4D PRINTED AUXETIC STRUCTURES WITH
TUNABLE MECHANICAL PROPERTIES

by

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Dedication

To my family and friends...

Abstract

Additive manufacturing (i.e., 3D printing) has revolutionized the entire design cycle from prototyping, machining, and assembly. The majority of the materials suitable for 3D printing result in rather rigid structures with a fixed set of properties. Four-dimensional printing (4D printing) addresses this limitation through the use of active and smart materials during 3D printing. The careful selection of structural design and suitable stimulus-activated smart material enables a unique set of shapes and properties to be programmed and achieved. This deviation from rigid structures with a fixed set of properties enables novel and unique applications in robotics, deployable structures, biomedical, and aerospace industries. The current work is focused on an interesting class of structures, 2D auxetic cellular solids, which exhibit distinctive mechanical properties (e.g., negative Poisson's ratio) due to their carefully designed porous structure. 4D printing of such structures using smart materials allows for changes in the unit cell shape and dimensions to be made and thus resulting in tunable structural stiffness, Poisson's ratio, and rigidity. This work aims to experimentally investigate the tunability of stiffness and Poisson's ratio in 4D printed auxetic structure through utilization of programming and shape recovery properties found in Shape-Memory Polymers (SMP). Auxetic honeycomb structures with tunable mechanical properties were successfully manufactured and evaluated in this work. The attained structural stiffness was tuned in the range of 0.179-0.242 kN/mm while the Poisson's ratio was controlled from -0.33 up to even positive magnitudes of +0.69. This wide range of elastic properties was obtained from a single structure programmed to different deformation levels. Experimental evaluation of 4D printed SMP structures under constant cyclic programming/recovery conditions induced residual strains which consequently affected the mechanical properties by degrading the Poisson's and structural stiffness. The magnitude of induced residual strains depend on the level of applied deformation during programming and the number of applied programming/recovery cycles. At the local level, full-field measurements revealed the localization of strains within the complex heterogeneous structure of the axially loaded auxetic honeycomb samples.

Keywords: *4D printing, Shape memory polymers, Recovery strains, Auxetic structures, Cellular solids, Tunable properties.*

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Chapter 1. Introduction

1.1. Overview

Four-dimensional (4D) printing is a new method of manufacturing that utilizes additive manufacturing (3D printing) with the integration of active smart materials. Since the invention of 3D printing in the 1980s, it was applied in wide range of applications from biomedical science to space science [1]. One of the main advantages of 3D printing over traditional manufacturing techniques, is that it enables building of complex geometries with no mold requirements and minimal wasted material utilizing a wide range of materials including polymers, metals and ceramics.

The interest in using active and smart materials in conjunction with 3D printing (i.e. 4D printing) is rather recent. The term “4D printing” was first introduced by a research group at MIT back in 2013 [2] and it depends on the rapid change of dimensions, 3D printing and mathematical modeling [3]. It is considered an evolution of 3D printing where dynamic structures are fabricated with tunable shapes, properties and functionality [4-6]. The ability to program functionality and tune properties after printing enables the design of novel and advanced applications.

Active or smart materials are a category of materials that can alter their physical properties in response to a stimulus. Shape memory materials (shape memory alloys ‘SMA’ or polymers ‘SMP’) are smart materials that can recover their original shape after a deformation when a stimulus is applied (i.e. heat or magnetic field) [7]. Thermoplastic shape memory polymers (SMP) possess an advantage over alloys (SMA) for being lighter in weight [8], cheaper and having higher recoverable strain [7]. They also possess tunable properties that may be achieved by forming composites [9] or by utilization in cellular solids. In addition, SMPs can be implemented in Fusion-Deposition Modeling (FDM) additive manufacturing [10, 11], which opens the door for wide range of applications where shape-memory effect is desired in complicated shapes.

The working mechanism of SMPs starts with programming the original materials into a different shape by deforming it under a temperature higher than the glass transition temperature T_g , followed by cooling the deformed sample, which preserves its dimensions (programmed shape). The programming process is followed

by recovery process in which the programmed sample is heated back to above T_g , thus triggering the sample to revert back to its original shape (recovered shape). Despite significant efforts in studying various aspects and properties of SMPs, their utilization in 4D printing is recent.

On a different topic, cellular solids are materials made up of an interconnected network of solid struts or plates which form the edges and faces of cells [12]. They permit the simultaneous optimization of stiffness, strength and low density in a given application [13]. A two-dimensional cellular solid is also called a honeycomb, and it can be made of different cell structures such as hexagonal or triangular [12]. Honeycombs, like majority of materials in nature, exhibit positive Poisson's ratio. However, a modified hexagonal honeycomb with re-entrant angle can exhibit negative Poisson's ratio, making it an auxetic structure.

When an axial load is applied to auxetic structures, elongation occurs in both the axial and the lateral directions as shown in Figure 1.1 [14]. Auxetic materials and structures are of interest owing to their mechanical and damping characteristics which is important in many engineering applications. For instance, a non-auxetic cellular solid subjected to compressive impact loading deforms in the lateral direction such that the structural density, and therefore resistance and load carrying capacity, is reduced. On the other hand, in auxetic cellular solids, the structural density increases under similar compressive loading condition. This densification process which is induced by the negative Poisson's ratio of the structure increases the stiffness resulting in better damping characteristics, as shown in Figure 1.2 [15, 16].

This work provides a comprehensive literature review where related previous studies are discussed, followed by the methodology section in which details are provided on the experimental setup and all the methods used and finally concluded with results, observations and future work.

1.2. Motivation and Aim

4D printing of complex structures allows for innovative designs with tunable and shape memory properties. Since its introduction in 2013, the unique properties that can be achieved by 4D printing has attracted significant interest in the research

community. A quick search on Google Scholar using the term “4D printing” indicates the growth of research on the topic as shown in Figure 1.3.

Figure 1.1: Auxetic honeycomb in the deformed and undeformed shape. Horizontal elongation is applied which cause a lateral expansion rather than shrinkage. This phenomenon results in negative poison’s ratio. Adapted from [14].

Figure 1.2: Non-auxetic vs Auxetic material in case of compression. Compressive load cause densification in Auxetic material, yielding higher stiffness and better damping characteristics. Reproduced from [15].

Various aspects are currently active research topics in 4D printing including printing technology and suitable active materials. To date, few studies have utilized SMP filaments in fused deposition 3D printing (Fused Filament Fabrication, FFF) of different structures with shape programming and heat activated recovery properties [10,

11]. Although these efforts proved the concept, they did not provide clear understanding on the dependence of shape memory properties on 3D printing parameters, which leaves unanswered questions regarding local measurements of shape memory properties and their relation to additive manufacturing. In a study done by Abuzaid et al, an experimental investigation was done on a single hexagonal honeycomb cell to study the local shape recovery properties with high levels of heterogeneity in stored strain fields [17].

Figure 1.3: Growth of research on "4D printing" past few years.

The study provided in-depth quantitative analysis on the local strain recovery, heterogeneous material response and dimensional stability. However, detailed and quantitative analysis of multi-cell structures has not been addressed and will be therefore a focus in the current work. In addition, the mechanical behavior of additively manufactured SMP samples (e.g. Stiffness, Poisson's ratio, etc.) which are of great importance in engineering applications especially when tunable mechanical properties are feasible through the programming of SMP, are quantitatively evaluated using different experimental measurements techniques.

Accordingly, this work focuses on the utilization of SMP filaments in 3D printing of multi-cell hexagonal auxetic honeycomb structures and investigate the achieved tunable mechanical properties (Poisson's ratio and Structural stiffness). Through the use of full-field measurement technique, the work sheds insight into the global as well as local accumulation of residual strain following multiple programming

and recovery cycles. The degradation in mechanical properties is thoroughly investigated in relation to the levels of accumulated residual strains under multi-cycle conditions (i.e. fatigue) and varying programming levels.

1.3. Research Objectives

The goal of this work is to investigate the recovery behavior and tunable mechanical properties of programmed SMP-based 3D printed multi-cell auxetic honeycomb structures using different deposition patterns and infill densities. Accordingly, the following objectives are to be achieved:

- Utilize numerical methods in the dimensional optimization of auxetic honeycomb structures to maximize the structure mechanical properties while accounting for the experimental setup limitations.
- Provide in-depth overview on the response of 4D printed structures to functional fatigue, in particular, the effect of residual strains accumulated during programming and recovery cycles.
- Evaluate the mechanical properties (e.g. Poisson's ratio and structural stiffness) of a multi-cell auxetic honeycomb that is programmed to different shapes.
- Develop a deep insight into the local strain response of complex structure such as multi-cell auxetic honeycombs through full-field measurement techniques.
- Investigate the recovery behavior of multi-cell honeycomb structure.
- Investigate the effect of different deposition patterns and infill densities on the shrinkage of 4D printed samples.

Chapter 2. Background and Literature Review

2.1. 4D Printing

4D printing consist of ‘3D Printing + Time’ where the 3D printed structure can change its shape, property and functionality as a function of time [2, 4, 5, 18-20]. 4D printing is a revolutionized 3D printed structure, in terms of functionality, property and shape. Figure 2.1 presents the main differences between 4D and 3D printing [1]. Various applications have been demonstrated based on 4D printing principles, e.g., self-healing, multi-functionality and self-assembly.

Figure 2.1: The key differences between 4D printing and 3D printing. Adapted from [1].

Through 4D printing, dynamic structures can be manufactured with configurable shapes, functionality and properties [4-6]. This ability is primarily owed to the appropriate combination of smart materials or active materials [6]. 4D printed structure can have at least two stable states or shapes at which structure can shift from one to another using a corresponding stimulus [21]. 4D printing is built on the following main blocks; 3D printing facility [22], stimulus-activated material [23-25], interaction mechanism and mathematical modeling.

2.1.1. Shape-shifting: shape-memory vs shape-changing. The main target of 4D printing is shape transformation with time, this is done through the use of shape-shifting materials which can be categorized into two types; shape-memory materials and shape-changing materials. Shape-changing materials alter its shape directly after the application of a stimulus, and directly returns to its original permanent shape when the stimulus is removed. This type of shape shifting is limited to simple affine alterations (i.e. shrinkage and linear volume expansion) [26]. On the other hand, shape-memory materials consist of two-step shape-shifting; 1- Programming or Fixing step at which the material shape is shifted from its original shape to a new programmed shape, 2- Recovery step at which the material is excited with an appropriate stimulus to initiate recovery back to its original shape [27]. Therefore, a shape-memory material can assume a temporary shape until a stimulus is applied, while shape-changing materials cannot.

2.1.2. Materials used in 4D printing. The main limitation of 4D printing is the lack of proper materials that can answer the needs of the applications in terms of mechanical properties. For example, 4D printing can produce artificial muscle tissue, however, the mechanical properties of current materials are not sufficient to result in a desired functions of an actual biomedical muscle tissue [28]. Thus, it is important to develop materials with desired properties that are compatible with 3D printers as well. The research on development of new and appropriate smart materials for 4D printing purposes is rapidly growing. Momeni et al. have summarized the up to date development efforts in 4D printing materials with more than 20 different combination of materials that exhibit shape-shifting properties and are 3D printable [1].

2.1.3. Potential applications of 4D printing. The ability to program functionality and tune properties after 3D printing enables the design of novel and advanced applications. Many potential 4D printing applications were proposed in various interesting applications such as drug delivery systems [29], smart textiles [30], adaptive pipes [31], self-healing hydrogels [32], self-assembly at large scales and harsh environment [18], self-robots [33, 34], tissue engineering [35-37], error-correct and self-repairing structures [18] and construction and contour crafting on Mars [38].

2.2. Working Principle of Shape Memory Polymers

The majority of the polymers in the early years were studied as rigid structures with no dynamic properties. However, in recent years, research on polymers have advanced and resulted in development of polymers with special functions that respond to external environmental conditions (i.e. stimuli-responsive polymers) [39]. Such polymers exhibit behavior similar to that observed in biological materials [40].

Shape memory polymers are a class of stimuli-responsive shape-shifting polymers with a programmable shape-shifting behavior. Specifically, SMP's are distinguished with their ability to be fixed at a temporary shape (programming) and to return to its original shape (recovery). A typical shape memory polymer exhibits the ability to attain one programmed shape (Dual-shape memory effect), while other shape memory polymers can exhibit two programmed shapes (Triple-shape memory effect) [41, 42].

The mechanism of thermally induced dual-SMP is shown in Figure 2.2 [40]. The SMP typically exhibits a rigid plastic solid at room temperature, and upon heating to a temperature above shape memory transition temperature (T_{trans}), a rubber elastomer and easily deformed material is obtained due to the increase in mobility of the molecular chains. T_{trans} in most of polymers is considered to be the glass transition temperature T_{glass} of that polymer.

Using an external force to macroscopically deform the rubber state of the SMP alters the molecular chain configuration. Upon cooling to a temperature lower than T_{trans} , the macroscopic deformation induced to the material is preserved despite the removal of the external force. The decrease in temperature reduces the mobility of the molecular chains within the SMP and therefore locks the shape at what we call *programmed shape*. The programmed shape remains in a stable state as long as the temperature is below T_{trans} in the absence of external force. Once the temperature is increased to T_{trans} , the SMP recovers back to its original shape (i.e. *recovered shape*). The recovery phenomenon occurs when the stored entropic energy, which was induced during the programming phase, is released and therefore moves the molecular chains back to the position of lowest entropic state (i.e. original molecular chain configuration).

Figure 2.2: The molecular configuration of dual-shape memory polymer cycle. The black dots are position track dots. Blue lines correspond to the molecular chains at low temperatures (i.e. T_{room}). Red lines correspond to the molecular chains at transition temperature (T_{trans}). Adapted from [44].

2.3. 3D Printing Parameters

The optimization of 3D printing process parameters is a major challenge and of great influence on the properties of printed structures. The important parameters include deposition velocity, layer thickness, nozzle tip size, filament diameter, extrusion temperature and deposition pattern [43, 44]. It was also reported that 3D printed thermoplastic polymers shrink upon exposure to heat, which influences the dimensions of printed components when used in their respective applications [10, 11-17].

2.3.1. Printing pattern. The mechanical properties of 3D printed components are strongly influenced by the deposition parameter, and in particular, the deposition pattern followed to print the structure. Multiple approaches were considered to investigate the anisotropy of 3D printed structures, for example, some efforts examined the effect of object orientation by printing it either horizontally, vertically or at an angle, on the mechanical properties such compressive strength and stiffness [44-48].

de Ciurana et. al. investigated the effect of printing path on biocompatible scaffolds' modulus of elasticity by programming the printer to follow certain printing

patterns (e.g., longitudinal, 45° angled and transverse) while having the samples oriented on the same direction, indicated in Figure 2.3 [43]. It was concluded that deposition patterns will yield different porosities within the structure, which as a result, will significantly influence the mechanical properties such as the modulus of elasticity.

Figure 2.3: 3D printing deposition patterns. “Longitudinal”, “45 degrees angled” and “Transverse” (from left to right). Reproduced from [43].

Cantrell et. al. extended the work on the effect of anisotropy in ABS/PC 3D printed tensile samples by applying full-field measurement technique to quantitatively study various as-printed properties such as Poisson’s ratio, Shear modulus, Young’s modulus, YS and UTS [48]. The full-field measurements allowed the observation of local response within the printed samples which clearly showed the significance of printing pattern on the local deformation behavior. Cantrell et. al. concluded that anisotropy isn’t evident in all properties, and the behavior will differ for different materials. In general, the previous studies shed important insight into the macro-scale variation in properties. However, the local changes induced by altering the deposition direction, which induce the different macro-scale response, cannot be quantified.

The anisotropy in properties, which is inherent to 3D printed structures as explained above, experiences an additional level of complication when an active material is used in the deposition process (4D printing). For example, a study by Abuzaid et. al. proved the dependency of the members orientation on the recovery strain rate of SMP 3D printed structures in the case of single honeycomb cell structures [17]. The study showed that the free strain recovery behavior is dependent on the orientation of the members within the structure and the deposition direction. Despite the effort, the effect of deposition pattern (e.g., longitudinal, 45° angled and crosswise) on the shape memory and mechanical properties of 4D printed honeycombs remain uninvestigated.

Taking previous studies into account, the deposition pattern is hypothesized to have significant influence on the 4D printed component's mechanical and shape memory properties due to the change of the solid material density and the different internal material interactions within the structure solid members, which will be targeted in this work.

2.3.2. Infill density. The infill density corresponds to the amount of material used in printing pattern where the number of deposition lines and spacings in between them are varied to vary the infill density. Changing the infill density in FDM process can result in reducing the amount of material used, and therefore reduced cost. However, the reduced cost comes in exchange to mechanical properties such as Elastic modulus, Strength [49-52], Impact absorption capacity [53] and ductility where the increased voids in the part will lead to lower the mechanical properties. Figure 2.4 demonstrates the infill density for the case of longitudinal printing pattern.

Figure 2.4: 3D printing pattern infill density for the case of longitudinal print pattern. (Infill density decrease from 100% to 25% from left to right).

Fernandez, M et al. experimentally investigated the influence of infill pattern and infill density on mechanical properties FDM 3D printing [50]. Results showed that a rectilinear print pattern with 100% infill density exhibits the highest strength among other 18 different combination of different printing patterns and infill densities.

Another study by Rodríguez, A. et al. experimented the effects of the 3D printing parameters such as infill density, layer height and printing pattern on mechanical properties [51]. For the case of ABS (A thermoplastic type widely used in FDM), their experimental results showed that increasing infill density by 50% caused an increase in strength by 25.2%.

Considering previous work on infill density effects on 3D printed parts, it is expected that infill density will play a role in deciding the shape memory properties such as total recovery and recovery rate, as well as mechanical properties.

2.3.3. Shrinkage due to heat exposure. 3D printed parts are typically not exposed to temperatures around the glass transition temperature of the deposition material. However, 4D printed structures manufactured using thermoplastic SMP materials require such heat exposure to induce functionality into the part. This heat exposure during programming of 4D printed SMP results in an undesired shrinkage in dimensions, which is further amplified during the recovery process. Shrinkage and the corresponding dimensional change are affected by the material properties and the 3D printing process parameters such as deposition orientation.

On this matter, Abuzaid et. al. conducted experimental analysis on 4D printed SMP dumbbell samples to capture the shrinkage strain upon exposure to temperatures of 45, 55 and 65 °C [17]. Results showed the dependency of total shrinkage strain on the temperature with the relation being nonlinear, for example, total shrinkage strain was reported to be 5% at 45 °C, 12% at 55 °C and 23% at 65 °C. In addition, it was shown that shrinkage starts at high strain rate before stabilizing with a much lower strain rate, which highlights the importance of time in the shrinkage phenomenon. Although the shrinkage behavior is not directly addressed in this work, it is instrumental to understand the mechanism behind it to prevent dimensional instability during programming and recovery phases.

2.4. Properties of Auxetic Structure

2.4.1. Effective in-plane mechanical properties of auxetic structures. Since cellular solids vary in shape, size and materials, it is convenient to establish general comparison rules to help generalize the properties of such structures. Similar to normal honeycombs, the main non-dimensional properties of interest in auxetic structures are relative density, effective modulus and Poisson's ratio.

Relative density is a property that relates the density of a honeycomb cell to the density of the solid material, it is the most important structural characteristic of a cellular solid and it is given by the following formula [12]:

$$Relative\ density = \frac{\rho^*}{\rho_s} \quad (1)$$

where ρ^* is the density of the structure and ρ_s is the density of the material it is made of. As discussed in the previous sections, the deposition pattern alters the density of the solid material, which will have a direct impact on the relative density of the structure.

Poisson's ratio defines the degree of lateral strain with respect to the axial strain, and is given by:

$$\nu = -\frac{\varepsilon_{lateral}}{\varepsilon_{axial}} \quad (2)$$

The effective modulus of elasticity relates the stiffness of the structure to the stiffness of the material it is made from, and it is given by the following formula [12]:

$$Effective\ modulus = \frac{E^*}{E_s} \quad (3)$$

where E^* is the elastic modulus of the structure and E_s is the elastic modulus of the solid material the structure is made from.

To evaluate the structural properties, it is important to understand that cellular solids are meant to replace conventional materials, thus, they should be treated as a solid material when taking the properties such as elastic modulus and Poisson's ratio. This however, does not eliminate the need of studying the local response in the cellular solids.

In the past years, several studies were conducted to investigate the influence of structure parameters and dimensions on the in-plane mechanical properties in auxetic structures [54-58]. In majority of the researches, efforts are spent in formulation of the structural properties as a function of the dimensions, followed by numerical and experimental validation.

The negative Poisson's ratio in auxetic honeycombs comes on the expense of the relative density of the structure. The re-entrant members result in smaller cell in comparison with the normal hexagonal honeycomb, which increases the structure density, and thus, the relative density. Li Z reported the effect of cell size and Poisson's ratio on the elastic modulus and shear modulus in hexagonal honeycombs, it was shown that negative Poisson's ratio yields not only higher relative density, but higher elastic modulus as well [59].

2.4.2. Influence of number of cells. Most of the application that utilizes honeycomb structures require the use of bulk number of cells, thus it is important to take the number of cells into consideration when evaluating the in-plane mechanical and shape-memory properties of honeycomb structures. The main question: how many cells does a sample require to be provide representative properties of bulk sample of the material?

It has been widely reported that the number of cells and structure size directly influences the in-plane mechanical properties such as Poisson's ratio, modulus of elasticity and strength [59-61]. Li K et. al. conducted a numerical study on the influence of hexagonal honeycomb cell numbers on mechanical properties by considering nine families starting from 20 cells and up to 504 cells [62]. The effort showed a notable decrease in the effective modulus of elasticity as number of cells goes up, while effective Poisson's ratio was insignificantly affected. Another numerical study by Li Z et. al. investigated the effect of cell size on the young's modulus and shear modulus in honeycomb structures.

Judging by the previous studies, it is evident that increasing the number of cells in investigation of in-plane properties of honeycomb is of significant importance. However, increasing the cells is not always feasible in experimental studies due to various limitations in the setup. The limitations include the space available in the load frame and dimensional restrictions due to use of full field measurement techniques which requires certain surface area.

Accordingly, this work will present dimensional optimization considering the experimental limitation to maximize the number of cells in the honeycomb structure and other properties including elastic modulus and Poisson's ratio.

2.5. Tunability of Mechanical Properties in Honeycombs

The unique properties of SMP can be utilized in cellular solids to alter their shape and therefore, their mechanical properties. Programming of SMP components will preserve its shape temporarily giving it new dimensions, this can be advantageous in the case of auxetic honeycombs since their mechanical properties depend on the structural dimensions. As a result, the mechanical properties can be tuned by programming the original structure to new shape that exhibit different properties.

Despite some efforts to tune mechanical properties of some auxetic structures [63, 64], there is a lack in literature on the employment of heat activated smart materials to tune the mechanical properties of cellular structures.

Chapter 3. Methodology

3.1. Dimensional Optimization and Sample Sketch

It is known that different parameters of auxetic honeycomb members can alter the properties of the structure such as the Poisson's ratio and the stiffness. Ultimately, the selection of the geometric properties dictates the resulting structural properties of the structural solid. However, from an experimental perspective, various factors will limit the choice of dimensions; for example, thickness of the members should be wide enough to allow space for the speckle pattern to be applied (needed for full field strain measurements using digital image correlation) and the overall width and height of the sample is limited by the access glass window of the universal testing machine (UTM) environmental chamber (to capture optical images, also needed for DIC measurements). Such problems will require an optimization tool that can iterate and optimize the calculations given different inputs and outputs.

To achieve that, Abaqus ISight, an optimization tool that can be integrated with Abaqus CAE, was used to provide dimensional optimization based on minimizing Poisson's ratio and maximizing the stiffness. A single representative auxetic cell was implemented in Abaqus CEA and optimized using Abaqus ISight. The limitations of the dimensions were determined experimentally and are as follows: 1- Strut thickness is fixed at 3 mm, 2- Overall sample height and width are 137 mm and 120 mm, respectively. As explained earlier, these limits were selected based on the limitations imposed by the experimental setup.

Results of the optimization showed that for the given limitation, the lowest Poisson's ratio accompanied with highest modulus of elasticity were achieved at tilt angle and vertical strut length of 60° and 9.3 mm, respectively. The optimization curves of Poisson's ratio and elastic modulus are presented in Figure 3.1. It can be concluded from the surface curves that the effect of vertical length on the mechanical properties is relatively negligible in comparison with the angle. The drawings of all samples used in this work were done using Solidworks as shown in Figure 3.2. The dimensions of the fixtures are neglected since it is not part of the sample.

Figure 3.1: Optimization curves generated using Abaqus ISight for (a) Poisson's ratio and (b) Elastic modulus.

3.2. Sample Preparation

3.2.1. Material and printing parameters. The material used in 3D printing of the samples is a shape memory polymer obtained from SMP Technologies, Japan in the form of filament rolls with diameter of 1.75 mm. Fusion deposition process was conducted using the commercial FlashForge Finder 3D printer with deposition temperature of 200 °C as recommended by the filament manufacturer. The printing parameter selected in the FlashPrint software are layer height of 0.1mm, 100% infill density and maximum number of perimeter shell count. A layer of glue was applied on the printer pad to prevent breakage of the first layer and the temperature was set manually in the printer at the beginning of the process. Figure 3.3 shows the proposed longitudinal printing pattern by the software in comparison with the resultant as-printed sample. The settings were adjusted to assure deposition direction parallel to the cell

walls. This was done to allow for local strain analysis in cell walls with known deposition direction (i.e. deposition pattern aligned with cell walls).

Figure 3.2: Bulk and local dimensions of all samples, dimensions in mm. (a) 3x4 multicell auxetic structure. (b) 2x2 single cell auxetic structure. (c) Dumbbell sample. The fixture assumes high stiffness which is considered relatively rigid.

3.2.2. Curing of as-printed specimens. In 3D printed SMP components, exposure to heat will result in shrinkage in the dimensions as explained in previous sections in the paper. To avoid this, a curing step is implemented on the as-printed sample to allow shrinkage to take place before the test. Curing consists of heating the

specimen to glass transition temperature T_g for an extended period of time. In this experiment, the as-printed sample was cured at 55 °C for 200 mins by placing it horizontally and freely on a smooth plate in a furnace. Introducing this step eliminates the relatively rapid dimensional changes that have been shown to occur following initial heat exposure. Although this process does not really assure 100% stability beyond curing, the additional changes during programming and recovery, which require heating, are insignificant and do not alter the observations and measurements conducted in this work.

Figure 3.3: Printing direction. (a) Proposed printing pattern and direction. (b) Optical image of the printed sample. The pattern indicated in the figure is “Longitudinal”, which goes parallel to the members’ length.

3.2.3. Applying speckle pattern. The utilization of full field measurement techniques requires very careful application of speckle pattern on the surface of interest. Due to the relatively large area of measurement, the pattern was done manually using a black marker with tip size of 1 mm. Prior to pattern application, the as-cured sample surface was polished using different grades of sandpapers and polishing clothes, to obtain a straight and smooth frontal surface. Figure 3.4 shows a zoomed-in optical image of a cell within the structure.

3.3. Experimental Procedure

The experimental procedure of a single SMP cycle in this work consists of 3D printing SMP structures to test the recovery properties and tunability of mechanical properties. To achieve that, experiments were conducted in different phases, as shown

in Figure 3.5. In all cases the elastic properties were initially evaluated post curing by applying tensile deformation at room temperature. The mechanical properties were then altered/tuned through a programming process. In this step, the sample is heated to $T_g = 55^\circ\text{C}$ and then deformed to the required shape. Once deformed, the sample is then allowed to cool to room temperature which locks the deformation and results in a programmed shape having different mechanical properties compared to the original, as-cured, shape.

Figure 3.4: Optical image of a single cell with polished surface and applied speckle pattern.

Tensile loading was again applied on the sample to evaluate the tuned mechanical properties of the programmed shape. Subsequently, the programmed shape will undergo stress-free recovery by heating the sample back to 55°C . The mechanical properties of the recovered shape will be compared to the original shape, on which they are expected to have the same behavior. During all stages, images are captured using the optical camera connected to *Vic Snap* software using a capture rate of 1 image every 2 seconds. The same steps will be repeated for different samples that will be subjected to different programming levels and repeated cyclic programming/recovery cycles. All tensile is done on a constant displacement rate of 1mm/min . Figure 3.6 shows one programming and recovery cycle.

Figure 3.5: Typical test procedure for a Shape Memory cycle. 1- Test start with elastic tension test to evaluate elastic properties, followed by 2- programming, which consist of heating the sample to T_g temperature and deforming it to a new desired shape, followed by cooling to preserve its shape. 3- Elastic property evaluation is done for the programmed shape. 4- Stress-free recovery by heating the sample back to T_g . 5- Cycle ends with elastic property evaluation of the recovered shape.

Figure 3.6: Programming and recovery cycle of SMP 3D printed 3x4 multicell auxetic structure. Programming is achieved by heating the sample to T_g and applying 15 mm deformation using a uniaxial tensile force followed by cooling down to room temperature (RT). Recovery requires the sample to be free from boundary restrictions and is achieved by heating the sample back to T_g .

Chapter 4. Experiment Setup

4.1. Digital Image Correlation

Digital Image Correlation (DIC) is an optical and non-contact technique used for full field measurements of displacements and strains. DIC is extremely useful when local measurement of strains is difficult through traditional techniques (i.e. strain gauges), especially in complex structures such as cellular solids. The working principle of DIC is done by using multiple high-resolution images of a sample with speckle pattern undergoing continuous deformation [65]. The test images are compared to a reference image by tracking groups of pixels to detect the fine motion in the pattern. A scale is used to convert the motion from pixels to a dimensional unit, and thus, displacements and strains for the full pattern are obtained. For DIC to run effectively, it is important to have a pattern that is as random as possible to create unique groups of pixels. A representative image showing the speckly pattern used in this study is shown in Figure 4.1.

Figure 4.1: Digital image correlation working principle. A red box represents a selected subset where pixels are tracked and compared to the reference image.

4.2. Experiment Tools

The main testing tool used is Instron Universal Testing Machine (UTM) with 100 kN capacity equipped with environmental chamber. A high-resolution optical camera (PointGrey Grasshopper 3) is used to capture test images and correlation of the

images is done using the commercial DIC software *Vic2D* from *Correlated Solutions*, USA. Figure 4.2 shows the main components of the experimental setup.

To apply a tensile load to a sample of large cross-sectional area, a fixture made from stainless steel was used as indicated in Figure 3.2a, and the sample was tightly secured to the fixture with bolts and nuts. The load is assumed evenly distributed across the width of the fixture due to its relatively larger stiffness. A steel rod was attached to the steel fixture to be gripped by the load frame.

Figure 4.2: Experimental setup with labels of important test tools.

Chapter 5. Results and Discussion

5.1. Tunability of Elastic Properties in Dumbbell Specimens

To shed insight into the inherent tunable properties of the 4D printed structures investigated in this work, samples with simple geometry (i.e., dumbbell shape) were studied under simple uniaxial loading conditions. Specimens were subjected to two different loading conditions; multiple cycles with similar programming levels and multiple cycles with incremental increase in the magnitude of programmed strains. The first set of experiments were conducted to study the functional fatigue effects on material recovery while the later were aimed to address the influence of programming level on the tunable and recovery properties of the 4D printed structures.

A typical programming cycle consist of axial loading after heating the sample above glass transition temperature ($T_g = 55^\circ\text{C}$) followed by air cooling while holding the applied deformation (i.e. extension) constant. This process locks the deformation generating the *programmed shape*. To initiate shape recovery, the programmed sample is heated above T_g . It should be noted that the recovery process is temperature and time dependent, therefore, sufficient hold time at T_g was necessary to achieve high levels of recovery. A representative programming/recovery cycle is shown in Figure 5.1.

Figure 5.1: A single programming and recovery cycle done on dumbbell specimen. Programming was conducted using extension rate of 1 mm/min, while recovery was conducted stress-free for 200 minutes.

The reported strains were strains captured from the programming and recovery phases for the case of constant $\Delta L = 2 \text{ mm}$. The time taken for recovery is much longer than the time needed for programming, thus, the time axis in Figure 5.1 has been re-scaled for each phase to clearly show the transient during programming and stress-free recovery. Ideally, the recovery strain is supposed to be equal to the programmed strains which would result in full shape recovery and zero residual strains. However, in this case, about 0.5% of strains remains accumulated in the sample after recovery. It is expected that the accumulation of residual strains due to incomplete recovery can also affect the mechanical properties leading to variation between the properties of the *original* and *recovered shapes*.

Figure 5.2 presents the results of the constant programming cycling test at $\Delta L_{prog} = 2 \text{ mm}$, which corresponds to programming strain of about $\varepsilon_{prog} = 6.25\%$. The first cycle in Figure 5.2a showed a noticeable tuning in elastic modulus E as it dropped from 2025 (original) to 1605 MPa (programmed). This difference indicates the ability to alter the mechanical properties in 3D printed SMP. Upon heat-induced recovery, the elastic modulus exhibits about 96% recovery in stiffness with E increasing from the programmed level of 1605 MPa to 1940 MPa (original E was 2025 MPa). When adding the rest of the cycles as shown in Figure 5.2b, it is observed that the programmed shapes exhibited very close values to each other, however the recovered moduli slightly decreased from the original magnitude of 2025 MPa with continued programming/recovery cycles.

A summary of the measured elastic moduli for the 3 programmed and recovered cycles is shown in Table 5.1. As no clear trends in the results were observed, the variation can potentially be due to the variability/sensitivity of the experiment. However, it should be pointed out that the sensitivity of this material to temperature is of great influence on the properties as a small change in room temperature will lead to a notable change of elastic modulus. The results presented in Figure 5.2 and summarized in Table 5.1 were collected while exercising extreme care to assure comparable loading conditions, in particular, the loading temperature. All in all, the effect of programming cycles on the elastic properties of 3D printed SMP is very

minimal and can be neglected in case of low values of programming levels as considered in Figure 5.2 (about $\epsilon_{prog} = 6.25\%$).

Figure 5.2: Elastic modulus, E , as a function of shape memory constant programming cycles with $\Delta L=2$ mm. (a) First cycle. (b) Three combined cycles.

In the previous set of experiments, the goal was to demonstrate tunability in properties and investigate the functional degradation in the attained modulus following

multiple loading cycles (i.e. fatigue). In the second set of experiments, the emphasis is on mechanical and recovery properties across a wide range of programming levels (0% to 30.75%).

Table 5.1: Summary of results in Figure 5.2.

<i>Cycle</i>	<i>Shape</i>	<i>Accumulated Programming / Recovery strain</i>	<i>Relative Residual strain</i>	<i>Absolute Residual strain</i>	<i>E (MPa)</i>
As-cured	Original	-	0 %	0%	2025
1	Programmed	6.25%	0.76%	0.76%	1605
	Recovered	-5.49%			1940
2	Programmed	6.19%	0.51%	1.27%	1648
	Recovered	-5.68%			1842
3	Programmed	6.27%	0.68%	1.95%	1637
	Recovered	-5.59%			1818

Six different sets of programming levels were conducted over a range of 10 mm. The tunability of elastic modulus is evident in the stress-strain curves shown in Figure 5.3a. A summary of the elastic modulus for each programming level ΔL is shown in Figure 5.3b. The results clearly highlight a non-linear relation between the elastic modulus and programming level with changes in moduli becoming less noticeable at the higher programming levels (i.e. $\Delta L_{prog} > 2mm$). It should be noted that the experiments reported in Figure 5.3 were collected in rather a random order (0 mm $\rightarrow$ 2 mm $\rightarrow$ 4 mm $\rightarrow$ 3 mm $\rightarrow$ 10 mm $\rightarrow$ 1 mm) in order to assure that any detectible correlations in properties are primarily attributed to different programming levels and not due to cyclic loading.

5.2. Effect of Constant Programming Cycles on Elastic Properties in 2x2 Structure.

The behavior of 3D printed shape memory polymer under uniaxial loading was studied in the previous section where specimens underwent multiple constant and

incremental programming and recovery cycles. This section will shed insight into the functional fatigue behavior of a complex 4D printed auxetic honeycomb structure. Unlike the previous set of dumbbell specimens, the stress state, deformation fields, programmed and recovery strains are all expected to be highly heterogeneous due to structure complexity.

Figure 5.3: Tunability of elastic modulus in 3D printed Shape memory polymer. (a) Stress-Strain curves. (b) Elastic modulus behavior as a function of programming displacement.

The focus is again on tunability of mechanical properties with focus on the effect of cyclic programming/recovery cycles and the level of programming. In this section, a structure with 2x2 cell dimension (shown in Figure 3.2b) was 3D printed and tested for multiple constant programming / recovery cycles with constant programming level of $\Delta L_{prog} = 10 \text{ mm}$, which corresponds to a global programming strain of $\varepsilon_{prog} = 13.1\%$. The structure of the sample was selected based on the optimization results shown in Section 3.1. However, as explained earlier, the work aims to control the mechanical properties of the 4D printed structure through programming the geometry into various temporary shapes with varying properties.

Due to structure complexity, localized deformation and heterogeneous accumulation of strain takes place during programming and mechanical loading. It's therefore important to clearly distinguish assess the local as well as the global sample's response. Figure 5.4 shows a representative strain contour plot for the 2x2 cell structure following programming. Clearly the sample exhibits localized response with strains ranging from negative to positive magnitudes in different regions. The global response in this case is defined using virtual extensometers extending between the edges of the sample as shown in Figure 5.4.

Figure 5.5 shows the experimental data of programming and recovery phases done using a programming level of $\Delta L_{prog} = 10 \text{ mm}$ on the 2x2 (single cell auxetic structure). A total of 4 consecutive cycles were applied on the sample. The results indicate almost constant strain levels $\Delta \varepsilon_N$ accumulated during the programming phase of each cycle, with average value of 13.1% at the end of each cycle.

The absolute residual strain $\varepsilon_{R, absolute}$ is a measure of the total non-recoverable strain accumulating in the sample as a result of programming and recovery cycles and it is calculated with respect to the initial strain (zero in this case). The residual strain, which is the strain measured for a cycle with respect to previous cycle, shows a constant increase in value as cycles carry on, which indicates a constant relative residual strain of about $\varepsilon_{R, relative} = 2.4\%$. The relatively constant level of residual strain ($\varepsilon_{R, relative} = 2.4\%$) is not surprising due to nature of programming cycles (i.e. all programmed to about 13% strain, regardless of the presence of any residual strain).

Figure 5.4: Representative strain contour plot of 2x2 sample structure post 10mm programming with selected DIC contour plots. (a) Whole view of the 2x2 structure. (b) Y-direction displacement. (c) Y-direction strains, ϵ_{yy} . (d) X-direction strains, ϵ_{xx} . (e) Shear strains, ϵ_{xy} .

Figure 5.5: Programming and recovery constant cycles done on 2x2 sample using programming level of 10 mm for all cycles. The programming phase is done using displacement rate of 1 mm/min, while recovery phase was conducted for 200 minutes.

The important outcome of this experiment is to show the significant effect of programming and recovery cycles on the dimensions and mechanical properties of a 4D printed structure. The unrecovered strain due to programming and recovery cycles

was also addressed in a previous research using Veriflex-E shape memory polymer where it has been confirmed that shape memory shape recovery of Veriflex-E SMP decreases with constant repeated cycles due to residual strains accumulated with cycle repetition [66].

It is clear that the phenomenon of residual strain accumulation during shape memory cycles is not bound to 3D printed shapes and structures, but it is a characteristic of the material itself, despite the method of manufacturing. To further understand the effects of such incremental accumulation of residual strain on the mechanical properties of the 4D printed structures, the Poisson's ratio was evaluated following each programming and recovery cycles. The Poisson's ratio was calculated using normal and transverse extensometer as previously shown in Figure 5.4.

Figure 5.6 shows linear increase of absolute residual strain as a function of repeated constant programming and recovery cycles and its effect of Poisson's ratio in 2x2 sample. The absolute residual strains are extracted from Figure 5.5 and Poisson's ratios were calculated using virtual extensometers applied on the middle cell of the 2x2 structure. Poisson's ratio for Programmed shape is always higher than the recovered shape, this is attributed to the opening of the inclined members in the auxetic structure as will be discussed in local response section later on.

The key point in Figure 5.6 is the slow increase (negative effect) of Poisson's ratio due to the accumulation of residual strains. The residual strains after programming phase indicate partial recovery where dimensions of the shape are left in between the programmed and previously recovered shapes. Partial recovery in shape results in partial recovery in Poisson's ratio as well, as shown in Figure 5.6. The progression of cycling increases residual strain accumulation (from $\varepsilon_{R,absolute} = 0\%$ to $\varepsilon_{R,absolute} = 8.7\%$) which leads to an increase in the Poisson's ratio in 4D printed structure. It is important to note that increasing the Poisson's ratio in a negative-Poisson's ratio material indicates degradation in the property.

In addition to the Poisson's ratio, the change in Structural stiffness k as function of accumulated residual strain in 2x2 4D printed auxetic structure was evaluated as shown in Figure 5.7. Similar to Poisson's ratio results, the effect of residual strains on

the stiffness is evident. Both programmed and recovered shape increase in stiffness with residual strain accumulation. Programmed shape exhibits lower stiffness than recovered shape which is in accordance to the simulation presented in Figure 3.1.

Figure 5.6: Summary of Poisson's ratio results of 2x2 sample for recovered and programmed shapes and the effect of absolute residual strain on the behavior of Poisson's ratio.

Figure 5.7: Summary of Structural stiffness results of 2x2 sample for recovered and programmed shapes and the effect of absolute residual strain on the behavior of structural stiffness.

It should be noted that the ability to alter the stiffness through programming is not as efficient as the Poisson's ratio. For example, through programming of the first cycles, the Poisson's ratio changes from -0.57 to -0.38 which corresponds to ~33%

change. The structural stiffness on the other hand experienced only ~2% change under the same conditions. This observation was surprising as the change in structure angle due to programming results in change in effective modulus.

Although not fully explored in the current work, it is expected that stiffness reduction due to programming (shown for simple geometry in Table 5.1) may exceed the predicted stiffening effects due to structural change following programming of the 2x2 cell structure.

5.3. Effect of Increasing Programming Levels and Cycles on Residual Strains in 3x4 Structure.

Prior to elastic property tuning in 4D printed multicell auxetic structures where incremental programming levels are achieved, the behavior of residual strains is studied and quantified as a function of incremental programming levels. This is achieved through multiple programming/recovery cycles conducted on 4D a printed auxetic structure with 3x4 cell configuration as shown in Figure 3.2a.

Figure 5.8 shows the global strain data (i.e. global DIC strains) of programming and recovery phases of 3x4 sample (multicell sample) with increasing programming levels of ΔL_{prog} 5, 10, 15, 20 and 40 mm, which corresponds to global programming strains ε_{prog} of 4.4%, 8.4%, 12.7%, 17.2% and 34.7%, respectively.

The main difference between the constant (shown in Figure 5.5) and incremental shape memory cycles is in the trend of the residual strains. In the shape memory cycles with constant programming levels where relative residual strain remains constant, the response with incremental increase in programmed strains exhibited amplification in the relative magnitude of residual strain with increasing degree of deformation. Consequently, the rate of absolute residual strain accumulation (i.e. total accumulative strain) occurred at a faster rate.

In summary it is important to note that the relative residual strain is found to be a function of programming level as well as number of cycles. Therefore, the relation between the relative residual strain and programming level presented in Figure 5.9 includes the influence of both of these factors, however, with level of programming having more influence on the trends in this case. That is the more programming you put

in the sample during programming phase, the more relative residual strain is accumulated.

Figure 5.8: Programming and recovery cycles for a 3x4 auxetic sample using programming levels of 5, 10, 15, 20 and 40 mm. The programming phase is done using displacement rate of 1mm/min, while recovery phase was conducted for 200 minutes.

Figure 5.9: Effect of programming level on the relative residual strain accumulation.

5.4. Tunability of Elastic Properties in 3x4 Structure

The tunability in auxetic honeycomb is done using the shape memory properties in shape memory polymer. To achieve that, the 3x4 multicell sample is programmed to

a temporary shape by vertically deforming it to certain programming level ΔL_{prog} , which as a result, will change the structural properties such Poisson's ratio and structural stiffness. The tuned mechanical properties at each programmed configuration were tested by applying an elastic tensile loading at room temperature (2 mm extension). The Poisson's ratio is calculated through the slope of the global Lateral Strain vs Axial Strain curves (along the loading direction) curves while the structural stiffness is the slope of the global Force vs Displacement curves.

5.4.1. Tunability of Poisson's ratio. Figure 5.10a shows the Poisson's ratio for the first programming and recovery cycle (i.e. $\Delta L_{prog} = 5mm$). Original, programmed and recovered shapes all exhibited a typical negative Poisson's ratio, however the values varied for each phase. For the same sample, the Poisson's ratio is changed from -0.42 to -0.33 after programming, and from -0.33 to -0.41 after recovery. It is evident that Poisson's ratio of auxetic structure can be tuned through the utilization of heat activated smart materials such as shape memory polymers.

At very high levels of programming, the structure can even be modified to exhibit a positive Poisson's ratio. For example, Figure 5.10b shows the Poisson's ratio for an extreme case of deformation (e.g. $\Delta L_{prog} = 40mm$). During this programming level, the auxetic structure is converted to a traditional honeycomb structure. The Poisson's ratio in this case is changed from -0.35 to +0.69 after programming, and from +0.69 back to -0.31 after recovery. This indicates the feasibility of obtaining Positive Poisson's ratio structure through programming a Negative Poisson's ratio structure using shape memory polymer.

A summary of the results obtained from all programming levels is shown in Figure 5.11 for the 3x4 multicell auxetic structure. The inset images show the programmed structure at 0, 15 and 40 mm programming levels. Clearly, the images show the progression of the inclined member angle as programming level increase. The resulting Poisson's ratios of the programmed shapes are in accordance with the predicted behavior for the effect of the angle on the Poisson's ratio as indicated in Figure 3.1a.

The programming stage changed the Poisson's ratio through the change of the inclined members' angles. The recovered shape which is supposed to be similar to the

original shape, exhibited a slightly lower Poisson's ratio than the original shape. This is owed to the residual strains accumulated during programming and recovery phases, which were observed previously in Figure 5.6.

Figure 5.10: The tunability of Poisson's ratio on 3x4 sample for (a) First cycle ($\Delta L_{prog} = 5 \text{ mm}$) and (b) cycle with most deformation ($\Delta L_{prog} = 40 \text{ mm}$).

5.4.2. Tunability of structural stiffness. This section focuses on assessing the structural stiffness at the different programming levels. Figure 5.12a presents the results of structural stiffness for the first programming and recovery cycle (i.e. $\Delta L_{prog} = 5 \text{ mm}$). Programming of the original sample by 5 mm had minimal effect on

stiffness as it decreased from 0.181 to 0.179 kN/mm . The recovered shape, which is expected to be equal to the original shape, increased to 0.185, which recorded higher value than the original stiffness. This further confirms the results obtained in the constant cycle test where stiffness constantly increased as a function of shape memory cycles due to the accumulation of residual strains.

Figure 5.11: Summary of Poisson's ratio tunability for multiple programming levels done on 3x4 multicell auxetic structure. The insets show a representative cell of the programmed shape for selected programming levels.

Stiffness results in the extreme case of 40 mm programming level are shown in Figure 5.12b. Stiffness increased from 0.177 to 0.242 kN/mm after programming, and it decreased from 0.242 back to 0.160 kN/mm after recovery. The stiffness trend of the programmed shape follows the simulation trend obtained in Figure 3.1b, where structure angles of 100° or higher will have a relatively higher stiffness.

Figure 5.13 shows a summary of structural stiffness as a function of programming level. The stiffness of programmed and recovered shapes showed minimal difference at low programming levels (i.e. 15 mm and less). The difference starts to deviate with higher levels of programming. The structural stiffness parabolic trend of the programmed shape followed the predicted behavior shown in Figure 3.1b for high values of inclined angle. Although programming to high inclined angles in honeycomb structures will lead to significant increase in structural stiffness, it comes with expense to the loss of Negative-Poisson's ratio property.

Figure 5.12: The tunability of Structural stiffness on 3x4 sample for (a) First cycle ($\Delta L_{prog} = 5 \text{ mm}$) and (b) cycle with most deformation ($\Delta L_{prog} = 40 \text{ mm}$)

5.5. Local Response of Multicell Structure

The results discussed in the previous sections focus on the global or average response of the 4D SMP structure. However, due to complexity in the configuration of auxetic structure, localizations in strains (programmed, deformed and recovered) will be developed. These localizations in strains relate to the accumulation of residual deformation and are therefore important to quantify and understand. The observations on local response were done during tensile loading and during programming and recovery cycles.

Figure 5.13: Summary of Structural stiffness tunability for multiple programming levels done on 3x4 multicell auxetic structure. The insets show a representative cell of the programmed shape for selected programming levels.

5.5.1. Local response due to axial tensile loading. To visualize and quantify the localization of multi strain components within the structure, local strain contour maps of multicell 4D printed auxetic structure are shown after the application of different levels of bulk vertical deformation. Furthermore, the heterogeneous member deformation behavior is quantified using virtual extensometers to better understand the effect of programming level on local deformation in differently oriented members within the structure.

Figure 5.14 shows the local strain response of 3x4 sample after the application of 5 mm bulk vertical deformation. It is notable that strain localization is heterogeneous within a single cell, however, the strains exhibit a consistent repetitive distribution when considering a multi-cell structure. The main regions of strain concentrations are found on the vertices and sharp edges in the structure as indicated in Figure 5.14b by horizontal strains accumulations.

It is also important to note that vertical members suffered small amount of strains, relative to vertices and edges, which is a strong indication that the bulk deformation of the whole structure, in the case of 5 mm extension, is mostly attributed to the structural mechanism of auxetic honeycomb and that the solid material properties

had minimal contribution to the bulk deformation. This, however, might not be the case for larger amounts of bulk deformation.

Figure 5.14: The local strain response of multicell SMP 3D printed structure to bulk deformation of 5 mm. (a) y-direction strains ϵ_{yy} (b) x-direction strains ϵ_{xx} (c) Shear strains ϵ_{xy} .

In order to better quantify the levels of deformation in the horizontal versus the vertical elements, virtual extensometers were implemented to capture the local axial

strain of the members of a selected cell. Auxetic honeycomb structures despite complex look, they consist of only few repetitive and symmetrical patterns. Accordingly, only two representative directions were selected for the analysis. The average of each set of extensometers (2x vertical and 4x inclined) was taken for the analysis.

Figure 5.15a presents the local member axial strain of a representative cell within the 3x4 multicell structure at 0 mm programming level (elastic loading). The results show tensile strain in the vertical directions peaking at 0.20% and compressive strain in the inclined directions peaking at 0.13%. The compressive strain in the inclined directions is an indicator of a negative Poisson's ratio. Despite the bulk tensile strain, auxetic structures exhibit complex combination of local tensile and local compressive strains.

Figure 5.15: Local member axial strain of a representative cell within the 3x4 multicell structure at (a) 0 mm programming level (b) 40 mm programming level.

Figure 5.15b shows the local member axial strain of a representative cell within the 3x4 multicell structure at 40 mm programming level which is the case of positive Poisson's ratio (elastic loading subsequent to programming). The vertical directions suffered tensile strain peaking at 0.21%, a close value to the 0 mm programmed shape. The response of the inclined directions also showed a tensile strain peaking at 0.09%. The local response can be used as an indicative to Poisson's ratio of the bulk structure, that it when inclined directions suffer compressive, Poisson's ratio is expected to be negative and vice versa.

Figure 5.16 shows a summary of local member axial strain of vertical and inclined directions as a function of programming level. The reported strains represent the peak strains at the highest applied load. The vertical directions barely suffer any change as programming level increases, which cancels their effect on the tunability of Poisson's ratio and structural stiffness in the bulk structure. On the other hand, inclined directions show a linear transition from compression to tension as programming level increase. This indicates that the main contributors to elastic property change are the inclined directions. Controlling the strain in inclined directions can also be used to control the elastic properties due to their direct relation with programming level.

Figure 5.16: Summary of local member axial strain of vertical and inclined directions as a function of programming level.

5.5.2 Local response due to incremental shape-memory cycles. Previous sections showed residual strain accumulation on the global level, which had significant effect on the degradation of mechanical properties in the 4D printed auxetic structure. To clarify the source the global residual strains, it is important to track down the residual strains on the local regions. Figure 5.17 addresses this aspect by clearly showing the local residual strains for a selected cell within the multicell structure. It is clear that after programming steps of 10mm or 40mm, a small local residual strain is present in the structure, most of it being accumulated in the inclined members. Therefore, it is necessary to quantify the accumulated strains to develop a clear understanding on the programming and recovery behavior of the local members.

To do that, extensometers were used on the main two directions present, vertical direction and inclined direction. The axial strains in the members were quantified during programming and recovery for the case of incremental shape-memory cycles with bulk programming levels of 5mm, 10mm, 15mm, 20mm and 40mm. The experimental data of programming and recovery strains for the vertical direction are presented in Figure 5.18 and summarized in Table 5.2. The bulk programming deformation induced axial tensile strains in the vertical members that are proportional to the bulk programming strain values.

The behavior of recovery in the vertical members show an over-recovery (further axial shrinkage) which is the opposite of that observed for the bulk recovery behavior as shown previously in Figure 5.5 and Figure 5.8. This is thought to be further shrinkage during stress-free heat exposure and is strongly dependent on the printing pattern used in the local members. A small peak at the beginning of each recovery curve indicates thermal expansion prior to recovery, which was not observed in the bulk recovery behavior. Although the bulk programming strain induce tensile strains in the vertical members, the absolute residual strain accumulated within the vertical members are compressive and will contribute to reducing the bulk vertical dimension of the structure. Results in Table 5.2 indicate the stability of the local absolute residual strain as number of cycles increase.

For the case of inclined members, the experimental data of programming and recovery strains are presented in Figure 5.19 and summarized in Table 5.3. For the same

bulk programming strains, inclined members suffer compressive strains that are higher in magnitude than the tensile strains found in the vertical members. This further proves the heterogeneity of the auxetic structure at the local level. The recovery behavior of the inclined members is also different than the usual bulk recovery behavior.

Figure 5.17: Local residual strain accumulation during programming of 10 mm and 40 mm. Higher programming level accumulates more local residual strain.

Typical recovery is observed at the beginning of the heating, however an observed relaxation takes place later on. It is clear that the relaxation is due to further

local member shrinkage which is owed to the printing pattern of the member. Table 5.3 indicates that further axial shrinkage in the local member tends to stabilize as number of cycle increase. The shrinkage in inclined members results in decreasing the unit-cell angle, and therefore increase the bulk vertical dimension of the structure.

Figure 5.18: Programming and recovery cycles for the vertical members of a selected cell within the 3x4 auxetic structure for programming levels of 5, 10, 15, 20 and 40 mm. The bulk programming phase is done using displacement rate of 1 mm/min, while recovery phase was conducted for 200 minutes. A small cell is shown to indicate the location of extensometers.

Table 5.2: Summary of local residual strain accumulation in the vertical members for the case of multicell auxetic structure with incremental programming levels.

<i>Programming level (mm)</i>	<i>Phase</i>	<i>Accumulated Programming / Recovery strain</i>	<i>Relative Residual strain</i>	<i>Absolute Residual strain</i>
5	Programming	0.52%		
	Recovery	-1.1%	-0.58%	-0.58%
10	Programming	0.86%		
	Recovery	-1.3%	-0.44%	-1.02%
15	Programming	1.1%		
	Recovery	-1.2%	-0.1%	-1.12%
20	Programming	1.6%		
	Recovery	-1.5%	0.1%	-1.02%
40	Programming	2.7%		
	Recovery	-2.8%	-0.1%	-1.12%

Figure 5.19: Programming and recovery cycles for the inclined members of a selected cell within the 3x4 auxetic structure for programming levels of 5, 10, 15, 20 and 40 mm. The bulk programming phase is done using displacement rate of 1 mm/min, while recovery phase was conducted for 200 minutes. A small cell is shown to indicate the location of extensometers.

Table 5.3: Summary of local residual strain accumulation in the inclined members for the case of multicell auxetic structure with incremental programming levels.

<i>Programming level (mm)</i>	<i>Phase</i>	<i>Accumulated Programming / Recovery strain</i>	<i>Relative Residual strain</i>	<i>Absolute Residual strain</i>
5	Programming	-0.53%	-0.45%	-0.45%
	Recovery	0.08%		
10	Programming	-1.0%	-0.2%	-0.65%
	Recovery	0.8%		
15	Programming	-1.5%	0.0%	-0.65%
	Recovery	1.5%		
20	Programming	-1.9%	0.0%	-0.65%
	Recovery	1.9%		
40	Programming	-3.8%	-0.2%	-0.85%
	Recovery	3.6%		

Although both directions were opposite in strain behavior, both resulted in shrinked members which indicates the dominance of shrinkage over the local residual strains. It can be concluded that the main influence of bulk residual strains in the structure is due to the local axial strain accumulation within the members which are influenced by the shrinkage during extended heat exposure.

5.6. Effect of Printing Parameters on As-Printed Sample Shrinkage

It was discussed earlier that FDM 3D printed shape memory polymers experience significant shape and dimensional changes when exposed to heat for the first time. This phenomenon is what we call “shrinkage” and it was first analyzed by Abuzaid et al in 2018 [17]. Many previous studies proved the direct effect of print pattern and infill density on the mechanical behavior of traditional FDM polymers. From this, it is predicted that printing pattern will have a direct influence on shrinkage behavior in FDM 3D printed shape memory polymers.

This section consists of two main studies; the effect of print pattern on shrinkage strains and the effect of infill density on shrinkage strains. The strain components of interest are the vertical component ε_{yy} , horizontal component ε_{xx} , and shear component ε_{xy} . A final summary is done on all results using the effective strain to compare the severity of each print pattern and infill density on the resulting shape stability during first-time heat exposure.

5.6.1. Effect of printing pattern. Shrinkage test was conducted using 4D printed shape memory polymer dumbbell specimens with four main printing patterns; Vertical lines (0 deg), Horizontal lines (90 deg), ‘+’ shaped grid lines (‘+’ grid) and ‘×’ shaped grid lines (‘×’ grid). The test consisted of holding the specimen vertically (stress-free) in an environmental chamber where temperature was held at 55 °C for 200 minutes.

The shrinkage results of 0 deg specimen are shown in Figure 5.20. Strain results show significant strain reduction in vertical component reaching -22%, and strain expansion in the horizontal component reaching 15%. Shrinkage showed minimal effect on the shear strain component.

Figure 5.21 shows the shrinkage test conducted for printing pattern of (90 degrees). This pattern consists of parallel lines directed 90 degrees from the (0 degrees) print pattern shown in Figure 5.20. Strains showed significant reduction in the horizontal component reaching -10% while vertical component experienced minimal shrinkage of -2%.

Figure 5.20: Shrinkage DIC strains averaged for a 3D printed shape memory polymer dumbbell specimen printed with (0 degree) printing pattern.

Figure 5.21: Shrinkage DIC strains averaged for a 3D printed shape memory polymer dumbbell specimen printed with (90 degree) printing pattern.

From Figure 5.20 and Figure 5.21, a conclusion can be made that shrinkage in 3d printed shape memory polymers strongly favors the directions of the printing lines. Another important observation in the pattern is that both the horizontal and vertical strain components showed reduction while the (0 degree) print pattern showed vertical reduction and horizontal expansion. Shrinkage had minimal effect on shear strain component with it being constant at 0%.

Shrinkage results of ('+' grid) printing pattern are presented in Figure 5.22. This print pattern consists of a combination of last two print patterns repeated layer by layer as shown in the pattern demonstration in Figure 5.22. In this case, vertical strain component showed more severe reduction than the horizontal component, which is the opposite to the results obtained in Figure 5.21 for the (90 degree) printing pattern. Both the vertical and horizontal strain components showed reduction reaching -13% and -3.5% respectively. Again, shear component was not changed during shrinkage test.

Figure 5.22: Shrinkage DIC strains averaged for a 3D printed shape memory polymer dumbbell specimen printed with ('+' grid) printing pattern.

Figure 5.23 shows the shrinkage results of ('×' grid) printing pattern. This print pattern is very similar to the ('+' grid) print pattern with the lines being rotated by 45 degrees with respect to reference coordinate system shown in the figures. The strains showed similar trends to ones for ('+' degree) printing pattern with slight difference in values. Vertical and Horizontal strain components reached -13.5% and -5.5%, respectively. Shrinkage had very small evident effect on shear strain component as it reached around -1%. This is the only print pattern to suffer this much shear strain.

5.6.2. Effect of infill density. Infill density is a parameter set by the slicer of the 3D printer that controls the number of lines that fills the core region of a part. It plays a vital role in deciding the mechanical properties and the final density of a 3D

printed part. For this experiment, the infill density of both printing patterns; the ('+' grid) and the ('×' grid) were altered to a lower value to study the influence of infill density on shrinkage response in 3D printed shape memory polymer dumbbell specimens. The two new specimens are called; ('+' low density grid) and ('×' low density grid). The infill density was not quantified, and the experiment is for qualitative purposes rather than quantitative.

Figure 5.23: Shrinkage DIC strains averaged for a 3D printed shape memory polymer dumbbell specimen printed with ('×' grid) printing pattern.

Figure 5.24 shows the shrinkage test conducted for printing pattern of ('+' low density grid). The shrinkage behavior of the ('+' low density grid) print pattern is very close to the original ('+' grid) print pattern shown in Figure 5.22 in terms of trends and values of the three strain components. The main observation is that the low density print pattern suffered slightly less strain accumulation compared to the original ('+' grid) print pattern, reaching a values of vertical and horizontal strain components of -11% and -2.5%, respectively, in comparison to -13% and -3.5%, respectively for the original ('+' grid) print pattern. The shear strain remained unchanged despite the change in infill density.

The shrinkage results of ('×' low density grid) printing pattern specimen are presented in Figure 5.25. Again, the shrinkage behavior of the ('×' low density grid)

print pattern is very close to the original ('×' grid) print pattern shown in Figure 5.23 in terms of trends and values of the three strain components. The low density print pattern suffered less strain accumulation in comparison with the original ('×' grid) print pattern, reaching values of vertical and horizontal strain components of -10.5% and -4.5%, respectively, in comparison to -13.5% and -5.5%, respectively for the original ('×' grid) print pattern. The shear strain component for the low density grid was recorded 0%, while the original density print pattern oddly reached -1%.

Figure 5.24: Shrinkage DIC strains averaged for a 3D printed shape memory polymer dumbbell specimen printed with ('+' low density grid) printing pattern.

5.6.3. Effective strain due to shrinkage. With the results of printing pattern and infill density completed, it is important to identify a proper method to compare the result to select which print pattern and infill density to implement in future applications. The shrinkage consists of a complex combination of vertical, horizontal and shear strains. Thus, it is instrumental to find a proper equation that combines the strains in order to compare between the printing patterns. For this work, the von-mises effective strain will be implemented to measure the severity of shrinkage in each printing pattern and infill density case. Von-mises effective strain is computed through Equation 4 and Equation 5:

$$\varepsilon_{1,2} = \frac{\varepsilon_{xx} + \varepsilon_{yy}}{2} \pm \sqrt{\left(\frac{\varepsilon_{xx} - \varepsilon_{yy}}{2}\right)^2 + \varepsilon_{xy}^2} \quad (4)$$

$$\varepsilon_{effective} = \sqrt{\frac{2}{3}(\varepsilon_1^2 + (\varepsilon_1 + \varepsilon_2)^2 + \varepsilon_2^2)} \quad (5)$$

where ε_1 and ε_2 are the principle strains, $\varepsilon_{effective}$ or ε_{eff} is the von-mises effective strain.

Figure 5.25: Shrinkage DIC strains averaged for a 3D printed shape memory polymer dumbbell specimen printed with ('x' low density grid) printing pattern.

Results of all printing patterns and infill directions were reproduced using the von-mises effective strain ε_{eff} , and are plotted in Figure 5.26. The criterion in which a printing pattern is consider “good” or “bad” is by looking at how “low” or “high” is its effective strain accumulation. This tells us that (0 degree) printing pattern showed the worst shrinkage response since it suffered the most distortion in shape by reaching a high effective strain value of about 37%. On the other hand, the ('x' low density grid) printing pattern showed the best resistance to shrinkage by reaching the lowest effective strain value of about 9%. The rest of the patterns came in between, much closer to the ('x' low density grid) printing pattern, with lower infill density printing patterns being less than the grids with original infill density.

Figure 5.26: A combined plot of all printing patterns and infill densities using the von-mises effective strain.

Another important observation in Figure 5.26 is the degree of stability in the steady-state region of the effective strain curves. The study of strain flow stability in shrinkage due to heat is crucial when implemented in practical applications. During application of shape memory cycling to a part, each shape memory cycle consists of heating the part to 55°C during programming and recovery for extended periods of time. The instability of a printing pattern will lead to further undesired shrinkage during the shape memory cycles. To measure the steady-state stability of a printing pattern, the time derivative is taken at the end portion of the curve.

For example, (0 degree) printing pattern showed a strain accumulation rate of 0.02%, while ('+' low density grid) printing pattern showed a strain accumulation rate 0.002%. This means for steady-state extra hold time of 100 minutes, (0 degree) sample

accumulates 2% extra effective strain, while ('+' low density grid) sample accumulates only 0.2%. Thus, the ('+' low density grid) is 10 times more stable than the (0 degree) sample.

Figure 5.27 provides a summary of the total effective strain accumulation during shrinkage for all printing patterns and infill densities. In general, all samples showed significant improvement in resistance to shrinkage in comparison with the (0 degree) printing pattern. The effect of infill density on effective strain accumulation is evident despite the insignificant difference in value. The shape of grid had barely any effect on effective strain accumulation. It can be concluded that grid structures exhibit higher resistance to shrinkage than the linear printing patterns.

Figure 5.27: Summary of the total effective strain accumulation during shrinkage for all printing patterns and infill densities.

The previous results gave an initial direction to which print pattern and infill density might be better upon implementation in 4D printing designs. However, there are other variables to consider that will affect the overall performance of the 4D printed shape memory part, such as strength, ductility and bulk part density and shrinkage aspect ratio. Thus, further investigation is required in order to make a more

comprehensive conclusion regarding the feasibility of each print pattern and infill density.

By shrinkage aspect ratio, we mean how homogeneous is the shrinkage effect on the part. For example, you would rather have your part shrink in all dimensions at the same rate, rather than having it shrink from one direction much more than another direction. This will basically preserve the identity of the part, regardless of dimensional reduction. The more the vertical and horizontal strain components are close to each other, the more the aspect ratio is conserved, and vice versa.

Chapter 6. Conclusion

This work studies the implantation of heat activated smart materials, e.g. Shape memory polymer, in additive manufacturing, to produce 4D printed structures with tunable mechanical properties. More specifically, the mechanical and recovery properties of multi-cell auxetic honeycomb cellular structures were investigated. The work supports the following conclusions:

- The use of SMP filaments in Fusion Deposition process successfully created complex structures such as auxetic honeycomb cellular solids. However, the as-printed shapes are not stable during the first-time heat exposure resulting in shrinkage in members and distortion in dimensions. This can be accounted for through the curing process which eliminates any further dimensional changes during heat-induced programming and recovery of the 4D printed structure.
- When implemented to structures with dimensional-dependent mechanical properties such as auxetic honeycomb, SMP enabled achieving tunable mechanical properties such as Poisson's ratio and structural stiffness. For the case of multicell auxetic honeycomb (negative Poisson's ratio structure), a global programming strain level of $\sim 35\%$ resulted in increase of Poisson's ratio from -0.35 to $+0.69$, and Structural stiffness from 0.177 to 0.242 kN/mm .
- Repeated constant programming and recovery cycles (functional fatigue) proved to have significant influence on property degradation due to accumulation of residual strains as function of number of cycles. Three cycles of constant programming strain of $\sim 6.3\%$ on a dumbbell specimen induced a residual strain accumulation of $\sim 2\%$, which resulted in degradation in elastic modulus from 2025 to 1818 MPa . Similarly, upon implementation on single-cell auxetic structure for four cycles using a constant global strain programming level of $\sim 13.2\%$, results showed a residual strain accumulation of $\sim 8.6\%$ which led to degradation in Poisson's ratio from -0.57 to -0.45% and increase in Structural stiffness from ~ 0.136 to ~ 0.150 kN/mm .
- Incremental programming levels during programming and recovery cycles showed dependence of residual strain accumulation on the programming level. In the case of multicell auxetic structure honeycomb, a global strain level of

~4.4% induced ~1% residual strain. Similarly, a global strain level of ~35% induced ~12% residual strain.

- Local response of auxetic honeycomb structure under tensile testing revealed strain accumulation on the vertices and sharp edges in the structure. The local heterogeneous nature of auxetic honeycomb showed dependence of the member orientation and dimensions on the local axial strains. For the inclined members in auxetic honeycomb, the transition from negative to positive Poisson's ratio during programming showed direct correlation with the strain sign in the members. Compressive strains in inclined members indicate Negative Poisson's ratio, and Tensile strains indicate Positive Poisson's ratio. Furthermore, it has been shown that the bulk residual strains are directly caused by local residual strains accumulated within the members of the auxetic structure.
- Effect of print pattern on shrinkage of as-printed dumbbell specimens showed major dependence of accumulated strains on the direction of filament deposition where the majority of strain accumulation during shrinkage is shown in the direction of the infill lines. The use of grid instead of longitudinal print pattern decreased the shrinkage von-mises effective strain from ~37% to ~13%, and showed a notable increase in steady-state shrinkage stability.
- The decrease of infill density in grid print pattern improved the resistance to shrinkage effective strain where it decreased from ~13% to ~11%.

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