Research Internship (II301)

Investigation and Validation of 4D Printing of Smart Materials Polymers for Engineering Applications

By

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1. INTRODUCTION

In the evolving landscape of additive manufacturing, **4D printing** marks a significant advancement, enabling the creation of dynamic systems that transform over time in response to environmental stimuli such as temperature, humidity, or light. Unlike traditional 3D printing, which builds static structures, 4D printing integrates smart materials that allow printed components to self-assemble, morph, or adapt post-fabrication—opening up innovative possibilities across multiple industries.

Among the commonly used materials, **Polylactic Acid (PLA)** stands out due to its biodegradability, ease of processing, and mechanical strength. In 4D printing, PLA is often enhanced with other responsive compounds to introduce shape-memory behaviour, enabling structures to change configuration when exposed to specific triggers.

Acrylonitrile Butadiene Styrene (ABS), widely used in Fused Deposition Modelling (FDM), can also be modified by blending with shape-memory polymers or thermally responsive fillers. These composites allow ABS to demonstrate programmable deformation and recovery, making it suitable for deployable components in robotics and aerospace applications.

In **vat photopolymerization (SLA)**, UV-curable resins offer excellent precision and surface finish. When formulated with smart chemistries like pH- or heat-responsive polymers, SLA resins can enable microscale actuation and shape transformation. This makes SLA an ideal platform for producing intricate 4D structures such as self-folding devices or biomedical scaffolds.

Beyond printing, **injection moulding** of smart materials presents a scalable manufacturing option, though it introduces challenges related to waste management. Smart-material scraps, often non-recyclable through conventional methods, require sustainable approaches such as chemical recovery, eco-blending, or biodegradable design strategies to reduce environmental impact.

Together, these material systems—PLA, ABS, SLA resins, and moulded smart composites—are driving the future of adaptive, functional, and sustainable design. Their integration into 4D printing technologies is expanding the boundaries of what materials can do, enabling smarter, more responsive solutions across biomedical, aerospace, and consumer sectors.

2. Literature Review

1) Background and Significance of 4D Printing with PLA, ABS, and SLA Resins-

4D printing extends conventional 3D printing by incorporating time as the fourth dimension, enabling printed objects to undergo shape or functional transformations in response to stimuli such as temperature, humidity, light, pH, or magnetic fields. PLA (Polylactic Acid) remains a front-runner due to its biodegradability, ease of processing, and abundance. Through additives, polymer blending, and post-processing, PLA can exhibit shape-memory and stimuli-responsive behaviour suited for 4D printing.

Similarly, ABS (Acrylonitrile Butadiene Styrene) has been enhanced by blending with materials like PETG and Fe_3O_4 nanoparticles, enabling magnetic and thermal responsiveness. For instance, PETG–ABS–Fe_3O_4 nanocomposites have shown improved tensile strength (from ~26 to 27 MPa) and magneto-thermal actuation capabilities, enabling shape recovery via alternating magnetic fields.

In vat photopolymerization (SLA/DLP), smart resins—such as silicone urethane methacrylate-based formulations—are tailored for high-resolution, stimuli-responsive 4D printing. These resins exhibit excellent shape fixity and recovery, with applications ranging from biomedical scaffolds to micro actuators.

2) Existing Approaches and Challenges-

- **PLA**: Studies have enhanced PLA's dynamic behaviour via thermal annealing, plasticizers, and blending with polymers or Fe_3O_4 nanoparticles. However, maintaining consistent transformation speed and durability across repeated cycles remains challenging.
- **ABS-based nanocomposites**: PETG–ABS–Fe_3O_4 composites demonstrate strong magnetically induced shape-memory properties, though high filler loading can impair print quality and mechanical homogeneity.
- **SLA Resins**: Multi-component photopolymer systems (e.g., incorporating CNTs, SiO_2, PCL) allow precise spatiotemporal tuning of shape-memory properties and even self-healing, but UV shielding and long-term cycle degradation remain concerns.

3) Injection Moulding and Smart-material Waste-

Beyond additive manufacturing, injection moulding of smart composites presents scalability but raises environmental concerns. Smart-material scrap—often unrecyclable via conventional routes—necessitates novel disposal strategies. Research suggests approaches like chemical recycling, eco-blending, and active disassembly using shape-memory adhesives to facilitate separation and reuse. These methods align with goals to integrate lifecycle considerations into component design.

4) Opportunities and Future Directions -

- **Biomedical**: PLA and ABS composites with shape memory and magnetic responsiveness enable applications like self-adjusting implants, dynamic scaffolds, and targeted drug delivery. SLA-fabricated micro actuators support high-resolution bio-interfaces.
- **Soft Robotics & Aerospace**: ABS-hybrid architectures offer thermally and magnetically activated deployable components. SLA printed bilayer or digitally patterned composites enable responsive grippers and morphing structures.
- **Sustainability and Manufacturing**: Advances in injection moulding of smart composites must integrate recyclability. Encouraging reuse via chemical depolymerization or disassembly aids closed-loop systems. Stimuli-responsive adhesives and design-for-disassembly tactics can enhance lifecycle sustainability.

3. Major Techniques

- **Fused Deposition Modelling (FDM) -**
  - Process: Feeds and melts thermoplastic filament through a heated nozzle, depositing layers onto a build platform.
  - Advantages: Low cost, wide material variety (PLA, ABS, PETG), easy to use.
  - Limitations: Visible layer lines, anisotropic part strength, moderate accuracy.
- **Poly-Jet -**
  - Process: Jets droplets of photopolymer onto a platform and cures each layer with UV light.
  - Advantages: High resolution, smooth surface finish, multi-material printing (including colour).
  - -Limitations: Resins can be costly; parts are typically more brittle than thermoplastics.
- **Stereolithography (SLA)-**
  - Process: A UV laser selectively cures liquid resin in a vat, solidifying each layer in turn.
  - Advantages: Excellent detail and surface finish, wide range of specialized resins.
  - Limitations: Requires post-curing, resins can be messy and relatively expensive.
- **Digital Light Processing (DLP)-**
  - Process: Uses a projector to cure entire layers of resin at once.
  - Advantages: Similar detail to SLA but can be faster; high accuracy.
  - Limitations: Limited build volume (projector resolution), requires post-curing and resin handling.
- **Selective Laser Sintering (SLS)-**
  - Process: A laser fuses polymer powder layer by layer.
  - Advantages: Good mechanical properties, no need for support structures (powder supports the part).
  - Limitations: Equipment and materials can be expensive; powder handling is complex.
- **Injection Moulding-**
  - Process: Molten polymer pellets are injected under high pressure into a precision mould cavity, then cooled and ejected as finished parts.
  - Advantages: Ideal for high-volume production with fast cycle times (10–60 s), low per-part cost, tight tolerances (±0.125 mm), complex geometry, wide material range, and minimal post-processing waste.
  - Limitations: Involves high upfront tooling costs ($1 k–$100 k+), long mould lead times (weeks–months), strict design requirements (uniform walls, draft, limited undercuts), defect risks (warping, sink marks, flash), and poor cost-effectiveness for small or oversized runs.

4. Smart Materials

1. Shape Memory Polymers (SMP) -

a) Polylactic Acid (PLA)

- Biodegradable, heat sensitive
- Low flexibility
- Melting temperature: 180–220°C
- Transition temperature: 60–65°C

b) Polyurethane (PU)

- Non-biodegradable
- Heat and moisture sensitive
- High flexibility
- Activation time: 5–120 seconds (heat), hours (moisture)
- Melting temperature: 200–230°C
- Transition temperature: –30–20°C

c) Polycaprolactone (PCL)

- Biodegradable, heat sensitive
- Shape memory effect
- Activation time: 30–180 seconds
- Melting temperature: ~60°C
- Transition temperature: –60°C

d) Acrylonitrile Butadiene Styrene (ABS)

- Non-Biodegradable thermoplastic
- Moderate flexibility and toughness
- Integrated in hybrid SMP composites (e.g., ABS–SMP honeycombs)
- amorphous- no distinct melting point
- Transition temperature: –105°C

2. Hydrogels -

a) Poly(N-isopropylacrylamide) (PNIPAM)

- Water temperature sensitive
- Transition temperature: ~32°C

b) Polyethylene Glycol (PEG)

- pH and temperature sensitive
- Melting temperature: 200–230°C
- Transition temperature range: 25–37°C
- Activation time varies from seconds to minutes

3. Vat-Polymerization Resin (SLA/DLP)-

- UV-curable photopolymers (e.g., acrylics, meth-acrylates, epoxies) tailored for shape-memory via crosslink design.
- Transition temps tunable between ~30–105 °C; exhibit good fixity/recovery and toughness.
- Custom resins (e.g., LC-doped acrylics) allow fast actuation and high-resolution 4D structures.

4. Natural Polymers - Examples include cellulose, chitosan, and starch, often used for eco-friendly or specialized applications.

5. Stimulus Of Smart Materials

- **Temperature:** Triggers shape memory or phase transitions in alloys and polymers.
- **Moisture:** Causes hydrogels and similar materials to swell or shrink.
- **pH:** Alters ionic interactions, affecting solubility or swelling behaviour.
- **Light:** Induces colour changes or structural transformations in photo responsive materials.
- **Electric Field:** Drives deformation or conductivity changes in electroactive polymers.
- **Magnetic Field:** Adjusts viscosity or mechanical properties in magnetorheological fluids and composites.
- **Mechanical Force:** Generates electrical signals (piezoelectric effect) or alters shape under stress.

6. Advantages & Applications of Smart Materials in 4D Printing

PLA (FDM-based)

- **Biodegradability**: Naturally breaks down, minimizing environmental footprint—ideal for sustainable applications.
- **Ease of Processing**: Compatible with standard FDM printers (melting point ~180–220 °C).
- **Shape-Memory Capability**: Modified PLA (e.g., PLA/TPU, PLA/CNT, PLA/graphene) displays responsive behaviour around ~60–65 °C.
- **Cost-Effective**: Affordable and widely available compared to other SMPs.
- **Reasonable Mechanics**: Offers fair tensile strength suitable for structural prototypes.

Applications:

- Self-folding components activated by heat
- Adaptive biomedical devices (e.g., stents, scaffolds)
- Responsive packaging & shape-changing wearables
- Low-cost soft robotic hinges

ABS (FDM & Injection-Moulded Blends)

- **Mechanical Resilience**: ABS/SEBS blends maintain tensile strength post-recovery, thanks to elastomeric modifications.
- **Thermal Stability**: Higher Tg (~105 °C) yields rigidity—fewer unintended deformations, but impedes thermal shape-memory behaviour.
- **Injection-Moulding Friendly**: Supports complex geometries, reusable scrap processing, and strong mechanical properties.

Applications:

- Durable and flexible shape-memory composites via injection moulding
- Smart actuators where rigidity and repeatable performance are required

SLA/DLP Resins (Vat Photopolymerization)

- **High Resolution & Precision**: Enables complex micro-structures with controlled crosslinking.
- **Advanced Shape-Memory Formulations**: UV-curable resins (e.g., liquid crystal/acrylate blends) deliver precise, tunable actuation and rapid recovery.
- **Minimal Waste**: Utilizes vat format with efficient material usage and scalable processes.

Applications:

- Precision 4D-printed micro-actuators
- Smart optical elements like morphable antennas
- Fine-feature soft robotics and biomedical devices

Injection-Moulded Smart Polymers & Composites

- **Robust Fixity & Recovery**: PLA/TPU and PLA/SEBS blends show excellent actuation (Rf ~87%, Rr ~99%) when processed under load.
- **High-Volume Production**: Ideal for scalable manufacturing of shape-memory parts.
- **Material Diversification**: Enables inclusion of additives (e.g., TPU, elastomers) while leveraging sustainable scrap.

Applications:

- Load-bearing shape-memory parts (e.g., adaptive fixtures)
- Sustainable, mass-manufactured adaptive components

7. Previously Conducted Research & Material Comparisons

- **PLA + Carbon Nanotubes (CNTs)**: Enhances thermal conductivity (~48% improvement) and mechanical strength, facilitating faster heat diffusion and quicker thermal response in shape-memory PLA composites.
- **PLA + Graphene Nanoplatelets (GNPs)**: Raises thermal conductivity (~181% increase at 6 wt% GNP), enabling swifter actuation by improving heat transfer.
- **PLA + TPU (Thermoplastic Polyurethane)**: Improves elasticity and shape fixity (recovery ~58–91%), though printability can decline at higher TPU ratios.
- **PLA + Natural Fibers (Hemp, Flax, Bamboo)**: Adds flexibility and moisture absorption, promoting faster moisture-triggered deformation.
- **PLA + Hydrogel Additives**: Enables humidity-responsive shape changes via rapid water absorption/release—ideal for smart humidity-sensitive applications.

- **ABS + SEBS (Injection-Moulded)**: ABS blended with SEBS shows excellent resilience, maintaining tensile strength after full deformation and showing stable recovery performance.
- **ABS + SEBS (FDM vs. Injection Moulding)**: ABS/SEBS exhibited consistent strength after recovery, regardless of manufacturing method, indicating flexibility in processing without performance loss.

- **Vat-Polymerization (SLA/DLP) Shape-Memory Resins**: Liquid crystal-doped and acrylate-based photopolymer resins exhibit controlled thermos-responsive behaviour and fast recovery, making them suitable for high-resolution 4D printing. Multi-material, dual-wavelength DLP allows spatially patterned SMP networks within a single print vat.

- **Injection-Moulded PLA/TPU & PLA/SEBS Blends**: Injection-moulded PLA/TPU demonstrates high shape fixity (~87%) and recovery (~99%). PLA/SEBS blends show reduced resilience, while ABS/SEBS counterparts maintain tensile properties after actuation, highlighting material blend and processing dependencies.

Improving PLA & ABS Shape-Memory Performance

PLA's shape-memory potential can be unlocked—and even rival ABS-based composites—through strategic design interventions:

- **Nanofillers**: CNTs and GNPs accelerate thermal response and actuation speed.
- **TPU blends**: Enhance elasticity and fixity for better recovery.
- **Natural fibres/hydrogels**: Enable moisture-based stimuli actuation.
- **Infill optimization**: Literature and our experiments show lower infill densities (15–30%) and open-pattern designs (grid/criss-cross, gyroid) accelerate response without sacrificing fixity.

8. Work Done

Comparing Shape Recovery of PLA and ABS with Infill and Pattern Variations

Phase 1 – Full-density PLA vs. ABS (100 % infill)

1. We began by fabricating two sets of rectangular samples: one from **PLA filament** at 100 % infill using an FDM printer, and the other from **ABS** produced via injection moulding using scrap ABS remelted and recast at 100 % infill.
2. Both sets were bent to a fixed angle and immersed in a beaker of deionized water heated to each polymer's glass transition temperature (T_g): ~60–65 °C for PLA, ~105 °C for ABS.
3. **Results**:
    - PLA showed slight bending reversal but only modest recovery.
    - ABS remained largely unchanged—its higher rigidity prevented significant shape restoration.

Phase 2 – 50:50 ABS/PLA Blend (100 % infill)

1. Next, we injection-moulded a **blended composite** (50 % ABS + 50 % PLA) at full density.
2. After bending, samples were heated in the same water bath near their T_g.
3. **Outcome**: Recovery remained unsatisfactory; neither material exhibited significant memory effect when combined at full density.

Phase 3 – Optimizing PLA Infill Density-

Given PLA's superior response, we tested lower-density samples—fabricated via FDM at **30 %**, **20 %**, and **15 % infills**—to assess how internal structure influences performance.

- **30 % infill**: ~35 % recovery toward the original flat shape
- **20 % infill**: ~50 % recovery
- **15 % infill**: ~63 % recovery

These results show a clear trend: **lower infill correlates with greater bending capacity and more substantial shape recovery** when heated to PLA's Tg.

Phase 4 – Exploring Infill Pattern Effects

Two infill patterns were compared at equivalent infill densities: **grid** versus **honeycomb**.

- **Grid infill**: delivered higher deformation and recovery due to greater flexibility.
- **Honeycomb infill**: printed samples were comparatively rigid, exhibited less bending, and recovered to a smaller degree.

Phase 5 – SLA Pattern and PLA Geometry Variations

1. An SLA-fabricated pattern was also tested, but recovery could not be evaluated because the material's T_g was unknown—preventing proper stimulus application.
2. Finally, various PLA geometries (e.g., strips, beams, curved shapes) were printed and tested under identical conditions. These tests confirmed that **geometry interacts with infill and pattern choices**, influencing both bending resilience and recovery speed.

9. Result & Discussions

ABS material

PLA material

Phase 1 – PLA vs. ABS (100 % Infill)

- **PLA (100 %)** showed slight bending reversal when heated at its glass transition temperature (~60–65 °C), but overall shape recovery was modest.
- **ABS (100 %)**, produced from injection-molded scrap and heated to ~105 °C, remained largely unchanged. Its higher rigidity and Tg prevented significant thermal shape recovery.

PLA (100% Infill)

ABS (100% Infill)

Discussion: These outcomes confirm the influence of material properties—especially stiffness and Tg. PLA's lower Tg provides some activation potential, while ABS is too rigid for effective recovery under these conditions.

Phase 2 – ABS/PLA 50:50 Blend (100 % Infill)

- The injection-molded composite blend displayed **poor recovery**, with neither polymer component responding substantially when heated near Tg.

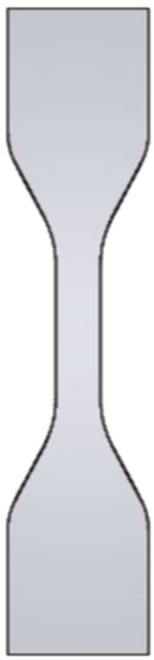
50% PLA + 50% ABS (100% Infill)

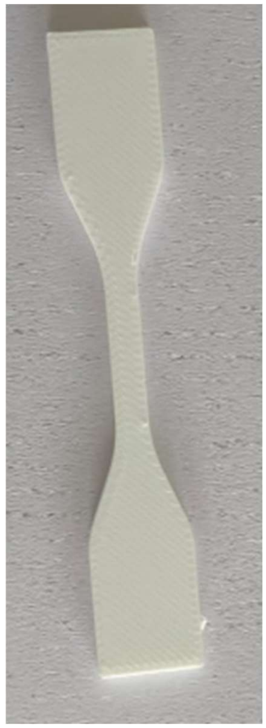
Discussion: Intended to fuse flexibility and durability, the blend appears to compromise both. When processed at full infill, neither component could facilitate shape recovery effectively.

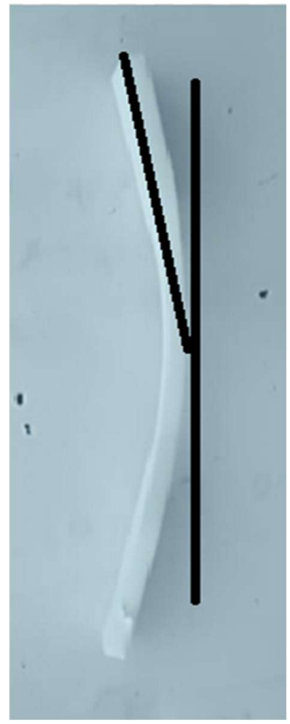
Phase 3 – Variable PLA Infill Densities

Infill Density	Recovery
30 %	~35%
20 %	~50%
15 %	~63%

Samples of 30% Infill –

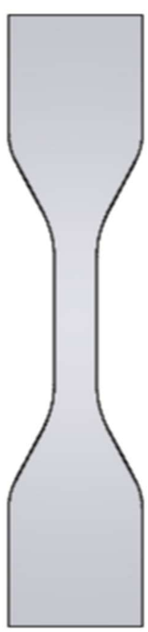


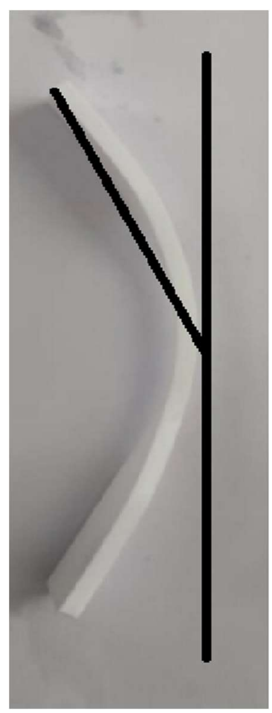




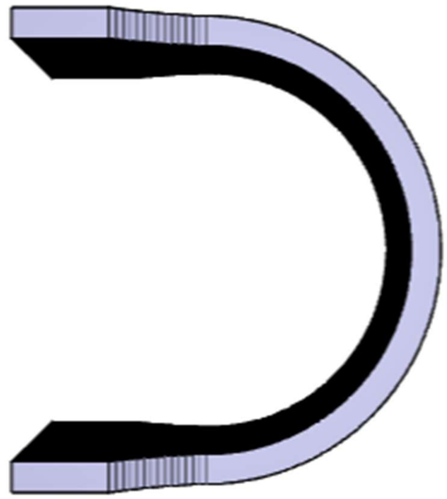
Initial Solid works file Printed sample After heated sample (12degree angle)

Samples of 20% Infill –





Initial Solid works file Printed sample After heated sample (25degree angle)

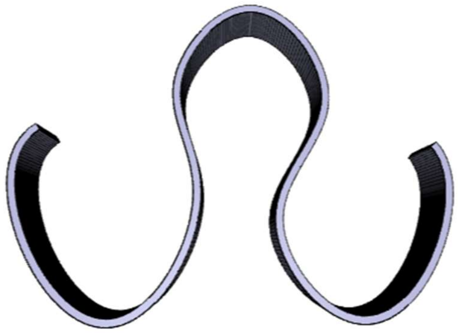


Initial Solid works file After heated sample

Discussion: Compared to the original sample, it shrank in thickness in the centre and spread outward from the sides.

Initial Solid works file After heated sample

Discussion: In contrast to the initial sample, it grew outward from the sides.



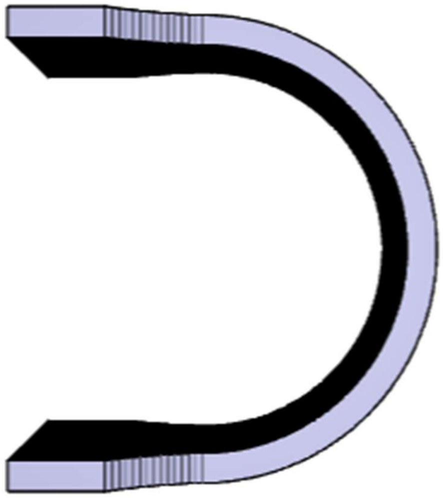
Initial Solid works file After heated sample

Discussion: In contrast to the original sample, it grew from the middle of the section.

Samples of 15% Infill –

Initial Solid works file After heated sample

Discussion: In contrast to the initial sample, it grew outward from the sides.




Initial Solid works file After heated sample

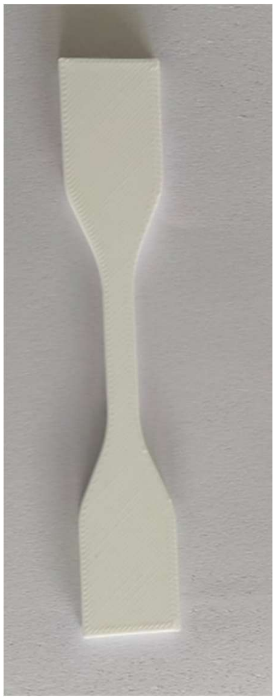
Discussion: Compared to the original sample, it shrank in thickness in the centre and spread outward from the sides.


Initial Solid works file After heated sample

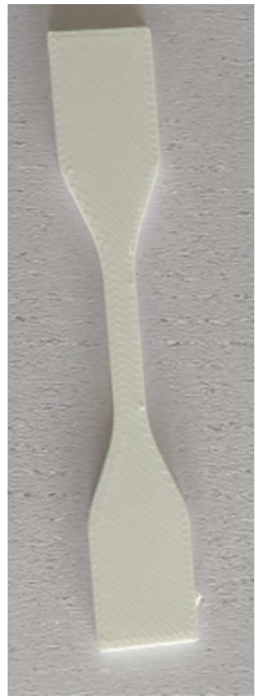
Discussion: In contrast to the original sample, it grew from the middle of the section. Results illustrate a clear trend: **lower infill density enhances bending capacity and shape recovery**. Open structure promotes swift heating and greater deformation, allowing more stored elastic energy to be released. Literature confirms that porous PLA structures (20–80% infill) recover ~80% in ~16–17 s, while 100% infill requires ~22 s due to slower heat penetration.

Phase 4 – Infill Pattern Differences

- **Grid infill** showed significantly greater deformation and recovery than **honeycomb infill**, which remained more rigid.

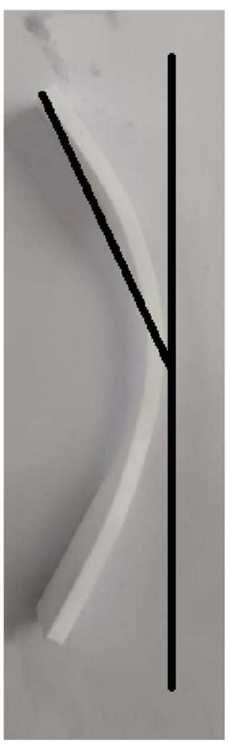


Grit Structure sample



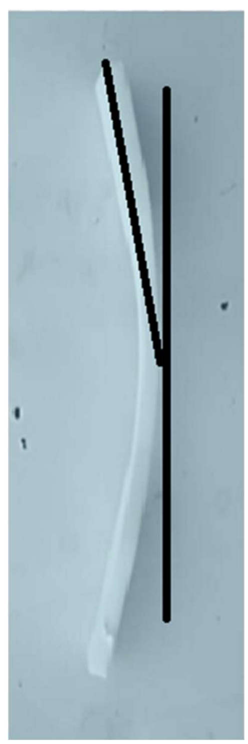
Honey-Comb Structure sample

Results –



Grit (after heated sample)

Angle = 25degree



Honey-Comb (after heated sample)

Angle = 12 degree

Discussion: Honeycomb structures prioritize strength over flexibility, hindering shape change. Studies suggest that while infill pattern has limited effect on recovery itself, it significantly influences mechanical rigidity and force distribution.

Phase 5 – SLA Infill & Geometry Variations

- **SLA samples** could not be evaluated due to unknown Tg.
- **Geometric variations** (strips, beams, curved shapes) in PLA demonstrated that shape recovery depends on a combination of infill, pattern, and overall design.

Sample from SLA Resin

Discussion: SLA offers high precision for SMP structures once Tg is known. Meanwhile, geometry interacts with infill to affect bending and recovery behaviour. These relationships align with studies highlighting the influence of porosity and internal structure on SME performance.

<u>Overall Discussion</u>

1. **Material is critical**: PLA, with its lower Tg and flexibility, outperforms ABS and blends in thermal activation.
2. **Infill density matters**: Decreasing infill from 100% to 15% boosts recovery from negligible to ~63%.
3. **Design structure is influential**: Grid patterns support deformation better than honeycomb, aligning with mechanical and SME data.

4. **Geometry and heat transfer**: Smaller, porous, and grid-structured samples reach recovery faster and more completely.

10. Conclusions

This This study demonstrates that **PLA**, more so than **ABS** or **ABS/PLA blends**, can reliably exhibit shape recovery via thermal activation. Initial trials with fully dense (100 %) PLA showed only modest restoration after immersion near its Tg (~60–65 °C), while injection-moulded ABS remained largely unrecovered due to its higher rigidity and Tg (~105 °C). A 50/50 ABS–PLA composite also failed to deliver satisfactory shape-memory performance.

Pivoting to lower-density PLA specimens produced dramatic improvements:

- **30 % infill PLA** achieved ~35% recovery.
- **20 % infill PLA** reached ~50% recovery.
- **15 % infill PLA** peaked at ~63% recovery.

These results highlight a clear trend: reducing infill markedly enhances bending flexibility and shape restitution. They align with literature showing that **lower infill densities favour faster and more substantial recovery**, while higher densities support more material volume but hinder responsiveness.

Further, infill pattern played a crucial role: **grid patterns**, with more flexible open architecture, facilitated superior bending and recovery compared to **rigid honeycomb structures**—mirroring existing evidence that infill geometry significantly influences shape-memory behaviour.

We also explored SLA-sourced patterns but lacked Tg information, preventing recovery analysis. Additional geometric variations of PLA samples further confirmed that **internal structure and external shape collaboratively determine performance outcomes**.

In summary:

1. **Material selection**: PLA is distinctly more amenable to low-temperature shape recovery than ABS or hybrid blends.
2. **Infill density**: Lower densities (15–30%) are optimal for achieving enhanced bending flexibility and recovery.
3. **Infill pattern**: Simpler grid designs outperform honeycomb in facilitating reversible deformation.
4. **Design synergy**: The interaction of geometry, density, and pattern dictates timing and extent of shape-change.

These findings underscore strategic design pathways—materials, infill, and shape—to engineer effective 4D-printed components. They also highlight the need for further research into infill combinations, alternative smart polymers, and thermal cycling protocols to optimize performance in adaptive systems across biomedical, soft robotic, and responsive-structure applications.

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