MECHANICAL ANISOTROPIES IN JETTED PHOTOPOLYMER STRUCTURES

by

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A thesis submitted in conformity with the requirements for the degree of Master of Applied Science
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University of Toronto

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Abstract

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Material jetting is a type of additive manufacturing that 3-D prints with a photopolymer resin through a series of small jets in a layer-by-layer process. Resulting structures were found to be anisotropic in nature with regard to print orientation due to differences in levels of bonding between successive print layers. The mechanical property anisotropies were quantified at both micro and macro length scales through nano- and micro- indentation and uniaxial tensile testing. The dependence and trends in anisotropy were found to be linked to the size scale of the testing method relative to the layer thickness. Under tensile testing, going from sample print orientations of 0° to 90°, approximately 20% drops in modulus and strength, and a 75% drop in ductility were seen. The effect of how these anisotropies are then exemplified in more complex architectures, such as a microtruss, was investigated and found to correlate with the tensile results.
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Chapter 1

Introduction

1.1 Motivation

Additive manufacturing is a process for producing physical products from a 3D model through the repeated joining of multiple layers to build a final part. As opposed to subtractive manufacturing, where material is successively removed to form a final part, additive manufacturing offers the advantage of being able to form parts of high complexity and with internal structure with near unlimited freedom as it is not limited by the ability of tooling to reach unwanted material [1, 2]. There are various types of systems that can be used in additive manufacturing ranging from solid-based (e.g. fused deposition modelling), powder-based (e.g. selective layer sintering), and liquid-based (e.g. stereolithography). These all work on the same basic concept of converting a 3D model into a series of layers and passing this information along to a machine which will deposit the printing material layer by layer while joining them using different techniques as required based on the method used.

Additive manufacturing has developed rapidly since its infancy when it was originally used as a technology for rapid prototyping, to its adoption for use in industry for component manufacturing [2–4]. In 2013, the aerospace industry utilized over 22,000 parts fabricated using additive manufacturing technologies [5]. The Boeing 787 Dreamliner is one example in the
aerospace industry of a commercial airliner which utilizes components fabricated using additive manufacturing, with approximately 30 printed parts used in its design [3]. Component part production for the aerospace and automotive industry combined to account for over 20% of the additive manufacturing market [5]. According to industry data published in Wohlers 3-D Printing and Additive Manufacturing State of the Industry Annual Worldwide Progress Report in 2016, the worldwide revenue from additive manufacturing products in 2015 was over $5.1 billion and forecast to surpass $8.8 billion by the end of 2017 [6]. The number of manufacturers which sold industrial-grade additive manufacturing systems (machines valued over $5,000) has doubled between 2011 and 2015 from 31 to 62 [6]. Not only has their been growth in industrial applications, the expiry of key additive manufacturing patents has also led to massive growth in the consumer sector as well, as costs have decreased [2, 5, 6]. Although the first major patent for stereolithography expired in 2004, it was not until after the expiry of the first fused deposition modelling patent in 2009 that the additive manufacturing industry took off [2]. This is due to the differences between the two technologies, most notably the technical complexities of the systems used in stereolithography compared to fused deposition modelling [2]. The average growth in the number of desktop class 3-D printers (machines under $5,000) sold worldwide per year have grown an average of 87.3% a year between 2012 to 2015 with increases in 2014 by 88% to 163,999 machines and 69.7% in 2015 to 278,385 machines (see Figure 1.1) [6].

One place where additive manufacturing is particularly appealing is in the development and design of lightweight hybrid materials. Figure 1.2 show the strength-density landscape of currently available materials. Lightweight hybrids (designed especially for the aerospace sector) would fill the upper left hand region of the diagram [7–10]. However, the manufacturing of these materials through conventional means is either not possible or prohibitively expensive [8, 11]. Additive manufacturing presents an opportunity to utilize its architectural freedom to design topologically optimized structural materials [7, 8, 12].

One of these optimized structural materials is the microtruss, a periodic cellular structural
Chapter 1. Introduction

Figure 1.1: The number of desktop class 3-D printers (machines under $5,000) sold per year for 2007 to 2015 [6].

hybrid of solid material and open space. These microtrusses, as a result of its stretch-dominated structures result in improved mechanical performance per a given weight compared to bending-dominated structures such as foams [14, 15]. Some examples of these hybrid are shown in Figure 1.3. Although additive manufacturing provides a method capable of fabricating these complex microtruss architectures, there are additional concerns that must be accounted for. Print orientation plays a large role in the fabrication of parts via additive manufacturing, with the effect of print orientation varying for the different types of printing processes. The print orientation will determine the cost of base and support material required, the time it takes to build, and most importantly the mechanical behaviour of the final structure. Due to joining between layers required in the manufacturing process, the bonding between layers will have reduced performance compared to the bulk material; this reduction as well, is dependent on the technology employed [16]. The bonding of layers in additive manufacturing is what leads to the inherent anisotropy of these parts. In order to build functional parts for any sort of load bearing application, be it for prototyping a design for testing or for building advanced architecture structures such as a microtruss, a solid understanding of the base material’s behaviour in
Figure 1.2: Ashby material selection chart for strength and density for current classes of materials. The void towards the top-left corner indicates a current void in the materials design space for high strength but low density materials [13].
 mechanical properties as it relates to print orientation is required. If these factors are not taken into consideration, unexpected failure of a part may occur as properties are overestimated. As the design and architecture of these structures are optimized for their particular application, different levels of anisotropy will be present for a given structure. Without a full understanding of the anisotropies present, any errors in the assumptions made about the base material will be compounded as the design complexity increases.

1.2 Thesis Objectives and Outline

The experiments in this thesis all utilize parts fabricated via material jetting by a 3D Systems ProJet HD3500 3-D Printer using VisiJet M3 Crystal and VisiJet S300 as the photopolymer part and liquid wax support material respectively as a basis for mechanical testing. The ProJet HD3500 is capable of printing at 750 dots per inch in the $x$-$y$ layer plane and 890 dots per inch in the vertical $z$ direction forming parts with 29 $\mu$m layers [18]. A scanning electron microscope (SEM) micrograph of one of the notched tensile coupon printed using the ProJet
Figure 1.4: A SEM micrograph of a notched tensile coupon printed by material jetting at a 45° angle. The lines running across the tensile coupon at a 45° angle is a clear indication of the layered structure. A schematic is shown to the left indicating the layering direction relative to the tensile coupon. The wax support material required to print the part is shown in blue.

HD3500 is shown in Figure 1.4. This sample was printed at a 45° angle relative to its length and the layered structure of the material jetting process is clearly visible in this micrograph. This layering acts as a potential site of mechanical inhomogeneity and anisotropy and is a concern that must be taken into consideration in the design of parts fabricated using material jetting and additive manufacturing in general.

This work presents the first systematic and comprehensive approach to investigating the anisotropies of additive manufacturing at different length scales in the case of material jetting. In this study, seven different print orientations between 0° to 90° will be looked at using multiple different test methods. This thesis aims to explore and build a connection between the anisotropies from the lower micron length scale at the layer interface level all the way up to the bulk tensile properties seen in uniaxial tension and then to the case of complex loading ge-
omtries and architectures in microtruss structures. The mechanical properties in relationship to orientation at each length scale will be quantified for a large range of orientations to provide a better understanding of failure and the optimization of print orientation of parts fabricated using material jetting.

This thesis is organized into five subsequent chapters. A background of necessary information will be presented in Chapter 2 with a focus on photopolymers in additive manufacturing, as well as additive manufacturing and its related anisotropy effects. Chapter 3 provides the details on the methodology employed for the experimental work done in this thesis and outlines the print orientations studied in this thesis. In Chapter 4, an extensive mechanical characterization of the anisotropic effects present in the jetted photopolymer structures is performed. This chapter is split up into three major sections. The first, Section 4.1, will explore the local properties of the photopolymer on the microscale through both nano- and micro- indentation testing. Section 4.2, which follows will look into anisotropies in tension and how the results from the indentation contribute and lead to trends seen. This provides the bulk of the analysis of this thesis as it explores a multitude of factors that effect the variability in properties and dwell time between printing and testing as it relates to the photopolymer system. In addition, the effect of introduced flaws by notching is explored and how the failure mechanism of the layering relates to unidirectional composite laminates is explained. Lastly for the results, Section 4.3 looks at the cumulation of anisotropies for the case of microtrusses loaded in three point bending. Chapter 5 provides a conclusion of the results and the main takeaways from this study and recommendations for future work are listed in chapter 6.
Chapter 2

Background Information

2.1 Photopolymers

The material used in this thesis is a class of photopolymer and as such, a background on the two main types of photopolymerization techniques used commercially in additive manufacturing and relevant post-cure behaviour of both are given. The amount of polymerization and crosslinking in the samples will determine the overall properties of the final part. Photopolymers are a class of light sensitive polymers which react when exposed to ultraviolet (UV) light. Typically used as a resin, these polymers are cured under UV radiation as the energy provided initiates a chemical reaction to form a chemical crosslink. Photopolymers have been used in industry since the mid 1970’s and are known for their rapid cure rates, low temperature operation, and versatility [19]. This makes it a prime choice for its use as a part material in additive manufacturing. Charles Hull developed the stereolithography process in the 1980s and patented the technology after experimenting with photopolymers and a scanning laser [20].

Photopolymers used in additive manufacturing typically consists of a blend of additives (flexibilizers, stabilizers, and diluents), photoinitiators, and liquid monomers/oligomers which polymerize either under free-radical or cationic photopolymerization [20]. This process occurs when the photoinitiators are chemically activated by the UV radiation and start undergoing
crosslinking reactions with the monomers and oligomers present to form a thermoset network. The most commonly used monomers and oligomers used due to their properties are acrylates and polyurethanes respectively [21]. For reference, a 14-24% blend of proprietary urethane acrylate oligomers are used in the VisiJet M3 Crystal photopolymer [22]. The type of photopolymerization reaction that occurs is dependent on the type of monomer and oligomers being considered. This also has a consequence in post-cure behaviour, a factor that has to be taken into consideration in additive manufacturing as additional crosslink reactions could occur after the printing process.

Epoxies and vinylethers are the most common types of cationic photopolymers where a cation generating photoinitiator leads to a ring opening reaction in the monomers [20]. Shama [23] noted that in the case of cationic photocuring for their study on vinyl ether end-capped urethane, post-curing can occur without the need of an active UV source as the protic acid formed by the photoinitiators are long lived species and continue the polymerization process.

Urethane acrylate photopolymers cure via the free radical mechanism where photoinitiators activated by UV light generates free radicals [23, 24]. These free radicals undergo crosslinking reactions with the oligomers present to form a thermoset network. The free radicals involved in the crosslinking reactions however are short-lived species that generally do not persist long enough to contribute to any post-cure reactions [23]. Although Shama [23] investigated both cationic and free radical photopolymerization and found both showed post-curing effects, a reason for the post-curing effect in free radical photopolymerization was not determined. Tey et al. [24] later studied the post-curing effects of a commercially available photocured polyurethane acrylate and found that the modulus and glass transition temperature did change over a period of ten days post-cure, thus showing additional crosslinking. This reaction was found to occur regardless of any additional UV or light exposure signifying that there was another curing reaction occurring. It was found that moisture in the air reacted with either isocyanate or cyanoacrylate groups present in the urethane acrylate in a two step process leading to further crosslinking [24]. This is of note as the VisiJet M3 Crystal photopolymer is based on urethane
2.2 Additive Manufacturing

3-D printing as mentioned in the introduction is an additive manufacturing technique that builds a final product directly as opposed to subtractive manufacturing which achieves its final shape by milling or removing parts from a starting material. This subsection will briefly cover some of the main technologies and methods of 3-D printing.

Fused deposition modelling (FDM) is an extrusion based deposition technique that passes a filament of material through a heated nozzle. Bonding in FDM is reliant on sufficient residual heat on the surfaces of the layer to allow for the two adjacent layers to bond. Without sufficient heat, adherence may be poor and result in a distinct boundary, and with too much heat, the deposited layer may flow out of the intended position [20]. Typically, the material is extruded at about 1°C above its melting temperature to allow for rapid solidification upon extrusion and welding of the layer [1]. The nozzle is capable of rastering in the horizontal plane to complete a layer before the building platform is lowered to allow for the next layer to print as seen in Figure 2.1.

Ahn et al. [26] looked at the anisotropies in FDM produced ABS P400 tensile coupons for 0°, 45°, and 90° cases and compared results to an equivalent injection molded sample. One immediate realization was that the FDM produced parts had significant issues in tensile testing due to stress concentration effects. The discretization of the shoulder region of the tensile specimen created an easy point of failure, see Figure 2.2, left. A second attempt was made by changing the raster pattern to follow the curvature but still resulted in premature failure as the gap in the raster pattern now acted as the stress concentrator, see Figure 2.2, right. This resulted in a change of standards of testing from ASTM D638 for polymer tensile testing to ASTM D3039 for tensile testing of polymer matrix composite materials which used rectangular shaped test specimens with no shoulder regions. This highlighted the initial difficulties of testing and acrylates.
considerations required for testing 3-D printed parts. In this study, all 3 orientations were printed flat, and focused on a single plane. Orientation effects studied here are seen through the rastering of the nozzle itself rather than through layering. The raster orientation and loading configuration along with results are shown in Figure 2.3. Note that the full tensile specimen required 12 layers to be printed with various raster patterns which is referred to in the naming convention. For example, the \([45^{\circ}/-45^{\circ}]_6\) referred to 6 repetitions of 45° and -45° layers. Ahn et al. [26] found that even in what was considered to be the ideal case, specimens failed at 73% of the injection molded value due to the raster patterning, and in a worse case scenario, down to 10% of the injection molded prints strength.

Domingo-Espin et al. [27] also looks at FDM anisotropies but for a polycarbonate system. In order to simulate FDM parts through finite element analysis (FEA), Domingo characterized the polymer with 0°, 45°, and 90° tensile coupons as well, in order to describe the compliance matrix for the material. Assuming that it was orthotropic, having three mutually perpendicular
Figure 2.2: Premature shear failure due to stress concentration effects in ASTM D638 standard tensile specimens as reported by Ahn et al. [26].

Figure 2.3: Tensile strength of specimens with various raster patterns printed via FDM compared to their injection molded counterpart as tested by Ahn et al. [26].
planes of symmetry, this is given by:

\[
\begin{pmatrix}
\varepsilon_x \\
\varepsilon_y \\
\varepsilon_z \\
\gamma_{yz} \\
\gamma_{xz} \\
\gamma_{xy}
\end{pmatrix}
= 
\begin{pmatrix}
1/E_x & -\nu_{xy}/E_x & -\nu_{xz}/E_x & 0 & 0 & 0 \\
1/E_y & -\nu_{yz}/E_y & 0 & 0 & 0 & 0 \\
1/E_z & 0 & 0 & 0 & 0 & 0 \\
sym & 1/G_{yz} & 0 & 0 & 1/G_{xz} & 0 \\
sym & 1/G_{xz} & 0 & 0 & 1/G_{xy} & 1/G_{xy}
\end{pmatrix}
\begin{pmatrix}
\sigma_x \\
\sigma_y \\
\sigma_z \\
\tau_{yz} \\
\tau_{xz} \\
\tau_{xy}
\end{pmatrix}
\]

where \( \varepsilon_i \) is for normal strain, \( \gamma_i \) is for shear strain, \( \sigma_i \) is for normal stresses, \( \tau_i \) is for shear stress, \( E_i \) the material’s modulus, \( \nu_i \) the material’s Poisson’s ratio, and \( G_i \), the material’s shear modulus. However, in this study, although clear that strain at fracture is limited in the 90° case, falling below 3% compared to the 10%+ ductility in the 0° for tension, these factors are not incorporated into their FEA model of the cantilever beam. The cantilever model was found to be off by an average of 7.3% with a range of 4.22 to 11.1% from experimental testing depending on the test orientation used. This showed that more factors need to considered in order to create a better FEA model.

Stereolithography (SLA) is one of the photopolymerization based techniques. This technique utilizes a vat of photocurable resin and a UV laser which traces out and individual layer in the surface of the vat. The stage is progressively lowered by a layer thickness after each trace to allow the following to be sketched. A schematic of the SLA process is shown in Figure 2.4 Bonding both within a layer and between layers are controlled by the power and rastering speed of the UV laser. In addition, by utilizing a laser, each individual layer is not bonded together simultaneously but instead by the raster pattern utilized to scan each individual layer. The choice in raster pattern was found to affect the bonding both within individual layers and between layers [28].

Puebla et al. [28] studied the mechanical anisotropies in stereolithography for the WaterShed 11120 photopolymer system used with a Viper si2 SLA system. This study focused
Figure 2.4: Schematic illustrating the SLA process obtained from [25].

mainly on the effect of the rastering in SLA and tested samples printed flat, on their side, or vertically, as shown in Figure 2.5. The results of the vertical printed sample actually performed on par with the edge print in terms of modulus and tensile strength, approximately 2650 MPa and 50 MPa respectively. It was the flat printed samples that had the worse performance with a modulus of approximately 2400 MPa and a tensile strength of 45 MPa. This was not expected by Puebla et al. [28] as it demonstrates that the bonding between successive layers was stronger than bonding inside a layer contrary to what has previously been seen in other studies. This was attributed to the scanning process of the UV laser used in SLA. Each layer is printed first with a slow, high intensity border vector which leads to higher degrees of curing, to create the surface boundary. The internal structure is then made up of fill vectors, approximately twice the speed of a border vector, for the bottom and top layers, and sandwiches the internal hatch vectors. These hatch vectors are scanned at six times the speed of the border vectors. It was found in SLA that trends in anisotropies may differ due to the rastering method used to cure the parts. From Figure 2.6, it can be seen that for the cross-sectional area that the tensile stress is acting upon, the flat, edge, and vertical samples have 20%, 80%, and 100% respectively of
Chantarapanich et al. [29] who later studied a similar Watershed 11122 epoxy photopolymer resin repeated a similar test comparing samples printed flat and on their sides but included the effect of angle by printing these two orientations at $0^\circ$, $45^\circ$, and $90^\circ$ as shown in Figure 2.7. Chantarapanich et al. [29] noticed the same effect of edge specimens being stronger than flat specimens for all orientations for the same reason as previously explained by Puebla et al. [28]. Differences as a result of the three different angles were seen for both cases and followed the same trend. It was found that the $45^\circ$ outperformed the $0^\circ$ and $90^\circ$ samples. For the edge case, the $45^\circ$ orientation had a modulus and tensile strength of 2400 MPa and 48 MPa respectively compared to values of 2340 MPa and 46 MPa respectively for both $0^\circ$ and $90^\circ$ cases. Similar trends of 2200 to 2100 MPa and 43 to 38 MPa were seen in modulus and tensile strength respectively for the flat $45^\circ$ to flat $0^\circ$ and $90^\circ$ cases respectively. This however, appears to occur
Figure 2.6: Schematic illustrating the different build vectors that are used to fabricate the (a) flat, (b) edge, and (c) vertical specimens. A cross-sectional view is provided on top. Obtained from [28].
because of their unconventional definition of the angles used. As the raster pattern employed in this study actually followed a $\pm 45$ pattern, it is more logical to define their naming convention differently. That is, the $45^\circ$ should be considered as a $[0^\circ/90^\circ]$ cross-hatched sample based on the raster direction relative to the tensile loading axis. Similarly, the $0^\circ$ and $90^\circ$ cases would behave more like a typically defined $\pm 45^\circ$ raster. Under this convention, the trend in anisotropy, are comparable to those previously discussed by Ahn et al. [26].

Material jetting is another of the photopolymerization based technique. This is the technique utilized by the ProJet HD3500 used in this thesis. Material jetting is comparable to an inkjet printer but rather than depositing ink, it deposits a photopolymer resin. The printer utilizes a series of jets to deposit the photopolymer part material in a series of fine liquid droplets. The jets are then run again to deposit liquid wax support material where required. Support ma-
Figure 2.8: Schematic illustrating the material jetting process obtained from [25].

terial is required in parts where there are overhangs or internal space as the part material cannot be printed in open space. The layer is then cured by a passing UV lamp to form a solid layer. A leveling blade is run through the layer to even out any height differences where droplets overlapped. The jets are raised by a single layer and the process is repeated until completion. A schematic of the material jetting process is shown in Figure 2.8. The final structure is then placed into the oven to remove any wax support material. The bonding strength between the layering here is determined by the level of crosslinking present at the layer interface.

Bass et al. [30] studied the anisotropies in material jetting utilizing six different composite blend ratios of VeroWhitePlus and TangoBlackPlus photopolymer that are rigid and flexible in nature respectively. ASTM D638 Type IV coupons were printed using a Stratasys Objet350 Connex at both $0^\circ$ and $90^\circ$. For their system, Bass et al. found that in general, parts oriented along the print direction, i.e. $0^\circ$ had better performance, compared to parts oriented in the layering direction, i.e. $90^\circ$. This is again due to the differences in interlayer strength, as expected for a material jetting system. Bass et al. also identified that higher levels of anisotropies were
seen with decreases in rigidity as the amount of TangoBlackPlus increased. They hypothesized this difference to be a result of the patterning required to print the polymers simultaneously. An interesting point in their results to note is that there were minimal changes in tensile strength with change in orientation for the most rigid polymer blend, both approximately 45 MPa, but drops in ductility were seen from approximately 13% to 3%. As no tensile curves were provided, it is assumed that the 90° failed at the yield point while orienting the part along the print direction allowed for significant deformation before fracture.
Chapter 3

Materials and Methodology

3.1 Material Jetting and Sample Orientation

Seven different print orientations, 0°, 11.25°, 22.5°, 33.75°, 45°, 67.5°, and 90°, were tested throughout this thesis under each testing technique. These orientations are given relative to the sample geometry for a particular test and the print direction of the printer jets. Schematics for each test are shown or described in their respective sections. The experiments completed in this thesis all utilize the same material and fabrication method. Parts were all printed using a 3-D Systems ProJet HD3500, shown in Figure 3.1, in ultra high definition mode at a resolution of 750 x 750 x 890 dots per inch. This forms a part with 29 \( \mu \)m layers with an accuracy of 0.001 - 0.002 inch per inch [18]. The net print volume is limited to 5 x 7 x 6 inches or 127 x 178 x 152 mm in ultra high definition mode. A standard high definition mode is also available for larger print volumes. This increases the net print volume to 11.75 x 7.3 x 8 inches or 298 x 185 x 203 mm at a resolution of 375 x 375 x 790 dots per inch. Layer thickness is also increased to 32 \( \mu \)m. The photopolymer resin part material and wax support material used for the work in this thesis were the VisiJet M3 Crystal and the VisiJet S300. The Projet HD3500 is capable of supporting two 2 kg cartridges each of part material and support material. A list of their properties as well as for other ProJet materials from the manufacturer as provided is
Figure 3.1: The ProJet 3500HD 3-D printer from 3D Systems utilized in this thesis [18].

shown in Figure 3.2. Note that the properties listed do not provide any information on the print orientation of the tested part.

As discussed later in Section 4.2.1, there are many factors that can affect the properties of the parts tested. To maintain consistency wherever possible, the same post-printing finishing procedure is performed on every sample. All printed parts are attached to the aluminum printer build plate by wax support material which provided a smooth and uniform base for polymer to be deposited compared to the aluminum plate which contains scratches and imperfections. This step is in addition to any wax support actually required for the printing and support of the structure. As such, all parts, regardless if they required any supports, require the additional finishing step to remove the wax present. To separate the parts from the aluminum build plate, the entire build plate along with all parts are removed from the printer and placed in a refrigerator set to 3°C for ten minutes. The difference in the thermal expansion coefficients of the wax and aluminum build plate allow for easy separation without unnecessary force. All samples were placed in a ProJet Finisher at 65°C and wrapped in tissues to melt, absorb and remove all wax support material. All samples were left in the finisher for 24 hours, and tissues were replaced as required.
Figure 3.2: The list of available photopolymers and support materials along with their properties available for use with the ProJet HD3500 as provided by the manufacturer[18]. VisiJet M3 Crystal and VisiJet S300 are used exclusively in the work of this thesis.

### 3.2 Indentation

#### 3.2.1 Nanoindentation

Nanoindentation was used to probe the hardness and elastic modulus on the lower micron size scale. Nanoindentation allows for direct determination of mechanical properties with the use of high-resolution test equipment to track the load-displacement information of the indenter on the \( \mu \text{N} \) and \( \text{nm} \) scale respectively. Schematics of an indentation cross-section and of a typical load-displacement curve are shown in Figure 3.3 and Figure 3.4. The hardness and modulus are measured based on the unloading process and determined using the Oliver and Pharr method as discussed below [31, 32]. The contact height, \( h_c \), is required to calculate the area of the contact, \( A \), where:

\[
A = 24.5h_c^2
\]  

(3.1)

The hardness is then defined as the max load divided by \( A \). The contact height is determined by subtracting the sink-in depth, \( h_s \), from the max indentation depth, \( h_{\text{max}} \), shown in Figure 3.3. The sink-in depth, \( h_s \), accounts for the loss in contact of the indenter due to sinking in of the
Figure 3.3: A schematic of the indentation cross-section during loading and unloading showing key parameters used in the Oliver and Pharr method [32]

material adjacent to it and is given by:

\[ h_s = \epsilon \frac{P_{\text{max}}}{S} \]  

(3.2)

where \( \epsilon = 0.75 \) for a Berkovich indenter and \( S \) is the contact stiffness, the tangent to the curve during the initial unloading, as shown in Figure 3.4. The contact height is then given by:

\[ h_c = h_{\text{max}} - \epsilon \frac{P_{\text{max}}}{S} \]  

(3.3)

The effective modulus, \( E_{\text{eff}} \), which accounts for both the tip and material stiffness, can then be calculated by:

\[ E_{\text{eff}} = \frac{\sqrt{\pi}}{2\beta} \frac{S}{\sqrt{A}} \]  

(3.4)

where \( \beta \) is taken as unity. The modulus of the material, \( E \), is then determined by:

\[ \frac{1}{E_{\text{eff}}} = \frac{1 - \nu^2}{E} + \frac{1 - \nu_i^2}{E_i} \]  

(3.5)

where \( \nu \) is the Poisson’s ratio of the material, \( \nu_i \) is the Poisson’s ratio for the indenter, and \( E_i \) is the modulus of the indenter.

The hardness values and elastic modulus of the material was measured using an Anton Paar UNHT Ultra Nanoindentation Tester with a diamond Berkovich tip with \( E_i = 1141 \) GPa and \( \nu_i \)
Figure 3.4: A typical load-displacement curve for a nanoindentation test [31]

= 0.07. The tip forms a triangular-based pyramid with a half angle of 65.27° from the indenter tip axis to one of the three pyramid flats and produces the same projected area to depth ratio as a Vickers indenter [33]. Nanoindentations were done at 10 mN with a loading rate of 60,000 µN/min, a 10 second hold, and an unloading rate of 60,000 µN/min. An additional set of nanoindentations were done at 20 mN using loading and unloading rates of 120,000 µN/min. Pucks of 20 mm diameter and 15 mm thickness were printed with the seven different print orientations listed at the start of this chapter. Indentations were made on the surface of these pucks, i.e. into a layer for a 0° puck, and between layers for a 90° puck. A schematic of the loading configuration is shown in Figure 4.1.

3.2.2 Microindentation

Microindentation was performed as a supplementary analysis to nanoindentation and only looked at the 0°, 45°, and 90° cases. Pucks of the same sample geometry and loading configu-
ration of the nanoindentation tests were used. Compared to nanoindentation, only a measurement of projected contact area after load removal is given as the instrumentation is much simpler and does not provide continuous load-displacement recording. All microindentation were completed using a Buehler Micromet 5103 Microindentation Hardness Tester with a Vickers indenter tip using a 50 gram force. Optical micrographs were taken using the microscope on the Anton Paar UNHT Tester to determine the length of the indent diagonals. The Vickers hardness number, $HV$ in kilograms force per square mm, is calculated using equation 3.6 with the average measured diagonal of the square indent, $d$, in mm, and the indentation load, $F$, in kgf. This Vickers number value can be converted into units of MPa by multiplying the $HV$ value by 9.81 $m/s^2$. In addition, the indentation depth, $h$, can also be calculated from $d$ using the indenter tip geometry and equations 3.7 and 3.8, where $A_p$ is the projected contact area.

$$HV = \frac{2F}{d^2} \sin(68^\circ)$$  \hspace{1cm} (3.6)

$$\frac{A_p}{h^2} = 4\tan^2(68^\circ)$$  \hspace{1cm} (3.7)

$$A_p = \frac{d^2}{2}$$  \hspace{1cm} (3.8)

### 3.3 Tensile Testing

For a polymer stress-strain curve, there are certain distinctions in terminology for yielding and tensile strength that must be noted for this analysis [34]. These designations are shown in Figure 3.5 for three typical polymer tensile stress-strain curves. The tensile strength of a polymer, similar to the ultimate tensile strength of a material, is defined as the maximum stress sustained during testing (points A, B, and E in Figure 3.5). However, for a polymer, there are certain distinctions for the tensile strength depending on when it occurs, particularly at the yield point or at fracture. The yield point is defined as the point at which the curve plateaus and has a slope of zero. That is, no further increase in stress is required for an increase in strain.
The yield strength for a polymer differs from the offset yield strength and is defined as the stress at the yield point. If this point is the maximum, it is also labelled as the tensile strength at yield (point B in Figure 3.5). However, it should be noted that not all materials will have a yield point. If the maximum stress occurs at fracture, the point is defined as the tensile strength at break (points A and E in Figure 3.5). As the type of curve will vary depending on the print orientation, the tensile strength may occur at various points. For this polymer, it was found that the stress value defined as the tensile strength could occur at the yield point, at fracture, and occasionally in between. For the sake of direct comparison in this thesis, the focus will be on points that are clearly defined. The four tensile properties focused on in the uniaxial tension testing results are:

- $E$ (MPa), modulus of elasticity, taken as the slope of the initial linear region
- $\sigma_Y$ (MPa), tensile stress at the yield point
- $\sigma_f$ (MPa), tensile stress at fracture
- $\epsilon_f$ (mm/mm), ductility or elongation at fracture

A Shimadzu EZ-L 5kN Universal Testing Machine was utilized for the bulk of the tensile testing except where explicitly stated. Testing was all done in accordance with a modified ASTM D638 standard [34]. Tensile coupons tested were printed at a quarter scale of the ASTM D638 Type I definition shown in Figure 3.6. This resulted in tensile coupons with a gauge length, width, and thickness of 12.5 x 3.25 x 1.75 mm respectively. The crosshead speed for testing was set to 1 mm/min for a strain rate of 8% per minute. There are two main reasons for the reduced sample size. The first is due to the limitation in print volume by the 3-D printer. The print volume of 127 x 178 x 152 mm greatly limits the placement and orientations possible of a full sized 165 mm long tensile coupon, i.e. samples like the 90° oriented case cannot be printed. The second limitation is from the $in situ$ tensile module. In order to compare future results between the Shimadzu load frame and the $in situ$ load frame, the tensile coupons tested were standardized to fit in both machines. It was found that ASTM D638 Type I tensile coupons
needed to be scaled down to 25% of its original size for this to occur. It should be noted that
the reduced gauge length should be reported with any of the tensile results. Samples were
also sanded using a P320 fine grit sandpaper to remove any stepped features present in the
shoulder region. This is done to remove any stress concentration effects at the shoulder due to
the printer’s approximation of the radius of curvature present in the shoulder. These defects are
removed in order to minimize premature failure in the shoulder region so that tensile results
are representative of the material in the gauge length. These effects are further discussed in
Appendix A. Samples tested here use the same print orientations outlined at the beginning of
this chapter. A schematic of the tensile coupons printed at these orientations illustrating the
layering structure is shown in Figure 3.7.

In addition to the Shimadzu load frame, a Kammrath Weiss 5 kN in situ tensile module
and heating stage was used for testing of tensile coupons with notches incorporated during
the printing process. These tensile coupons were still of the same quarter scale models of the
Figure 3.6: Dimensions for tensile coupons with thickness values of 7mm or under from ASTM D638 [34]
Figure 3.7: A schematic illustrating the seven different print orientations used in tensile testing. These are, from the back left to front right, the 90°, 67.5°, 45°, 33.75°, 22.5°, 11.25°, and 0° oriented tensile coupons. Each tensile coupon has an overall length of 41.25 mm.
ASTM D638 Type 1 standard but included various shaped notches in order to study the effect of stress concentrators in regards to print orientation. A preliminary proof of concept experiment was also done using the heating stage to test samples at an elevated temperature of 65°C. All heated samples tested were left on the heating stage for 20 minutes before testing to allow for consistent temperature through the entire gauge. The sample temperature was confirmed using both an infrared thermometer and a thermocouple. All work done in situ utilized a Hitachi SU3500 Scanning Electron Microscope for imaging. The loading of the in situ module and heating stage in the Hitatchi SU3500 is shown in Figure 3.8. Serial connection settings for receiving data output from the in situ tensile module via COM port for use with either PuTTY or HyperTerminal are provided in Figure 3.9. Various difficulties were encountered with the handling and processing of the data output from the in situ module. These issues are discussed in Appendix B and a MATLAB script is provided for the batch processing and correction of this data output.
Figure 3.8: The *in situ* tensile module and heating stage being loaded into the Hitachi SU3500.
Figure 3.9: Serial connection settings required for PuTTY or HyperTerminal software programs in order to receive data output from the *in situ* tensile module via COM port. Note that COM port values may differ.
3.4 Three Point Bending

Three point bending was performed on microtrusses (see Figure 3.10) with the seven print orientations outlined at the start of this chapter in order to expand the study of anisotropy from point loads in indentation and uniaxial loading in tension. A schematic of the microtrusses printed with the seven different orientations tested and their layering structure are shown in Figure 3.11. Due to the geometry of the struts in the microtruss and the three point loading configuration, anisotropies of the part can be analyzed for a more complex loading condition. A Shimadzu AG-I with a 500 N load cell was used for three point bend testing. An effective crosshead displacement rate of 1 mm/min was used. The microtrusses utilized in this study are based on an optimized design by Lausic [35]. The samples tested were a 11 x 3 pyramidal unit cell microtruss. Individual unit cells were 6.25 mm in size with a strut radius of 625 µm and a slenderness ratio of 20. Note that the full length of the microtruss is 68.75 mm but a span length of 56.25 mm was used as the two lower support pins for three point bending were placed between the first & second, and tenth & last unit cells as shown in Figure 3.10.

Figure 3.10: A schematic of a microtruss and the relative locations of the loading and support pins for three point bend testing. The microtruss has an overall length of 68.75 mm.
Figure 3.11: A schematic illustrating the seven different print orientations of microtrusses tested in three point bending. These are, from left to right, the $0^\circ$, $11.25^\circ$, $22.5^\circ$, $33.75^\circ$, $45^\circ$, $67.5^\circ$, and $90^\circ$ oriented microtrusses. Each microtruss has an overall length of 68.75 mm.

3.5 Digital Image Correlation

Digital image correlation (DIC) is a non-contact method used to track displacement and strain of 3-D surfaces. This technique uses a two camera system to capture images during testing which are processed by a computer to give 3-D surface information. Samples are typically given a speckle pattern by spray painting it with a non-reflective matte white coating followed by a mist of black spray paint. This creates a random speckled pattern of black dots on a white background, an example is shown in Figure 3.12. The speckle pattern is used to increase the number of unique features that can be tracked by the image recognition software of the DIC system. The local strain values on the sample surface is then calculated by tracking the relative movements of each of these unique points to one another.

A GOM 5M DIC camera system was used in conjunction with the ARAMIS software package to capture and process the results. This method is used extensively throughout this thesis not only for observing strain localization and distribution during mechanical testing but as a tool to measure the actual applied strain in the sample. Force and displacement data from
Figure 3.12: Tensile coupon with a spray painted speckle pattern prepared for DIC analysis.

tensile testing are recorded from the load cell and crosshead displacement respectively. However, due to the compliance of the load frame, additional displacement is measured resulting in an overestimation of the applied strain in the samples. DIC was used to measure the actual displacement in the sample gauge length without the added effects of machine compliance. Compliance corrected values for both the Shimadzu 5kN EZ-L Universal Testing Machine and the Kammrath Weiss in situ tensile module are provided in Appendix C. All tensile results in this thesis utilize compliance corrected displacement data to calculate strain.
Chapter 4

Characterization of Mechanical Anisotropies in Jetted Photopolymer

The following chapter discusses the testing and analysis performed to determine the significance of print orientation on the inherent mechanical anisotropy of the jetted photopolymer due to the layering process. The testing done looked at variations in material properties of the seven different print orientations outlined in Section 3.1. The changes in properties with print orientation for each type of experiment will be compared with the base case where the sample is oriented in line with the print direction, i.e. the 0° orientation. To determine if these anisotropies held true at different length scales, experiments were chosen to characterize both bulk and local properties. These experiments can be divided into the two types of length scale investigated:

- Microscale
  - Through nanoindentation for characterization of individual layers and their local interlayer interactions.
  - Through microindentation to observe the above-mentioned interactions over a larger scale of multiple layers.
• Macroscale

  – Through tensile testing for characterization of bulk interlayer effects under uniaxial loading.

  – Through three point bend testing of architectured microtruss structures to observe orientation effects in more complex shapes. Different individual struts of the microtruss will have varying print orientations and be subjected to different loading conditions.

### 4.1 Micro- and Nano- Indentation

This subsection of the chapter will characterize the properties and anisotropies of material jetted parts on the micro length scale. It will also be used as a base of comparison for the bulk properties results in the subsections to follow. The effect of orientation will be looked at on a local layer-level scale by testing individual layers and their interlayer interactions via indentation.

#### 4.1.1 Nanoindentation

Nanoindentation testing was done on the seven print orientations to determine the extent of anisotropies present at the lower boundaries of the micro length scale level. The direction of loading relative to these seven layering directions can be seen in Figure 4.1. This figure shows to scale, the indenter tip, indentation depth, layer thickness, and approximate interaction volume of the indent which will all be further discussed in this subsection as it relates to the nanoindentation results. As illustrated in this figure, the size of the indent relative to the layer thickness, approximately 29 µm, is small. This allowed for the probing of the sample at the length scale of one to two layers to observe if the effects of orientation are present at the micro level. Ten indents each at two different loads, 10 and 20 mN, were done on each sample orientation for a total of twenty indents for each orientation. The reason for the two
different loads is to confirm if the same results are seen at different indentation depths and hence different interaction volumes. The interaction volume of the indent refers to the volume of material that acts upon the load to resist deformation. Although the size of the volume is not clearly defined, a typical rule of thumb taken is that a sample must be at minimum ten times thicker than the indentation depth to avoid contribution effects from outside of the sample [36]. In this study, for the simple purpose of understanding the size scale of the interaction volume to the layer thickness, this volume is approximated using this rule of thumb. A semicircle around the indent with a radius $10 \times$ the indentation depth is shown in the schematics of Figure 4.1 to give a rough visual estimation of the approximate volume contributing to the values being measured.

The load versus indentation depth curves for all 140 nanoindentation tests are shown in Figure 4.2. A representative curve for each of the seven orientations is shown in Figure 4.3 for clarity. From Figure 4.2 and Figure 4.3, there is a clear orientation effect seen in the 90° samples. For a given load, either 10 mN or 20 mN, the indentation depth is typically more than two times as high compared to the other six different samples. This is a sign of significantly lower hardness as the sample is unable to resist the indentation. Measurements of contact depth, hardness values, and elastic modulus are extracted from the load-indentation depth curves for further analysis. These results are given in Table 4.1. A polar plot of these results is shown in Figure 4.4 for easier visualization of the trends. The contact depth (see Figure 3.3) defines the depth of material under contact with the indenter accounting for sink-in adjacent to the indent. The projected area of contact are determined from the contact depth of the indent based off the indenter geometry. Indents at 10 mN resulted in indentation contact depths, $h_c$ of approximately 1.6 to 3.0 $\mu$m and an equilateral triangle shaped indent area with side lengths between 12 to 22 $\mu$m depending on the sample print orientation. Similarly, indents at 20 mN resulted in indentation contact depths, $h_c$ of approximately 2.2 $\mu$m to 4.6 $\mu$m and triangular shaped indent areas with equal side lengths between between 17 to 35 $\mu$m, again dependent on orientation.
Figure 4.1: A schematic diagram illustrating the direction of loading for the (a) 0°, (b) 11.25°, (c) 22.5°, (d) 33.75°, (e) 45°, (f) 67.5°, and (g) 90° print orientations. The typical indentation size is highlighted in green and the estimated interaction volume of the indentation is circled in red for a 10 mN indent. The indenter tip, indentation depth, and layer thickness are to scale but the overall size of the samples is much larger than depicted. Note that the 90° orientation has a slightly deeper indent and larger interaction volume.
Figure 4.2: All 140 nanoindentation load-displacement curves for the seven different orientations tested at 10 and 20 mN loads.

Table 4.1: Summary of results from nanoindentation of samples of various print orientations under 10 and 20 mN load indentations. Both hardness and elastic modulus are given in units of MPa.

<table>
<thead>
<tr>
<th>Print Orientation (°)</th>
<th>Contact Depth, $h_c$ (nm)</th>
<th>Hardness, $H_{IT}$ (MPa)</th>
<th>Elastic Modulus, $E$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>10 mN</td>
<td>20 mN</td>
<td>10 mN</td>
</tr>
<tr>
<td>0.00</td>
<td>1790 ± 30</td>
<td>2530 ± 40</td>
<td>126 ± 4</td>
</tr>
<tr>
<td>11.25</td>
<td>1590 ± 50</td>
<td>2240 ± 30</td>
<td>161 ± 11</td>
</tr>
<tr>
<td>22.50</td>
<td>1700 ± 50</td>
<td>2370 ± 40</td>
<td>141 ± 10</td>
</tr>
<tr>
<td>33.75</td>
<td>1710 ± 40</td>
<td>2380 ± 40</td>
<td>138 ± 6</td>
</tr>
<tr>
<td>45.00</td>
<td>1710 ± 70</td>
<td>2400 ± 50</td>
<td>139 ± 12</td>
</tr>
<tr>
<td>67.50</td>
<td>1650 ± 50</td>
<td>2350 ± 50</td>
<td>153 ± 6</td>
</tr>
<tr>
<td>90.00</td>
<td>2990 ± 50</td>
<td>4640 ± 70</td>
<td>47 ± 2</td>
</tr>
</tbody>
</table>
Figure 4.3: Representative nanoindentation load-displacement curves for the seven different orientations tested at 10 and 20 mN loads.
Figure 4.4: Polar plots of the elastic modulus and hardness versus orientation for the 10 and 20 mN load nanoindentation results.

Outside of the 90° case, the load-displacement curves for orientations between 0° to 67.5° shown in Figure 4.2 produced a consistent set of curves. This gave values of hardness and modulus ranging from 126 to 161 MPa and 2260 to 2670 MPa respectively. These values are well within range to one another when compared to the outlier, 90° case. The low hardness and stiffness of the 90° sample, approximately 33% and 25% respectively of the other orientations, is a clear indication of the weakness in the polymer at the interface of the different layers due to the reduced amount of crosslinking achieved between successive print layers. The low hardness and stiffness of the 90° sample, approximately 33% and 25% respectively of the other orientations, is a clear indication that there is a significant difference in the interlayer strength.

From Figure 4.1 (g), it can be seen that the indenter on a 90° sample is able to effectively wedge between individual layers as it is the area of least resistance to deformation, analogous to trying to stick a pin into the side of a stack of paper. For the other orientations, there was also not much variation seen between the 10 and 20 mN loading conditions, except in the case of the 90° orientation. It showed a further drop in its hardness from 47 to 37 MPa and its modulus from 530 to 370 MPa as it is easier for the indenter tip to further propagate the splitting
of the two layers up until this point. The loss in properties measured in the transverse direction of the layering can be compared to the loss of mechanical performance in unidirectional composites laminates loaded in the transverse direction. Similar to transverse matrix failure in these composites as opposed to fiber fracture, the $90^\circ$ required less load to initiate deformation through the interlayer interface than in the layer itself. Outside of this $90^\circ$ case, it is noted that the modulus values were vastly overestimated from the manufacturer quoted tensile modulus value of 1463 MPa [18]. As polymers are more strain rate sensitive and as a strain rate cannot be defined for indentation, the modulus values extracted from indentation are more qualitative and require a correction factor [37]. Typically, nanoindentation appears to overestimate the modulus, with larger correction factors being required the smaller the actual modulus value is [37]. A more quantitative study on the effects of orientation on tensile modulus is given in Section 4.2.

4.1.2 Microindentation

In the previous subsection, nanoindentation was done to look at the effect of orientation on layer thickness scale properties to compare to bulk properties measured through macroscopic testing methods. It was found that the $0^\circ$ and $90^\circ$ cases were dominated by different local interlayer interactions while the other cases showed more consistent results with minimal anisotropy. Microindentation was done to probe a larger interaction volume consisting of multiple layers as opposed to individual layers, to observe more bulk microscale property of the material compared to that of the nanoindentation results. Three indents each at 50 gram force load were conducted on $0^\circ$, $45^\circ$, and $90^\circ$ orientation samples. These indents left square based pyramidal indentations with diagonals of approximately 81 $\mu$m and indentation depths of approximately 12 $\mu$m. These indents are on the scale of 3 to 8x deeper than the nanoindentation results depending on the orientation considered, and thus takes a larger interaction volume into account. Figure 4.5 is a schematic showing to scale, the indentation depth, indenter tip, layer thickness, and approximate interaction volume of the indent. Results from the microindentation tests are
given in Table 4.2.

The results of the microindentation showed no anisotropies between the three tested orientations, with practically the same indent size and hardness values seen between the three. There is no longer the significant drop in hardness at 90° as seen in the nanoindentation results. The reason for this is the previously mentioned larger interaction volume being tested with the larger load. The indent now probed a group of layers together and the interaction volume is large relative to the individual layer thickness, as seen in Figure 4.5. This gave properties more reflective of the material itself without orientation effects and interlayer interactions affecting the results as a larger volume is sampled.

The Vickers hardness value for the three different orientations of 137 to 139 MPa correlate well to the hardness values seen in nanoindentation of 139 to 141 MPa for the 22.5° to 45° orientations, which were more representative of the material hardness as discussed in the previous subsection. This confirmed that the microindentation provides a hardness value more consistent with the material itself without orientation effects. In addition, the yield strength of the material can be estimated from the Vickers hardness value using Tabor’s relation. Typically, $\sim 0.33 \times$ the Vickers hardness value in MPa, gives a good estimation of the material’s yield strength according to Tabor’s relation [38, 39]. As the Vickers hardness value was not affected by significant interlayer effects such as the ones seen in the 90° case in nanoindentation thus producing a constant hardness, it is possible to estimate the bulk material yield strength from this value. Assuming this relationship holds true, an estimated yield strength of 46 MPa is calculated from the microindentation results. This is well in the ballpark of the yield strength values seen later for uniaxial tension in Section 4.2.2 of $\sim 40$ MPa. Note that deviations from this correlation are possible and this calculated yield strength value is only provided to confirm that the indentation results are within reason. Koch and Seidler [40] who studied the correlation between indentation hardness and yield stress in a variety of thermoplastic polymers found that for crystalline and amorphous polymers, the correlation factor ranged from 2.8 to 6 and 2.3 to 5.1 respectively.
Figure 4.5: A schematic diagram illustrating the direction of loading for the (a) 0°, (b) 45°, and (c) 90° print orientations. The typical indentation size is highlighted in green and the estimated interaction volume of the indentation is circled in red. Note that the indenter tip, indentation depth, and layer thickness are to scale but the overall size of the samples is much larger than depicted.

Table 4.2: Summary of microindentation results which includes the average diagonal length of the indent’s square cross section, Vickers hardness (HV) calculated from the average diagonal, the estimated yield strength assuming 0.3x HV, and the calculated indentation depth.

<table>
<thead>
<tr>
<th>Print Orientation (°)</th>
<th>Average Diagonal Length, ( \bar{d} ) (μm)</th>
<th>Vickers Hardness, ( HV ) (MPa)</th>
<th>Estimated Yield Strength, ( \sigma_y ) (MPa)</th>
<th>Indentation Depth, ( h ) (μm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0°</td>
<td>81.5 ± 2.0</td>
<td>137 ± 7</td>
<td>~46</td>
<td>12</td>
</tr>
<tr>
<td>45°</td>
<td>81.0 ± 2.5</td>
<td>139 ± 8</td>
<td>~46</td>
<td>12</td>
</tr>
<tr>
<td>90°</td>
<td>81.5 ± 1.5</td>
<td>137 ± 5</td>
<td>~46</td>
<td>12</td>
</tr>
</tbody>
</table>
4.2 Uniaxial Tension

Tensile testing was conducted on tensile coupons printed in the seven different orientations to determine the extent of tensile anisotropies in parts fabricated via material jetting. The change of layering orientation within the part resulted in a large degree of change in the overall stress-strain response of the material.

Over the course of this thesis, 210 tensile coupons for the seven different print orientations were tested. The stress-strain curves for all 210 samples were plotted as shown in Figure. 4.6. Although cluttered, there are two main points that can be extracted from these plots:

- The anisotropy with varying print orientation is clear as the samples fracture under smaller stress and strains with increasing deviation from the print axis.

- The set of stress-strain curves for each particular print orientation span a wide range of the stress axis. There is a clear variation in the yield points and stress at fracture for a given orientation.

4.2.1 Variability of Properties

Before the trend in anisotropy can be analyzed and discussed, the complicating factors of batch to batch variance must be accounted for. The data was looked at in further detail to determine the reasons for this large spread in values in order to deconvolute external contributions from the analysis of print orientation. It was determined that there are two main factors that must be taken into consideration when comparing the mechanical properties.

- Tests from different print jobs showed significant variation in properties and samples from different batches cannot be directly compared. The variations in properties, which includes modulus, strength, and ductility, are generally unpredictable due to a large quantity of external factors. There are many possible combinations of causes that could alter
Figure 4.6: All 210 stress-strain curves collected for the seven different print orientations over the course of the study. Variation is seen between different print orientations as expected, but also within a particular print orientation.
the performance of the sample in different amounts, both positively and negatively, between printing and testing. This could be due to fluctuations or deterioration of the UV lamp over its lifetime, changes in the liquid polymer resin over time with regards to its stability, amount of time spent in the finisher, and cooling rate after removal from the finisher. Although attempts were made to keep these factors as consistent as possible, such as using identical printer and finishing settings, there are factors which cannot be directly controlled.

- Even after data from different print jobs were isolated and compared exclusively, the variance between samples, although decreased, was still present. This variation was determined to be related to the dwell time between printing and testing. Tests at different dates after the printing and finishing process showed a trend of increasing strength and stiffness with time. When the curves were categorized by the number of days from the time they were finished to the time they were tested, and for the specific print job, it was seen that there was generally good repeatability in the data on the order of 5% or less.

Over the course of this thesis, there have been external factors that could have led to batch to batch variations that cannot be controlled. One being the replacement of the consumable resin and wax support print cartridges as they were depleted, and the other being the replacement of the UV bulb as the UV intensity deteriorated over time. There is the possibility of minor chemical variations between resin cartridges manufactured at different times, and losses in intensity of the UV bulb over its lifespan can become significant [41]. The variation between batches can be significant as seen in Figure 4.7 which shows tests done on parts 8 days removed from finishing for two different print jobs before and after the replacement of the UV bulb. A newer bulb provided UV light at its full expected intensity leading to more crosslinking and thus stronger parts. Although the batch and time factors contributed to large variances and must be deconvoluted for more accurate analysis, it should be noted that the general trend of anisotropy with print orientation does hold true regardless of which print job the part is coming from and when the part is being tested.
Figure 4.7: Stress-strain curves for the seven different orientations being tested eight days after being removed from the finisher. Data is being shown from two different print batches before and after replacement of the UV bulb as noted by the solid and dotted lines respectively. Tensile coupons with the same layering structure show good repeatability within the same print job.
4.2.2 Anisotropies in Uniaxial Tension

This section will characterize the mechanical anisotropies in uniaxial tension from a single print job to ensure reliable comparisons. It will also look at results with regards to dwell time between finishing and testing to see the effect of post-curing over time. For this, over 70 samples were tested with a minimum of three repeats for each orientation and day parameter. All results for the 0°, 11.25°, 22.5°, 33.75°, 45°, 67.5°, and 90° are given in Figure 4.8. Representative results from Figure 4.8 are shown in Figure 4.9 for clarity. Each plot in these figures include the 0° results for direct comparison. A schematic demonstrating the orientation of the applied force relative to the print orientation and layer structure is also shown with each plot. Prior to the discussion of the material properties extracted from these curves, a few obvious observations can be made from these figures.

- Repeats of tests produced similar results with minimal deviation as compared to tests results under the same condition from other print batches.

- The same general trend of increasing peak stress and decreasing elongation at break is seen in all sample orientations as the number of days post-finishing increased.

- For 0° to the 90° print orientations, as the loading axis deviates from the layering direction, a clear sign of anisotropy is seen as the curves both deviate from linearity and terminate at earlier strain values.

As seen in Figure 4.8, repeats of tests for a particular set of testing conditions led to similar results. Minor deviations in stress values and the modulus between tests can be attributed to slight deviations in alignment of the sample in the tensile grips relative to the loading axis, and minor sample to sample variability. However, larger deviations in the strain at fracture values can be seen between samples within a set and can be attributed to the jetting process used in the manufacturing of these tensile coupons. As the coupon is being printed layer by layer, any curvature in the part will be approximated by steps. These steps, if not completely removed,
Figure 4.8: Stress-strain curves are shown for the seven different print orientation tested 1, 2, and 8 days after being removed from the finisher. The $0^\circ$ print orientation curves are overlaid in each graph for comparison. Increasing sample print orientations are shown in graphs (a) to (e) and labelled accordingly in the legends. A schematic diagram of the layered structure for each orientation is shown.
Figure 4.9: A representative stress-strain curve for each print orientation from Figure 4.8 tested 1, 2, and 8 days after removal from the finisher are shown in graphs (a) to (e) as solid lines. The $0^\circ$ print orientation is overlaid in each graph as dotted lines for comparison.
will act as stress concentrators and can lead to premature fracture. This effect will be discussed in further detail in Appendix A. In addition to these step features, the surface of any sample is also prone to surface roughness and other stochastic flaws from the layered printing process. Depending on the size of these flaws, they may not play a role in fracture until higher strain values well past the yield point if they are small enough as failure would first be dominated by the weaker interlayer interfaces discussed later in Section 4.2.4. The variability in strain at fracture is thus more prevalent in the more ductile print orientations, namely the 0° and to a certain extent the 11.25° print orientation. This is shown more clearly in Figure 4.10 (b). The error bars present on the plots for the 0° print orientations are noticeably larger relative to the other six orientations.

Table 4.3: Summary of mechanical properties from tensile coupons printed in various orientations. Values for Young’s modulus, strain at fracture, stress at yield point, and stress at fracture with their standard deviations are given for each sample orientation tested at 1, 2, and 8 days after sample finishing.

<table>
<thead>
<tr>
<th>Print Orientation (°)</th>
<th>Young's Modulus, $E$ (MPa)</th>
<th>Strain at Fracture, $\epsilon_f$ (%)</th>
<th>Stress at Yield Point, $\sigma_Y$ (MPa)</th>
<th>Stress at Fracture, $\sigma_f$ (MPa)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Day 1</td>
<td>Day 2</td>
<td>Day 8</td>
<td>Day 1</td>
</tr>
<tr>
<td>0.00</td>
<td>1690 ± 70</td>
<td>1670 ± 20</td>
<td>1750 ± 50</td>
<td>18.0 ± 2.1</td>
</tr>
<tr>
<td>11.25</td>
<td>1640 ± 90</td>
<td>1650 ± 20</td>
<td>1740 ± 10</td>
<td>14.4 ± 1.2</td>
</tr>
<tr>
<td>22.50</td>
<td>1590 ± 20</td>
<td>1680 ± 60</td>
<td>1700 ± 30</td>
<td>10.7 ± 1.0</td>
</tr>
<tr>
<td>33.75</td>
<td>1500 ± 20</td>
<td>1610 ± 80</td>
<td>1630 ± 50</td>
<td>8.2 ± 0.5</td>
</tr>
<tr>
<td>45.00</td>
<td>1450 ± 60</td>
<td>1580 ± 50</td>
<td>1670 ± 90</td>
<td>7.3 ± 0.7</td>
</tr>
<tr>
<td>67.50</td>
<td>1400 ± 50</td>
<td>1490 ± 20</td>
<td>1610 ± 40</td>
<td>5.9 ± 0.3</td>
</tr>
<tr>
<td>90.00</td>
<td>1390 ± 40</td>
<td>1460 ± 80</td>
<td>1540 ± 40</td>
<td>5.2 ± 0.8</td>
</tr>
</tbody>
</table>
The aforementioned trends with regards to sample dwell time before testing, and anisotropy with print direction seen in Figure 4.8 can be further expanded by comparing the material properties extracted from the stress-strain data. The results of the data from these tensile tests are summarized in Table 4.3. Due to the large amount of test data, the results can be summarized in a variety of ways to extract and visualize different trends.

### 4.2.3 Effect of Dwell Time

The effect of dwell time prior to testing is first examined by plotting the changes in mechanical properties as a function of time for the seven different print orientations and is shown in Figure 4.10. The dwell time refers to the period of time between the sample being removed from the finisher and when it is tested. Note that this does not include the amount of time spent in the finisher, approximately 24 hours, after printing is completed. The increase in Young’s modulus, stress at yield, stress at fracture, and a corresponding decrease in strain at fracture seen in this figure makes it clear that the material is becoming stiffer, stronger, and less ductile. This is in agreement with what was expected from the stress-strain curves previously seen in Figure 4.8. A plateau in the changes of these four properties is seen as there is a more notable difference between dwell times of one and two days compared with two and eight days. Overall, these trends appear to hold true regardless of the orientation they were printed. This is seen in each of the four graphs as the results of the seven different orientations appear to be nearly identical but shifted with respect to the y-axis. This signifies that the effect of dwell time is not connected to the orientation effect but is rather intrinsic to the photopolymer itself. Although it can be seen from this figure that mechanical performance is clearly effected by orientation, it will be left as another point of focus to be discussed later in this section.

A point to note in Figure 4.10 (c) is that, as the material stiffened and lost ductility over time, it caused some of the less ductile print orientation cases to fracture before they yielded. This explains why there are missing data points for some of the the 67.5° and 90° tensile coupons in that figure.
Figure 4.10: (a) Young’s modulus, (b) strain at fracture, (c) stress at yield point, and (d) stress at fracture in relationship to the number of days post finishing before testing for seven different print orientations.
Figure 4.11: Compressive modulus as a function of sitting time (referred to as dwell time in this thesis) between removal of part from the finisher to testing in accordance to ASTM standard D695-15 obtained from Bird [42].
As the effect of time appears to be independent of the orientation, the reason for this occurring will be considered from another standpoint. Previous studies by Bird [42] that looked at the same jetted photopolymer system, but in compression, recommended that parts be allowed to sit for six hours prior to testing to allow for the properties to plateau. He found that the compressive modulus started at 640 MPa when tested four minutes after removal from the finisher and rapidly increased and plateaued at around 1500 MPa when tested after an hour. This occurred as the samples cooled down from the 65°C of the finisher to room temperature. The results from this study are shown in Figure 4.11. The results of the present study in contrast, indicate that the mechanical behaviour of the jetted polymer is more sensitive to dwell time in tension than compression. This is shown by the large changes in mechanical properties over a span of eight days in Table 4.3 and Figure 4.10. This was the initial cause of difficulty in the understanding of the tensile results as large variations in properties were not expected beyond a day of dwell time and thus not taken into consideration.

The change of properties, particularly softening, due to elevated temperatures is expected for a polymer. Contrarily, stiffening of the polymer upon removal from the finisher at 65°C as it cools to room temperature is to be expected as well. The large 2.5x increase in stiffness after an hour of cooling from 65°C as seen by Bird [42] also correlates well with the glass transition temperature of the material of 64°C. As the finisher temperature is just above the glass transition temperature of the photopolymer, it is not unusual to see significant drops in the modulus [43]. A typical plot of modulus as a function of temperature for polymers is shown in Figure 4.12. The lack of stiffness is also observed physically in the tensile coupons as they are removed from the finisher.

However, the photopolymer was still found to increase in strength even after days and cannot be explained by the temperature effect. Thus, other aspects of the photopolymer must be considered. The photopolymer system being used consists of proprietary blend of ten or more precursors and thus complicates and convolutes different effects in play. The only known components of the system is that it is based on proprietary urethane acrylate oligomers combined
with ethoxylated bisphenol A diacrylate as a diluent, and tripropyleneglycol diacrylate as a photoinitiator [22].

From previous literature review on the post curing behaviour of urethane acrylate photopolymers in Section 2.1, it was shown by Tey et al. [24] that the modulus and glass transition temperature of photocured polyurethane acrylates did show increases over a period of ten days post cure indicating additional crosslinking. This was a result of moisture in the air reacting with isocyanate or cyanoacrylate groups present in the oligomer chains in a two step process to form further crosslinking. The increase in the material properties seen by Tey et al. [24] was large from day one to two, and had plateaued going from day two to ten, similar to the trend seen in Figure 4.10. Tey et al. [24] also found that regardless of the initial properties of their samples, which varied depending on the processing conditions they were subjected to during initial curing, the samples all followed the same trend and plateau over the ten days in ambient humidity. This explanation can similarly be applied to the tensile coupons being analyzed in this study which also show a post-cure increase and plateauing of properties over time.
Figure 4.13: (a) Young’s modulus, (b) strain at fracture, (c) stress at yield point, and (d) stress at fracture in relationship to print orientation for three different dwell periods of 1, 2, and 8 days post finishing before testing for seven different print orientations.
4.2.4 Effect of Print Orientation

While Figure 4.10 is able to effectively present the effect of dwell time on the mechanical properties, the effect of print orientation is obscured due to the fact that there is no orientation axis. Thus, although a loss of stiffness, ductility, and strength can be seen as the orientation varied from 0° to 90°, details and trends in the results are lost. As such, the data from Table 4.3 is plotted as a function of print orientation in Figure 4.13 to better highlight this. Contrarily, Figure 4.13 now washes out the plateau effect previously seen in Figure 4.10. Although it is clear for example that yield strength goes up from one to two to eight days of dwell time, the difference between one and two days, and two and eight days appear to increase by approximately the same stress value at each respective print orientation.

Overall, it can be seen that the mechanical performance decreases across the board as you deviate from printing parallel to the loading axis, confirming the presence of anisotropy. The cause of this performance loss was determined to be the result of the layer by layer method of manufacturing utilized in jetted photopolymers which creates inherently weaker interlayer interfaces.

During the jetting process, a layer of liquid photopolymer resin droplets are deposited onto the surface from the inkjet heads. This layer is then cured by a passing UV lamp that activate the photoinitiators initiating the polymerization reactions. These steps are repeated as necessary to build up the final part. However, each successive layer being printed will have a higher density of crosslinks laterally compared to the bonding to the underlying layer in the transverse direction that had already begun the curing process during the last UV pass. The lower degree in crosslinking between layers is a result of the limited amount and lifetime upon activation of the photoinitiators, and the number of sites still available for where a crosslink can be made to the underlying layer. This leads to a structure that is inherently anisotropic with strong intralayer performance and weaker interlayer bonding. The poor interlayer performance relative to the bulk of the material was previously shown in the nanoindentation indents between the layers of the 90° sample in Section 4.1.1. How loads are applied and resolved in a sample
relative to the layering direction will determine the overall performance.

The results discussed in the following paragraphs will focus on the samples tested after eight days of dwell time, shown by the green lines in Figure 4.13. The trends discussed will be applicable to both the one and two day samples as well but with different absolute numbers. The photopolymer is at its stiffest and strongest when the uniaxial tensile load was applied parallel to the layering direction (0° print orientation), as the tensile load is not acting to split the layer interfaces. The inverse is seen when the tensile load was applied perpendicular to the layering direction (90° print orientation) as now the interlayer bonds are subjected to the entire load. A schematic of the loading and layering directions was shown in Figure 4.9 for visual clarity. The 0° and 90° print orientation cases are analogous to loading in isostrain and isostress conditions respectively in fibre and layered composites. The comparison to fibre composites will further be expanded in Section 4.2.6.

The Young’s modulus and stress at fracture dropped from approximately 1750 MPa to 1540 MPa and 39.8 MPa to 32.9 MPa respectively in a relatively linear manner from 0° to 90° as more of the loading is being applied to the weaker and less stiff bonds between the layers within the sample. The same effect is seen in the stress at yield point, which dropped from 40.4 MPa to 38.1 MPa from 0° to 45°. Note that samples with print orientations higher than 45° tested after eight days of dwell time did not have yield points anymore as they fractured before that point. The drop in the stress at yield point with orientation can be attributed to the change in stiffness. As this photopolymer yields at approximately the same strain values regardless of orientation, the loss of stiffness going from 0° to 90° resulted in lower stress values at the same strain levels.

One of the details not previously seen in Figure 4.10 that is now clear in Figure 4.13 is the non-linear effect of orientation on strain seen in Figure 4.13 (b) and the aforementioned linear decrease in strength and stiffness with respect to orientation. There is a large drop-off in ductility with a small deviation from 0°, going from 14 to 6% elongation at fracture over a span of 33.75°. However, between 33.75° to 90° there is only a further drop of 3% total elongation
at fracture from 6% to 3% over the remaining span of 67.5°. The plateauing of ductility in the samples occurs at a strain level close to the yield point on the stress-strain curve showing the limited capability for plastic deformation between layers. In this plateau region, the ability to plastically deform is governed and dominated by the fracture of the weaker interlayer crosslinks. Thus ductility is limited as fracture initiated by the weak interface occurs before or shortly after the yield point. This change in failure mechanism from intralayer dominated to interlayer dominated was further explored in Section 4.2.6.

SEM micrographs of the tensile coupon at failure and their respective fracture surfaces for the 0°, 45°, and 90° print orientations shown in Figure 4.14 and Figure 4.15. The fracture surfaces shown are all brittle in nature. Although larger strain values are seen in the 0° orientation, showing more ductile behaviour, no necking occurs as the plastic deformation occurs globally within the entire gauge length. Fracture in these tensile coupons occur at a site of initiation and propagates perpendicular to the loading direction in a brittle manner. The fracture initiation site for the 0° and 45° orientations are apparent in Figure 4.14 and appears to propagate from the bottom right and bottom left corners respectively. The 90° orientation produced a much smoother fracture surface in comparison as the interlayer crosslinks were cleaved straight across.

Results for the four mechanical properties from Figure 4.13 are normalized with respect to the 0° print orientation in order to gauge the percentage performance loss with respect to orientation and presented in Figure 4.16. It also allows for the changes of the different properties to be compared to each other as change of 200 MPa in modulus cannot be compared to a 7 MPa drop in stress at fracture from 0° to 90°. By normalizing the results with respect to the number of days of dwell time, it is also seen that the drops in performance with increasing deviation from the print axis follow a near identical trend regardless of dwell time. That is, the percent loss in Young’s modulus, elongation at fracture, stress at yield point, and stress at fracture relative to the ideal 0° case should follow a similar trend to that presented in Figure 4.16 regardless of the number of days of dwell time from the finisher to testing. A rough rule of thumb for
Figure 4.14: SEM micrographs of tensile coupons at fracture for (a) $0^\circ$, (c) $45^\circ$, (e) $90^\circ$ print orientations, and their respective fracture surfaces (b), (d), and (f). A schematic diagram of the layered structure for each orientation is shown on the right.
Figure 4.15: SEM micrographs of the tensile coupon fracture surface from an angle for (a) 0°, (b) 45°, and (c) 90° print orientations. The layering structure is clearly visible on the bottom surface of each sample. A schematic diagram of the layered structure for each orientation is shown on the right.
the Young’s modulus, stress at yield point, and stress at fracture under a uniaxial tensile load for this photopolymer system appears to be a 0.2% loss per degree deviation from 0°. This allows for rough estimates of expected properties for different orientations at a specific dwell time if a 0° tensile coupon is tested at the same time. The same principle can be applied to the elongation at fracture of the material as well but with a non-linear fit as the ductility loss plateaus around 33.75°.

The four mechanical property plots in Figure 4.16 for samples tested with eight days of dwell time between finishing and testing are summarized in a polar plot in Figure 4.17. This allows for direct comparison of the four different properties to each other with respect to orientation. Polar plots for dwell times of one and two days will be nearly identical for the reasons previously mentioned and are not plotted for clarity. Figure 4.18 mirrors the polar plot of Figure 4.17 for the other three quadrants and is presented for visualization purposes. It can be seen in these polar plots that the stiffness and strength behaviour with respect to orientation both behave similarly and form a slightly eccentric oval as stiffness and strength at a 90° orientation dropped to approximately 80% of the 0° orientation value. The elongation at fracture curve however forms a much more eccentric oval as it drops below 25% of the initial value, highlighting the severity of the loss in ductility.
Figure 4.16: Normalized (a) Young’s modulus, (b) strain at fracture, (c) stress at yield point, and (d) stress at fracture in relationship to print orientation with respect to its $0^\circ$ value for three different dwell periods between finishing and testing.
Figure 4.17: A polar plot combining the normalized values of Young’s modulus, stress at yield point, stress at fracture, and elongation at fracture to the $0^\circ$ print orientation for tensile coupons tested with eight days of dwell time between finishing and testing.
Figure 4.18: A full 360° polar plot of Figure 4.17 for visualization purposes created by mirroring the figure onto the other three quadrants.
4.2.5 Notched Tensile Testing

One of the major advantages of 3-D printing is the ability to form complex architectures that might not be possible through conventional manufacturing methods. An example of this are the microtrusses discussed in the following subsection. However, with increasing complexity, there is usually more potential for stress concentration sites, as more features, discontinuities, or joints are incorporated. For example, going from a smooth solid structural beam for bending to a more structurally optimized and material efficient microtruss leads to many nodal points between the struts of the microtruss. These are areas that would need to be considered in its design as it would be more susceptible to local stress concentrations. It is important to understand how the role of orientation contribute to potential failure at these sites.

Tensile coupons of $0^\circ$, $45^\circ$, and $90^\circ$ print orientations were printed with simple right-angled triangular notches placed 25% into the gauge width to create a site of stress concentration in the middle of the gauge length. In addition, the $0^\circ$ coupon was printed in two different ways, one with the notch oriented upwards and one downwards. The different configurations are shown in Figure 4.19. The reason for the two differently positioned notches is to observe any effects of the wax support material deposited in the notch, as illustrated by the blue sections in the figure. In the material jetting process, samples are printed up vertically and require some sort of support underneath any regions with no material present. A low melt temperature wax is used as this support material as it can be melted off in post processing. However, as the wax and polymer resin are deposited as liquids, there is the potential for the two to mix when deposited adjacent to one another. This could lead to more surface roughness where the two meet as the polymer seeps away from its intended position or wax seeps into the area where the polymer is suppose to be. There is also potential for mixing when the two are deposited in successive layers but is less likely due to the cooling and solidification of the wax after deposition, and the hardening of each successive polymer layer by the scanning UV bulb prior to the next layer is printed.

The stress-strain curves for the notched tensile coupons are shown in Figure 4.20 and then
Figure 4.19: A schematic showing the print, layering, and notch orientation of the four types of samples tested in this subsection (a) $0^\circ$, (b) $180^\circ$ (0° with notch down), (c) $45^\circ$, and (d) $90^\circ$. The location of the wax support is shown in blue.

Table 4.4: Summary of mechanical properties from notched tensile coupons. The $180^\circ$ orientation refers to the $0^\circ$ sample with the notch facing downwards.

<table>
<thead>
<tr>
<th>Print Orientation ($^\circ$)</th>
<th>Young’s Modulus, $E$ (MPa)</th>
<th>Stress at Fracture, $\sigma_f$ (MPa)</th>
<th>Strain at Fracture, $\epsilon_f$ (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0</td>
<td>$1480 \pm 45$</td>
<td>$21 \pm 1$</td>
<td>$2.0 \pm 0.2$</td>
</tr>
<tr>
<td>180</td>
<td>$1470 \pm 40$</td>
<td>$21 \pm 1$</td>
<td>$1.7 \pm 0.1$</td>
</tr>
<tr>
<td>45</td>
<td>$1500 \pm 40$</td>
<td>$20 \pm 1$</td>
<td>$1.6 \pm 0.1$</td>
</tr>
<tr>
<td>90</td>
<td>$1460 \pm 20$</td>
<td>$13 \pm 1$</td>
<td>$1.0 \pm 0.1$</td>
</tr>
</tbody>
</table>

compared to their un-notched counterparts in Figure 4.21. The tensile property results are summarized in Table 4.4. From Figure 4.20 and Table 4.4, the notched samples have fairly similar ductility levels, failing between 1 to 2% strain. However, when compared to their un-notched counterparts, the loss in ductility for the notched $0^\circ$ sample from 15 to 2% is most apparent due to its high initial ductility. It effectively loses its entire plasticity range as it fails prior to reaching its yield point. Although the other samples did not show a similarly large drop in ductility as they were already typically below 5%, there is still a concern regarding the reduced stress at fracture. However, that is to be expected as the effective stress at the crack tip will be larger than the global stress. Interestingly, the $0^\circ$ with the notch facing downwards
Figure 4.20: Stress-strain curves for the four different notch orientations. The notch (wax) 0° line refers to the 0° coupon with the notch facing downwards.

Figure 4.21: The stress-strain curves from Figure 4.20 are replotted against a representative result of their un-notched counterpart to highlight the difference in performance.
Figure 4.22 shows the strain map from the digital image correlation test results for the four different types of samples tested just prior to fracture. The strain distribution of each type is plotted to the left of the scale bar indicating the percentage of the sample under a specific strain level. In the 90° sample, the strain concentration around the notch tip has barely formed as most of the remainder of the tensile coupon is at the same levels of strain away from the notch. However, it is already enough to initiate fracture across the interlayer interface at the notch. Any notch that is oriented parallel to the layering, thus leading directly into the layer interfaces, will severely compromise the overall strength as stress concentrates in an already susceptible area. Compared to the very local area of strain concentration at the notch tip seen in this sample, a large area of strain concentration was able to be formed at the notch tip and nearly covers the entire gauge width before fracture for the 0° case. This is evident by the large red butterfly shaped region around the notch tip in Figure 4.22 (d). There is strain build up as it is more difficult for fracture to occur across multiple different layers than it is for it to cleave across the interface between layers. The 45° has a strain distribution between the two extremes, along with the 0° notch down sample which was nearly identical. As previously mentioned, they both fail at roughly the same overall strain value, and as confirmed by the strain distribution, the same amount of strain at the notch. SEM micrographs were taken of the samples to observe and compare the differences in and around the notch to further explain the difference in strain at fracture for the two 0° cases.

SEM micrographs of the different samples at the notch are shown in Figure 4.23. Additional micrographs of the tensile coupon face in the notch region after fracture are shown in Figure 4.24. Due to both the effect of layering, and the location of the wax, different features can be observed in these micrographs. For the 0° orientation, a subtle difference can be seen between the notch up and notch down configurations. As both of these notches are printed as upwards and downwards pointing triangles, the notches are printed as staggered layers as seen in the schematics of Figure 4.19, thus producing stepped like features. A more detailed
Figure 4.22: Strain maps from digital image correlation results of a notched (a) $90^\circ$, (b) $45^\circ$, (c) $0^\circ$ with notch down, and (d) $0^\circ$ sample just prior to fracture. The strain distribution of each sample is plotted on the left of the strain scale bar with the coloured lines corresponding to the samples in the legend.
Figure 4.23: SEM micrographs of the notches from different print orientations (a) 0°, (b) 0° with downwards facing notch, (c) 45°, and (d) 90° before testing.
Figure 4.24: SEM micrographs of the notches from different print orientations (a) 0°, (b) 0° with downwards facing notch, (c) 45°, and (d) 90° after fracture.
explanation of these stepped features is provided in Appendix A. These stepped features are visible as the horizontal lines inside the notch in Figure 4.23 (a) and (b) but is more clearly distinguishable in the latter. The reason for this is due to the aforementioned wax deposition in the $0^\circ$ sample with the notch facing downwards. As the gauge is no longer a flat surface with a uniform layer of polymer being deposited onto a flat wax support layer, seen in Figure 4.19 (a), the notch is now printed as layers consisting of both liquid wax and polymer resin deposited in the same layer. Mixing at the interface created rougher stepped features than if there was no polymer/wax interface. This extra surface roughness would lead to slightly higher stress being exerted on the notch compared to the smoother stepping approximation seen in the notch without wax and is the reason for the poorer relative performance. Comparatively, the $45^\circ$ notch had no visible stepped features as the notch was oriented in such a way that the sides of the notch were either parallel or perpendicular to the layering, as seen in Figure 4.19 (c). The $90^\circ$ notch was also printed as a stepped approximation with wax support leading to large amounts of surface roughness as well. The roughness in the faces of the notch can also be seen from the sample side profiles in Figure 4.24. These micrographs were taken right after fracture during in situ SEM tensile testing. The samples on the right in Figure 4.24 (b) and (d), with wax incorporated into the notch have more distinct and rough edges whereas the edges of the notch on the left images of Figure 4.24 (a) and (c), are notably smoother. A rough estimation of the total ductility can also be made from the distance between the two fractured ends.

### 4.2.5.1 Notched Tensile Testing at Elevated Temperatures

To better understand the effect of orientation on failure through the layered structure, a preliminary proof of concept was developed for tracking the propagation of cracks through material jetted structures. The tensile testing in this subsection was completed utilizing an in situ tensile load frame and heating stage. The first set of testing done was completed on $0^\circ$ and $90^\circ$ tensile coupons with a center notch printed in it. The inserted notch had dimensions of 0.65mm (20% of the gauge width) by 0.16mm (25% of the notch width). The tensile curves from these
tests and a schematic of the notch and loading configuration are shown in Figure 4.25. The original goal of this test was to demonstrate the ability to effectively track crack size and rate of propagation via \textit{in situ} tensile testing. However, at room temperature, crack propagation occurs too rapidly to be imaged. Samples were further tested above the material glass transition temperature at 65°C in an attempt to slow the rate down. SEM micrographs from \textit{in situ} tensile loading are shown for the 0° and 90° notched examples are shown in Figure 4.26 and Figure 4.27 respectively. Preliminary results for the measurements of crack length for the 90° case versus strain are shown in Figure 4.28. Current limitations of this technique involve the scanning rate and resolution of the SEM. As the image raster process must be done fast enough to capture rapid changes in the sample during testing, the quality of the micrographs suffer and make it more difficult to visualize the actual crack length.

It was previously shown in Section 4.2.4 that under tensile loading, all samples fractured perpendicular to the loading direction regardless of orientation. It was to be determined if the print orientation played a role in direction of crack propagation and failure. As another proof of concept, it was shown that by leveraging the capabilities of additive manufacturing to introduce controlled flaws, it was possible to influence the direction of fracture. Tensile coupons were printed with a series of five diamond notches spread evenly at a 15° through the center of the gauge length. The notches were 0.325 mm (10% of the gauge width) by 0.160 mm (50% of the notch width). Again, these samples were tested at both room temperature and at 65°C. The tensile curves from these tests and a schematic of the notch and loading configuration are shown in Figure 4.29. These two cases were also studied using DIC to confirm if strain concentration followed the 15° line of notches. This is shown and confirmed in Figure 4.30. It is also noted that in the case of heated tensile testing, there is a strain gradient present under the heating stage as it only covers an area of 1x1 cm. This is present in Figure 4.30 (b), where there is a distinct change in strain from green in the center of the gauge length to the two blue shoulder regions. This is also confirmed by the strain distribution graph in the legend which shows two peaks in strain. The fracture of these samples produced a stepped surface in line
Figure 4.25: Stress-strain curves of the center notch tensile coupons tested at both room temperature and 65°C. A schematic of the notch and print orientations is provided as a reference. Note that the layering thickness is not to scale.
Figure 4.26: SEM micrograph of the $0^\circ$ print orientation tensile coupon with center notch just before failure. Crack growth can be seen in the top-left, top-right, and bottom-left corners of the notch.

Figure 4.27: SEM micrograph of the $90^\circ$ print orientation tensile coupons with center notch at the start of testing and prior to fracture. Crack growth can be seen in the top-right, bottom-left, and bottom-right corners of the notch. The crack lengths at the sites circled are tracked in Figure 4.28.
Figure 4.28: The stress-strain curve for the 90° print orientation tensile coupon with center notch tested at 65°C (black) and the crack length at the four corners as a function of strain. Crack a, b, c, and d, refer to the cracks forming in the top-left, top-right, bottom-left, and bottom-right corners respectively of the notch seen in Figure 4.27. Note that the layering thickness is not to scale.
Figure 4.29: Stress-strain curves of the diamond notched tensile coupons tested at both room temperature and 65°C. A schematic of the notch and print orientations is provided as a reference. Note that the layering thickness is not to scale.

with the introduced notches as shown by the SEM micrographs in Figure 4.31. In order to observe the entire gauge length and all five notches, the magnification was reduced, resulting in poor resolution of the crack length.
Figure 4.30: DIC strain map results showing the concentration of strain at a $15^\circ$ down the center of the gauge as a result of the five notches introduced under (a) room temperature and (b) $65^\circ$C, see Figure 4.29. The strain distributions for both the room temperature and heated samples are shown above the strain scale bar by the blue and red lines respectively.

Figure 4.31: SEM micrographs of a diamond notched tensile coupon (a) before and (b) after failure showing a stepped fracture surface.
4.2.6 Failure Mechanism and Comparison to Unidirectional Composite Laminates

Unidirectional composite laminates provide a good analogy to the effects of orientation seen in these tensile coupons as they too show large losses in mechanical performance when subjected to loading in the transverse direction. An example of this is shown for a glass fibre/epoxy unidirectional lamina as a function of fibre orientation in Figure 4.32. This failure strength envelope was generated under the maximum stress theory and takes the lowest values of predicted strength for each of the different failure modes. This plot has a very similar curve to that of the ductility versus orientation plot in Figure 4.13 (b) for the material jetted samples. The change of strength with orientation follows a sharp drop as it changes from fibre fracture to shear failure of the matrix and the matrix/fibre interface as it deviated from 0° [44]. A similar large drop in ductility was seen in the material jetted samples as it deviated from the 0° print orientation as well. At 0° the tensile load acts to fracture the individual layers, analogous to fibers in a laminate, and as you deviate from the optimal loading condition, the weaker interlayer crosslinks are acted upon more heavily which act as a point of failure. Following the shear failure regime in the laminates, there is a plateau into the transverse failure, as the matrix and matrix/fibre interface fail in tension. A similar effect in the ductility of the material jetted samples was seen as fracture became dominated by the weaker interlayer bonding at higher print orientations and the ductility drop plateaus as well.
4.3 Microtrusses in Three Point Bending

In the previous section, the effect of print orientation in jetted photopolymer was observed under the case of uniaxial tension. Change in mechanical performance was seen as the print orientation deviated from that of the loading direction resulting in losses in ductility and strength. As this change is inherent to this fabrication method, this effect will be observed in any part that is fabricated via material jetting. To further study this effect in other more complex structures and loading geometries, microtrusses structures were subjected to three point bending. In the case of a tensile coupon under uniaxial tension, the layering orientation remained consistent with regards to the loading direction. Conversely, with the microtruss architecture, struts are oriented in different direction leading to different layering orientations depending on the position of the individual struts. The different layering structure present in the struts is shown in Figure 4.33. A look at how all seven different microtrusses orientations are printed was previously shown in Figure 3.11. Furthermore, under a three point bending load, the location of a particular strut will play a role as well as the structure is subjected to tensile, compressive, and shear forces throughout, depending on location relative to the three points of loading.
Figure 4.33: (a) A model of a microtruss and representations of the layering structure seen in unit cells from microtrusses printed in (b) $0^\circ$, (c) $45^\circ$, and (d) $90^\circ$ orientations. Each unit cell is 6.25 mm in size. The overall length of the microtruss is 68.75 mm.
4.3.1 Three Point Bending Results

External factors effecting the variability in properties of the jetted photopolymer material such as batch variability and dwell time previously discussed in regards to the tensile results in Section 4.2.1 which are now understood were taken into consideration ahead of time. This allowed for a direct analysis of the effect of orientation on the properties of the microtrusses. In this study, a single batch of 35 microtrusses with five repeats for each of the seven orientations were tested. Representative force-displacement curves for the $0^\circ$, $11.25^\circ$, $22.5^\circ$, $33.75^\circ$, $45^\circ$, $67.5^\circ$, and $90^\circ$ orientations are shown in Figure 4.34. From first glance, there is also clear signs of anisotropy going from the $0^\circ$ to the $90^\circ$ samples in that the curves both deviate from linearity and end at earlier strain values.

![Force-displacement curve](image)

Figure 4.34: A representative force-displacement curve for seven different print orientations. A schematic diagram of the loading configuration is shown.
The results of the force-displacement data from the three point bending tests are summarized in Table 4.5 with the following four extracted parameters being used as the basis of comparison:

- $k$ (N/mm), stiffness, taken as the slope of the initial linear region
- $F_p$ (N), peak force supported
- $d_p$ (mm), midspan deflection at point of peak force
- $d_f$ (mm), midspan deflection at fracture

Table 4.5: Summary of results from microtrusses printed in various orientations loaded under three point bending. Values for stiffness, force supported at peak load, midspan deflection at peak load, and midspan deflection at fracture with their standard deviations are given for each sample orientation.

<table>
<thead>
<tr>
<th>Print Orientation $(^\circ)$</th>
<th>Stiffness, $k$ (N/mm)</th>
<th>Peak Force, $F_p$ (N)</th>
<th>Midspan Deflection at Peak Force, $d_p$ (mm)</th>
<th>Midspan Deflection at Fracture, $d_f$ (mm)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.00</td>
<td>20.9 ± 0.4</td>
<td>61.5 ± 0.8</td>
<td>4.9 ± 0.1</td>
<td>5.4 ± 0.1</td>
</tr>
<tr>
<td>11.25</td>
<td>21.2 ± 0.3</td>
<td>62.2 ± 1.0</td>
<td>4.8 ± 0.2</td>
<td>5.2 ± 0.1</td>
</tr>
<tr>
<td>22.50</td>
<td>20.8 ± 0.8</td>
<td>59.6 ± 0.5</td>
<td>4.7 ± 0.1</td>
<td>5.0 ± 0.1</td>
</tr>
<tr>
<td>33.75</td>
<td>20.7 ± 0.2</td>
<td>57.3 ± 0.5</td>
<td>4.4 ± 0.1</td>
<td>4.5 ± 0.1</td>
</tr>
<tr>
<td>45.00</td>
<td>21.4 ± 0.5</td>
<td>54.6 ± 0.4</td>
<td>3.9 ± 0.1</td>
<td>4.0 ± 0.1</td>
</tr>
<tr>
<td>67.50</td>
<td>21.5 ± 0.2</td>
<td>50.8 ± 0.5</td>
<td>3.6 ± 0.1</td>
<td>3.6 ± 0.1</td>
</tr>
<tr>
<td>90.00</td>
<td>21.5 ± 0.5</td>
<td>50.1 ± 0.8</td>
<td>3.4 ± 0.1</td>
<td>3.4 ± 0.1</td>
</tr>
</tbody>
</table>

4.3.2 Location of Fracture

Before further analysis of the three point bending results are done, the location of fracture in these microtrusses was first considered. By identifying the strut or node that leads to fracture, it is easier to compare the effects of orientation in the microtrusses with the previous tensile coupon results. Digital image correlation (DIC) was used to visualize the strain profile of a $0^\circ$ printed microtrusses at various deflection values as shown in Figure 4.35. It is apparent that there are areas of max compressive and tensile strains in the centers of the top and bottom face.
sheets respectively. Due to the colour scale of the strain values to better show the strain profile under lower deflection values of Figure 4.35 (a) and (b), extreme values are washed out in (d). A magnified and rescaled version of Figure 4.35 (d) is shown in Figure 4.36. The strain value of selected struts, labelled in Figure 4.36, are plotted as a function of midspan deflection in Figure 4.37. These includes a center strut in tension (points $a$ and $b$), strut in tension close to the center (point $c$), strut in tension far from the center (point $d$), core strut (point $e$), and strut in compression (points $f$ and $g$).

The points and struts of interest discussed in this paragraph refer to DIC figures 4.35 to 4.37. From the graph of strain versus deflection, it is seen that the center strut of the bottom face sheet is subjected to the highest amount of strain. It was confirmed from the test samples that this was the location at failure as all samples had failed right down the center unit cell, at either the left or right node. This will be a major point of consideration in the following section when comparing the effects of print orientation in three point bending to the effects in tension. The strain graph shows that the strut reaches 6.5% strain at point $b$, and 9% strain at the node at point $a$. As you move away from the center loading pin, the amount of strain with respect to deflection experienced by the structure gets progressively lower as seen by point $c$ one unit cell away and point $d$ three unit cells away. These struts experienced approximately 2.6 and 0.6% tensile strain at the point before fracture respectively. From the strain map, it appears all the core struts are under approximately the same strain. In one of the center core struts at point $e$, there is negligible strain experienced in the strut with respect to deflection. On the top face sheet, the strain at point $g$ deviates from point $f$ at about 2.5 mm deflection as the microtruss bends and the loading pin rolls slightly off center. The center region of the top face sheet is exposed to the highest compressive loading with approximately 3.2 and 5.5% compressive strain at points $f$ and $g$ respectively prior to fracture.

It is interesting to note how the strain at points $a$ and $b$ appear to deviate from linearity at approximately 3% tensile strain as it is around this strain value in the tensile stress-strain curve where it starts to yield. That means the strain being experienced in the center strut is
Figure 4.35: Digital image correlation (DIC) measurements of strain profiles in the struts of a microtruss under three point bending corresponding to deflection values of approximately (a) 3.5 mm, (b) 4.0 mm, (c) 4.5 mm, and (d) 5.0 mm. Note that there was a slight loss of data in the strut directly underneath the loading pin resulting in the black spot and discontinuity seen in the strain map at that location. This is due to the fact that that particular spot was either blocked out of view to one of the two DIC cameras by the loading pin or the shadow caused by the pin made it difficult for the DIC software to track.
Figure 4.36: A magnified and rescaled DIC image of Figure 4.35 (d) highlighting particular struts tracked in Figure 4.37. The strain distribution of the entire microtruss is plotted.

Figure 4.37: Strain values of struts corresponding to Figure 4.36. The four vertical dotted lines from left to right correspond to DIC images (a) to (d) in Figure 4.35 respectively.
not linearly proportional to the midspan deflection. As mentioned in the previous paragraph, the bottom center strut was determined as the point of failure. As point \( a \) tracks the maximum tensile strain in the microtruss with regards to deflection, an approximate maximum tensile strain sustained in microtrusses of different orientations can be estimated. This is done by looking for the particular value of strain for line \( a \) in Figure 4.37 at the expected deflection at fracture for that orientation. This value can be compared to the expected strain at fracture for a tensile coupon of the same orientation. This is summarized in Table 4.6. It can be seen that the two strain values are comparable for a given orientation between the three point bending and tensile testing results. This is in agreement with the layering direction of the struts in the bottom face sheet. For example, the center strut in the bottom face sheet of 0\(^\circ\) microtruss would experience a tensile strain parallel to the layering direction, thus behaving similarly to a 0\(^\circ\) tensile coupon. In contrast, the center strut of the bottom face sheet in a 90\(^\circ\) microtruss would have layering perpendicular to the tensile strain. This is shown in Figure 4.38. This particular strut in the microtruss will be one of the main points of discussion in the following section in regards to the effect of orientation and the similarities in performance to the tensile results.

Table 4.6: Approximate values from Figure 4.37 of max tensile strain expected in microtrusses of various print orientation at fracture based on its midspan deflection at fracture. The average elongation at fracture for tensile coupons of the same orientation is included for comparison. Estimated tensile strains marked with an asterisk are extrapolated values.

<table>
<thead>
<tr>
<th>Print Orientation (^\circ)</th>
<th>Three Point Bending</th>
<th>Tensile Strain at Fracture, ( \epsilon_f ) (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Midspan Deflection at Fracture, ( d_f ) (mm)</td>
<td>Estimated Max Tensile Strain @ ( d_f ) (%)</td>
<td></td>
</tr>
<tr>
<td>0.00</td>
<td>5.4 ± 0.1</td>
<td>13*</td>
</tr>
<tr>
<td>11.25</td>
<td>5.2 ± 0.1</td>
<td>11*</td>
</tr>
<tr>
<td>22.50</td>
<td>5.0 ± 0.1</td>
<td>9</td>
</tr>
<tr>
<td>33.75</td>
<td>4.5 ± 0.1</td>
<td>6</td>
</tr>
<tr>
<td>45.00</td>
<td>4.0 ± 0.1</td>
<td>5</td>
</tr>
<tr>
<td>67.50</td>
<td>3.6 ± 0.1</td>
<td>4</td>
</tr>
<tr>
<td>90.00</td>
<td>3.4 ± 0.1</td>
<td>3</td>
</tr>
</tbody>
</table>
Figure 4.38: The blue and red lines represent the direction of tensile loading in blue and direction of fracture respectively for microtrusses with print orientations of (a) 0° and (b) 90°.

4.3.3 Effect of Print Orientation

Results from Table 4.5 are plotted as a function of orientation in Figure 4.39. Although obvious signs of anisotropy can be seen in this figure in regards to peak force and midspan deflection, the trends have notable differences than those seen under uniaxial tension in Figure 4.13. In order to better understand the effect of orientation on these microtrusses, analogous properties to the tensile case will be grouped and compared. In order to do this, results from both studies were normalized and put into three groups:

- Stiffness parameter - Stiffness, $k$ (N/mm), for three point bending, and Young’s modulus, $E$ (MPa), for tensile loading
- Strength parameter - Peak load, $F_p$ (N), for three point bending, and stress at fracture, $\sigma_f$ (MPa), for tensile loading
- Ductility parameter - Midspan deflection at peak force, $d_p$ and fracture $d_f$ (mm), for three point bending, and elongation at fracture, $\epsilon_f$ (%), for tensile loading

The normalized results are shown in Figure 4.40. From this figure and Figure 4.39, the difference in performance versus orientation can be compared and contrasted with the tensile coupons. Under three point bending, the microtrusses did not suffer any notable losses in stiffness averaging values of 20.7 to 21.5 N/mm through the entire span of 0° to 90°. Under the
Figure 4.39: (a) Stiffness, $k$, (b) force at peak load, $F_p$, and (c) midspan deflection at peak load, $d_p$, and at fracture, $d_f$, in relationship to print orientation for microtrusses loaded in three point bending.
Figure 4.40: Normalized properties with respect to the $0^\circ$ print orientations for the microtruss three point bending samples (dark lines) compared with similar properties from the tensile coupons (light grey lines). (a) Stiffness, $k$, and Young’s modulus, $E$, (b) force at peak load $F_p$, and stress at fracture, $\sigma_f$, and (c) midspan deflection at peak load, $d_p$, at fracture, $d_f$, and elongation at fracture, $\epsilon_f$.

same span in tensile loading, losses of approximately 20% in Young’s modulus were seen. The insignificant changes in stiffness can be attributed to how the load is distributed and resolved through the network of struts. As opposed to the case of uniaxial tension where the load is always acting in the same direction relative to the layering direction, this is not the case in the microtruss. As seen in the DIC results in Figure 4.35, the strain distribution forms a gradient with max compressive and tensile strain in the centers of the top and bottom face sheets respectively. These strain values fall to zero moving away from the center loading pin in both directions. In addition, struts in the core have different layering directions than struts in the face sheets. As a result, the initial linear region of the curve does not noticeably deviate for the different orientations.

Anisotropy is more apparent in terms of strength and ductility, both of which decrease as the print orientation changes from $0^\circ$ to $90^\circ$. The max load supported by these microtrusses decreases almost linearly between $11.25^\circ$ to $67.5^\circ$ from approximately 62 to 50 N with the exception of the two extremes $0^\circ$ and $90^\circ$ which taper off. With the exception of these two points, the changes in strength correlate well with the tensile results both seeing loses of approximately 80% of their max strength. It is hypothesized that the reason for the tapering in
the microtruss case is due to the fact that under a bending load the center strut of the bottom face sheet is slightly angled in such a way that the tensile strain is no longer parallel to the layer orientation. This is shown in Figure 4.41 where there is approximately $6^\circ$ of deflection. That means that the direction of maximum tensile strain is parallel to neither the $0^\circ$ or $11.25^\circ$ layering in the bottom face sheet struts. Compared to the stress at fracture in tension, both suffer approximately a 20% reduction in strength going from $0^\circ$ to $90^\circ$ orientations.

![Figure 4.41: A magnified DIC image of Figure 4.36 showing the amount of deflection present in a microtruss under three point bending at 5 mm of midspan displacement. There is approximately $6^\circ$ of deflection at the center.](image)

It is more difficult to compare the deflection in three point bending with strain at fracture in tension. That is because as previously mentioned, the strain in some struts are not linearly proportional to deflection. However, from the estimated values of peak tensile strain at fracture for the microtrusses in Table 4.6 these values are more comparable to the strain at fracture seen in tension. When the expected strain value at fracture in the microtruss is plotted as a function of orientation, the same large loss in ductility from $0^\circ$ to $33.75^\circ$ and plateau afterwards in Figure 4.13 is seen. In Figure 4.39 (c), the difference between midspan deflection at peak load and at fracture gradually goes to zero from $0^\circ$ to $67.5^\circ$. This is due to the center strut in the bottom face sheet as it now fractures before it can yield, plateau, and continue to strain. This is the same effect seen in the tensile coupons which no longer show a yield point at print orientations of $67.5^\circ$ or higher.

The normalized parameters for the microtruss results were plotted into a single polar plot in Figure 4.42 to allow for direct comparison of the four different parameters. Figure 4.43 mir-
Figure 4.42: A polar plot combining the normalized values of stiffness, peak load, midspan deflection at peak load, and midspan deflection at fracture to the $0^\circ$ print orientation for microtrusses tested in three point bending.
Figure 4.43: A full 360° polar plot of Figure 4.42 for visualization purposes created by mirroring the figure onto the other three quadrants.
rors the plot into the three other quadrants and is presented for visualization purpose. Unlike
the previous polar plot for the tensile results, the stiffness and strength behaviour of the mi-
crotrusses with respect to orientation do not behave similarly. In this case, this is due to there
being minimal effect on stiffness as previously discussed. In regards to peak load and deflec-
tion, they both form slightly eccentric ovals as they both drop in performance going towards
90° with orientation having a bigger influence on both deflection parameters.

In this particular case of the microtruss in three point bending, it was seen that the trend of
orientation effects were similar to the trends seen in tensile for the most part. This is due to the
fact that in this particular case, the critical failure mode within the complex geometry is due to
tension. In addition, the loading direction in relationship to the layering direction is the same in
this case and for the tensile coupons. That is, for the 0° case, although the load is being applied
perpendicular to the bottom strut of the microtruss, the tensile force is acting on the same axis
as the strut and thus parallel to the layers. Similarly in a tension test, the tensile force would be
parallel to the loading direction and thus the layers. In the 90°, the tensile load would be acting
perpendicular to the layers in both tension and three point bending. A schematic of this for the
microtruss orientations is shown in Figure 4.38 and for the tensile orientations in Figure 4.8.

4.3.4 Effects of Scaling and Orientation

This section briefly discusses the possible expected effect of orientation on scaling as an ex-
tension to a previous study by Lausic [35] who looked at the effects of scaling on microtrusses
fabricated via material jetting. In that study, it was seen that microtrusses when scaled down
below a certain size showed a discrepancy between the predicted results and failed at a lower
load than expected as seen at $s = 1$ in Figure 4.44 [35]. It was hypothesized that when struts
were shrunk below a certain size, stochastic size effect would become relevant as the inherent
flaw length associated with the material jetting process approaches the strut size. This effect
was noticed up to a strut radius of 156 $\mu$m, about 5.4x the minimum layer thickness. If orien-
tation were to be taken into account, it is believed that moving towards a 90° print orientation
would prevent this discrepancy. As previously discussed in Section 4.2.2 the effect of these inherent flaws were more apparent in the 0° tensile coupons. This is not because they are more prone to these flaws than the other orientations but because the weakness due to the layering becomes less apparent. That is, when a stress is applied perpendicular to the layers, i.e., the 90° case, that becomes the weak link as opposed to any present flaws up to a certain size. For the case of a 90° printed microtruss, although the line for predicted strength based on size effect would be lower than that of the solid line in Figure 4.44, the experimental data is expected to match more closely at smaller size scales. However, this may cause another discrepancy at larger size scales. The initial reason for the size scale effect and hence the change from the dotted to solid line in Figure 4.44 is due to deterministic energy effects. As scale increases, the energy to extend a crack increases linearly with scale, whereas the energy released by stress relaxation has a quadratic relationship resulting in less stress required for fracture [35]. However, when the layers are closer to being perpendicular to the tensile direction, e.g. the 90° case, the energy to extend a crack is expected to be lower. That is, it requires less stress to fracture as it is easier for a crack to propagate between layers as opposed to propagating through multiple layers. A schematic of the direction of fracture and crack propagation for the 0° and 90° cases can be seen in Figure 4.38 (a) and (b) respectively.
Figure 4.44: Deterministic energetic size effect in polymer microtrusses obtained from [35]. Dashed lines represent the maximum strength of the material and the solid line represents the predicted strength accounting for size effect.
Chapter 5

Conclusion

This thesis explored the inherent anisotropies of material jetted structures through a wide range of length scales for seven different print orientations and how these anisotropies build up from one scale to the next. Starting at the interface level on the micron scale, the weakness in the interlayer bonding manifests itself all the way up into the materials tensile properties and in architectural structures fabricated with this manufacturing method. The poor interlayer performance is a result of the layer by layer printing process in which successive photopolymer layers are not as strongly bonded with each other as there is limited crosslinking compared to within and individual layer. This performance was investigated using nanoindentation to probe the hardness, and by correlation, the relative strength of the interlayer bonding. It was found that the hardness between the layers was approximately one third of the hardness of any other orientation. Microindentation was used to confirm the accuracy of the nanoindentation results by probing a larger volume of layers, thus giving hardness values that were not selectively influenced by the weak interlayer bonding. These hardness values were consistent across all print orientations and consistent with the nanoindentation results that did not demonstrate a relation to print orientation. The hardness values from microindentation also correlates well with the tensile yield strength later determined.

From the micro length scale results, the anisotropies here perpetuate upwards to the macro
length scale. Tensile coupons of the seven different orientations were tested and tensile properties demonstrated a strong relationship to the print orientation. Large drops in ductility were seen when the layering orientation deviated from being parallel to the tensile axis but the losses tapered off after a change larger than $33.7^\circ$ in the tested orientations. This trend and the change in failure mechanism from layer fracture to interface fracture is analogous to fiber and matrix fracture respectively in unidirectional laminate composites. In addition, near linear losses in modulus and strength were found in relationship to changes in orientation. Tensile properties were found to have a strong reliance on both batch variations and the dwell time between finishing and loading, but the effect of orientation was found to be the same regardless of amount of dwell time when normalized by the $0^\circ$ print orientation. That is, the percentage drop in tensile properties from $0^\circ$ to $90^\circ$ should be similar regardless of dwell time between testing. It is then recommended that extra $0^\circ$ tensile specimens be printed and tested with each batch of testing as a baseline for tensile properties. As a preliminary proof of concept for future work, notched tensile coupons were tested and the ability to measure and control crack propagation via in situ SEM mechanical testing was shown to be possible. Finally, how the various anisotropies found on the different length scales cumulated into the testing of a structure with higher internal complexity than that of a simple uniaxial tension test. The effect of orientation was explored in microtruss structures and showed that although the trends in anisotropy for the microtruss differed than for that of uniaxial tension, the failure mode is still controlled by the print orientation.
Chapter 6

Future Work

At the start of this thesis, it was hypothesized that the weak interface between the jetted layers would play a larger role in tension than compression in the same way that brittle ceramics are more sensitive to flaws in tension than compression. With the completion of the uniaxial tensile testing study and having sorted out all external effects affecting property variations, testing of anisotropy in compression and even shear testing is suggested for future work in order to fully understand the anisotropy of material jetted structures. In addition, it would also be interesting to note the differences in buckling performance between the different orientations. In addition, the focus of this thesis has been solely on the mechanical property of the material. Another aspect that can be investigated from the chemical side is the quantification of crosslinking through either dynamic mechanical thermal analysis or through swelling experiments an approximation of the percentage crosslink as a function of orientation and dwell time.

In this thesis, the preliminary work in utilizing the in situ SEM tensile module and it’s differences has been ironed out for future use. A preliminary proof of concept demonstrating the ability to investigate crack behaviour and fracture mechanics of the polymer has also been done. The advantages of being able to fabricate complex geometries and notches via material jetting can be leveraged and utilized to investigate how orientation effects manifests itself in areas of stress concentration. As additive manufacturing is typically used to fabricate complex
structures not possible by other means, stress concentration effects in relationship to orientation represent an area that warrants further work. This could range from more classical examples of fracture mechanics testing in accordance to ASTM or ISO standards such as a compact tension specimen or through various unique geometries, such as variations of the series of notches tested in this thesis, to control the direction of fracture. A couple of examples of unique notch patterns that could initiate atypical fracture paths are shown in Figure 6.1. Features such as these could allow for more detailed study of crack propagation with regards to layering and orientation in these materials.

Figure 6.1: Ideas for example tensile coupons with unique notching configurations.
Bibliography


als: a current state of the art on manufacturing, mechanical properties, and applications.  


Appendix A

Effect of Sample Curvature

Due to the inherent stepping of any curved surfaces in the layer by layer printing process, these areas may be more prone to failure as they can act as stress concentration sites. In the case of tensile testing, as the shoulder region which connects the gauge to the grip region is curved, the shoulder in some cases may be more prone to failure as it is approximated by the printer as steps. This can be seen in Figure A.1. Ten tensile coupons of 90° were printed and tested under the same methodology described in Section 3.3 but half were left with the shoulder region unsanded to observe the effects of the stepping. In Section 4.2, all samples were sanded in the shoulder region to remove these steps as the focus was on the determination of the orientation effect of printing. The 90° orientation was chosen as a basis of comparison as it puts the weak interlayer interface in the most susceptible loading condition, perpendicular to the applied stress. As the interlayer interface was the dominant mechanism leading to failure, it was to be observed if the stepped feature would further contribute to earlier failure. Stress-strain curves for this test is given in Figure A.2. Tensile strengths of 27 ± 3 MPa and 30 ± 2 MPa and elongation at fracture values of 4 ± 1% and 5 ± 1% were seen for the unsanded and sanded coupons respectively. As not all printed samples are ideally shaped for sanding, e.g. microtrusses, this is a factor that should be taken into consideration when testing material jetted parts.
Figure A.1: SEM micrograph illustrating the stepped approximation of the shoulder region.

Figure A.2: Stress-strain curves for the case of tensile coupons with the shoulder region sanded to remove any stepped features and for tensile coupons as printed.
Appendix B

Correction of *In Situ* Tensile Stage Output

The current *in situ* tensile machine dumps a constant stream of force-displacement data through a terminal console via the COM port. This output is difficult to work with, prone to errors, and requires corrections to ensure that digits are not dropped. The code provided automates this procedure by cycling through all .txt files in a local folder and delimits the columns and accounts for incorrectly outputted delimiters, trims unnecessary data points before and after the tests, and corrects for dropped or incorrect digits in the displacement data. The displacement data outputted can show spikes of ± 0.010, 0.100, or 1.000 mm in value not consistent with the actual crosshead movement. This script can be used for the entire range of the machine testing speeds of ± 0.2 to 1.3 mm/min and assumes a stream of 3.14 lines of data per second outputted from the terminal console. This script will output individual .xlsx files with columns for time in seconds, force in newtons, and displacement in mm. The required .txt input files can be simple copy and paste of the outputted terminal console window for a single test and does not require the manual trimming of data from before the tests and post fracture. Examples of data before and after correction for three tensile coupons, converted to stress-strain, are shown in Figure B.1.
clc
clear
close all
tic

% importing outputted serial port data
files = dir('*.txt');
errorfiles = [];
% note files that were not imported or processed correctly
delimiter = {'\t', ',', ';', 'N', 'mm', 'K1'};
formatSpec = '%s %s %s %s';

% looping through all .txt files and collecting values into dataArray, a 1x4 cell of +/-, force(N), +/-, displacement (mm)
try
    for i=1:length(files)
        fileID = fopen(files(i).name,'r');
        fprintf('%d of %d
',i, length(files))
        fileName = files(i).name;
        dataArray = textscan(fileID, formatSpec, 'Delimiter', delimiter,
            'MultipleDelimsAsOne', true, 'ReturnOnError', false);
            %dataArray is a 1x4 cell of +/-, force(N), +/-, displacement (mm) columns
        fclose(fileID);
        % find first point where data starts and clear text before this point
        arraystart = min(find(strcmp(dataArray{1}, '-') == 1, 1),
            find(strcmp(dataArray{1}, '+') == 1, 1));
        if isempty(arraystart)
            arraystart = find(strcmp(dataArray{1}, '+') == 1, 1);
        end
        if arraystart ~= 0
            for j = 1:length(dataArray)
                dataArray{1,j}(1:arraystart-1)=[];
            end
        end
    end

    % create Fddata with columns time (s), force (N) with proper polarity, and displacement (mm) with proper polarity)
    time = linspace(1,length(dataArray{1}),length(dataArray{1}));
    Fddata = [time cellfun(@str2num, strcat(dataArray{3},dataArray{4}))
        cellfun(@str2num, strcat(dataArray{1},dataArray{2}))];
    Fddata_precorrect = Fddata;

    % trimming Force
    for k=2:length(Fddata)
        deltaF(k-1,1) = [Fddata(k,3)-Fddata(k-1,3)];
    end

    % find where force drops by >10N, trim post fracture output
    endFindex = find(deltaF < -10);
    if isempty(endFindex) ~= 1
        Fddata = Fddata(1:endFindex-1,:);
    end

    % if force started in the negative, trim data to start at ON
    if Fddata(1,3) < 0
        startIndex = find(Fddata(:,3) >= 0,1);
        Fddata = Fddata(startIndex:end,:);
    end

    % correcting displacement values with incorrectly recorded values (+/-1.000mm, +/-0.100mm, or +/-0.010) due to machine error
    % loop to identify points where displacement jumps backwards as a result of a spike in displacement at the previous reading
errorindex=[1];
while isempty(errorindex) ~= 1
for l=2:length(Fddata)
    deltad(l-1,1) = [Fddata(l,2)-Fddata(l-1,2)];
end

errorindex = find(deltad < 0); % find index where displacement value is spiked causing a drop in displacement in the following index
% displacement should change by ~0.0008 to 0.007 mm/line normally but can have their values off by 0.010, 0.100, or 1.000mm due to a machine output error
for m=1:length(errorindex)
    if deltad(errorindex(m)) < -0.08 && deltad(errorindex(m)) > -0.5
        % deltad can range from ~ -0.093 to -0.100 mm for +/- 0.100mm output error
        Fddata(errorindex(m),2) = Fddata(errorindex(m),2) - 0.1;
    elseif deltad(errorindex(m)) < -0.001 && deltad(errorindex(m)) > -0.05
        % deltad can range from ~ -0.003 to -0.010 mm for +/- 0.010mm output error
        Fddata(errorindex(m),2) = Fddata(errorindex(m),2) - 0.01;
    elseif deltad(errorindex(m)) < -0.9 && deltad(errorindex(m)) > -1.5
        % deltad can range from ~ -0.993 to -1.000 mm for +/- 1.000mm output error
        Fddata(errorindex(m),2) = Fddata(errorindex(m),2) - 1;
    end
end

% shifting data so displacement value starts at 0.000 mm
lastzeroindex = find(Fddata(1:(find(Fddata(:,3) >= 10, 1)),3) == 0); %finding last ON value before 10N is reached, this index signifies the start of loading)
if isempty(lastzeroindex)
    lastzeroindex = 1;
end
Fddata = Fddata(lastzeroindex:end);end,:);
Fddata(:,2) = Fddata(:,2) - Fddata(1,2);
Fddata(:,1) = linspace(0, length(Fddata)/3.14, length(Fddata))'; % updating time column based on data output at 3.14 lines per second

% plot corrected and uncorrected force vs displacement
figure('visible', 'off')
hold on
plot(Fddata_precorrect(:,2), Fddata_precorrect(:,3),'r');
plot(Fddata(:,2), Fddata(:,3), 'b');
hold off

filename = [files(i).name(1:3),'.xlsx'];
saveas(gcf, strcat(filename(1:3),'.png'));
xlswrite(filename,Fddata);

% Clear temporary variables
clearvars dataArray arraystart Fddata Fddata_precorrect deltad deltaF
end

errorindex fileID filename j k l m lastzeroindex startindex time;
fclose('all');
end

catch ME
    errorfiles=[errorfiles files(i).name];
    rethrow(ME)
end

errorfiles % display files with errors
toc
Figure B.1: Stress-strain curves for three tensile coupons using the outputted force-displacement data from the in situ tensile machine before correction in black, and after correction in red, blue, and green. The corrections noted are the shifts to zero strain, removal of data after fracture (green curve), and the removal of different levels of introduced jaggedness in the displacement data for the blue and red curves. Note that the jaggedness before correction in the curves is a result of errors in the displacement data discussed in this section.
Appendix C

Tensile Machine Compliance Correction

With any tensile testing, the use of machine crosshead displacement as a measure of sample extension typically overestimates the true extension of the sample. The additional extension is contributed from the lack of stiffness in the machine, known as the machine compliance, and must be accounted and deducted for to determine the true extension in the sample. Without correction, the extra displacement recorded by the machine crosshead will result in reduced modulus and increased ductility values calculated due to the apparent extra deformation. Digital image correlation (DIC) was used to determine the true amount of displacement within the sample gauge to calculate the contribution from the machine compliance. Correction factors were determined for both the Shimadzu 5 kN EZ Test universal testing machine and the Kammrath Weiss in situ tensile module. Different correction factors are required for the two machines as the compliance values of each are not identical. An example of two stress-strain curves before and after correction are shown in Figure C.1. There is an obvious distinction between the two curves before correction due to the differences in machine compliance.

From the DIC results, it was found that the rate of elongation in the gauge was not linear and below the machine set rate of 1 mm/minute or 8% strain/minute. It was determined that the rate of elongation followed a bi-linear relationship with an increase in rate at the point of yielding in the sample. This is shown in Figure C.2 for three representative cases. It was also found
that the rate of elongation was also affected by the print orientation of the sample. This can be attributed to the difference in stiffness values of the different print orientations, as seen in Section 4.2. This is because the effect of machine compliance is increased when testing stiffer samples. To determine the compliance correction factors, DIC results from tensile testing of different print orientations were fitted by two linear approximations. Figure C.3 shows the corrected strain rates as a function of print orientation for both tensile machines used for both before and after yielding. In the case of samples where fracture occurs before yielding, only one strain rate correction factor is needed. Note that these corrected values are based off of and only valid for a machine set rate of 8% strain/minute. A table summarizing the corrected strain rates is provided in Table C.1.
Figure C.2: Actual elongation values in the gauge length for representative tensile coupons of print orientations: 0°, 45°, and 90°. A bi-linear fit is applied to approximate the rate of elongation in the two regions. Values were extracted from DIC.

Figure C.3: The strain rate in the tensile coupon gauge length as measured by DIC for both before (rate 1) and after yielding (rate 2) for the Shimadzu 5 kN universal tester and the in situ tensile module.
Table C.1: Compliance corrected strain rates for tensile loading at 8% strain/min for both the Shimadzu 5 kN universal tester and the *in situ* tensile module, for before (rate 1) and after (rate 2) yielding. Values marked with an asterisk represent extrapolated values.

<table>
<thead>
<tr>
<th>Print Orientation (°)</th>
<th>5kN Universal Tester (%/min)</th>
<th>In Situ Tensile Module (%/min)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Rate 1</td>
<td>Rate 2</td>
</tr>
<tr>
<td>0.00</td>
<td>3.29</td>
<td>5.00</td>
</tr>
<tr>
<td>11.25</td>
<td>3.32</td>
<td>4.95</td>
</tr>
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<td>22.50</td>
<td>3.36</td>
<td>4.90</td>
</tr>
<tr>
<td>33.75</td>
<td>3.39</td>
<td>4.85</td>
</tr>
<tr>
<td>45.00</td>
<td>3.43</td>
<td>4.80</td>
</tr>
<tr>
<td>67.50</td>
<td>3.49</td>
<td>4.72</td>
</tr>
<tr>
<td>90.00</td>
<td>3.56</td>
<td>4.63*</td>
</tr>
</tbody>
</table>