direction is perpendicular to the \(i\) direction and the stress is applied in the \(i\) direction.

If the transverse isotropy is assumed for the short-fibre layered composite (i.e., properties invariant with rotation about the layer-plane normal), the compliance matrix is simplified to 5 independent parameters. Assuming the 1-2 plane is the plane of isotropy (i.e., the layer plane), the compliance matrix is given by
Since the properties are invariant in the 1-2 plane, the subscripts 1 and 2 are interchangeable, so that \( E_1 = E_2 \), \( G_{23} = G_{31} \), and \( v_{13} = v_{23} \).

\[
S = \begin{bmatrix}
S_{11} & S_{12} & 0 & 0 & 0 \\
S_{12} & S_{13} & 0 & 0 & 0 \\
0 & 0 & S_{33} & 0 & 0 \\
0 & 0 & 0 & S_{44} & 0 \\
\text{sym} & \text{sym} & \text{sym} & \text{sym} & 2(S_{11} - S_{12})
\end{bmatrix}, \tag{6.30}
\]

Figure 6.8. Principal directions and orthogonal planes of an orthotropic material.

6.3.2 Predictions of direction-dependent mechanical properties

Classifying the layered composites as orthotropic materials, to obtain a complete description of their elastic behaviour, one would need to obtain estimates for all 9 engineering constants in Equation (6.28). However, the main indicators of material behaviour are the tensile moduli. Thus, to evaluate the variation of the material properties with direction, the tensile moduli are estimated below for each of the three principal directions. Also estimated are the tensile strengths along each direction.

The three directions are defined in Figure 6.8. The fibre orientation measurements indicate that the wiping process affects fibre orientation within layers. Thus, one of the principal directions (X) is defined as parallel to the wiping direction. The Z axis is normal to
the layer plane. Therefore, the Y axis lies in the layer plane, perpendicular to the wiping direction.

The tensile modulus is estimated in each direction by evaluating Equation (6.10). This requires calculation of the orientation-efficiency ($\chi_1$) and fibre-length correction ($\chi_2$) factors using Equations (6.14) and (6.22), respectively. The fibre-orientation and fibre-length distributions used to evaluate these factors are obtained from specimen section examination described in Chapter 5, Section 5.4.

For data given in Table 6.3 and Table 6.4, observations for the specimen identified as “SOR” (“Small Offset Rivets”) were used (Table 6.1). Additionally, the required material properties of the constituent components and their interface are presented in Table 6.2. All the properties are from material specifications provided by the manufacturer or reference sources, except the pure-resin modulus and strength and the interfacial shear strength. The pure resin properties used are from the experimental results for the layered pure-resin specimens. The interfacial shear strength is from the fibre pull-out tests described in Chapter 2.

Figure 6.9 shows the resulting predictions for modulus and strength along the three principal directions. The orientation particularly impacts the material modulus, as it decreases by about 33% from the modulus along the X axis (wiping direction) to modulus along either of the two other principal axes. The equivalent effect on strength is not as strong (about 10%).

<table>
<thead>
<tr>
<th>Name</th>
<th>ID</th>
<th>Fibre Content (%)</th>
<th>Modulus (GPa)</th>
<th>Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>SOR</td>
<td>403Cb</td>
<td>17.9</td>
<td>2.43</td>
<td>66.9</td>
</tr>
</tbody>
</table>
Chapter 6. Mechanical Properties of Layered Composites

Table 6.2. Constituent component properties used for composite material property predictions.

<table>
<thead>
<tr>
<th>Material</th>
<th>Property</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Matrix (SL5170)</td>
<td>Tensile modulus, $E_m$ (GPa)</td>
<td>1.37</td>
</tr>
<tr>
<td></td>
<td>Tensile strength, $\sigma_m$ (MPa)</td>
<td>62</td>
</tr>
<tr>
<td></td>
<td>Poisson’s Ratio, $\nu_m$</td>
<td>0.35</td>
</tr>
<tr>
<td>Fibre (737BD)</td>
<td>Tensile modulus, $E_f$ (GPa)</td>
<td>72</td>
</tr>
<tr>
<td></td>
<td>Tensile strength, $\sigma_f$ (GPa)</td>
<td>3.4</td>
</tr>
<tr>
<td></td>
<td>Poisson’s Ratio, $\nu_f$</td>
<td>0.22</td>
</tr>
<tr>
<td>Interface</td>
<td>Interfacial shear strength, $\tau_{iu}$ (MPa)</td>
<td>40</td>
</tr>
</tbody>
</table>

Table 6.3. Predicted tensile modulus dependence on orientation.

<table>
<thead>
<tr>
<th>Direction</th>
<th>Orientation-Efficiency Factor ($\chi_1$)</th>
<th>Fibre-Length Correction Factor ($\chi_2$)</th>
<th>Product ($\chi_1\chi_2$)</th>
<th>Predicted Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>X</td>
<td>0.340</td>
<td>0.291</td>
<td>0.0988</td>
<td>2.40</td>
</tr>
<tr>
<td>Y</td>
<td>0.136</td>
<td>0.291</td>
<td>0.0395</td>
<td>1.63</td>
</tr>
<tr>
<td>Z</td>
<td>0.118</td>
<td>0.291</td>
<td>0.0342</td>
<td>1.57</td>
</tr>
</tbody>
</table>

Table 6.4. Predicted tensile strength dependence on orientation.

<table>
<thead>
<tr>
<th>Direction</th>
<th>Orientation-Efficiency Factor ($\chi_3$)</th>
<th>Fibre-Length Correction Factor ($\chi_4$)</th>
<th>Product ($\chi_3\chi_4$)</th>
<th>Predicted Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>X</td>
<td>0.191</td>
<td>0.105</td>
<td>0.0201</td>
<td>63.4</td>
</tr>
<tr>
<td>Y</td>
<td>0.098</td>
<td>0.105</td>
<td>0.0103</td>
<td>57.4</td>
</tr>
<tr>
<td>Z</td>
<td>0.091</td>
<td>0.105</td>
<td>0.0095</td>
<td>57.0</td>
</tr>
</tbody>
</table>
6.4 Tensile tests of layered composites

A number of tensile tests were conducted throughout the period of development of the RLCM (Rapid Layered Composites Manufacturing) process. These tests served to provide feedback for process modifications. This section will report primarily on a series of tensile tests conducted after substantial completion of the process design phase, Chapter 3.

The test objectives were to determine (1) the degree of improvement in the material properties of pure resins which can be achieved by the addition of reinforcements, (2) the effect of alternative layer-scanning styles (rivets vs. no rivets), and (3) the effect of part-building orientation (horizontal vs. vertical). Tests were conducted on dogbone-shaped specimens built on the RLCM prototype system. The specimens were tested in tension until failure, with the main parameters observed being the tensile modulus and strength.
6.4.1 Procedure

6.4.1.1 Specimens

The dimensions of the dogbone-shaped tensile test specimens were set according to the guidelines given in ASTM Standard Test Method D638-91a (Tensile Properties of Plastics) for specimen type M-III (Figure 6.10).

All the specimens were fabricated on the RLCM prototype system over a two-week period. The pure-resin specimens were made from SU170 resin (Ciba-Geigy), and the composite specimens were made by adding 737BD 1.6-mm glass fibres to this resin.

![Figure 6.10. Dogbone-shaped tensile test specimen (all dimensions are in mm).](image)

The tests were performed on seven sets, each comprising four specimens. The set names and descriptions are given in Table 6.6. Two sets of pure-resin and four sets of composite horizontally oriented specimens were built. The two pure-resin sets comprised specimens with Non-Riveted building style (HP_NR) and Large In-line Rivets building style (HP_LIR) to test for the effect of rivets. The four composite sets comprised one with Non-Riveted building style (HC_NR) and three sets of alternate rivet building styles: Large In-line Rivets, Small Offset Rivets, and Large Offset Rivets (HC_LIR, HC_SOR, and HC_LOR). The latter three sets intended to determine the effect of the rivet style. Rivet parameter specifications are listed in Table 6.5. (See Chapter 3 for parameter definitions.) Figure 6.11 shows rivet locations within the specimens ((d) shows a layer from the mid-section of the vertically oriented specimen).
All horizontally oriented specimens were fabricated two-at-a-time, at locations within the processing vat shown in Figure 6.12(a). They were arranged symmetrically with respect to the centre-line of the vat to equalize the effect of building location on each specimen. The specimens contained thirteen layers each.

All four vertically oriented specimens were produced simultaneously. To prevent specimens from toppling, they were interconnected at the side edges of the end-tabs, as indicated in Figure 6.12(b). The specimens contained 200 layers each. After completion of the building process, the vertical specimens were cut away from each other.

<table>
<thead>
<tr>
<th>Rivet Parameter</th>
<th>H(P/C)_LIR</th>
<th>HC_SOR</th>
<th>HC_LOR</th>
<th>VC_SOR</th>
</tr>
</thead>
<tbody>
<tr>
<td>Size</td>
<td>X 3.50</td>
<td>Y 2.0</td>
<td>X 1.25</td>
<td>Y 1.25</td>
</tr>
<tr>
<td>Spacing</td>
<td>X 7.50</td>
<td>Y 5.0</td>
<td>X 3.5</td>
<td>Y 2.50</td>
</tr>
<tr>
<td>Shift</td>
<td>X 3.75</td>
<td>Y 0.0</td>
<td>X 1.75</td>
<td>Y 1.25</td>
</tr>
<tr>
<td>Origin</td>
<td>Y 0.00</td>
<td>Y 0.0</td>
<td>Y -0.25</td>
<td>Y -0.25</td>
</tr>
</tbody>
</table>

Table 6.6. Description of tensile test specimens.

<table>
<thead>
<tr>
<th>Set</th>
<th>Specimen Description</th>
<th>Specimen Build Time</th>
</tr>
</thead>
<tbody>
<tr>
<td>HP_NR</td>
<td>Horizontal, Pure-resin, No-Rivets</td>
<td>2 h</td>
</tr>
<tr>
<td>HP_LIR</td>
<td>Horizontal, Pure-resin, Large In-line Rivets</td>
<td>2 h 20 m</td>
</tr>
<tr>
<td>HC_NR</td>
<td>Horizontal, Composite, No-Rivets</td>
<td>2 h 20 m</td>
</tr>
<tr>
<td>HC_LIR</td>
<td>Horizontal, Composite, Large In-line Rivets</td>
<td>2 h 50 m</td>
</tr>
<tr>
<td>HC_SOR</td>
<td>Horizontal, Composite, Small Offset Rivets</td>
<td>3 h</td>
</tr>
<tr>
<td>HC_LOR</td>
<td>Horizontal, Composite, Large Offset Rivets</td>
<td>2 h 50 m</td>
</tr>
<tr>
<td>VC_SOR</td>
<td>Vertical, Composite, Small Offset Rivets</td>
<td>17 h 10 m</td>
</tr>
</tbody>
</table>
Figure 6.11. Rivet locations within (a) HP_LIR and HC_LIR, (b) HC_SOR, (c) HC_LOR, and (d) VC_SOR specimens (rivets from two consecutive layers are distinguished by the solid and dashed lines).

Building parameters common to all specimens are shown in Table 6.7, and those specific for each set are shown in Table 6.8. After layered fabrication, the parts were post-cured for two hours using an ultraviolet lamp.

Fibre content of the composite specimens was measured via ASTM Standard Test Method D792-91 (Density and Specific Gravity (Relative Density) of Plastics by Displacement).
Figure 6.12. Location of (a) horizontal and (b) vertical dogbone specimens within the vat (top view). Two arrows indicate the direction of wiper motion.

Table 6.7. Common processing parameters for all specimens.

<table>
<thead>
<tr>
<th>Parameter</th>
<th>Value</th>
</tr>
</thead>
<tbody>
<tr>
<td>Laser Power (mW)</td>
<td>13-15</td>
</tr>
<tr>
<td>Scanning Speed (mm/s)</td>
<td>1.5</td>
</tr>
<tr>
<td>Hatch Line Spacing (mm)</td>
<td>0.3</td>
</tr>
</tbody>
</table>

6.4.1.2 Setup and tests

The tensile testing equipment is located in Room WB22, Department of Chemical Engineering and Applied Chemistry, University of Toronto. The specific machine utilized in this thesis was Sintech 20 (S/N 657200) with a 500-kg loadcell (Model 3132-149, S/N 10625). The grips were standard screw-action type, with the upper grip attached to a 50-cm extension bar connected to the cross-head via a universal joint. Grip separation distance (and thus the gauge length) was set to 20 mm. The cross-head speed was set to 1 mm/min, as recommended by the ASTM test method specifications, which state that the extension rate should be set so that the specimen will break within 0.5 to 5 min from start of the test. The load versus extension data were collected using the proprietary digital data acquisition hardware and software.
The tests were conducted ten days after all the parts had been fabricated. The specimens were tested in tension until failure. They were centred and clamped into the upper grip first, and then let hang freely before clamping into the bottom grip. The specimens were visually aligned with the central axis of the two grips.

### 6.4.2 Results

Figure 6.13 displays a Scanning Electron Microscope image of a typical fracture surface of a composite specimen. The holes and the protruding fibres indicate that the fibre pull-out (not breakage) was the prevailing failure mechanism. Table 6.9 summarizes the test results. Figure 6.14 shows representative stress-strain curves obtained for the pure and composite specimens. One can note the high degree of plastic deformation of the pure-resin specimens and the brittle fracture of the composite specimens.
Figure 6.13. Fracture surface of a mould-prepared specimen (SL5170 resin and 737BD 1.6-mm fibres, 15% volume fraction).

Figure 6.15 displays the tensile modulus and Figure 6.16 the tensile strength observed. Figure 6.17 shows the strain-at-break observations. (The error bars indicate a range of ± one standard deviation.) Because of the curved nature of the stress-strain plots, the moduli were calculated by a linear fit to the initial rising portion of the curve (between strains of 0.1 and 0.5%).

Table 6.9. Tensile test results for layered composites.

<table>
<thead>
<tr>
<th>Set</th>
<th>Fiber Content (%)</th>
<th>Modulus (GPa)</th>
<th>Strength (MPa)</th>
<th>Strain at Break (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>HP_NR</td>
<td>0.0</td>
<td>0.0</td>
<td>1.32</td>
<td>0.12</td>
</tr>
<tr>
<td>HP_LIR</td>
<td>0.0</td>
<td>0.0</td>
<td>1.42</td>
<td>0.10</td>
</tr>
<tr>
<td>HC_NR</td>
<td>15.9</td>
<td>1.5</td>
<td>1.79</td>
<td>0.12</td>
</tr>
<tr>
<td>HC_LIR</td>
<td>16.9</td>
<td>0.3</td>
<td>2.26</td>
<td>0.22</td>
</tr>
<tr>
<td>HC_SOR</td>
<td>17.0</td>
<td>1.1</td>
<td>2.49</td>
<td>0.19</td>
</tr>
<tr>
<td>HC_LOR</td>
<td>16.0</td>
<td>0.4</td>
<td>2.48</td>
<td>0.10</td>
</tr>
<tr>
<td>VC_SOR</td>
<td>10.7</td>
<td>0.8</td>
<td>1.63</td>
<td>0.15</td>
</tr>
</tbody>
</table>
Figure 6.14. Sample stress-strain curves for (a) pure (HP_LIR) and (b) composite (HC_SOR) layered specimens. Straight line indicates the slope used for modulus calculation.

Figure 6.15. Tensile modulus of the layered specimens.

The primary findings of the tests are as follows. For composite layered parts, the riveted building style provides a significant improvement (almost 40%) in modulus compared with the non-riveted style; however, rivets do not affect the modulus or strength of the pure-resin layered parts. The improvement in modulus achieved by the introduction of 16% by volume of reinforcements into the pure horizontally oriented layered parts is 81%. Vertical orientation of the specimens reduces their modulus by about 20% and strength by 30%.
Detailed discussion of the test results is presented below, focusing on the three test objectives. First, the effect of the rivets on pure-resin and composite specimens is evaluated. Second, the degree of improvement due to reinforcements is determined. Third, the effect of the part-building orientation is quantified.

Alternate layer-scanning styles (rivets vs. no rivets)

For pure-resin specimens, there is no statistically significant difference between the modulus or the strength values observed for the non-riveted (HP_NR) specimens and the riveted (HP_LIR) specimens. Thus, one can conjecture that rivets do not affect the mechanical properties of pure-resin parts.
Before comparing the non-riveted and riveted composite parts, results for three different rivet styles (differing by the rivet dimensions or relative positioning) are analyzed. A single-factor analysis of variance on the modulus observations for the three rivet styles revealed that there is no significant difference (at 5% level) between them. However, a direct comparison of the LIR- and LOR-type rivets showed that the modulus of the HC_LOR specimens is greater than that of the HC_LJR specimens at a significance level of just over 5%. The observed variability of the HC_LOR modulus data is smaller than for the data collected for the other two rivet styles. However, comparison tests of variance ratios at 5% level did not find this difference statistically significant.

Regarding the strength data, clearly, the means for all rivet styles are equal within the statistical error. However, the variability of the HC_SOR strength results was found to be significantly higher than that of HC_LOR.

Thus, it can be concluded that the modulus of the HC_LJR specimens is lower than that of the HC_LOR specimens and that the HC_LOR strength observations have lower variability than those of the HC_SOR specimens. The HC_LOR set can thus be selected as having the best characteristics of highest modulus and strength, and lowest variability.

For the comparison of riveted vs. non-riveted composite specimens, the best representative of the riveted composite data sets, HC_LOR, was compared against the non-riveted composite set, HC_NR. There is a clear increase in modulus (38.6%). As for strength, however, the data showed no significant difference, even at 10% level. Thus, it can be concluded that the rivets indeed increase the modulus of the layered composites, but not their strength.

**Pure-resin versus composite parts**

Comparing the best representative of the composite parts, HC_LOR, (fibre volume fraction of 16.0±0.4%) with the combined data for both pure-resin sets (HP_NR and HP_LIR), a modulus increase of 81%, from 1.37±0.12 to 2.48±0.10 GPa, was obtained. For strength, the corresponding increase is 15.2%. The strain at break is reduced, however, with the addition of the fibres, from about 8% to 5%. 
Vertically versus horizontally built parts

Direct comparison of parts with horizontal and vertical orientation is not possible, since building parts with a vertical orientation (i.e., with the layers lying transverse to the testing direction) resulted in a notably reduced fibre content (from 17.0% for HC_SOR to 10.7% for HV_SOR, even though the same fibre content was present in the liquid raw material of both specimen types). However, by linear interpolation between the observed pure-resin-specimen properties and the composite HC_SOR properties, the equivalent values for modulus and strength at 10.7% fibre content for the horizontally built HC_SOR-type specimens are estimated to be 2.06 GPa and 68.6 MPa, respectively. In comparison to these estimated values, the values observed for the vertical specimen were 21% lower for modulus (1.63±0.15 GPa) and 30% lower for strength (47.8±3.8 MPa).

Thus, as expected, a significant reduction in modulus and strength results when parts are built so that the primary loading direction is transverse to the layer planes. This observation confirms the predictions made earlier in Section 6.3.2, which indicated an expected reduction of 35% in modulus and of 10% in strength.

6.4.3 Mechanical property predictions based on the observed fibre-length and fibre-orientation distributions

The mechanical-property prediction method in Section 6.3.2 was applied to predict the modulus and strength values for selected composite specimens. The specimens chosen were those for which the cross-sectional examinations supplied the information about their microstructure in the form of the fibre-length and fibre-orientation distributions. For brevity, the letters “HC” will be dropped from the specimen designations since all the specimens discussed in this section are horizontal and composite.

The constituent component properties used are those listed in Table 6.2 in Section 6.3.2. Experimental fibre content and mechanical property observations for each specimen are summarized in Table 6.10. Table 6.11 shows the predicted modulus values together with the length and orientation correction factors used to predict them.

The predictions and experimental observations are displayed for comparison in Figure 6.18. The error bars are the standard deviations for the corresponding specimen’s set.
Predictions match experimental observations fairly well (within 10%) for the riveted specimens (LOR and SOR). The model also predicts a significant decrease in modulus of the second non-riveted specimen (NR2) (within 20% of observed value). The prediction for the first riveted specimen (NR1), however, significantly deviates from the observed value (it is about 85% higher). The high predicted modulus value is due to a good alignment with the testing direction observed in the sampled cross-section of the specimen. It may be conjectured that this was a localized phenomenon. The particular specimen examined was from the first batch built for the test series. It is possible that some fibre clumping (insufficient fibre dispersion) has occurred, leading to localized volumes of aligned fibres. As the raw material was recycled for subsequent specimens, the mixture would be expected to become more homogeneous.

Table 6.12 shows the predicted strength values as well as the correction factors used to obtain them. Figure 6.19 displays the strength predictions and experimental observations (with the error bars from the corresponding sets). Predictions match observations fairly well for all specimens.

Table 6.10. Observed mechanical properties for the specimens with the collected microstructure data.

<table>
<thead>
<tr>
<th>Specimen Name</th>
<th>ID</th>
<th>Fibre Content (%)</th>
<th>Modulus (GPa)</th>
<th>Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LOR</td>
<td>407Ca</td>
<td>16.4</td>
<td>2.46</td>
<td>72.1</td>
</tr>
<tr>
<td>SOR</td>
<td>403Cb</td>
<td>17.9</td>
<td>2.43</td>
<td>66.9</td>
</tr>
<tr>
<td>NR1</td>
<td>401Cb</td>
<td>16.9</td>
<td>1.66</td>
<td>73.1</td>
</tr>
<tr>
<td>NR2</td>
<td>408Cb</td>
<td>14.7</td>
<td>1.94</td>
<td>70.8</td>
</tr>
</tbody>
</table>

Table 6.11. Predicted tensile modulus.

<table>
<thead>
<tr>
<th>Name</th>
<th>Orientation-Efficiency Factor ($\chi_1$)</th>
<th>Fibre-Length Correction Factor ($\chi_2$)</th>
<th>Product ($\chi_1\chi_2$)</th>
<th>Predicted Modulus (GPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LOR</td>
<td>0.266</td>
<td>0.365</td>
<td>0.0971</td>
<td>2.29</td>
</tr>
<tr>
<td>SOR</td>
<td>0.340</td>
<td>0.291</td>
<td>0.0988</td>
<td>2.40</td>
</tr>
<tr>
<td>NR1</td>
<td>0.313</td>
<td>0.442</td>
<td>0.1383</td>
<td>2.82</td>
</tr>
<tr>
<td>NR2</td>
<td>0.210</td>
<td>0.159</td>
<td>0.0334</td>
<td>1.52</td>
</tr>
</tbody>
</table>
Figure 6.18. Comparison of modulus predictions and observations.

Table 6.12. Predicted tensile strength.

<table>
<thead>
<tr>
<th>Name</th>
<th>Orientation-Efficiency Factor (χ₃)</th>
<th>Fibre-Length Correction Factor (χ₄)</th>
<th>Product (χ₃χ₄)</th>
<th>Predicted Strength (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td>LOR</td>
<td>0.157</td>
<td>0.138</td>
<td>0.0216</td>
<td>64.1</td>
</tr>
<tr>
<td>SOR</td>
<td>0.191</td>
<td>0.105</td>
<td>0.0201</td>
<td>63.4</td>
</tr>
<tr>
<td>NR1</td>
<td>0.183</td>
<td>0.172</td>
<td>0.0315</td>
<td>69.9</td>
</tr>
<tr>
<td>NR2</td>
<td>0.139</td>
<td>0.068</td>
<td>0.0094</td>
<td>57.8</td>
</tr>
</tbody>
</table>

Figure 6.19. Comparison of strength predictions and observations.
6.5 Summary

The chapter provides background information about the mechanics of short-fibre composites. A modified rule of mixtures model is used to predict their properties. This model relies on fibre-length correction factor to account for the effect of short fibre length and orientation-efficiency factor to account for the misalignment of fibres with respect to the loading direction. Both factors are estimated for the three principal directions of the composite test specimens using the fibre-orientation and fibre-length distributions obtained in Chapter 5. The moduli and strengths along the three orthogonal principal directions are predicted and the properties are found to be best aligned in a horizontal (layer) plane, along the direction of wiping.

The chapter then reports on the mechanical test results for the specimens fabricated on the experimental RLCM system. The tests demonstrated that a significant improvement in modulus (80%) is possible with the addition of short reinforcing fibres. This result was achieved when the specimens were built using a "riveted" building style, which itself showed a nearly 40% increase in modulus compared with the "non-riveted" building style for the composite specimens. The test results also confirmed the predictions made earlier in the chapter that the vertical part-building orientation leads to weaker material properties in the testing direction.
Chapter 7. Conclusions and Recommendations

7.1 Conclusions

This thesis has described the development of a novel process for a fully automated, rapid layered composites manufacturing (RLCM). As a raw material, the process uses a composite liquid comprising short glass fibres and a liquid photopolymer resin. The process employs a UV laser for the selective layer-by-layer solidification of the liquid raw material to form composite parts. The principal distinguishing features of this process are:

(1) Separation of the fibre-resin mixing function into an external raw material supply source.

(2) New layer formation by precisely controlled (in terms of location and volume delivered) deposition from above of the composite liquid.

(3) Selective solidification by a scanning pattern with the “rivet” features.

The proposed RLCM process design overcomes the three problems brought about by the introduction of the fibres into the photopolymer liquid:

(1) Difficulty of layer formation due to the high viscosity of the composite liquid.

(2) Fibre settling during the building process.

(3) Lack of inter-layer fibre penetration.

To arrive at the proposed design solution, first, the relevant properties of the raw materials were experimentally investigated; second, the process design was iteratively improved while the process performance was evaluated by analyzing its output (i.e., the fabricated parts).

The material properties of the constituent components were studied primarily to evaluate the interaction between the fibres and the photopolymer. The strength of the interface bond between the glass fibres and the solid photopolymer was measured by the single-fibre pull-out test. The results indicated that a strong bond is formed between the two materials. The information provided by this test was further used in the predictions of the mechanical properties of the finished product.
Interaction of the fibres and the liquid photopolymer was observed through an extensive series of rheological measurements. The experiments assessed the relationship between the composite liquid's viscosity and (a) the fibre concentration and (b) the fibre aspect ratio. Both higher fibre concentration and greater aspect ratios resulted in a significant rise in the liquid's viscosity. While the pure liquid photopolymer was found to have a shear-rate-independent viscosity within the tested range (i.e., it behaved as a Newtonian fluid), the composite liquid displayed pronounced shear-thinning behaviour (i.e., the viscosity decreased significantly with the increasing shear rate, as is characteristic of a non-Newtonian fluid).

Based on these observations, an appropriate fibre concentration and fibre aspect ratio were selected for the raw material. The choice involved maximizing the fibre concentration and aspect ratio while maintaining acceptable viscosity level. The observed shear-rate-viscosity relationship was subsequently employed in the rheological modelling of the layer-formation process.

In designing the RLCM process, Axiomatic Design Theory was employed. The theory states through an Independence Axiom that any proposed design solution expressed by a set of Design Parameters (DPs) must maintain the independence of the Functional Requirements (FRs). The DPs and FRs are identified in a hierarchical sequence, from the highest to the lowest level. The Information Axiom further adds that among the designs satisfying the Independence Axiom, the one with the least information content should be selected. The present design is the result of several cycles of evaluation and modification which took into account the above-stated principles.

To assist with the design improvement process, parts produced by the RLCM process were examined in terms of their geometric quality and mechanical properties. The geometric quality examination concentrated on quantifying the quality of individual layers, since they form the building blocks from which the whole parts are created. Continuous process improvement has been tracked by monitoring three layer quality parameters of the test parts: (1) the average layer thickness, (2) the within-layer variability of the layer thickness, and (3) the between-layer variability of the layer thickness. To interpret certain patterns in layer-to-layer thickness variation, fluid-mechanical layer formation models have been employed.
Modelling mechanical properties of the short-fibre composites requires information about the fibre orientation and length characteristics. Fibre orientation particularly influences the mechanical properties. Fibre alignment along any particular direction raises the modulus and strength along that direction. Uniformly distributed fibre orientations result in isotropic material properties.

The fully three-dimensional Fibre Orientation Distribution (FOD) was measured for the parts built on the RLCM prototype. To accomplish this task, a novel experimental methodology has been developed in this thesis. The approach consists of observing microscopically two closely spaced consecutive cross-sections of the samples (i.e., the “two-section method”). For each cross-section, the parameters of the elliptical fibre intersections with the cutting plane were obtained. The fibres were matched between the cross-sections, allowing accurate and unambiguous estimation of each fibre’s spatial orientation. The method’s novelty lies in finding a way of accurately calculating the transformation parameters relating the two sets of the cross-sectional data.

Additional information derived from the two-section method of specimen examination is an estimate of the average fibre length. As the fibre length affects the mechanical properties, such information is valuable for property predictions. While the length prior to part building can be obtained by direct fibre length measurements, this length is normally reduced due to fibre breakage in processing. Thus, the information about the fibre length within the part itself is of interest. Observations indicated significant reduction in the average fibre length between the fibres prior to part fabrication and the fibres within the test specimens.

Mechanical properties of the layered composite parts are of importance since these parts are intended to serve not only for form-and-fit testing but also as functional prototypes or even small-batch products. The mechanical properties of the short-fibre composite layered parts were modelled in the thesis using a commonly used approach called “modified rule of mixtures.” In this approach, the composite properties are estimated by combining the properties of the constituent components according to the volume fraction of each. The effects of finite fibre length and distribution of fibre orientations are accounted for by multiplication factors modifying the fibre’s contribution. In calculating these factors, the
information from the previously conducted FOD, fibre length, and interface strength measurements was used.

The model predictions were compared and showed good agreement with the tensile properties obtained from the test specimens fabricated on the RLCM prototype system. The tests demonstrated that a significant improvement in modulus (80%) is obtainable by the addition of the short fibres to pure photopolymer. The tests also showed that the proposed "riveted" layer scanning method results in parts with nearly 40% greater modulus when compared with the equivalent parts made using the "non-riveted" (i.e., complete-layer) scanning style. Tensile testing along the direction transverse to the layer planes confirmed the anisotropic nature of the layered composites, with about 20% reduction in modulus and 30% reduction in strength compared with the equivalent in-the-layer-plane properties.

7.2 Recommendations

While this thesis has established the scientific background information and the major elements of the RLCM process, there remain several issues which can be investigated to further improve the process. The recommendations given below are classified by three categories: (1) process/prototype system improvements; (2) further experimental evaluation of the process; and (3) improvements in theoretical modelling related to the process.

7.2.1 Process/prototype system improvements

For any manufacturing process, it is important that the quality of fabrication is not affected by the geometry of the part to be built or that the process parameters can be easily and predictably adjusted to accommodate different part geometries. Several aspects of the RLCM process may need to be improved to achieve such part-geometry independence.

The RLCM process delivers composite liquid by simultaneous translation and deposition technique. To accomplish this task, the pump is activated at the same time as the movement of the nozzle begins. However, there is a delay between the activation of the pump and the time sufficient pressure builds up for the liquid to be expelled from the nozzle. Thus, direct deposition onto small solid areas is not effective, as the deposited material is likely to miss a small target. For example, for the vertically built tensile-test specimens fabricated by the
current RLCM prototype, the experimentally observed ratio between the fibre content of the liquid delivered into the vat \((v_{f1})\) and the fibre content in the narrow waist of the dogbone \((v_{f2})\), \(v_{f2} / v_{f1}\), was only 0.64±0.04. On the other hand, for the horizontally built specimens, which have larger cross-sectional area, this ratio was very close to 1. The low fibre content can be attributed to the small cross-sectional areas of the vertically built parts.

As has been discussed in Chapter 4, the layer thickness is affected by the geometry of the underlying solidified part areas and the depth of the uncured liquid within the vat. Adjustment of the wiping speed can be used to control the final layer thickness. Thus, it would be desirable to implement accurate control of the wiping speed, so that the speed can be adjusted from layer to layer and even during each wiping stroke.

### 7.2.2 Process scale-up

The current prototype system, which served as a testbed for the process development, can build only relatively small parts, being limited by its workspace of 93×93×70 mm \((L\times W\times H)\). The limited workspace volume facilitated experimentation with different fibre-resin combinations while minimizing the amount of raw material required for each build. The commercial utility of the RLCM process would be significantly extended by increasing the workspace volume. However, there are several issues which would have to be addressed in order to scale up the process successfully.

First, increase of the workspace volume would require correspondingly larger supply of the raw material. The fibre-resin mixing subsystem, which supplies the raw material, keeps the fibres in suspension by agitation of a single batch. For completely automated system operation, it would be desirable to avoid manual refilling of the mixing container during the part building. Thus, the size of the mixing container would have to increase with the workspace volume. Increasing the mixing container dimensions may require adding extra agitators, changing the impeller size, and increasing its rotation speed.

Second, the RLCM process requires generation of the deposition path trajectory for delivering the composite liquid directly onto the previously solidified layer surfaces. Currently, the trajectory is a single line bisecting each contour's bounding box along its longest dimension. Such an approach will suit only narrow (on the order of 0.5-2 cm) and
elargated shapes (such as the tensile test dogbone-shaped specimens). To make this stage of the process perform correctly for a wide range of part shapes and larger part sizes, a more generic approach to deposition path generation must be developed.

Third, the area to be covered by the deposition and by the laser-scanning will increase with a greater workspace volume. This increase would lead to a longer time delay between the deposition and the curing of the composite liquid. As the fibres continuously settle until the liquid is cured, the effect of a greater delay would have to be assessed by observation of the part cross-sections and mechanical testing. If found to be significant, possible ways of reducing the delay are by increasing the speed of the deposition-nozzle translation and by increasing the laser scanning speed. These measures may be also required to decrease the part building times, which may become impractically long if the currently used deposition and scanning speeds are employed for building of larger parts.

However, to increase the laser scanning speed while achieving the required exposure of the photopolymer requires corresponding increase in the laser power. Given sufficient laser power, higher scanning rates can be achieved by using galvanometrically actuated mirrors, as opposed to the X-Y translation table currently used.

7.2.3 Further experimental evaluation of the RLCM process

As mentioned above, the manufacturing process should not be affected by the product's geometry (within the process limits). Experimental studies should be conducted to verify that this is the case for the RLCM process. For example, the effects of the part size and horizontal aspect ratio on the fibre content and layer profile shape need to be examined. If there exists a dependence on these factors, either the process should be improved to eliminate the effect or automated adjustments should be implemented to account for the part geometry.

When depositing new-layer material, the volume for the area deposition and the flow rate per unit distance travelled for the direct deposition have been determined by trial-and-error. It was found that different deposition volumes were required for pure and composite liquids to produce parts with correct layer thickness. Thus, it would be desirable to investigate and to quantify the effect that the deposition volume has on the fabricated parts.
Microscopic cross-sectional examinations of the composite parts produced by RLCM can provide important information regarding the orientation and length of the short reinforcing fibres. Both parameters can be affected by the fabrication process. For example, the way the fibres are handled during mixing and deposition can affect the degree of fibre breakage, and thus the average length. The wiping process can affect the fibre orientation. In this thesis, a methodology has been developed to evaluate the fibre orientation and length characteristics of the short-fibre layered composites. However, it would be desirable to speed up the current process of data acquisition so that more samples can be collected, and thus the process could be evaluated more frequently and accurately. To speed up data collection, the process should be automated. Specifically, the tasks which are the most time-consuming, and thus would benefit the most from automation, are the image processing for identifying the fibre elliptical cross-sections and the section-to-section matching of ellipses required to implement the “two-section” method (Chapter 5).

7.2.4 Theoretical modelling related to the RLCM process

Several simplifying assumptions have been made in this thesis when modeling the liquid layer formation by wiping. For example, the channel length under the wiper blade is not sufficiently long to fully form the flow velocity profile of the assumed steady-state conditions. The liquid “bulge” accumulating in front of the wiper as it traverses will change in height. Also, the bulge height will depend on whether the wiper traverses over a solid substrate or over the liquid area. For better understanding of this important part of the RLCM process, it would be desirable to develop a more accurate model of the liquid layer formation by wiping. Such model should represent the transient behaviour of the system and take into account the wiper shape.

A commonly used, but relatively simple, model of the mechanical properties of short-fibre composites was used in this thesis. In particular, simplifying assumptions were made when accounting for the effect on the strength of the fibre orientation and length distributions. A more complex model, such as generalized Mori-Tanaka mean-field approach (see Chapter 6), may offer a more accurate prediction of the short-fibre composite properties.
Appendix A: Bias in Misalignment-Angle Estimates

In order to find the expected error in the fibre misalignment angle \( \phi \), which is calculated as the inverse cosine of the observed minor to major radii ratio, let

\[
A = A_0 + \varepsilon_A \quad \text{and} \quad B = B_0 + \varepsilon_B
\]

where \( A_0 \) and \( B_0 \) are the respective true values of the ellipse major and minor radii, \( A \) and \( B \) are the observed radii, and \( \varepsilon_A \) and \( \varepsilon_B \) are respectively the random variables, \( N(0,\sigma_A) \) and \( N(0,\sigma_B) \), representing the measurement noise. Also, let \( B_0 = A_0 - \Delta R \), where \( \Delta R \geq 0 \) and \( \Delta R \ll A_0 \).

Then, the true misalignment angle, \( \phi_0 \), is given by

\[
\phi_0 = \cos^{-1}(B_0/A_0)
\]

In case of a nearly circular ellipse, \( \phi_0 \rightarrow 0 \), and therefore one can use the Taylor series expansion to approximate \( \cos(\phi) \). Considering the first two terms only:

\[
\cos(\phi_0) = \frac{B_0}{A_0} \approx 1 - \frac{\phi_0^2}{2}.
\]

Rearranging the above:

\[
\phi_0^2 = 2 \left(1 - \frac{B_0}{A_0}\right) = 2 \frac{\Delta R}{A_0}.
\]

Since one cannot distinguish \textit{a priori} the minor radius from the major radius, the misalignment angle is calculated by taking the ratio of the smaller over the larger of the two radii,

\[
\phi_A = \cos^{-1}(B/A) \quad \text{for} \quad B \leq A \quad \text{and}
\]

\[
\phi_B = \cos^{-1}(A/B) \quad \text{for} \quad B > A.
\]

For \( B \leq A \):

\[
\phi_A^2 = 2 \left(1 - \frac{B_0 + \varepsilon_B}{A_0 + \varepsilon_A}\right).
\]

Assuming \( \varepsilon_A \ll A_0 \) and \( \varepsilon_B \ll B_0 \), and using the Taylor series expansion for \((1+\delta)^{-1}\), where \( \delta \rightarrow 0 \), (A.6) becomes:
Substituting the above result back into (A.6):

\[
\phi_A^2 = 2\left(1 - \frac{A}{A_0} + \frac{A}{A_0} \frac{B_0}{A_0} - \frac{B_0}{A_0}\right) = 2\left(1 - \frac{A}{A_0} - \Delta R + \frac{A}{A_0} \frac{B_0}{A_0} - \frac{B_0}{A_0}\right).
\]  

The above is further approximated as:

\[
\phi_A^2 = 2\left(\frac{\Delta R + \frac{A}{A_0} - \frac{B_0}{A_0}}{A_0}\right) = \frac{2}{A_0} \left[\varepsilon_A - (\varepsilon_B - \Delta R)\right].
\]  

By similar arguments, for \(B > A\):

\[
\phi_B^2 = \frac{2}{B_0} \left[(\varepsilon_B - \Delta R) - \varepsilon_A\right].
\]

Expressions (A.9) and (A.10) are approximately equal since \(A_0 = B_0\) and the bracketed terms only differ by a sign. Thus, squaring the right-hand side of both produces equivalent expressions. Then, to evaluate \(\phi\) for both cases (\(B \leq A\) and \(B > A\)):

\[
\phi^4 = \left(\frac{2}{A_0} \left[\varepsilon_A - (\varepsilon_B - \Delta R)\right]\right)^2.
\]

Calculating the expected value of the term \(\left[\varepsilon_A - (\varepsilon_B - \Delta R)\right]^2\):

\[
E\left[\left(\varepsilon_A - (\varepsilon_B - \Delta R)\right)^2\right] = \Delta R^2 + 2\Delta R[E(\varepsilon_A) - E(\varepsilon_A)] + E(\varepsilon_A^2) + E(\varepsilon_B^2) - 2E(\varepsilon_A\varepsilon_B)
\]

\[
= \Delta R^2 + \sigma_A^2 + \sigma_B^2,
\]

where the two variables are assumed to be independent and thus \(E(\varepsilon_A\varepsilon_B) = \text{cov}(\varepsilon_A, \varepsilon_B) = 0\).

Assuming \(E(\phi) = (E(\phi^4))^{1/4}\),

\[
E(\phi) = \left[\frac{4}{A_0^2} \left(\Delta R^2 + \sigma_A^2 + \sigma_B^2\right)\right]^{1/4}.
\]

Note that the above is not equal to \(\phi_0\) in (A.4), i.e., the ellipse-based misalignment angle estimate is biased.
Appendix B: Simulation of the Error Propagation in the Fibre-Orientation Estimates for the Single-Section Method

Numerical simulations were performed to observe the effect of random measurement errors in the position of individual ellipse curve points on the ellipse-based fibre orientation, i.e. on the azimuth angle $\theta$ and the misalignment angle $\phi$. The simulations consisted of the following steps:

1. Generate 8 points lying on an ellipse;
2. Add normally distributed, zero-mean random errors with the standard deviation $\sigma_p$ to the $X$ and $Y$ point coordinates;
3. Fit an ellipse to the noisy data to get $\theta$, $A$, and $B$, which are, respectively, angle of the ellipse major axis, the major and the minor radii; and,
4. Calculate $\phi = \text{acos}(B/A)$.

Two cases were considered: an exact-circle case, where the true angle of $\phi$ is zero, and a near-circle case, where the true $\phi$ is 20 degrees. In each case, the means and standard deviations of $\phi$ and $\theta$ estimates were recorded for a range of $\sigma_p$ from 0.1 to 0.5 image pixel units. The actual measurement error is expected to be on the order of 0.5 pixels.

Case 1: Exact circle

Figure B.1 shows the dependence of the mean and standard deviation of 500 simulated angle $\phi$ estimates on the point position measurement noise, $\sigma_p$. The simulations were based on the true parameter values of $\theta = 0$, $A = 10$, $B=10$, $\phi = 0$. Also plotted (as the “Model mean”) are the predictions of the expected value of $\phi$ based on the expression in (A.13). Good agreement between the model and the simulated data is obtained, considering the approximations involved in deriving the relationship.

Representative histograms for $\sigma_p = 0.5$ are displayed in Figure B.2 for $\theta$ and $\phi$. Note the uniform $\theta$ distribution. This is expected as the $(X, Y)$ errors result in random rotation of the ellipse major axis. The angle $\phi$ appears to be normally distributed over the range of 10 to 30 degrees.
Figure B.1. Variation of $\phi$ mean and standard deviation as a function of measurement error standard deviation (Case 1).

Figure B.2. Histograms of $\theta$ and $\phi$ (Case 1).

**Case 2: Near circle**

This case shows how the $\phi$ estimation bias obscures the true variation in the angle. The only difference between this case and Case 1 is that the true $\phi$ is 20° instead of zero. Comparing Figure B.3 for Case 2 with Figure B.1 for Case 1 shows that, as the measurement noise decreases, the estimate mean approaches its true value of 20 degrees, as expected. Standard deviations of estimates have similar relationships for both cases.
Representative histograms for $\sigma_\theta = 0.5$ are displayed in Figure B.4 for $\theta$ and $\phi$. Note that the $\theta$ histogram shows a normally distributed data, unlike Case 1 for the exact circle. Also note that the $\phi$ histograms are very similar for Case 1 and 2, making it impossible to distinguish between a fibre oriented at a true angle $\phi=0$ and $\phi=20$ degrees based solely on the single-section data.

Figure B.3. Variation of $\phi$ mean and standard deviation as a function of measurement error standard deviation (Case 2).

Figure B.4. Histograms of $\theta$ and $\phi$ (Case 2).
Appendix C: Fibre-Data-Collection Procedure

Following are detailed instructions on collecting the fibre orientation data and deriving from it a fibre orientation distribution. The programs mentioned below are to be executed, unless otherwise specified, in the MATLAB® Version 4.0 environment.

1. Polish specimen section.
2. Acquire images (.tif format).
3. Determine image-to-world scaling by measuring distances on the fibre sections between identifiable features using a microscope.
4. Measure same feature dimensions on the image (in pixels).
5. Determine scaling factors based on the above measurements.
6. Convert acquired images from .tif to .raw format required by MATLAB® Version 4.0. From the DOS command prompt, type: alchlong *.tif OutputPath\ -r. The program “alchlong” is an image conversion utility available from www.handmadesw.com. A free demo limited to images of up to 640x480 pixels is available. Produce file names like 407a093.raw, 407a094.raw etc.
7. Collect fibre orientation data from a set of images for one specimen by manually tracing each ellipse border (run "fibmeas"). Produce fibre data files, e.g., f407a093.txt, and marked image files, e.g., f407a093.tif.
8. Collect layer boundary curve data from each image (to be used for display purposes and for image-to-image orientation adjustment for paired section images). (Run "get_crv"). Produce files such as c407a093.txt.
9. Identify matching fibres between adjacent images in order to obtain image-to-image transformations. Produce files such as f1407a.m which contain IDs of the matching fibres.
10. Combine fibre-orientation data from individual images into a single data file and rescale the data from image to world coordinates (run "fdatcomb"). Produce files such as fr407a.txt.
(11) Combine individual layer boundary curve data into a single file (run "crvcomb").
    Produce files such as: `c407a.txt`.

(12) Identify matching pairs of fibres between Section 1 and 2 (use "fibmmtch"). As
    input, use two files, one from each section, e.g., `fr407a1.txt` and
    `fr407a2.txt`. Also, use a file that lists matching image pairs, e.g.,
    `mi407a.txt`. Produce files such as `mf407a.txt` - a list of matching fibre pairs.

(13) Estimate parameters for section-to-section transformation \((x, y, \Delta d)\) (use
    "fibmmtch") Produce files such as `st407a.txt` with a set of section-to-section
    transformation parameters, one for each image pair.

(14) Calculate the fibre orientations for the matched fibres based on the two-section data.
    (use "fibmmtch"). Produce files such as `ft407a.txt`, which contain the modified
    fibre data set for all section 1 images combined with the two-section data.

(15) Obtain fibre orientation distribution (use "f_dist"). Produce files such as
    `fod407a.txt` which contain the probabilities for each bin and the bin ranges.
Appendix D: Estimation of Ellipse Parameters

**General case**

Ellipse fitting is a frequent problem encountered in image processing applications (Safaee-Rad, 19??). An ellipse can be described by:

$$ax^2 + bxy + cy^2 + dx + ey + 1 = 0.$$  \hfill (D.1)

To estimate the five parameters of the ellipse equation, one can use the least-squares fit that minimises the function

$$Q(a, b, c, d, e) = (ax^2 + bxy + cy^2 + dx + ey + 1)^2.$$ \hfill (D.2)

The least-squares solution leads to a set of five normal equations solved by matrix inversion:

$$\begin{bmatrix}
\sum x^4 & \sum x^3y & \sum x^2y^2 & \sum x^3 & \sum x^2y \\
\sum x^3y & \sum x^2y^2 & \sum xy^3 & \sum x^2 & \sum xy \\
\sum x^2y^2 & \sum xy^3 & \sum y^4 & \sum xy^2 & \sum y^3 \\
\sum x^3 & \sum x^2y^2 & \sum xy^3 & \sum x^2 & \sum xy \\
\sum x^2y & \sum xy^2 & \sum y^3 & \sum xy & \sum y^2 \\
\end{bmatrix}
\begin{bmatrix}
a \\ b \\ c \\ d \\ e \\
\end{bmatrix}
= \begin{bmatrix}
-\sum x^2 \\
-\sum xy \\
-\sum y^2 \\
-\sum x \\
-\sum x \\
\end{bmatrix}, \hfill (D.3)

where \((x, y)\) are the ellipse-boundary points.

To obtain the desired ellipse parameters \((x_0, y_0, \theta, A, B)\) from the five coefficients of Equation (D.1):

$$x_0 = \frac{2cd - be}{b^2 - 4ac}, \quad y_0 = \frac{2ae - bd}{b^2 - 4ac}, \quad \theta = \tan^{-1} \left( \frac{-b}{c - a + \sqrt{(c-a)^2 + b^2}} \right),$$

$$A = \sqrt{\frac{2(1-F_s)(c + a + \sqrt{(c-a)^2 + b^2})}{b^2 - 4ac}}, \quad B = \sqrt{\frac{2(1-F_s)(c + a - \sqrt{(c-a)^2 + b^2})}{b^2 - 4ac}}, \hfill (D.4)$$

where \(F_s = \frac{bde - ae^2 - cd^2}{b^2 - 4ac}\).

To improve the numerical conditioning of the equations in (D.3), the ellipse boundary-point data is transformed in the following manner:

$$\begin{bmatrix} x' \\ y' \end{bmatrix} = \frac{1}{K_s} \begin{bmatrix} x \\ y \end{bmatrix} - \begin{bmatrix} \bar{x} \\ \bar{y} \end{bmatrix}, \hfill (D.5)$$
where \((\bar{x}, \bar{y})\) are the averages of the original data coordinates and \(K_s\) is a scale factor which makes the parameters \(A\) and \(B\) of approximately same magnitude as the angle \(\theta\) expressed in radians. After solving the equations with the transformed data, the correct parameter values are recovered as:

\[
\begin{bmatrix}
  x_0 \\
  y_0
\end{bmatrix} = K_s \begin{bmatrix}
  x' \\
  y'
\end{bmatrix} + \begin{bmatrix}
  x \\
  y
\end{bmatrix} = K_s \begin{bmatrix}
  A' \\
  B'
\end{bmatrix} \quad \theta = \theta'.
\] (D.6)

The above transformation reduces by several orders of magnitude the condition number of the linear system in (D.3). For example, for a set of parameters \([x_0=400, y_0=200, \theta=90, A=12, B=10]\), the condition number is reduced from \(4.12 \times 10^{12}\) to 5.00.

**Robust ellipse parameter estimation for nearly perpendicular fibres**

**Algorithm**

The problem may be stated as: "Reliably estimate curve parameters for highly elongated ellipses based on incomplete boundaries and noisy boundary points." The above situation occurs when a section plane intersects a fibre nearly parallel to it. If a complete ellipse outline is available, the standard estimation procedure described in the preceding section can be used. However, in most cases, the plane intersects one or both of the fibre's ends, resulting in an incomplete outline (see cases A and C in Figure 5.12). In such cases, using the standard parameter-estimation procedure leads to a very high sensitivity to measurement noise, frequently producing imaginary parameter estimates. In the following development, several approximating assumptions are made to achieve reliable estimation of the ellipse parameters in such extreme cases.

First, let us assume that the ellipse boundary points will be located in a coordinate system \(t-w\). This system is centred at one end of the ellipse major axis, with the \(t\)-axis aligned along the major axis and the \(w\)-axis aligned along the minor axis (Figure D.1). Thus, by definition, the ellipse centre is located in these coordinates at \((A, 0)\). Such an ellipse can be described by an equation:

\[
\frac{(t-A)^2}{A^2} + \frac{w^2}{B^2} = 1,
\] (D.7)
where $A$ and $B$ are the major and minor radii, respectively. Rearranging this equation and normalizing $w$ as $v = w/B$ produces:

$$v^2A^2 - 2tA + t^2 = 0.$$ \hspace{1cm} (D.8)

To proceed with the derivation, it is assumed that the minor radius $B$ is known. This parameter is equal to the fibre radius and can be obtained from the average of the minor radii of other ellipse observations. Then, (D.8) can be solved as a quadratic equation, with $A$ as an unknown:

$$A = \frac{t}{v^2} \left( 1 \pm \sqrt{1 - v^2} \right).$$ \hspace{1cm} (D.9)

A negative square root in the above equation corresponds to the boundary points such that $t > A$. It is assumed that only points with $t < A$ are used and thus positive roots will be chosen when estimating $A$. Equation (D.8) provides an estimate of $A$ for every pair of ellipse boundary coordinates ($t_i$, $v_i$). Using $n_p$ boundary points, an estimate of $A$ is obtained by averaging as follows:

$$\overline{A} = \frac{1}{n_p} \sum_{i=1}^{n_p} t_i \left( 1 + \sqrt{1 - v_i^2} \right).$$ \hspace{1cm} (D.10)

Figure D.1. Nomenclature of parameter estimation for elongated ellipses.
Boundary point collection

To acquire the ellipse boundary points in this special case, the operator is asked to select \(2n_p + 1\) points in the specific sequence shown in Figure D.1. The data consist of \(n_p\) point pairs plus a single point marking an approximate location of the ellipse major axis end-point.

The points must be selected such that the line segment connecting points in each pair is perpendicular to the \(t\)-axis. The \(t\)-axis, lying along vector \(e\), is then found as the direction from the last point picked to a point midway between the first two points:

\[
\varepsilon = \frac{1}{2}(\mathbf{p}_1 + \mathbf{p}_2) - \mathbf{p}_{2n_p+1}\cdot
\]

The ellipse boundary points in the \(t\)-\(w\) coordinates are found by:

\[
w_i = \frac{1}{2}\left|\mathbf{p}_{2i} - \mathbf{p}_{2i-1}\right| \quad \text{and} \quad t_i = \frac{1}{2}\left((\mathbf{p}_{2i-1} + \mathbf{p}_{2i}) - \mathbf{p}_{2n_p+1}\right), \quad \text{where} \quad i = 1, \ldots, n_p.
\]

Only the points with \(w_i < B\) are used in estimating \(A\); others are discarded.

Ellipse centre is located as:

\[
\begin{bmatrix} x_0 \\ y_0 \end{bmatrix} = \mathbf{p}_{2n_p+1} + A\frac{\varepsilon}{|e|}.
\]

Finally, the major axis rotation is determined by:

\[
\theta = \tan^{-1}\left(\frac{e_y}{e_x}\right).
\]

Numerical simulations have shown that the above method produces reliable and accurate estimates of \(A\) in the presence of artificially added measurement noise. The method has also proven itself reliable with actual image data.
Appendix E: Simulation of the Estimation Process for Section-to-Section Transformation Parameters

Objective

The simulation described herein is used to verify the correct functioning of the estimation algorithm and to assess its ability for accurate estimation of the required parameters, given the noisy nature of the fibre orientation data.

Method

The simulation comprised the following steps:

1. Generate 25 fibre data sets with varying orientations from Section 1;
2. Generate corresponding Section 2 data sets by shifting the Section 1 data according to $x_n$, $y_n$, and $\Delta d$ parameters;
3. Generate eight equally distributed boundary points for each ellipse in both sections;
4. Add normally distributed zero-mean noise to the coordinates of each point;
5. Calculate the ellipse parameters from the above boundary points and obtain the misalignment angle $\phi$ from these parameters; and,
6. Estimate the section-to-section transformation parameters based on the two-section simulated data.

A set of 25 ellipse data sets representing fibre cross-sections were generated with their centres lying on a grid, with overall dimensions of 300 by 200 (X×Y). The ellipses were further specified by giving the fibre azimuth angle ($\theta$) and the misalignment angle ($\phi$), and the minor diameter (equal to the fibre diameter) of 16. The $\theta$ angles were distributed evenly within the range [-175 175] degrees. Two cases of $\phi$ angle distributions were looked at: [0 60] and [30 60].

Within each angle distribution, 25 values were sampled. One value of $\theta$ and one of $\phi$ for each ellipse were then randomly picked for each of the 25 ellipses. Once picked, the value was not used for the other ellipses.
Having defined the ellipse parameters, two sets of data are assembled: one for Section 1 and another for Section 2, which lies at a depth $\Delta d$ further into the specimen. The only difference between them is the location of the ellipse centres. Given the ellipse centres of Section 1 image, $(x_{o}^{(1)}, y_{o}^{(1)})$, the Section 2 image ellipse centres are then calculated by first expressing their centre positions in Section 1 frame (Figure 5.6):

$$
x_{o}^{(21)} = x_{o}^{(1)} + \Delta d \tan \phi \cos \theta \quad y_{o}^{(21)} = y_{o}^{(1)} + \Delta d \tan \phi \sin \theta.
$$

(E.1)

Next, the above points are translated to section 2 frame:

$$
x_{o}^{(2)} = x_{o}^{(21)} - x, \quad y_{o}^{(2)} = y_{o}^{(21)} - y,
$$

(E.2)

Thus, the Section 2 ellipses are described by the centre coordinates $(x_{o}^{(2)}, y_{o}^{(2)})$ and the same fibre orientation angles and ellipse diameters as the Section 1 data.

For each ellipse in the above two data sets, eight points equally distributed along the ellipse boundary were generated and a random, normally distributed error was added to the X and Y coordinates of these points. A minimum of five points is required to define an ellipse. Typically, 8-12 are used when acquiring data from images.

Next, the algorithm used on the image data is applied here to estimate the five ellipse parameters from the artificially generated noisy data. Simulated ellipse-parameter-data sets for Sections 1 and 2 are produced in the above manner. These two sets are then used to estimate the transformation parameters between the two sections.

As the ellipse boundary points are randomly generated, each simulation run produces different values for these parameter estimates. To evaluate the reliability of the estimates, the simulation was rerun ten times for each setting of the simulation parameters.

**Results**

First, to verify the correct recovery of the section-to-section transformation parameters from the simulated data, the simulation was run with no error added and $0^\circ < \theta < 60^\circ$. Figure E.1 shows the ellipses generated. Lighter-shaded ellipses belong to the Section 2 and the darker-shaded ones to the Section 1. Their positions correspond to $(x_{o}^{(1)}, y_{o}^{(1)})$ values for Section 1 and $(x_{o}^{(2)}, y_{o}^{(2)})$ values for Section 2. In other words, these are the ellipse centre positions observed from the images, expressed in each image's frame.
The first step, which uses the linear regression, is represented in Figure E.2. The plot shows $\Delta \bar{x}$ plotted against $c_{\theta \phi}$ parameter. Note that the actual data obtained can only produce positive $c_{\theta \phi}$ values. However, to implement the linear fit, the data below the inflection point are reflected about the Y axis. The reflected data points correspond to fibre orientations with $-180^\circ < \theta < -90^\circ$ and $90^\circ < \theta < 180^\circ$. For these orientations, $\Delta x = x^{(2)}_\phi - x^{(1)}_\phi$, is negative. Circles over the "x" marks designate these points on Figure E.2.

Since one does not know a priori which points have negative $\Delta x$, the data points are analyzed, and a value of $\Delta \bar{x}$ is determined below which the $c_{\theta \phi}$ values are reflected. This process produces exact results in the absence of noise. Thus, for the noise-free data, the section-to-section transformation parameters were recovered exactly.

Next, two simulation cases were run with noise standard deviation (SD) set to 0.5, one with $0^\circ < \phi < 30^\circ$ and a second one with $30^\circ < \phi < 60^\circ$. The intention was to compare the accuracy of the estimates between the two cases. Figure E.3 shows the linear regression data from one of the runs for each case. Points below the horizontal dashed line where reflected about the Y axis. Since this is a simulation, it is known which points were supposed to be reflected. To verify the correct operation of the algorithm, these are marked on the figure by circles. Only four of these marked points are not reflected for first case plot. Their effect should be minimal as they lie very close to the fitted line.

Table E.1 shows the simulation results for both cases. In the first case, while for the first two parameters the error of estimate mean from the actual value is close to the sample SD, the estimate error for $\Delta \bar{d}$ is significantly greater than the SD (3.6 compared to 0.55). The estimates are also consistently lower than the actual $\Delta \bar{d}$ value. The second case results, with $30^\circ < \phi < 60^\circ$, are clearly superior to those of the first case. The SD of all parameters is reduced by about a factor of two and the absolute error of the estimate mean is smaller as well, especially for $\Delta \bar{d}$ (0.13 vs. 3.6).
Table E.1. Simulation results (Noise SD = 0.5, all units are pixels).

<table>
<thead>
<tr>
<th>Run #</th>
<th>$x_1$</th>
<th>$y_1$</th>
<th>$\Delta d$</th>
</tr>
</thead>
<tbody>
<tr>
<td>1</td>
<td>-9.76</td>
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Mean: -9.38, 19.91, 6.43
SD: 0.33, 0.37, 0.55
Error: 0.62, -0.09, -3.57

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Mean: -9.96, 19.98, 9.87
SD: 0.17, 0.15, 0.21
Error: 0.04, -0.02, -0.13

Act. Val: -10, 20, 10

Figure E.1. Ellipses used in generation of the simulated fibre orientation data.
Figure E.2. Linear regression used to estimate $x_r$ and $\Delta d$ parameters (noise-free data): (a) data prior to reflection; (b) data after reflection and linear fit. Circles indicate those points known to have negative $\Delta x$ values.

Figure E.3. Linear regression used to estimate $x_r$ and $\Delta d$ parameters (data with noise added): (a) $0<\phi<30$; (b) $30<\phi<60$. Linear fit shown by a straight line. Circles indicate those points known to have negative $\Delta x$ values. Estimated parameters: (a) $x_r = -9.21$, $y_r = 19.47$, $\Delta d = 6.90$; (b) $x_r = -10.04$, $y_r = 19.75$, $\Delta d = 10.19$.

Conclusions

Since the estimate reliability decreases notably for fibres with $0<\phi<30^\circ$, these points should not be used for the estimation. Only the fibres with $\phi>30^\circ$ should be selected to obtain the section-to-section transformation parameters.
Appendix F: Fibre Diameter Observations

The minor diameter of any fibre cross-section ellipse is equal to the fibre diameter itself. Figure F.1 shows distribution of the fibre diameters observed for two specimen cross-sections. One can note good consistency between the two sets of observations. The diameters appear to be normally distributed. The overall mean and standard deviation are 16.4 μm and 1.7 μm, respectively.

Figure F.1. Histograms of fibre diameters obtained from the fibre ellipse minor axis measurements: (a) mean dia = 16.5 micrometers, SD = 1.76 and (b) 16.2 micrometers, SD = 1.73.
References


Adamson, A. W., 1976, Physical Chemistry of Surfaces, 3rd edition, Wiley, N.Y.


References
References


Owens-Corning Fibreglas Corp., 1994, Product Data Sheet for 408BC Chopped Strands.


