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# 4D Printing of Stimuli-Responsive Materials

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**Abstract**

Not only can additive manufacturing technology print complex geometries with remarkable accuracy, but it can also precisely program and control the geometry, materials, structures, and properties of printed objects. This allows for great design flexibility, high customizability, and significant material savings for the products. But one significant drawback of traditional additive manufacturing technology, commonly referred to as three-dimensional (3D) printing, is that the printed parts are inert, immobile, and stiff. Stated differently, the structures that are 3D printed are not able to adjust or adapt in response to modifications in the dynamic surroundings. Due of this, 3D-printed parts are unable to satisfy the growing needs of devices for intelligent functions including self-adaptation, self-assembly, self-repair, self-learning, self-sensing, and decision making. In this regard, an inventive subset of additive manufacturing known as four-dimensional (4D) printing, an innovative branch of additive manufacturing, emerges based on the multidisciplinary integration of materials, machinery, mechanics, information, and so on.Using additive manufacturing, 4D printing creates dynamic components with forms, qualities, and/or functions that can be controlled and changed automatically over time and/or space in response to preset stimuli such heat, moisture, light, pH, magnetism, and electricity. This concept emphasizes that 4D-printed devices' stimuli-responsive behaviors extend beyond simple form modifications to encompass property and functionality changes as well..

**Keywords:** Stereolithography Apparatus (SLA); Additive Manufacturing (AM); Ultra Violet (UV); Solid Creation System (SCS); Electro-Opt ical Technology (EOT); Fused Deposition Model (FDM); and Continuous Liquid Interface Production (CLIP)

## Introduction

In addition to its exceptional ability to print complex geometries, additive manufacturing technology can precisely program and control the geometry, materials, structures, and properties of printed objects, providing products with great design flexibility, high customizability, and substantial material savings. But one major drawback of traditional additive manufacturing technology, commonly referred to as three-dimensional (3D) printing, is that the printed parts are inert, immobile, and stiff. Stated differently, 3D-printed structures are not able to adapt to changes in the dynamic environment around them. Because of this, 3D-printed parts are unable to fulfill the growing expectations for intelligent device performances, including Self-adaptation, self-assembly, self-repair, self-learning, self-sensing, and decision-making. In this setting, a novel area of additive manufacturing known as four-dimensional (4D) printing develops as a result of the interdisciplinary integration of materials, equipment, mechanics, information, and other fields. [1-8]

### Definition of 4D Printing

In 2013, Skylar Tippets, the head of the Self-assembly Lab at the Massachusetts Institute of Technology (MIT), demonstrated an additively created soft strand that could become the automatically formed the letters "MIT" when submerged in water. By means of this exhibit, he initially put out the phrase "4D printing." The shape change over time is how the fourth dimension is defined here. The technique of 4D printing began

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with this. The quarterly American journal Foreign Affairs released an article headlined "Get ready for the 4D printing revolution" in October 2014, sparking interest in 4D printing among people from all walks of life. Since then, both academia and industry have seen a rise in 4D printing research. [9, 10]

"3D printing plus the fourth dimension of time" was the original definition of 4D printing. Furthermore, the shape change that occurs in 4D-printed components over time is the primary transformation. However, as 4D printing technology progresses, the idea behind it has continued to develop and get better. The first academic symposium on 4D printing technology was held in Wuhan, China, in 2017 thanks to the efforts of Yusheng Shi, a professor at Huazhong University of Science and Technology, and other pertinent professionals in China. He put up a definition of 4D printing that was more thorough. [11-15]

**Prospect of 4D Printing**

In conventional manufacturing technologies, material preparation, structural design, and functional realization are often three separate processes. On the other hand, 4D printing enables the integrated manufacture of material, structure, and functionality and streamlines the process from concept design to finished goods. It also incorporates the design of intelligent behaviors into the fabrication processes. [16-19]

It holds the potential to transform conventional design ideas and practices, bringing about a significant revolution in conventional manufacturing technology. Because 4D printing allows for the direct implantation of intelligent performances into printed items without the need for external electromechanical systems, it is also anticipated to bring about an innovative development in high-end and microscale intelligent devices. [20, 21]

It offers an alternative to sophisticated intelligent devices that are challenging to produce using standard manufacturing techniques, in addition to having the ability to eliminate the inherent weakness of electromechanical systems in conventional intelligent devices. Furthermore, by employing the stimuli-responsive shape-morphing capability of 4D-printed components, the items may be created in a small, easily stored configuration before being activated to their full volume when needed. [2]

## Materials of 4D Printing

Using additive manufacturing techniques, D printing creates structures that are sentient and have tunable responses to inputs. Since the fourth dimension of 4D-printed components relates to the stimuli-responsive properties rather than the forming processes, the majority of forming techniques employed in 4D printing are now typical 3D printing techniques. The ability of 4D-printed components to respond to stimuli is often attained by the thoughtful design, use, mixing, and positioning of stimuli-responsive materials. [22]

The capacity to print well is the primary need for stimuli-responsive materials intended for 4D printing. In other words, additive manufacturing methods allow for the effective fabrication of materials. This is necessary in order to accomplish 4D printing. At the moment, the standards used to assess the printability of 4D printing materials are essentially the same as those used to assess traditional 3D printing materials, with the primary difference being the methods used to create the material during printing. For instance, photosensitive liquid materials with quick curing times, little curing shrinkage, low viscosity, and suitable projection depths should be utilized for 4D printing based on stereolithography equipment (SLA). Powder materials with tiny, homogeneous particle sizes, good sphericity, high fluidity and high apparent density should be utilized for selective laser melting (4D) printing. [23]

Furthermore, following additive manufacture, the materials used in 4D printing can exhibit controlled stimuli responsive behaviors. This is the cornerstone and secret to achieving 4D printing since the qualities of the materials employed, as well as their combination and arrangement in three dimensions, primarily determine how stimuli-responsive 4D-printed things are. A class of materials known as "intelligent materials" are ones that have the ability to perceive changes in their surroundings and adapt by changing their forms, physical characteristics, or chemical makeup. [24]

As a result, the most obvious choice for stimuli-responsive materials for 4D printing is intelligent material. But not all intelligent materials can be utilized in 4D printing; some cannot be produced using additive manufacturing technologies, while others may lose their ability to respond to stimuli as a result of additive manufacturing. However, 4D printing can also be accomplished by using some non-intelligent materials, or traditional materials, which do not have any controllable stimuli-responsive capacity of their own. These materials can, however, be made to respond to stimuli through proper programming of their composition and structure, presetting of a stimulus signal, or combining different materials. This is true even though the majority of current reports on 4D printing are based on intelligent materials. [14]

Despite the fact that hundreds of materials for 4D printing have been created in the last ten years, the majority of them are only now able to

demonstrate phenomena or match the performance criteria of 4D-printed structures. The biggest obstacle to the development and use of 4D printing is the absence of high-performance stimuli-responsive materials. It is therefore imperative to carry out research and create novel stimuli-responsive materials for 4D printing in order to enhance the functionality of these components and encourage real-world uses. Reviewing the scientific developments in stimuli-responsive materials for 4D printing in a timely manner is crucial. At the moment, polymers and their composite materials, metals and their composite materials, and ceramics according to their chemical materials can be categorized as stimuli-responsive materials for 4D printing. [25-34]

## 4D Printing of Polymers and Their Composite Materials

The most popular class of materials for 4D printing is Stimuli-responsive polymers and their composites since they are easy to build using additive manufacturing methods and have a wealth of material systems. Furthermore, in comparison to metals and ceramics, stimuli-responsive polymers and their composite materials provide the benefits of cheap cost, low density, and straightforward design. Direct ink writing (DIW), fused deposition modeling (FDM), selective laser sintering (SLS), Poly Jet, stereolithography apparatus (SLA), and digital light processing (DLP) are the additive manufacturing processes most often utilized in 4D printing. The stimuli-responsive polymers that are now on the market, together with their composite materials for 4D printing, may be broadly categorized into five categories: thermo-responsive, moisture-responsive, magnetism-responsive, electricity-responsive, and light-responsive polymeric materials.

### *Thermo-responsive Polymers and Their Composite Materials*

Thermo-responsive polymers and the composite materials made of them are able to sense changes in ambient temperature and adapt by changing their functions, forms, or characteristics. Currently, liquid crystal elastomers (LCEs), thermos-responsive gel materials, and thermos-responsive shape-memory polymers (SMPs) are the most common types of thermos-responsive polymers and their composite materials. [6, 35-68]

### *Thermo-responsive Shape-memory Polymers*

One class of smart polymer materials known as thermo-responsive SMPs is distinguished by its capacity to regain its original form after being heated to a transient state. Advantages of thermo-responsive SMPs include great recovery strain, easy shape-programming, low cost, light weight, and plentiful raw materials. These thermos-responsive materials have been the most widely used ones for 4D printing. Through physical and chemical cross-links relating to the transition temperature Tt—typically the melting temperature Tm for a semicrystalline polymer and the glass transition temperature Tg for an amorphous polymer thermos-responsive SMPs react to variations in the surrounding temperature. They typically consist of a reversible phase that can perform a reversible transformation between two states in response to external inputs and a stationary phase for remembering the starting shape. [69]

The thermos-responsive SMPs often have high Tt values and are cross-linked structures, either chemically or physically. When the SMPs are deformed under external stresses, they are strong enough to prevent permanent plastic deformation and only cause elastic deformation of the molecular chains. Physically or chemically cross-linked structures with low Tt values are often the reversible phases. With changes in temperature, they may reversibly transition between the soft and stiffening states. [70]

The shape-memory process of thermo-responsive SMPs contains three steps [39, 71]

- To soften the reversible phase, the thermos-responsive SMP is first heated above the Tt of the reversible phase but below the Tt of the stationary phase. At this temperature, the external tension is applied to the SMP in order to bend it into the necessary temporary shape. In this instance, the stationary phase is stretched and elastic.
- Next, with the applied stress, the deformed SMP cools to a temperature lower than the Tt of the reversible phase. The phase that is reversible solidifies. Additionally, the entropy elasticity is stored in the stationary phase due to the frozen stretched condition of the stationary phase's molecular chains. Following the removal of the external load, the SMP is fixed into a temporary configuration.
- Ultimately, the reversible phase becomes softer when the deformed SMP is heated over its Tt. Additionally, the stationary phase's molecular chains unfreeze and recover due to entropy elasticity. The link between the stationary and reversible phases causes the reversible phase's molecular chains to deform. The distorted SMP therefore returns to its original form.

Thermo-responsive SMPs and related composite materials have been widely employed to construct 4D-printed structures with thermos-responsive

shape-changing properties because of their substantial recovery strain and changeable Tt values.

Yang et al. used polyurethane SMP and FDM technology to print a variety of shapes, such as a gripper, rocket, flower, and airplane. After heating, the printed flower has the ability to spontaneously fold from a two-dimensional (2D) flat structure to a three-dimensional one. By printing the polycaprolactone SMP using SLA technology, Zarek et al. created the cardiovascular stent, Eiffel Tower, and bird models (Figure 1). The printed gripper could conduct a grabbing action when heated to temperatures over the Tt. When heated, these models may return to their initial, temporary forms automatically. [72]

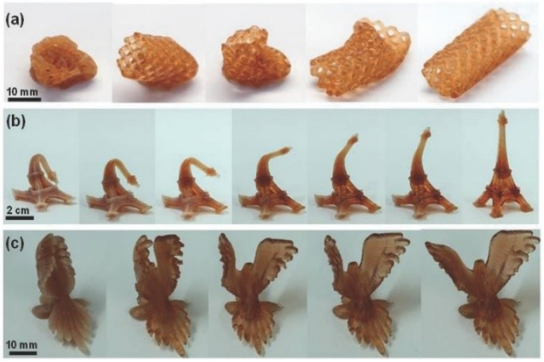


Figure 1: Cardiovascular stent, Eiffel Tower, and bird models

Using high-resolution projection micro-stereolithography (PμSL) technology, Ge et al. were able to print photo-curable methacrylate-based SMPs with varying Tt values in a single build, thereby completing the 4D printing of multi-material thermo-responsive structures. At 50 °C and 70 °C, respectively, the printed multi-material flowers could open their outer and interior petals in turn (Figure 2). [73]

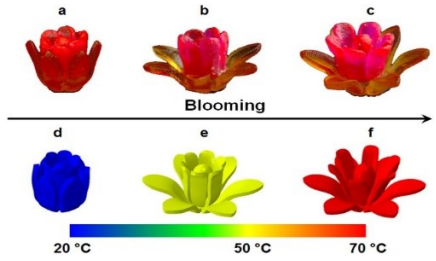


Figure 2: Blooming Flowers

As has been extensively documented, when the molten polymer filaments are extruded and stretched during printing, the homogeneous internal strain will be retained in the SMP structures created using FDM technology. When the FDM-printed SMP structures are heated above their Tt, the internal strain can be released, causing the SMP filaments to shrink in the direction of extrusion but expand in the direction of thickness. It is possible to develop 4D-printed structures that can accomplish controlled thermo-responsive shape-changing behaviors without the requirement for the shape programming phase by taking use of the anisotropy of FDM-printed SMPs. For example, Zhang et al. used FDM and polylactic acid (PLA) SMP to create 2D lattice materials. When heated, the circular rings in the printed lattice materials may transform into hexagons or quadrangles due to PLA filament heat shrinkage along the extrusion direction. [74]

Wang and colleagues used fused deposition modeling (FDM) to print polyester (PE) SMP on a paper substrate, resulting in the fabrication of thermos-responsive shape-changing bilayer structures. In this case, the paper layer functioned as a passive strain-limiting layer and the polymer layer as an active layer that responded to stimuli. The PE layer would shrink along the filament orientation when the bilayer structures were heated over the Tt of PE. This would cause the bilayer structures to distort because the paper layer would resist the PE layer's contracting deformation. The bilayer constructions could produce bending, twisting, and spiraling deformations by varying the angle between the length direction of the paper sheet and the contracting direction of the PE layer (Figure 3). [75]


Figure 3: Stimuli-responsive paper sheet

*Liquid crystal elastomers*

LCEs, or liquid crystal materials with cross-linked main-chain or side-chain segments, are a type of flexible polymeric material with low cross-linked density. A significant anisotropic contraction deformation will occur on a macroscopic level as a result of the LCEs changing from the liquid crystal phase with elongated molecular chains to the isotropic phase with contracted molecular chains when heated. Structures with thermos-responsive shape-changing properties that are produced by 4D printing have been made possible by the special heat-shrinkage capabilities of LCEs. [76, 77]

A high-operating temperature DIW technique for LCEs was created by Kotikian et al. By manipulating the DIW print route, they were able to program the anisotropic heat contraction of LCEs and produce a range of LCE designs that exhibited shape-morphing behaviors during heating, both in

two dimensions and three dimensions (Figure 4). Similar to this, Ren et al. suggested a 4D printing programming technique for fabricating single-material constructions of LCEs. By locally adjusting the printing route and speed during the DIW process, this approach might demonstrate a variety of shape-changing behaviors to program the alignment degree of molecules. Using DIW technology, López-Valde Olivas et al. achieved digital control over the local anisotropy of LCEs and produced thermo-responsive LCE structures. [78]

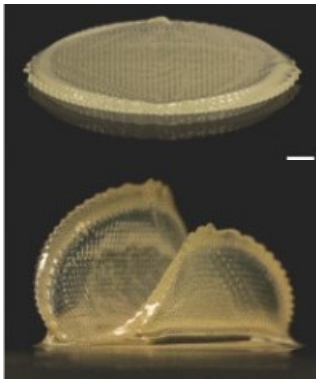

Figure 4: Programmable shape morphing liquid crystal elastomers

*Thermo-responsive Gel Materials*

Thermo-responsive gel materials are networks of self-adoptable macromolecules that are physically or chemically coupled. They have the ability to expand and contract, changing their structure in response to changes in temperature. Another class of appealing polymeric materials for 4D printing are thermo-responsive gel polymers and their composites. [79]

By printing orthogonally continuous thermo-unresponsive poly(ethylene glycol) (PEG) components in a thermo-responsive poly(N-isopropyl acrylamide) (PNIPAm) matrix, Arslan et al. created bilayer gel actuators. The passive anisotropic PEG pattern inhibited the heat-swelling and shrinking of the active isotropic PNIPAm matrix along the direction of continuous PEG filaments. Bending and twisting were among the actions that the printed multi-material constructions might execute by varying the shape and orientation of the PEG parts. [80]

Chen et al. created porous PNIPAm substrates with randomly oriented fibers by using the electrospinning process. Afterwards, to produce programmed anisotropic swelling performances, several well-designed stiff PNIPAm/clay patterns were manufactured using 3D printing technology on the porous substrates. By doing this, they produced a line of gel actuators that are thermos-responsive and have improved designability in three-dimensional forms in addition to rapid deformation. In a separate study, Chen et al. printed a bilayer leaf made of two distinct gel materials PNIPAm gel and polyacrylamide (PAAm) gel using DIW technology. The gel leaf twisted as a result of the PNIPAm layer contracting and the PAAm layer expanding when submerged in 50 °C water. By designing the printing path to program the anisotropy of the printed structures, various shape-changing behavior could be achieved in an identical structure. [81, 82]

The 4D-printed gel tubes made of a thermo-responsive swelling PNIPAm gel and a thermo-nonresponsive PAAm gel were conceived and manufactured by Liu et al. Axial elongation, radial expansion, and bending are just a few of the thermos-responsive shape changes that these gel tubes may accomplish by adjusting the symmetric and segmented arrangements of the passive PAAm gel and active PNIPAm gel within the tubes. Jin et al. created a composite gel by incorporating Laponite nano clay into the PNIPAm gel, which improved its self-supporting printability, and a composite gel by including graphene oxide (GO) into the PNIPAm gel, which improved its temperature responsiveness. The PNIPAm-Laponite and PNIPAm-GO composite gels have distinct shrinkage ratios and mechanical characteristics, which might cause the multi-material constructions to deform predictably when heated. [79]

By in-situ polymerizing acrylamide in an agarose matrix, Guo et al. created a shape-memory gel material that could be printed in the air. Based on the sol-gel transformation characteristic of agarose, this gel material may process a reversible transition between soft and hard states upon heating and chilling, permitting a thermos-responsive shape change from 3D structures into diverse patterns (Figure 5). [83]

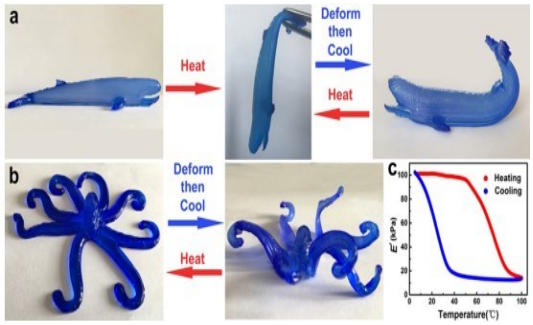


Figure 5: Hardening cycle of a whale and octopus like gel

***Moisture-responsive Polymers and their Composite Materials***

One type of exceptional hydrophilic polymer is a hydrogel, which is a three-dimensional network structure created by chemically and physically crosslinking monomers containing hydrophilic

groups. After absorbing water, they may grow up to 200% of their original volume while still keeping their original architecture intact. On the other hand, typical single-component hydrogels often have an isotropic swelling capability. That is, the single-component hydrogel structure experiences simply a linear expansion. In order to accomplish intricate and manageable moisture-responsive shape-changing actions such as bending, twisting, folding, and so forth, the hydrogel frameworks had to incorporate programmed anisotropic swelling performances.

By adjusting the monomer conversion and crosslinking degrees, one method for producing asymmetrical swelling in a single hydrogel structure is to produce zones with varying crosslinking densities. A photopatterned bilayer made of two PEG hydrogels with various molecular weights was created by Jamal et al. Due to the distinct swelling of the two PEG bilayers, this PEG-based bilayer might exhibit a self-folding tendency when submerged in aqueous solutions. [84]

Zhao et al. created programmed continuous adjustments in crosslinking density in the single-hydrogel structures using a grayscale photo-printing technique based on DLP. When the printed structure was submerged in water, the uncured oligomers inside the low crosslinking density regions diffused out of the structure, causing a contraction in these areas. The structure then experiences a folding deformation as a result (Figure 6). They created and developed a number of intricate origami constructions that could self-fold from flat configurations into three-dimensional forms using this idea as its foundation. [85-87]

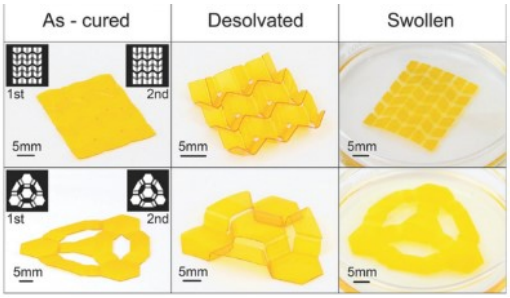


Figure 6: Desolvation and Swelling

Furthermore, by constructing programmable secondary structure grooves on one side of the highlighted structures, Ji et al. suggested a unique method to produce controlled asymmetrical swelling in the hydrogel architectures. Depending on the groove patterns, the asymmetrical swelling brought on by the structural asymmetry would produce local curvatures and cause the hydrogel structures to bend, twist, or even combine. The printed structures may process reversible and regulated moisture-responsive shape changes from 1D strips, 2D sheets, and even 3D structures to 3D morphologies by coding the groove patterns. The benefits of this strategy were simple material compositions, quick production, and flexible design. [88]

Adding anisotropic particles to the hydrogels is another method for producing anisotropic swelling performances. Gladman et al. used DIW technology to develop and create a hydrogel composite made of cellulose fibrils and acrylamide hydrogel, drawing inspiration from botanical systems. This biomimetic composite's anisotropic swelling behavior in water was caused by the nano-fibrillated cellulose's shear-induced alignment. By adjusting the printing parameters to modify the cellulose fibril orientation and subsequently programming the localized swelling anisotropy, they produced a range of bilayer architectures capable of handling water's varying shape changes and producing desired, intricate three-dimensional morphologies. [89]

Mulakkal et al. used a similar method to produce programmed swelling mismatch by carefully aligning the pulp fibers obtained from cotton in a carboxymethyl cellulose hydrogel utilizing the DIW technology. By using this technique, they created a petal architecture that could be hydrated to take on a planar shape then dehydrated to return to its original petal structure. [90]

Anisotropic swelling may also be achieved by combining many materials with various swelling tendencies into a single structure. Raviv et al. coded the proportion, form, size, and location of the two materials to print a hydrophilic acrylate hydrogel and a stiff polymer in a single structure that exhibited a variety of moisture-responsive shape-changing behaviors. [9]

Origami-inspired trilayer structures with hydrophilic cores and hydrophobic skins were shown by Baker et al. In this case, polyurethane elastomer served as the skin and polyurethane hydrogel as the core. In a single additive manufacturing structure, they integrated localized bilayer areas (active hinges) with a global trilayer composition (passive). Ultimately, they were able to accomplish predictable shape changes by strategically planning the bilayer regions' spatial locations inside the structures. [91]

Using DLP technology, Zhao et al. created a bilayer structure made up of a hydrophilic poly (ethylene glycol) dimethacrylate layer and a hydrophobic poly (propylene glycol) dimethacrylate layer. The strain mismatch between the two layers during swelling would cause a deformation of the bilayer structure when it was submerged in water. [92]

***Light-responsive Polymers and Their Composite Materials***

Light has a captivating quality. Its benefits include rapid switching, easy trigger, clean/no pollution, accurate focusing, and non-contact control. One type of polymeric material that may process chemical and/or physical processes in response to changes in light is light-responsive polymers and related composite composites. The polymers and their composites often change in form, property, or functionality as a result of these reactions. The use of light-responsive polymers in composite materials and 4D printing has grown in popularity.

Wales et al. created a unique photochromic nanostructured polyoxometalate that, when exposed to visible light, could transform from colorless to blue and, when exposed to oxygen, could revert to colorless through a redox process. In order to create a photochromic polymeric material suitable for additive manufacturing and produce macroscopic functional devices with reversible photochromism, this photochromic molecule was introduced to a custom polymeric ionic-liquid matrix. [93]

Gelebart et al. described the use of liquid crystal polymers containing a pH- and light-responsive purple azomerocyanine (1-Am) dye to create an actuator with rewritable and reprogrammable light-responsive features. By employing light irradiation with varying colors, it was possible to programmatically fold the actuators due to the dyes' sensitivity to varying wavelengths of light. [94]

Liu et al. patterned the polymer sheets using various colored inks. The light absorbance of these inks varied based on the light's wavelength. The printed color inks selectively produced heat in response to differing amounts of external light because of their varying absorption of light. This sequentially relieved local strain and caused sequential folding deformation in the areas covered with ink. This idea allows these colored ink-covered patches to function as a programmed light-responsive hinge. Liu et al. used this process to produce 2D polymer sheets that, when stimulated by light from the outside, could successively self-fold into 3D designs. [95]

Another well-liked method for creating light-responsive materials for 4D printing is to combine materials having photo-thermal effects, such as graphene, carbon nanotubes, carbon black, and gold nanorods, with thermo-responsive polymeric polymers. When exposed to light, these photo-thermal particles can generate heat, which in turn causes the thermo-responsive polymeric matrix to alter in form, property, and/or functionality. By incorporating photo-thermal carbon black (CB) into a thermo-responsive polyurethane (PU), Yang et al. created a light-responsive SMP. With the use of this composite material and FDM technology, they produced a light-responsive sunflower that could open its petals in response to 87 mW/cm2 of light. [96]

Hua et al. constructed flexible light-responsive shape-changing actuators by printing a light-responsive composite material made of PLA thermal SMP and multi-walled carbon nanotubes (MWCNTs) on paper substrates using FDM technology. [97]

Zhang et al. combined the agent aniline trimer (AT) with a thermo-reversible shape-memory polymer (PU) to create a light-responsive polymeric composite. By using DIW technology to print this photothermal DA-reactive PU, they were able to create things that resembled hands and butterflies and had shape-memory properties that could be programmed in response to light. [98]

The PNIPAm ink containing photothermal multiwalled MWCNTs was printed on a polydimethylsiloxane (PDMS) substrate by Cheng et al. using DIW technology. Because of the asymmetric thermal reactivity of the PNIPAm and PDMS layers, this bilayer structure would bend when heated. An artificial tendril capable of rotating bending under rotating light irradiation was constructed by strategically placing and forming PNIPAm and PDMS elements inside a single framework. [99]

Furthermore, Luo et al. described a straightforward method for printing alginate/gelatin methacryloyl (GelMA) and alginate/polydopamine (PDA) bio-inks to create near-infrared-triggered cell-laden scaffolds with programmable shape modification. The light intensity, length of irradiation, and alginate/PDA strut design patterns inside the scaffolds may all be used to control the deformation of these orthogonal bilayer scaffolds. [100]

Zhao et al. created a simple process for creating light-responsive origami structures by taking advantage of the volume shrinkage of acrylate resin during photopolymerization. The addition of photo-absorbers to the polymer resin resulted in an attenuated light field and non-uniform curing in the thickness direction. Consequently, a nonuniform volume shrinkage was created, which in turn created a nonuniform stress field and caused the sheet to bend into a three-dimensional structure. [101]

### *Magnetism-responsive Polymers and Their Composite Materials*

The external magnetism field triggers the form, property, and/or functionality change behaviors of magnetism-responsive polymers and related composite materials. At the moment, thermo-responsive polymers constitute the foundation for the development of various magnetism-responsive polymeric materials for 4D printing. In essence,

they are creative augmentations of thermos-responsive polymers. By incorporating magnetic particles (such as Fe3O4, NdFeB, Fe, and Ni nanoparticles) into thermos-responsive polymers, this type of magnetism-responsive polymer is created. The oscillating magnetic field outside would cause these magnetic particles to reciprocate. Heat will be produced by their friction and collisions with molecules, raising the temperature and, as a result, altering the structure, characteristics, and/or functioning of the thermos-responsive polymeric matrix. Wei et al. created a magnetic-field-responsive SMP by adding Fe3O4 nanoparticles to the PLA SMP, based on the above-mentioned approach. With this SMP composite, they produced a range of biological scaffolds that respond to magnetism. Similar to this, Lin et al. used thermo-responsive PLA materials combined with $Fe_3O_4$ nanoparticles to create the shape-memory occluders that were 4D printed. Under the influence of an external magnetic field, these occluders may return from a distorted shape to their original shape after implantation. Zhu et al. created a composite ink using PDMS and iron nanoparticles that was used to create quickly responding, magnetism-responsive 4D-printed objects. [102]

Kim et al. created a DIW printer with a magnetic field surrounding the dispensing nozzle and a unique composite ink that included fummed silica nanoparticles and NdFeB microparticles. The manufactured filaments have the designed magnetic polarity because the nanoparticles were reoriented along the applied field during the printing process. With the use of this technique, they were able to program ferromagnetic domains in intricate soft material systems and produce a variety of structures that showed intricate form changes in response to applied magnetic fields. [103]

Shinoda et al. used an external magnetic field to incorporate a programmed magnetic anisotropy into UV-curable gel material that was mixed with two types of magnetic particles: carbonyl iron powders and strontium-ferrite particles. Under an external magnetic field, aligned magnetic particles in various printed structure parts produced distinct rotational moments, resulting in a predetermined movement. They developed a range of biomimetic actuators with a magnetic field response by employing this technique. Using SLS technology, Wu et al. created 4D-printed grippers that respond to magnetism by utilizing a composite powder made of thermoplastic polyurethane and magnetic NdFeB. Remotely controlled deformation of the printed grippers was seen under an external magnetic field. [104]

Layer by layer, Bastola et al. created a novel class of 4D-printed magnetorheological (MR) hybrid elastomer structures by using DIW to encapsulate an MR fluid within an elastomer matrix. Here, the MR fluid and the elastomer made up each layer of the composite design. The mechanical characteristics of these printed MR-elastomer composite structures may be controlled by varying the external magnetic field. [105, 106]

On the basis of a related idea, Jackson et al. also introduced a novel architected material, whose mechanical properties that respond to magnetism may be dynamically controlled and temporally altered by careful material composition and structure selection and design. They created intricate structures via printing polymeric tubes filled with MR fluid. Through manipulation of the applied magnetic field's intensity, the printed composite structures have the potential to display significant, fast, and reversible variations in stiffness. [107]

**4D Printing of Stimuli-responsive Metals and Their Composite Materials**

The superior carrying and actuation capabilities, better high/low temperature performances, and faster response rate of stimuli-responsive metals and their composites are drawing more attention in 4D printing, despite the fact that they are heavier and more expensive than stimuli-responsive polymers and their composite materials. Currently, shape-memory alloys (SMAs) and related composite materials are the primary stimuli-responsive metals and materials for 4D printing. One class of functional metal materials that exhibits super-elasticity (SE) and the shape-memory effect (SME) is called SMAs. The SME phenomenon refers to the ability of an alloy that has been temporarily shaped by applied stress or a magnetic field to revert to its original shape in response to certain stimuli, such as temperature changes or magnetic field fluctuations. [108]

The phenomenon known as the SE occurs when an external force causes the alloy to temporarily change shape; upon unloading, the alloy returns to its previous shape without the need for additional stimulation. The austenite (stable at high temperature) and martensite (stable at low temperature) phases undergo a reversible transition that gives rise to both the SME and SE. The combinational functions of sensing, driving, controlling, carrying, energy conversation, self-assembly, and self-adaptation are realized by SMAs because of their distinct SME and SE.

SMAs have a lot of potential uses in 4D printing as a result. Nowadays, wire and arc additive manufacturing (WAAM), binder jetting, laser engineered net shaping (LENS), selective laser melting (SLM), selective electron beam melting (SEBM), and wire and arc additive manufacturing (WAAM) are the most widely utilized additive manufacturing methods in the 4D printing of

SMAs. In 4D printing, several SMA systems have been employed. They may be divided into two groups based on the types of stimuli: thermo-responsive and magnetism-responsive SMAs. [108]

### *Thermo-responsive Shape-Memory Alloys and Their Composite Materials*

*Mechanisms of thermal shape-memory effect and super-elasticity*

Heat activates the SME of thermos-responsive SMAs. The thermo-responsive SME is characterized by two mechanisms: (a) thermal martensite reorientation produced by stress, and (b) martensitic transformation induced by stress. The SMAs in mechanism (a) are at the deformation temperature in a martensite condition. External stress will cause the self-accommodating thermal martensite (twin martensite) variations to reorient to the martensite variant that is beneficial for the direction of the stress, resulting in the detwinned martensite. This will cause a macroscopic distortion in the SMAs. Furthermore, because the distorted martensite is preserved, the deformed shape endures when the applied force is withdrawn. The detwinned martensite reverts to the original austenite upon heating above the completion temperature of the reverse martensitic transition Af, resulting in recovery from the distorted shape to the original one. The SMAs in mechanism (b) are at the deformation temperature in an austenite condition. The stress-induced martensitic transformation assumes the deformation of the SMAs under external stress. The distorted shape is maintained because the stress-induced martensite remains at this deformation temperature after unloading. The stress-induced martensite turns back into the original austenite when the SMAs are heated over Af. As a result, the distorted alloy takes on its original form. In contrast to SME, where heating or altering the magnetic field is required for the deformed alloy to regain its original shape, shape recovery in SE occurs automatically upon the removal of applied stress and doesn't require any further stimuli. The stress-induced martensitic transformation that results from applying external stress to SMAs above Af but below M is the process responsible for the macroscopic deformation known as SE. When the external load is released, the stress-induced martensite will retransform into austenite since it is unstable at this deformation temperature. Consequently, after unloading, the macroscopic distortion returns. [109]

### *NI-TI-Based shape-memory alloys*

The most researched and advanced thermo-responsive SMAs now fall into three categories: Ni–Ti–based, Cu–based, and Fe–based. More specifically, because Ni–Ti alloys have the best SME and SE together with strong biocompatibility, high recovery stress, remarkable corrosion resistance, outstanding wear resistance, good functional stability, and exceptional mechanical characteristics, they are the major focus of current investigations on 4D printing of SMAs. [110-113]

The basis for 4D printing of Ni-Ti SMAs is the production of high-quality, high-performing additive manufacturing. As a result, a lot of research has been done with an emphasis on process optimization and performance management for Ni-Ti SMAs that are built additively.

The impact of laser power and scanning velocity on the microstructures, behavior of martensitic transformation, and SE of Ni–Ti SMAs produced using SLM technology were methodically examined by Saedi et al. It was discovered that the combinations of low laser power with a low scanning velocity or high laser power with a high scanning velocity were recommended in order to create totally dense Ni–Ti components. Compared to Ni-Ti components made with high laser parameters, those made with low laser parameters process with a superior SE. They were successful in producing dense Ni–Ti with a high relative density of almost 99% and a high SE recovery strain of 5.77% by using an optimal combination of 100 W and 125 mm/s. [111]

The impact of laser volumetric energy density on the mechanical characteristics, SME, and martensitic transformation behaviors of Ni–Ti SMAs produced using SLM technology was investigated by Lu et al. The findings demonstrated that when the laser's volumetric energy density is lowered, so are the martensitic transition temperatures. While the Ni-Ti SMAs printed with a laser volumetric energy density of 155 J/mm3 showed the maximum recovery strain of 4.99%, the Ni-Ti SMAs printed with a laser volumetric energy density of 222 J/mm3 revealed a high tensile strength of 776 MPa and a considerable elongation of 7.2%. [114]

The SME in the SLM-printed Ni–Ti SMAs made with mechanically mixed powders and high laser volumetric energy densities of 375–675 J/mm3 was measured by Zhao et al. utilizing a bending test. When the Ni–Ti samples with 675 J/mm3 were bent 6% at room temperature, the recovery strain increased to 5.17%. [115]

A U-shaped Ni–Ti component with multi-stage shape recovery characteristics was produced by Ma et al. using SLM. It was accomplished by adjusting the hatch spacing at various U-shaped component sections to regulate the temperatures at which martensitic transformation occurs, resulting in an active reaction that is location-dependent. [116]

### *Cu-based shape-memory alloys*

Cu-based SMAs are inexpensive (about one-tenth of the cost of Ni–Ti-based alloys), offer excellent thermal and electrical conductivity, strong damping performance, good SME and SE (recovery strain up to 8%), and a tunable martensitic transformation temperature range (−90–120 °C). As a result, there is growing interest in Cu-based SMAs for 4D printing. Cu-based SMAs may be broadly categorized as Cu-Zn–Al, Cu–Al–Mn, and Cu–Al–Ni based SMAs based on their chemical composition. Cu-Al-Ni-based SMAs have garnered the most interest among them because of their effective stability against aging-related phenomena. [109]

The Cu–Al–Ni–Mn alloy was initially made by Mazzer et al. utilizing SLM technology. Single monoclinic martensite microstructures, no fractures, and very dense Cu-Al-Ni-Mn materials with fine grains (10–100 μm) were produced. [117]

The impact of processing settings, scanning techniques, and extra re-melting stages on the microstructures and mechanical characteristics of the Cu–Al–Ni–Mn alloy made using SLM technology was then investigated in more detail by Gustmann et al. [104–106]. The findings demonstrated that between 30 and 40 J/mm3 is the ideal laser volumetric energy density. A complete martensite microstructure and a high relative density of 99% could be seen in the printed samples when a laser power of 300 W and a scanning speed of 700 mm/s were used. The SLM printed samples had a fracture strength of 1515 ± 50 MPa and a fracture strain of 18 ± 1.7%, respectively. These values were similar to dense suction-cast sample fracture strain (16 ±0.2%) and fracture strength (1500 ± 30 MPa). [118-120]

A Cu–Al–Ni–Ti alloy was also successfully produced by Tian et al. using SLM technique. The Cu–Al–Ni–Ti samples printed by SLM had an average grain size of 43 μm and a relative density of 99.5%, respectively. These samples were nevertheless inferior to cast samples, with an elongation of just 7.6% and an ultimate tensile strength of 541 MPa. The aforementioned studies demonstrate that thick and fine Cu-based SMAs can be manufactured by additive manufacturing. However, the few available publications mostly concentrate on the process parameter optimization that leads to the development of the mechanical properties and microstructures of Cu-based SMAs that are made using additive manufacturing. However, there haven't been many studies published on the shape-memory capabilities of Cu-based SMAs produced additively. To fully realize the promise of Cu-based SMAs in 4D printing, more research is still desperately needed. [121]

***Fe-based shape-memory alloys***

Fe-Mn-Sibased, Fe-Mn-Al-based, and Fe-Ni-Co-Al-based SMAs are the three primary categories of thermos-responsive Fe-based SMAs. The lowest cost, superior formability, and outstanding mechanical characteristics are the benefits of Fe-based SMAs, despite their marginally lower SME and SE when compared to Ni-Ti-based and Cu-based SMAs. They provide a wide range of potential applications in the large-scale, 4D printing of intelligent structural elements. The first Fe–Mn–Al–Ni alloy was created using SLM technique by Niendorf et al. in 2016. When compressed by about 11% at −100 °C, the printed sample that underwent simple solution treatment and age treatment showed a high SE recovery strain of 7.5%. This work shows that additive manufacturing technologies may be used to produce Fe-based SMAs with high shape memory performance. To the best of the authors' knowledge, no other relevant research have been published. There is much more to be discovered about the possibilities of Fe-based SMAs in 4D printing. [122]

***Magnetic Shape-Memory Alloys and Their Composite Materials***

Another name for magnetic SMAs is ferromagnetic SMAs. The change in the magnetic field activates their SME. By virtue of being activated by magnetic fields rather than the comparatively sluggish heat transfer process, magnetic SMAs have a substantially greater actuation frequency than thermo-responsive SMAs. Like the thermal SME, the magnetic SME has two mechanisms: (1) martensite reorientation produced by magnetic field; and (2) martensitic transformation induced by magnetic field. Currently, the two primary types of magnetic SMAs are Co-Ni and Ni-Mn-based SMAs. Because of their high magnetic-field-induced strain, the Ni–Mn–Ga SMAs are now the most researched magnetic SMAs in the 4D printing sector. [123]

Ternary iron-based Among the several types of unique magnetism-responsive SMAs, Ni–Mn–Ga Heusler structured alloys have garnered a lot of attention because single Ni–Mn–Ga crystals were shown to exhibit a significant magnetic field-induced strain of up to 10%. [124]

Caputo et al. used binder jetting method to effectively produce complicated Ni–Mn–Ga magnetically sensitive SMA sections with varying porosities. Following a thermo-magneto-mechanical training procedure, the Ni-Mn-Ga printed samples may display a reversible strain that is sensitive to magnetism of around 0.01%. [125]

This work demonstrated that magnetism-responsive SMAs may be printed in four dimensions. The impact of sintering temperatures on the microstructures, martensitic transformation

features, and magnetism-responsive behaviors of Ni–Mn–Ga SMAs produced using binder jetting technique were then methodically examined by Mostafaei et al. [126]

Using SLM technology, Laitinen et al. created the Ni–Mn–Ga SMAs and looked at how different process variables affected their microstructures. It was discovered that a major worry for the SLM production of Ni–Mn–Ga SMAs was the loss of Mn, which corresponded to a fluctuation in Ni concentration. To offset the observed loss, a small over-alloying of Mn into the original powder was therefore necessary. The loss of Mn increased as the volume energy density rose. Furthermore, the results showed that the applied process parameters had very different impacts on the composition and relative density of the material as it was created, with a distinct optimum where high relative density material could be made with little Mn loss. The SLM Ni–Mn–Ga magnetism-responsive SMAs with homogeneous compositions and a high relative density of 98.3% were produced by using the ideal process settings. [127]

A novel approach to additive manufacturing was presented by Taylor et al. for the fabrication of porous polycrystalline Ni–Mn–Ga micro-architectures. The three primary processes in this process are as follows: (1) using extrusion-based 3D printing to create microstructures; (2) annealing treatment to remove residual solvents and binder; and (3) homogenizing composition using high-temperature sintering. The Ni–Mn–Ga micro trusses made with this technique have reversible martensitic transition temperatures between 45 °C and 90 °C, a saturation magnetization of up to 56 Am2/kg, and a Curie temperature of 85–90 °C. [128]

### 4D Printing of Stimuli-responsive Ceramics and Their Composite Materials

The physical and chemical qualities of ceramics and their composite materials are stable, they resist wear and corrosion well, and they perform exceptionally well in electrical insulation. Numerous industries, including aerospace, biology, architecture, automotive, communication, environmental protection, energy-saving, and others, demonstrate their considerable potential for use. Nevertheless, it is challenging to create controlled stimuli-responsive changes in forms, properties, and functions in conventional ceramics and their composite materials due to their extreme brittleness and stable physical and chemical characteristics. Consequently, they are seldom used in 4D printing. Lu et al. created a unique ZrO2 nanoparticle-reinforced poly (dimethylsiloxane) matrix composite material in order to get around this restriction. The DIW technique made it simple to print the nanocomposites on a precursor substrate that had already been stretched. Motivated by the elastic energy stored in the precursor substrate, the printed structures may deform into complex ceramic precursors, which with heat treatment will become elastomer-derived ceramics with strong mechanical characteristics. Through pattern design, the ceramic ancestors were able to execute programmable self-shaping. The creation of intricate ceramic origami and the 4D printing of ceramic structures are made possible by this effort. [32]

## Future look and Summary

The updated definition broadens the meaning of 4D printing, which will hasten its progression from a mere display of a phenomena to real-world use. Note that 4D printing is still in its early stages at this time. 4D printing will continue to grow and evolve in the future as this technology progresses.

The great advantages make 4D printing attractive in numerous fields including aerospace, biomedicine, automobile, robotics, and architecture. Therefore, 4D printing has become the forefront and hot spot of research at home and abroad. The market research predicts that the market share of 4D printing in the global industry will reach 555.6 million in 2025.

## Conflict of Interest

The authors declared no competing interests in the publication of this article

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**الطباعة رباعية الابعاد للمواد المستجيبة للمحفزات**

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***المستخلص:***

ليس فقط بإمكان تقنية التصنيع الإضافي أن تطبع هندسات معقدة بدقة ملحوظة، بل يمكنها أيضًا برمجة ومراقبة بدقة هندسة ومواد وهياكل وخصائص الأجسام المطبوعة. وهذا يتيح مرونة كبيرة في التصميم وقدرة عالية على التخصيص وتوفير كميات كبيرة من المواد للمنتجات. ولكن هناك عيب مهم في تقنية التصنيع الإضافي التقليدية، المعروفة أيضًا بطباعة ثلاثية الأبعاد، وهو أن الأجزاء المطبوعة غير عرضة للتغير وغير قابلة للتكيف في مواجهة التعديلات في البيئة المحيطة. بسبب ذلك، لا يمكن للأجزاء المطبوعة ثلاثية الأبعاد تلبية الاحتياجات المتزايدة للأجهزة التي تتطلب وظائف ذكية بما في ذلك التكيف الذاتي، والتجميع الذاتي، وإصلاح الذات، والتعلم الذاتي، والاستشعار الذاتي، واتخاذ القرارات. في هذا الصدد، تظهر مجموعة ابتكارية من تقنية التصنيع الإضافي المعروفة باسم طباعة ثلاثية الأبعاد في أربعة أبعاد، وهي فرع مبتكر من تقنية التصنيع الإضافي، وذلك بفضل التكامل المتعدد التخصصات بين المواد والآلات والميكانيكا والمعلومات وما إلى ذلك. باستخدام التصنيع الإضافي، تقوم طباعة الأبعاد الأربعة بإنشاء مكونات ديناميكية تتحكم وتتغير تلقائيًا مع مرور الوقت و/ أو في الفضاء بناءً على المحفزات المضبوطة مسبقًا مثل الحرارة والرطوبة والضوء والحموضة والمغناطيسية والكهرباء. يؤكد هذا المفهوم أن سلوك أجهزة الطباعة في الأبعاد الأربعة تستجيب للمحفزات تتعدى تعديلات الشكل البسيطة لتشمل تغيرات الخصائص والوظائف.

***الكلمات المفتاحية:*** جهاز التصوير بالليزر الصلب (*SLA*)؛ التصنيع الإضافي (*AM*)؛ التقنية الكهرضوئية (*EOT*)؛ طراز الترسيب الانصهار (*FDM*)؛ وانتاج الواجهة السائلة المستمرة (*CLIP*).