OPTIMIZATION OF 3D PRINTING OF SHAPE MEMORY POLYMER

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DECLARATION

This work has not previously been accepted in substance for any degree and is not being concurrently submitted in candidature for any degree.

Signed……………………………yin…………………………………(GUN YONG YIN)

Date……………………24/7/2022……………………………………….(24/7/2022)

STATEMENT 1

This thesis is the result of my own investigations, except where otherwise stated. Other sources are acknowledged by giving explicit references. Bibliography/references are appended.

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Last but not least, I would like to express my deep gratitude to my family, friends, and classmates for their support and motivating me to carry on whenever I faced obstacles. Their support both morally and physically motivated me greatly in completing this project.

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LIST OF ABBREVIATION

3D	Three-dimensional
SMP	Shape memory polymer
SME	Shape memory effect
PLA	Polylactic Acid
PVA	Polyvinyl Alcohol ().
HIPS	High Impact Polystyrene
ABS	Acrylonitrile Butadiene Styrene
CT	Computed Tomography
FFF	Fused Filament Fabrication
4D	Four-dimensional
LENS	laser engineering net shape
FDM	fused deposition modelling
SLA	Stereolithography
SMM	Shape memory material
CAD	Computer-aided design
CNC	computer numerical control
TPU	Thermoplastic-urethane
SMM	Shape memory materials
SMA	Shape memory alloys
Tg	Glass transitions
Tc	temperature of crystallization
Tm	melting transition
PFTE	polytetrafluoroethylene

EVA	ethylene–vinyl acetate
DSC	Differential scattering calorimetry
DMA	Dynamic mechanical analysis
SEM	Scanning Electron Microscope
HA	hydroxyapatite
CS	chitosan
PBS	Polybutylene succinate
ASTM	American Society for Testing and Materials
UTM	Universal testing machine
AM	Addictive manufacturing

ABSTRAK

Percetakan 3D ialah teknologi popular dalam persekitaran pembuatan semasa kerana ia membenarkan fabrikasi fleksibel reka bentuk produk yang rumit pada harga yang berpatutan. Ia adalah proses pembuatan ketagihan yang membina sesuatu dengan melapisi filamen cair. Kesan ingatan bentuk ialah sifat polimer memori Bentuk di mana ia dapat mengingati bentuk utamanya, mengekalkan bentuk sekunder selepas terdedah kepada perubahan terikan, dan kemudian kembali kepada bentuk utama apabila rangsangan luar digunakan. Dalam beberapa tahun kebelakangan ini, polimer memori bentuk adalah salah satu topik yang dibincangkan secara meluas di bawah bidang penyelidikan percetakan 3D. Dalam kertas ini, kesan ingatan bentuk polimer bercetak 3D dengan 75% asid polilaktik dan 25% termoplastik-uretana telah disiasat. Masa kitaran untuk spesimen kembali kepada bentuk utamanya selepas dipanaskan telah direkodkan dan keputusan menunjukkan bahawa faktor yang paling ketara mempengaruhi kesan ingatan bentuk ialah suhu cetakan dan ketinggian lapisan. Ujian tegangan menunjukkan ketinggian lapisan adalah faktor yang paling ketara mempengaruhi tindak balas. Oleh itu, sebagai cadangan untuk kerja masa hadapan, ketumpatan infill boleh digunakan untuk menggantikan kelajuan pencetakan untuk mengkaji potensi polimer memori bentuk.

ABSTRACT

3D printing is a popular technology in current manufacturing environment since it allows flexible fabrication of complicated product design at a reasonable price. It is an addictive manufacturing process which construct things by layering melted filament. Shape Memory Effect (SME) is a property of Shape Memory Polymer (SMP) where it was able to remember their primary shape, maintaining a secondary shape after being exposed to a strain change, and then returning to the primary shape when external stimulation is applied. In recent years, SMP is one of the widely discussed topics under the 3D printing research field. In this paper, the SME of 3D printed polymer with 75% polylactic acid (PLA) and 25% thermoplastic-urethane (TPU) was investigated. The cycle time for the specimen to return to its primary shape after heated was recorded and the results showed that the most significant factors affecting the SME were the printing temperature and layer height. The tensile test showed that layer height is the most significant factor affecting the responses. Therefore, as suggestions for future work, infill density can be used to replace the printing speed to study the potential of SMP.

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Chapter 1 Introduction

1.1 Introduction

3D printing is a type of addictive manufacturing (AM) or additive layer manufacturing process, which is also a type of computer-controlled process that can fabricate three-dimensional object by depositing material layer by layer. The material being used as filament in 3D printing is usually made out of High Impact Polystyrene (HIPS), Acrylonitrile Butadiene Styrene (ABS), Polylactic Acid (PLA), or Polyvinyl Alcohol (PVA). The filament will be melted at the high temperature nozzle and then be extruded onto the bed of the 3D printing machine. 3D printer currently has increased demand not only among manufacturers, but also home users due to its low-cost in fabricating custom product, its user-friendliness as low level of technical skills is needed and its high reliability.

In 3D printing, defect is one of the important elements as it will reduce the mechanical performance of the part produced. The presence of pores or void in the 3D printed part as a result of incorrect parameters selected during the operation of machine will reduce the final density of the specimen. Thus, minimizing voids is an essential part of 3D printing as sintering process will only reduce the size of voids, but not eliminating the void that is present in the structure. In addition, the selection of printing strategy will lead to the presence of voids for example in places where two perimeters joined or where the infill started or jointed. This sequence of pores is observed in the Computed Technology (CT) scans of specimens produced by Fused Filament Fabrication (FFF) process with other highly filled filaments and it can weaken the mechanical properties of specimens (Ferretti et al., 2021).

The addition of several smart materials to the industry evolved the concept of 3D printing. Smart materials are materials that can be controlled with an external stimulus to generate response. Among the smart materials that are currently available in the market, Shape Memory Polymer (SMP) captured great interest of many researchers to investigate deeper into the potential of the material (Garcia Rosales et al., 2019).

Shape Memory Effect (SME) is a property of SMP which is able to remember its primary shape, maintain a secondary shape after being exposed to a strain change, and then returning to the primary shape when external stimulation is applied. Internal stress is created through the programming process, the part-specific process of transitioning from a fundamental shape to an altered shape (or vice-versa) in the presence of stimulus. The stored energy from the process may be released as a driving force during the recovery phase. Changing the temperature, known as transformation temperature, is a common way to produce these shape changes. SMP is programmable, and its shape can be changed depending on printing procedures or mechanical deformation of the manufactured product.

The benefits of using materials with SMP include the lower cost and density of the material, enabling greater recovery deformation, being biodegradable, and responding to more diverse stimuli. Besides, it has potential to produce part that can respond automatically to environmental stimuli without the help of other power or hardware. However, material with SMP have limitations such as long cycle time to return to its primary shape and low actuation stress (Yang et al., 2016).

The rapid advancement in the 3D printing technology had led to the invention of 4D printing, or more precisely controlled 3D printing. For this new technology, the material may deform when subjected to an external stimulus. Although 3D printing techniques have been applied using a variety of materials, including plastics, metals and many other materials, most of the materials are not suitable for 4D printing because these materials do not change shape sufficiently when exposed to stimuli such as humidity, temperature change or an external magnetic field. Efforts to discover such smart materials to accelerate the development of 4D printing technology are still continuing (Mehrpouya et al., 2021).

1.2 Project Background

In the early 1980s, 3D printing had steady popularity grow. Since then, 3D printing has been utilized in a variety of industries, including manufacturing, product development and prototyping. Meanwhile, new applications for 3D printing processes employing sophisticated materials have proliferated at a fast pace. In current technology, few 3D printing techniques that are used include laser engineering net shape (LENS), fused deposition modelling (FDM), Stereolithography (SLA) and other printing processes. (Mehrpouya et al., 2021)

Shape memory material (SMM) is a topic that piques the attention of both academics and industry. It is a fascinating topic that poses a technical challenge while appealing to the imagination. SMMs alter their forms in response to given stimuli. The important aspect of this behaviour is that the material can "memorize" the original shape and reconstructed without further mechanical effort following deformation. Changes in ambient temperature are the most common triggers for shape change, although material stress, magnetic fields, electric fields, pH values, UV radiation, and even water can be activating stimuli. Many technical parameters in smart materials systems in response to environmental changes, such as shape, stiffness, friction, position, damping, strain, and water vapor penetration which are controlled by SMMs which is because of variance activation stimuli or some other predetermined (Chakraborty et al., 2017)

SMP may deform from an initial shape to a stress-free, temporarily sustained shape until an additional stimulus triggers shape recovery. Internal stress is created throughout the programming process, and the stored energy may be released and used as a driving force during the form recovery phase. Transformation temperature will occur when the temperature is changing. This is a common way to produce these shape changes. 'Programming' refers to the part-specific process of transitioning from a fundamental shape to an altered shape (and vice versa) in the presence of a stimulus. SMPs can produce desired shape transformations via printing processes or mechanical deformation by programming to created product. Polymer selection is crucial in developing programmable 4D printed structures in this aspect. PLA type of polymer which has emerged as a promising contender for 4D printing due to its wide range of uses in 3D printing and inexpensive cost. Although PLA's shape-memory capabilities have been studied and documented, there is little study on its use in shape distortion following 3D printing**.**(Mehrpouya et al., 2021)

In terms of the extra dimension- time, which adds stimuli-responsive self-evolving property to the printed objects, 3D printing of shape memory polymers is also known as 4D printing. Smart sensors, electrical gadgets, soft robotics, medicinal devices, deployable structures, and other applications are possible with 4D printing. However, due to the lack of mechanical strength and stiffness in present 3D printable shape memory polymers, 3D printed shape memory polymers for advanced structural applications has not be implemented primarily. (Li et al., 2019)

1.3 Problem Statement

Shape memory polymer is type of smart materials which has the ability to return to its original form and shape upon release of load by an external stimulus, such as temperature change. One of the techniques that can use to produce shape memory polymer is 3D printing. However, extend of shape memory effect from 3D printed polymer and the relationship to the 3D printing parameters is not well-known. This project is to study the various SME polymer and to determine the best 3D printing parameters for optimum shape memory property of polymer (Polylactic Acid Polymer).

1.4 Objectives

The purposes to carry out this project are:

1. To investigate the shape memory effect of the polymer when different parameter has been set during 3D printing.
2. To achieve optimal parameter 3D printing on shape memory polymer (Polylactic Acid Polymer)

1.5 **Scope of Work**

The scope of this study is to determine the method and prescribed that involve in the listed below:

1. A product will be design at Parasolid file and save as the finalized drawing and will import into the 3D printer.
2. Optimization the parameter of 3D printer by study to produce quality part and overcome defect.
3. To generate the parameter that are most significant software of Minitab will be used.

Chapter 2 Literature Review

2.1 3d printing

3D printing is a technology that was first invented in 1987, and it has attracted researchers, governments, and businesses to use it to manufacture diverse products based on 3D models using computer-aided design (CAD) software. Several advantages of 3D printing over traditional production methods including extrusion and injection moulding have prompted this endeavour. 3D printer can be used to produce a 3D structure with a complex geometry, which is difficult using injection moulding. Furthermore, it is built on additive manufacturing technology, which reduces waste by layering feedstock elements to produce an object. In contrast, subtractive manufacturing, such as computer numerical control (CNC) machining, manufactures a component by lowering the amount of material utilized. (Nugroho et al., 2021)

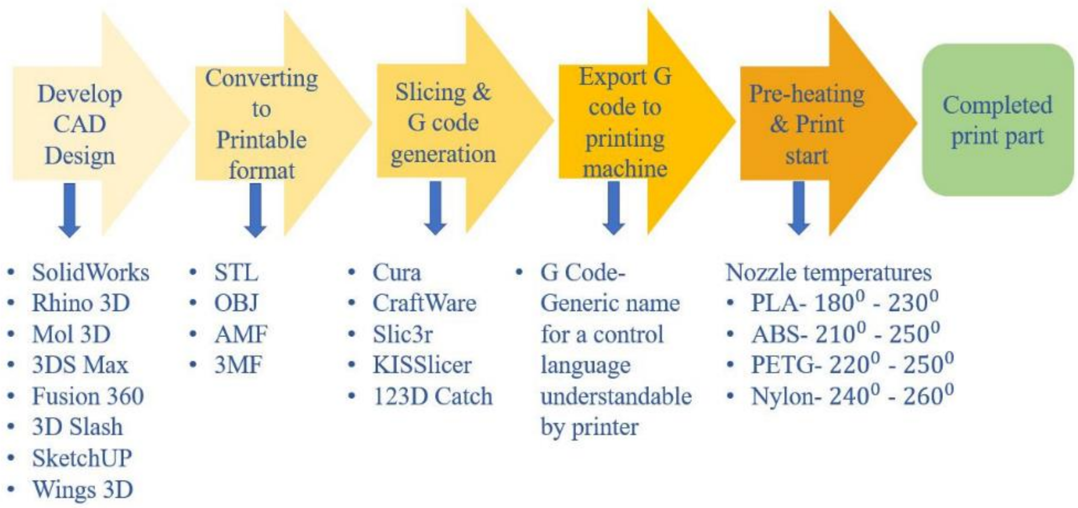


Figure 2.1 Simplified process flowchart of FDM including different types of software used in the 3D printing industry(Wickramasinghe et al., 2020)

Time, stimuli, programming, and smart materials may all be used to turn 3D printing into 4D printing. ‘Time’ in this context does not refer to the time taken to make a part which refers to suggest that following manufacture process, the printed components continue to alter over time and restore their original form when exposed to environmental stimuli such as heat, applied loads, light, pH level, evaporation, magnetic and electrical fields. Besides that, traditional 3D printing can only generate a static item. 4D printing, on the other hand, combines meticulously specified geometries with precisely controlled deposition of active (smart) materials to induce shape change in response to external stimuli. The word "programming" refers to the shape-setting of printed components, in which the 3D printed object may be deformed by an external force in response to specific external stimulus, for example, heat, light, magnetic, and electrical fields. ((Nugroho et al., 2021))

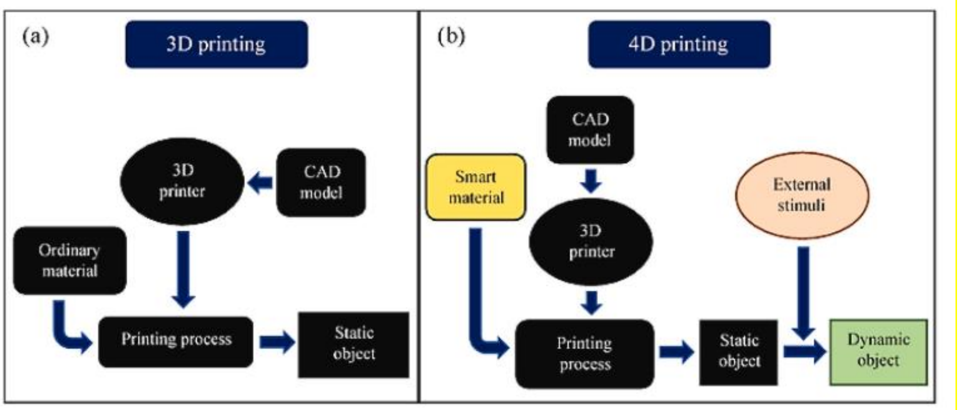


Figure 2.2:Basic concept of (a) 3D printing and (b) 4D printing.

Under the management of heat, strain (applied stress), or environmental factors, SMM have the potential to remember a form that is significantly different from their inherent shape. (Ochoński, 2010)

Through software tuneable settings, various printers offer varied degrees of user control over the printing process. The infill pattern, layer thickness, extruder speed, build plate temperature, cooling fan speed, and various outline shells are a few examples of such factors. Based on the few research that had been done on the effects of processing, despite a large body of literature on the effects of parameter selection on print quality and mechanical qualities, it shows that quality of the print can be customised in accordance with the desired characteristics of a product by iteratively adjusting these parameters. For instance, raising infill density can boost a part's effectiveness while decreasing the layer thickness can enhance the quality of the design's tiny details (Subash & Kandasubramanian, 2020)

During the process of manufacturing of a product with a 3D printer, there are a few types of defects that will be produced caused by a few factors, including:

-High temperature of extrusion nozzle: The suitable melting temperature for all type of material is different. Hence, when the temperature set is higher than the general melting point of the material, it will cause the flow rate to be extremely high, leading to a deformed structure.

-Distance between the nozzle tip and the printing bed: During the beginning phase of the printing process, if the distance between the two components is not accurate, it leads to improper printing. If the distance is too close, it may cause the nozzle to come in contract with the printed layer. Inversely, if the distance is set too far, it will cause damage during the new layer creating process and the strength of the product will become suboptimal.

-Misalignment of bed and nozzle: It can cause the most severe problem leading to material wasting during printing process. Since the movement of the nozzle is limited, to prevent misalignment, some parts of the object may lie outside the reach of the nozzle.

-The other factors that will cause defect to happen include blocked nozzle, runout filament, and heating element problems. (Farhan Khan et al., 2020)

2.2 Material Selection

PLA is a material that has a higher tensile strength compared to ABS. By comparing ABS and PLA, it is shown that ABS demonstrates better impact strength, but the tensile strength for PLA is higher. PLA degrades quickly in the right conditions, whereas other polymers can only be discarded or recycled. Parts constructed of PLA retain their plasticity and toughness for an extended period of time. Several studies have been carried out to determine the best conditions to improved mechanical qualities in PLA. The raster angle has the greatest impact on tensile strength, followed by the raster width and later height. The majority of studies concluded that increasing the layer height of a print causes large voids in the microstructure, lowering the print part's tensile strength. (Wickramasinghe et al., 2020)

The PLA filament that has been used for 3D printing usually consisted of 90 wt% PLA and 10 wt% of Thermoplastic-urethane (TPU). Besides that, the dimension of the filament is usually in 1.75 mm. Figure 2.3(a) shows an example of the filament while Figure 2.3(b) is the observation of the filament at the cross-section's phase morphology when 210 ◦C and 190 ◦C has been set for preheating and extrusion head temperature respectively. Since the TPU presence is 10 wt% in blends, so form of the island appeared. (Dong et al., 2021)

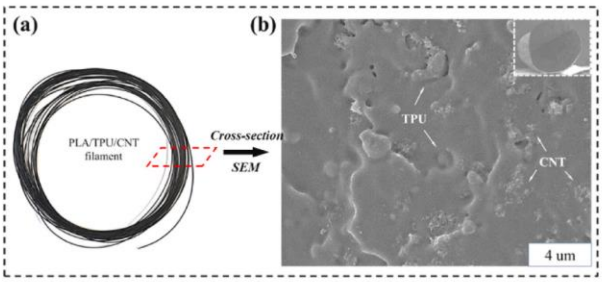


Figure 2.3:(a) Filament (b) The phase morphology of cross-section of the filaments

2.3 Shape memory polymer

One of the important elements for SMM is SME, which after being colossally and quasi-plastically twisted, may recuperate their normal form in the presence of the correct stimulus. From the research by Chang and Read it shows that the first SMM which they discovered having SME property is Gold-Cadmium alloy in 1951 and later in Indium-Titanium alloy in 1953. The application of these shape-memory materials was described in 1963. SMM and systems that are antiphon to extramural inputs have a huge impact on daily life and may be found in a wide range of industries, including smart robotics, drug delivery, tissue engineering scaffolds, and self-healing systems. These materials, which are dubbed super elastic, pseudo-elastic, and viscoelastic, exhibit exceptional thermomechanical behaviour and recuperate its original form following the SME. The glass transition and martensitic transformations (in alloys) axioms – possibility of retrieving large deformations and existence of transformation stress plateau, accompanied by substantial shear-like distortions associated with a diffusion less change through configuration. The external elements that cause SMM to change the most in a legion of materials accessible are chromaticity and design variations. The figure below shows the general classification of SMM. (Subash & Kandasubramanian, 2020)

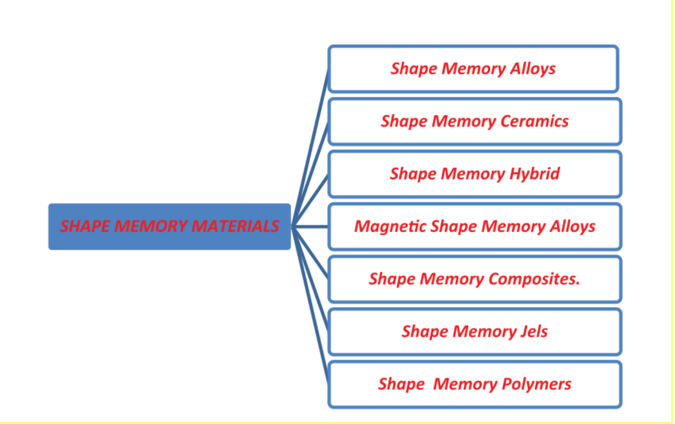


Figure 2.4: Classification of shape memory material(Chakraborty et al., 2017)

SMPs are an emerging class of smart polymeric systems that have the ability to recall either programmed or permanent temporary shapes when exposed to exogenous stimuli such as temperature, light, and so on. As a result, they are being used for functions such as smart fabrics, smart medical devices, self-deployable sun sails in spaceships, implantations, or minimally invasive operations, and heat shrinkable turbine for electronics. (Korde & Kandasubramanian, 2020). Based on the research that had been done it was stated that SME in the polymer, which are methacrylic ester resins. SMPs are double-shaped entities capable of actively modifying from shape I to shape II, as shown in Figure 2.6 where shape I is an interim structure obtained as a result of mechanical contortion causing shape change, and shape II is the permanent shape**.**(Subash & Kandasubramanian, 2020)

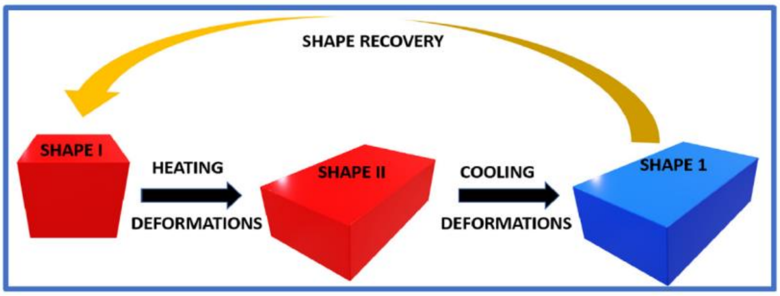


Figure 2.5:Schematic illustration of transition in shape memory polymers

SMP is considered as a type of smart material that made by polymer which can be restored from a transitory shape to their basic shape in response to an external signal. SMPs can recover up to 150% of their elongation, which is substantially more than the greatest form recovery reported in SMAs, which is around 8%. Furthermore, SMPs are usually cheaper than SMAs, and the cost required for producing various shapes and sizes is lower. Textiles, medicinal devices, tissue engineering, and aerospace are just a few of the industries that could benefit from these materials. Aside from that, there are several drawbacks to SMP. SMPs have a lower recovery stress which is less than 10MPa.

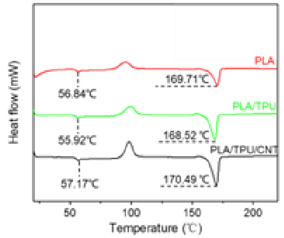
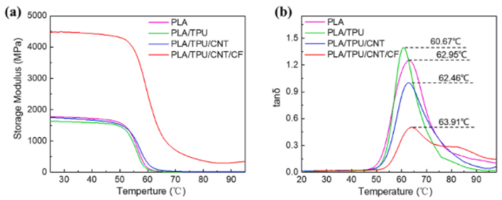
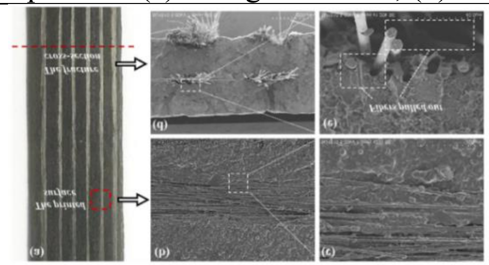
Besides that, the life cycle of SMPs will also become lesser. Another drawback is the time it takes for them to recuperate (Chakraborty et al., 2017)

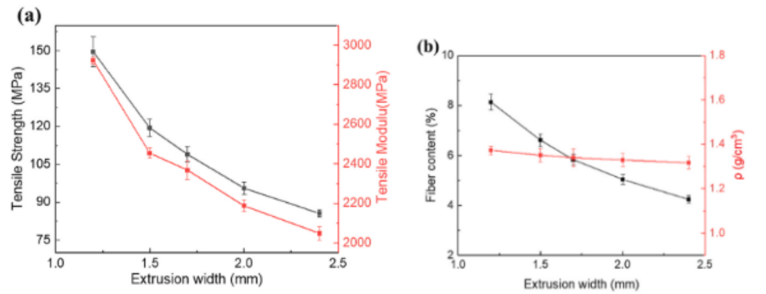
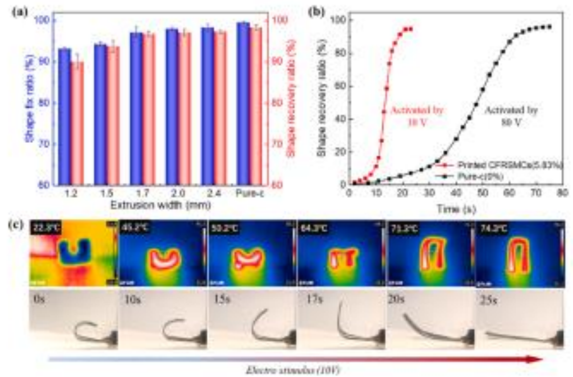
Despite the fact that combining "3D printing technology" with SMP allows for additional shape-controllable changes to meet real-world requirements such as 3D configuration restoration and target deformation, developing 3D printed shape memory devices with quick responses, strength, precision, and excellent durability remains a challenge. SMP can only be printed in three-dimensional structures in a restricted number of ways. SMPs are polymer-based materials that are "intelligent" and have excellent shape recovery capabilities. These materials alter shape as a result of magnetic and electric fields, temperature, mechanical stress, and other external and internal stimuli. SME is one of the phenomena of produce a produce for SMP. SMPs take on the required form when temperature and mechanical force are applied, but when the force is withdrawn and the temperature is increased it will return to their original shape. Glass transitions (Tg), crystallization temperature (Tc), melting transition (Tm), elongation hardness, and tensile power are the "shape memory effect" factors for polymers. Trance can be known as if a polymer's real shape can be regained when heated above a shape recovery temperature. Polyurethanes, (meth)acrylates, polyurethane-polyvinylchloride blends, and other polymers can display shape memory effect. Engineering polymers that can display the shape memory effect include Polylactide acid (PLA), polytetrafluoroethylene (PFTE), and ethylene–vinyl acetate (EVA). (Verma & Kumar Verma, 2021)

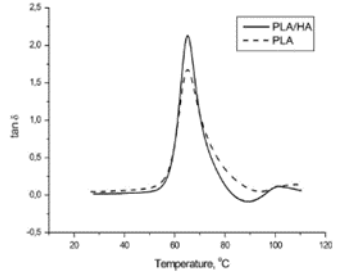
2.4 Previous study

Studies about the analysis experiment of previous researchers are focused in this section. The summaries of the previous studies are listed at Table 2.1

Table 2.1:Summary for literature review

Journal	Material	Analysis method	Result
Electro-induced shape memory effect of 4D printed auxetic composite using PLA/TPU/CNT filament embedded synergistically with continuous carbon fibre: A theoretical & experimental analysis(Dong et al., 2021)	Polylactic acid (PLA) Thermoplastic-urethane(TPU)	-Differential scattering calorimetry (DSC)	 DSC thermogram of the filament
		Dynamic mechanical analysis (DMA)	 DMA results for the printed samples as a function of temperature (a) storage modulus; (b) tanδ.
		Scanning electron microscopy (SEM)	 (a) The printed CFRSMCs specimen, (b–c) printed surface of the PLA/TPU/CNT blends embedded with carbon fiber (d–e) fractured cross-section of the printed CFRSMCs specimen after the tensile measurement.

		Mechanical properties	 Tensile performance of the printed CFRSMCs as a function of extrusion width (a) tensile strength and Young's modulus (b) fiber content and density
		Shape memory properties -Thermal-induced shape memory effect -Electro-induced shape memory effect:	 Shape memory performance of the printed CFRSMCs (a) thermal shape memory performance (b) shape recovery ratio of printed CFRSMCs and Pure-c under electrical stimulus (c) electro-induced behavior of the printed CFRSMCs

<table>
<tr>
<td rowspan="3">Mechanical properties and shape (Dong et al., 2021) of 3D-printed PLA-based porous scaffolds(Senatov et al., 2016)</td>
<td rowspan="3">polylactide (PLA)
hydroxyapatite (HA)</td>
<td>Dynamic mechanical analysis (DMA)
.</td>
<td>

Dependence of the internal friction (tan δ) on temperature for PLA and PLA/HA.</td>
</tr>
<tr>
<td>Mechanical testing</td>
<td>Table 1 – Mechanical properties in compression of PLA-based porous scaffolds obtained by 3D-printing.
<table>
<tr><th></th><th>Number of cycles</th><th>Shape recovery, %</th><th>Strength at 15% strain, MPa</th><th>Yield strength, MPa</th><th>Young's modulus, MPa</th></tr>
<tr><td>PLA</td><td>0</td><td>–</td><td>25.8±2.7</td><td>21.3±2.0</td><td>1439±179</td></tr>
<tr><td>PLA</td><td>1</td><td>99.1</td><td>18.9±2.1</td><td>17.1±1.8</td><td>1229±72</td></tr>
<tr><td>PLA</td><td>2</td><td>Destruction</td><td></td><td></td><td></td></tr>
<tr><td>PLA/HA</td><td>0</td><td>–</td><td>47.0±9.2</td><td>34.3±8.0</td><td>2091±470</td></tr>
<tr><td>PLA/HA</td><td>1</td><td>98.2</td><td>34.6±4.8</td><td>26.0±5.7</td><td>1690±238</td></tr>
<tr><td>PLA/HA</td><td>2</td><td>96.3</td><td>30.9±3.5</td><td>30.9±3.5</td><td>1360±226</td></tr>
<tr><td>PLA/HA</td><td>3</td><td>Destruction[a]</td><td></td><td></td><td></td></tr>
</table>
[a] delamination of several parts.</td>
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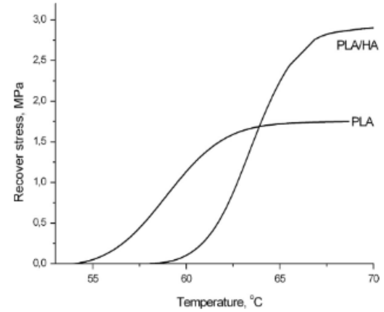
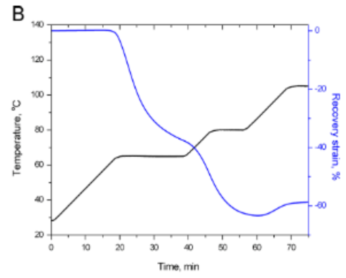
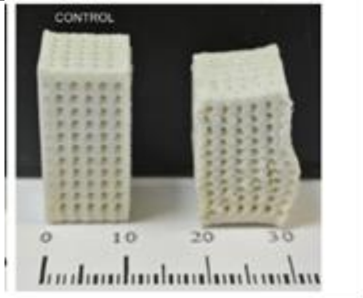
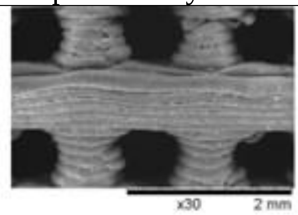
<tr>
<td>Recovery strain</td>
<td>

Dependence of recovery stress on temperature above Tg for PLA and PLA/HA.</td>
</tr>
</table>

		Non-isothermal recovery stress	 Dependence of recovery strain on temperature for PLA/HA
		PLA-based porous scaffolds obtained by 3D-printing	 Figure: porous scaffolds after the 2nd and 3rd heating-compression cycle Shape recovery = 96%
		Study compsites and 3D porous scaffolds	 Structure of PLA-based porous scaffolds obtained by 3D-printing. Magnification of 30x

<table>
<tr>
<td></td>
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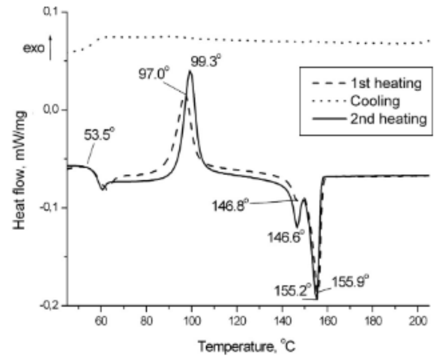
<td>Differential scanning calorimetry (DSC)</td>
<td>

DSC curves for the PLA/15% HA composite (first heating, cooling, second heating)</td>
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<tr>
<td rowspan="2">3D printed biodegradable functional temperature-stimuli shape memory polymer for customized scaffoldings(Pandey et al., 2020)</td>
<td rowspan="2">Polylactic acid (PLA)
chitosan (CS)</td>
<td>Recovered length through annealing</td>
<td>Observations of shape memory recovery.
<table>
<tr><th>S^N</th><th>Gauge length (mm)</th><th>Pre-elongated length (mm)</th><th>Recovered length through annealing (mm)</th><th>F (%)</th><th>Signal/noise (S/N) ratio, dB</th></tr>
<tr><td>1</td><td>28.50</td><td>28.93</td><td>28.46</td><td>18.8</td><td>25.4832</td></tr>
<tr><td>2</td><td>28.50</td><td>28.91</td><td>28.57</td><td>13.6</td><td>22.6708</td></tr>
<tr><td>3</td><td>28.50</td><td>28.99</td><td>28.69</td><td>12</td><td>24.0824</td></tr>
<tr><td>4</td><td>28.50</td><td>28.95</td><td>28.55</td><td>16</td><td>24.0824</td></tr>
<tr><td>5</td><td>28.50</td><td>29.00</td><td>28.64</td><td>14.4</td><td>23.1672</td></tr>
<tr><td>6</td><td>28.50</td><td>28.86</td><td>28.67</td><td>7.6</td><td>17.6163</td></tr>
<tr><td>7</td><td>28.50</td><td>28.94</td><td>28.59</td><td>14</td><td>22.9226</td></tr>
<tr><td>8</td><td>28.50</td><td>28.93</td><td>28.73</td><td>8</td><td>18.0618</td></tr>
<tr><td>9</td><td>28.50</td><td>28.98</td><td>28.86</td><td>4.8</td><td>13.6248</td></tr>
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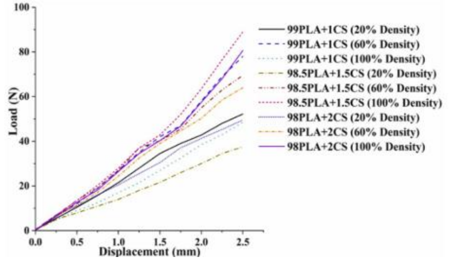
Observations of shape memory recovery.</td>
</tr>
<tr>
<td>Pre-elongation of the scaffoldings</td>
<td>

Load vs. displacement plots of 3D printed PLA/CS scaffolds.</td>
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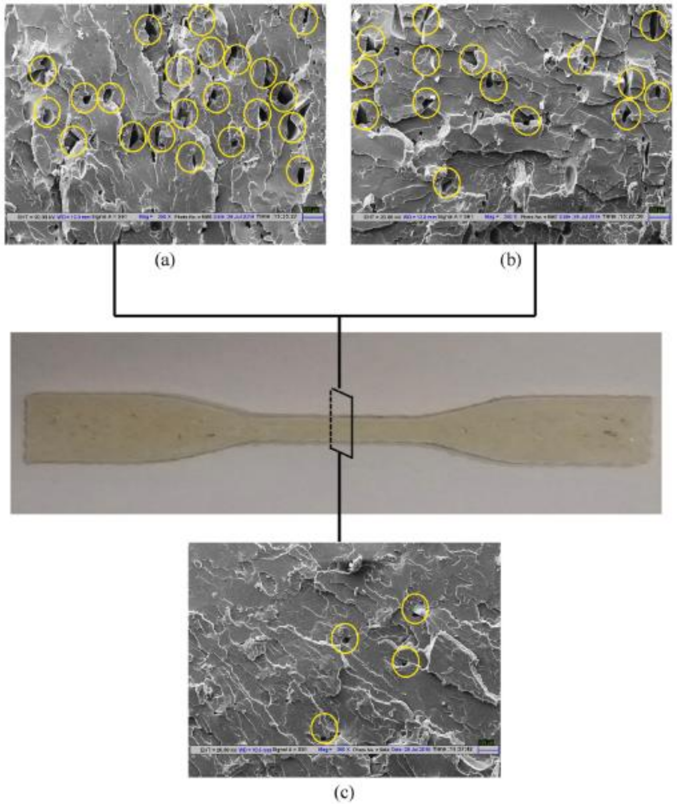
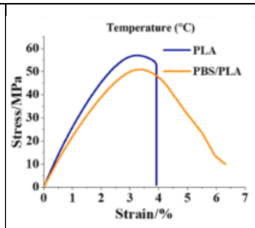
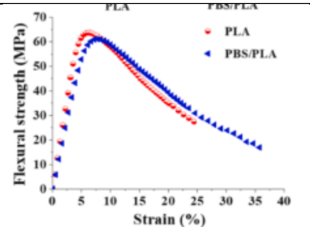
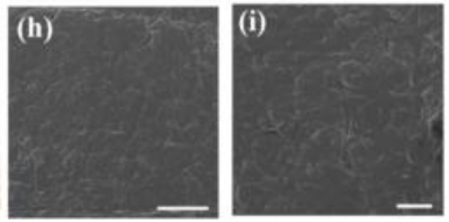
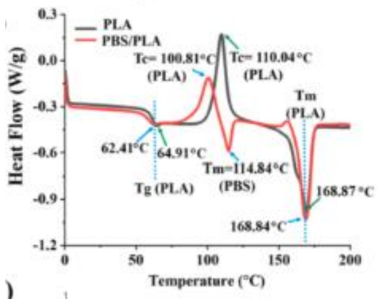
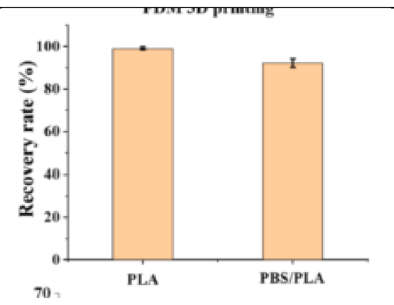
		Scanning electron microscope	 Cross-sectional SEM images of shape recovered PLA/2%wt. CS scaffoldings: (a) 20%, (b) 60%, and (c) 100% infill density. Note: The yellow mark shows the distorted pore geometry because of the pre-straining of the samples.

4D printing of shape memory polybutylene succinate/polylactic acid (PBS/ PLA) and its potential applications(Lin et al., 2022)	Polylactic acid (PLA) Polybutylene succinate (PBS)	Tensile/flexural properties .	 Uniaxial tensile tests Three-point bending tests
		Surface Morphology	 Surface morphology of specimens with scale 500 µm(h); 200 µm(i)
		Differential scanning calorimeter (DSC)	 DSC patterns

		Shape memory performances	 Shape memory performances Shape recovery rate =92.10 %

Chapter 3 Methodology

3.1 Introduction

In this chapter, every single detail of the processes will be explained. This indicates how the experiment was conducted. Besides that, in this chapter all the solutions to specific problems that arise during the experiment will also be recorded. Figure 3.1 shows the flow chart which explains about the overall processes

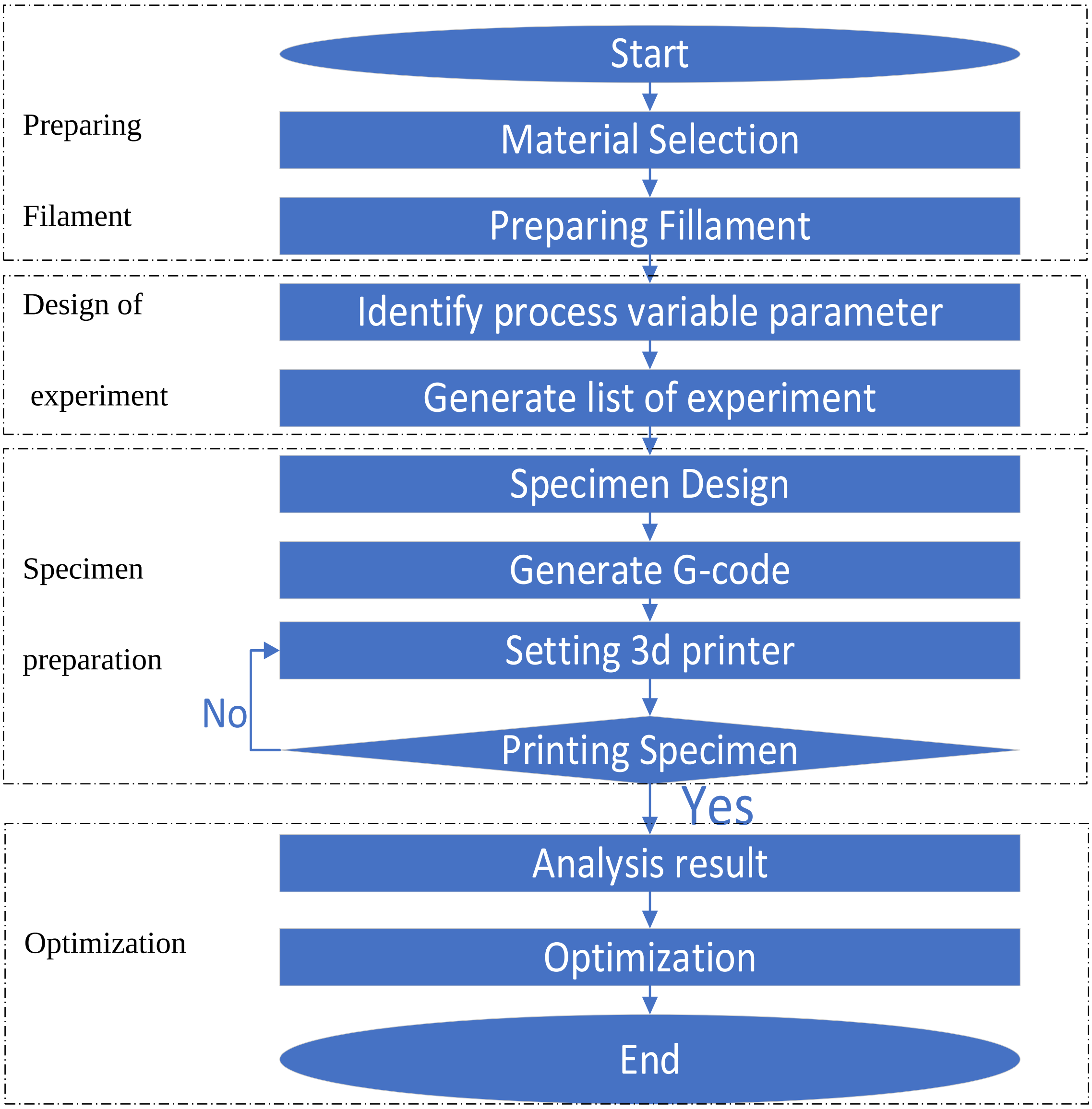


Figure 3.1:Flow chart about overall process

To start the experiment filament, need to be prepared. To prepare the filament the first thing required is to select the material that need to be used. After the material has been selected, the filament can be prepared with the aid of the machine.

After preparing the specimen, the next step that need to be done is to design the experiment. In this stage, the variable parameter will be decided based on the parameters for 3d printing. After the parameter is determined, the list of experiment will be generated by Minitab.

CAD design is created using the Solidwork programme. A STL file is then created from a CAD file for 3D printing slicing. The Ultimaker Cura 5.0.0 slicing programme will generate the G code based on the parameter that has been determined. After the G-code had been generated, it will be installed into 3d printer and the specimen will be printed.

To analyse the result, little amount of filament will be needed to test the Tg of the composite by using DSC. After getting the result, it is taken to test the SME. SEM is carried out to investigate the microstructure of specimens. To examine the specimens' tensile properties, a tensile test is then conducted.

Lastly, the obtained recommended parameter is sorted and let the results being auto generated by using software Minitab in this study. The combination of parameters that are most significant will be found and the expected result will be recorded.

3.2 Material Selection

The plastic material that has been used in this study is Polylactic Acid Polymer (PLA) and Thermoplastic polyurethane (TPU). The two types of material properties are shown in Table 3.1 and 3.2. The ensure the shape memory effect can be tested, PLA and TPU with the ratio of 75% and 25% being selected.

Table 3.1:Material properties of Polylactic Acid Polymer (PLA)

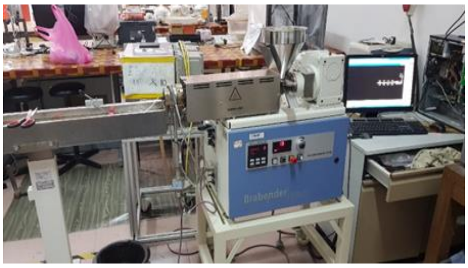
Parameter	Detail
Plastic Material	Polylactic Acid Polymer (PLA)
Specific gravity	1,24 g/cm3
Tensile modulus	3120 MPa
Melting temp.	115±35°C
Glass transition temp.	57 ºC
Extruder temperature	190 °C - 220 °C
Bed temperature	65 °C
Speed	10-70 mm/s

Table 3.2:Material properties of thermoplastic polyurethane (TPU)

Parameter	Detail
Plastic Material	Thermoplastic polyurethane (TPU)
Specific gravity	1,22 g/cm3
Tensile modulus	26 Mpa
Melting temp.	220°C
Glass transition temp.	-24 ºC
Extruder temperature	220 -240 ºC
Bed temperature	30-50 °C
Speed	20-50 mm/s

3.3 Filament preparation

The 2 raw materials PLA and TPU were inserted into the oven to get the materials dry subsequently at 90°C for 8 hours, with the aims to remove moisture from the materials. After the duration of 8 hours reached, the materials were taken out from the oven and arranged according to the ratio set at 75% PLA and 25% TPU using the electronic balance. The raw materials were then mixed and shaken inside a container to ensure it mixed well before fed it into the torque rheometer set at 50 rpm and 180 °C. Figure 3.2(a) shows the machine of torque rheometer. The material will then be crushed into a pellet form using the grinder after melted and was dried again using oven. Finally, it will be fed into the underwater extrusion machine (Brabender Kompaktextruder KE19) with temperature ranging from 155°C to 165°C and will be rolled with the filament winder machine. Figure 3.2(b) and 3.2(c) shows the extrusion machine (Brabender Kompaktextruder KE19) and filament winder.



(b)

(a) (c)

Figure 3.2:(a) Torque rheometer machine (b)extrusion machine (Brabender Kompaktextruder KE19) (c)filament winder machine