Additive Manufacturing from the Point of View of Materials Research

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1 INTRODUCTION

Over the course of history, there have been three major industrial revolutions, each of them powered by the technological advances of the time and characterized by an increased productivity of industrial processes. Industry 1.0 incorporated the use of hydropower, steam power, and the

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development of machine tools that enabled the mechanization of manufacturing processes; Industry 2.0 introduced mass production assembly lines that were powered by electrical energy; and Industry 3.0 introduced production automation, robots, and computer systems [1, 2]. The key aspect of the ongoing industrial revolution, Industry 4.0, relates to the cyber-physical production systems that consist of physical machines controlled and interconnected by collaborating computational elements. In fact, Industry 4.0 is strongly influenced by our ability to process data, which has phenomenally increased over the past 15 years. In parallel with Industry 4.0, there also exists the concept of Materials 4.0 (or big data materials informatics), which incorporates the tools of cyber-physical space and materials informatics to enhance the design of materials and devices with targeted functionalities in a virtual environment through computational synthesis or reverse engineering from existing knowledge on materials [3, 4]. This approach aims at a higher efficiency in synthesizing and testing novel material compositions and allows shorter lead times from conceptualization to production. However, as the concept of Materials 4.0 has been extensively reviewed in a recent article by [3], it is not discussed further in this chapter. Instead, we focus on the emerging topic of the additive manufacturing (AM) of metal-based stimuli-responsive materials and emphasize possible future directions for the additive manufacturing of metallic materials in general.

‘Smart manufacturing’ (later Manufacturing 4.0) is one of the primary concepts under Industry 4.0, and it can be described as an adaptable manufacturing system where production processes can adjust automatically for multiple types of products or changing conditions [1]. Manufacturing 4.0 incorporates a large group of base technologies, such as robots and other manufacturing automation, artificial intelligence, the internet of things, analytics and big data [2]. Additive manufacturing, also known as 3D printing, is without a doubt one of the key technologies empowering manufacturing under Industry 4.0. Additive manufacturing is a general term for technologies that are based on the layer-by-layer deposition of material according to a digital model of the object to be manufactured. Additive manufacturing offers many advantages, such as mass customization, reduced tooling costs, on-demand manufacturing, shorter lead times, reduced material waste, and the application-oriented optimization of geometries. In principle, additive manufacturing facilitates a greater freedom of design compared to traditional manufacturing technologies, which has opened up new ways to conduct engineering design. One of the central aspects in this development has been design for additive

manufacturing (DFAM), which is a method that aims to consider additive manufacturing processes and material-related constraints in the design of components for additive manufacturing [5].

Besides freedom of design and enhanced shape complexity, another advantage of additive manufacturing relates to the materials themselves. Additive manufacturing is already today suitable for realizing complex geometries using several engineering materials, such as polymers, metals, ceramics, and composites [5–8]. Additive manufacturing has proven to be feasible for the processing of metallic materials, such as tungsten, which have been considered difficult to work with using conventional methods because of their high hardness and low ductility. In fact, for the last few years, pure tungsten has been commercially available for use in additive manufacturing systems made by EOS GmbH. Additionally, some additive manufacturing processes may introduce new options for metallic materials and enable the engineering and manufacturing of materials that are difficult or nearly impossible to synthesize using conventional methods. A good example of such materials are the so-called functionally graded materials, in which tailored properties can be obtained through a spatial gradation of chemical composition (gradient materials) and/or a 3D structure (hierarchical metamaterials). In addition, the size of these compositional or structural features can span multiple orders of magnitude. Furthermore, the introduction of new materials allows an expansion of the design space for additive manufacturing, which is interconnected with another interesting concept under Industry 4.0: the so-called ‘smart materials’ [9, 10].

Because materials themselves cannot be smart but can rather only exhibit certain intrinsic characteristics, the expressions ‘smart materials’ or ‘intelligent materials’ are typically (but not exclusively) used as an analogy to stimuli-responsive materials that can change their physical properties in response to external stimuli, such as a temperature change, mechanical stress, a magnetic field or an electrical current. In the scientific literature, stimuli-responsive materials are often divided into different classes based on their responses to an applied stimulus. Here, we entertain a similar approach and divide the stimuli-responsive materials into the four classes listed below.

- *Stimuli-responsive actuator materials*—materials that produce strain in response to the applied stimuli.
- *Stimuli-responsive energy conversion materials*—materials that exhibit an electric current, electrical resistance, magnetic field or temperature change as a primary response to the applied stimuli.

- ***Stimuli-responsive optical materials***—materials that exhibit an optical response, such as light emission or a change in optical properties, as a response to the applied stimuli.
- ***Stimuli-responsive state-changing materials***—materials that alter their physical properties, such as viscosity, in response to the applied stimuli.

Examples of stimuli-responsive materials and some of their applications are listed in Table 1, based on research by [11–88]. Applications of stimuli-responsive materials under Industry 4.0 range from small actuators, sensors, and signalization devices all the way to photovoltaic materials used in the production of electricity from sunlight. In general, stimuli-responsive materials may yield a multitude of enhanced capabilities and functionalities for many products as these allow an active response to be achieved in a product that would otherwise lack it. Some examples of applications for stimuli-responsive materials under Manufacturing 4.0 are listed below; refer to Table 1 for specific examples and references.

- Materials that can generate significant mechanical motion with almost no other components besides the material itself have a high potential for replacing traditional mechanical components, such as the gears, shafts, and pulleys that are used to **generate motion** in conventional machines. Some of these materials, such as thermally activated shape memory alloys (SMAs) or magnetic shape memory alloys (MSMAs), can still produce motion below the size threshold where mechanical components or traditional mechanisms can no longer be used, thus offering a feasible application in different types of **microelectromechanical systems**. Additionally, some of these materials, such as the shape memory alloy Ni-Ti or some of the shape memory polymers, are highly appreciated due to their biocompatibility for medical applications. Stimuli-responsive actuators can also be practical in any **soft robotics** that may be required for the handling of delicate or brittle materials or even living organisms.
- Some stimuli-responsive materials, such as magnetorheological liquids or the magnetic shape memory alloy Ni-Mn-Ga, may be practically useful in **shock absorption** and **active vibration damping**, for example in high-precision devices.
- Shape memory polymers can be used in **active disassembly** systems that are triggered at specific temperatures.
- Magnetocaloric materials can be used for high-efficiency **magnetic cooling and refrigeration** systems.

Table 1 Classification of different types of stimuli-responsive materials and examples of their applications

<i>Class</i>	<i>Group</i>	<i>Stimulus</i>	<i>Response</i>	<i>Example materials</i>	<i>Example applications</i>
Stimuli-responsive actuator materials	Chemomechanical polymers	Chemical reaction	Strain	Hydrogels [11]	Delivery of drugs such as insulin, artificial muscles, tissue engineering [11]
	Electroactive polymers	Electric current	Strain	Polyaniline, Nafion, polythiophenes, poly(vinyl alcohol) gel with dimethyl sulfoxide, poly(acrylonitrile) with conductive fibers [12], polypyrrole, poly(3,4-ethylene dioxythiophene) [12, 13]	Robotic applications, space applications, biomimetic applications [12], wearable sensors, prosthetic applications, haptic sensing, pulse rate monitoring, muscle movement detectors [13]
	Electrostrictive	Electric current	Strain	Lead magnesium niobite [14, 15], barium titanate [16]	Piezo-actuator applications [14, 15], actuators, transducers, energy harvesters [17]
	Magnetostrictive	Magnetic field	Strain	Metglas, Terfenol-D [18–20]	Microwave devices, sensors, transducers [18], sonar, ultrasonic cleaning [20]
	Photostrictive	Light	Strain	PLZT [21, 22]	Non-contact actuation [21], wireless remote control [21, 22]
	Shape memory alloys	Shape memory effect Temperature	Strain	Ni-Ti [23, 24], Fe-Mn-Si, Cu-Zn-Al, Cu-Al-Ni [23]	Thermal actuators, couplings [23], dental implants, artificial heart valves [23, 24], active disassembly [25, 26]
	Shape memory polymer	Shape memory effect Temperature, electric current, light	Strain	Polyurethane [27–29]	Active disassembly [30], ocular implants, vascular stents, sutures [27], self-tightening sutures [28], autochoke elements, intravenous cannula [29]
	Magnetic shape memory alloys	Shape memory effect Magnetic shape memory effect Magnetic field	Strain	Fe-Pd [31, 32], Ni-Mn-Ga [33–35], Ni-Mn-Al [36],	Micropumps [33], magnetic sensors, energy harvesters [32, 35, 37], actuators [34, 36, 38]

(continued)

Table 1 (continued)

<i>Class</i>	<i>Group</i>	<i>Stimulus</i>	<i>Response</i>	<i>Example materials</i>	<i>Example applications</i>
Stimuli-responsive energy conversion materials	Magnetoelectric	Magnetic field	Electric current	Multiferroics [39, 40] Cr2O3 [41, 42]	Information storage [40], energy harvesting, resonators [42]
		Electric current	Magnetic field		optoelectronics, transducers, radioelectronics [43] microwaves [44], sensors, optical components [45, 46], spintronics and medicine [46]
	Photovoltaic	Light	Electric current	Silicon [47], gallium arsenide [48], Chalcogenide [49]	PV solar applications [47]
	Magnetocaloric	Magnetic field	Temperature change	Ni-Co-Mn-Sn [50]	Magnetic refrigeration [50]
	Pyroelectric	Temperature	Electric current	Barium strontium titanate [51]	Infrared detector [51]
	Piezoelectric	Mechanical stress	Electric current	Gallium arsenide [52], lead-zirconate-titanate [53]	Microactuators [52], biosensors [54]
	Piezoresistive	Mechanical stress	Electrical resistance	Silicon [55, 56]	Pressure sensors [55] accelerometers [56]
	Thermoelectric	Temperature	Electric current	Silicon/SiGe [57], bismuth telluride, PbTe, SrTiO3 [58]	Engines, refrigeration, air conditioning [57], automotive HVAC [58]

Stimuli-responsive optical materials	Electrochromic/Electroreflective	Electric current	Color/optical property change	Poly(indole-6-carboxylic acid) [59], polyacrylate [60], 3,4-ethylenedioxythiophene [61, 62]	Energy-saving, light control [59] supercapacitors, transistors, diodes [60], switchable mirrors [63], optical devices [61], optical recordings, forensics, smart windows [62] Hydrogen gas detectors/sensors [64, 65]
	Chemochromic	Chemical reaction	Color change	Tungsten oxide [64, 65]	Optical data storage, optical switching [66], drug delivery systems [67], gel-glasses [68], holographic storage [69], image processing, laser resonators [70]
	Photochromic/photorefractive	Light	Color change	Azobenzene, dithienylethene [66] spiropyrane [67, 68] LiNbO ₃ :Mn [69] BaTiO ₃ [70]	Energy saving [71, 72]
	Thermochromic	Temperature	Color change	Vanadium dioxide [71, 72] titanium nitride [72]	Healthcare [73], protective helmets, biosensors [74]
	Mechanochromic	Mechanical stress	Color change	Poly(1,4-butylene succinate) [73], polydiacetylenes, spiropyrans [74]	Warning signs, dial plates, escape routes [75], footpaths, lanes [76]
	Photoluminescent	Light	Light emission	Strontium aluminate [75, 76], zinc sulfide [76]	Medical dosimetry [77]
	Thermoluminescent	Temperature	Light emission	Lithium borate, beryllium oxide, lithium fluoride, calcium sulphate [77]	Emergency signage, lighting, backlights [78, 79], detection of electric/magnetic fields [80]
	Electroluminescent	Electric current	Light emission	Zinc sulfide doped with terbium or manganese [78], zinc sulfide doped with copper [79]	Dynamic pressure mapping, stress sensing [80], optoelectronic devices, security papers [81]
	Mechanoluminescent	Mechanical stress	Light emission	Zinc sulfide doped with copper, zinc sulfide doped with manganese [80]	

(continued)

Table 1 (continued)

<i>Class</i>	<i>Group</i>	<i>Stimulus</i>	<i>Response</i>	<i>Example materials</i>	<i>Example applications</i>
Stimuli-responsive state changing materials	Shear-thickening	Mechanical stress	State change (fluid/solid)	Silica particles in a polyethylene glycol solution [82, 83], amphiphilic polymer, hydrophilic particles and polyethylene-oxide [82]	Adaptive stiffness, damping, smart structures [82], protective equipment, seismic isolation of buildings/bridges, shock loading applications [83]
	Electrorheological	Electric current	State change (fluid/solid)	Aluminosilicate, biopolymers (silicone oil/hydrocarbon), oxides (surfactant) and TiO ₂ , mineral oil, water [84, 85], zeolite and silicone oil [84]	Damping devices (shock absorbers, bearing dampers), clutches, mechanical polishers [84], haptic sensors, force feedback systems, anti-lock braking systems [85]
	Magnetorheological	Magnetic field	State change (fluid/solid)	Carbonyl iron particles and carrier liquids (silicone oils, polyesters, synthetic hydrocarbons) [86] hydrocarbon-based magneto-rheological [87]	Biomedical applications (prosthetic knees) [87], shock absorbers and dampers, brakes, clutches [86-88]

- Stimuli-responsive materials also have a high potential in different types of signalization devices, such as **displays** or **haptic** (sense of touch) technologies. In fact, haptic devices provide a unique interface between humans and machines, allowing remote distance operators to receive force feedback from the operated machines. For example, operators could receive information about the weight or resistance of lifted objects or be alerted when there is an issue with the operated machine.
- Another group of applications for stimuli-responsive materials under Industry 4.0 are different types of **sensors**, such as the ones used for failure detection and predictive maintenance in manufacturing systems. Additionally, **wearable sensors** are a prominent group of applications for many stimuli-responsive materials.

2 ADDITIVE MANUFACTURING OF STIMULI-RESPONSIVE MATERIALS

When it comes to stimuli-responsive materials, additive manufacturing is often referred to as 4D printing, which may refer to either the stimuli-responsive properties of the additively manufactured material in general or the ability of some of the materials (stimuli-responsive actuator materials) to change their physical shape in response to an applied stimulus. However, here we employ the term ‘additive manufacturing of stimuli-responsive materials’ instead of 4D printing as the usage of the former aligns better with the existing standardized terminology for additive manufacturing.

The additive manufacturing of different stimuli-responsive materials has gained significant interest in the past few years as this technology could facilitate a higher freedom of design concerning the stimuli-responsive properties of the manufactured objects. Tremendous advantages can be gained when devices can be optimized to fulfill the requirements of the intended application, instead of designing within the limits of the used manufacturing process. Thus, additive manufacturing may also accelerate the adoption of stimuli-responsive materials or expand their possible applications. Additionally, a combination of structural and stimuli-responsive materials under a single additive manufacturing process could enable the manufacturing of entire devices with integrated stimuli-responsive sections. In this case, certain functional characteristics or properties would be obtained locally in certain sections of the additively manufactured device. For example, in the case of stimuli-responsive actuator materials, the stimuli-responsive material would replace the traditional mechanisms within the manufactured device. These ‘active regions’ of the device could be

actuated using a passive source of energy, such as a magnetic field in the case of magnetic shape memory alloys or heat in the case of thermally activated shape memory alloys. Additionally, additive manufacturing could allow a localized tailoring of properties (as in Fig. 1) within a single device,

Fig. 1 (a) A location-dependent active response generated by temperature-dependent multi-stage shape recovery in a U-shaped Ni-Ti component deposited using L-PBF; (b) effect of the L-PBF process parameters on the transformation temperatures and active responses at different sections of the build. Reproduced from [151] under Creative Commons Attribution 4.0 International License

for example by inducing local differences in composition or microstructure in the processed stimuli-responsive material. Overall, these developments could facilitate the additive manufacturing of entire devices with embedded actuators or sensors, which could act as functional parts in existing systems, such as in soft robotics or pneumatics.

The majority of the published reviews on the additive manufacturing of stimuli-responsive materials have focused on shape memory polymers [152–161], while a few articles [162–167] have discussed aspects of expanding the DFAM method towards the adoption of these materials in additively manufactured components. Although some reviews have also discussed the additive manufacturing of thermally activated shape memory alloys, reviews concerning other metal-based stimuli-responsive materials, such as magnetic shape memory alloys or magnetocaloric materials, are sparse to non-existent. The popularity of polymer-based materials is expected because they are more feasible for low-cost additive manufacturing in comparison to metal-based materials, which are more difficult to manufacture additively without defects. Hence, this chapter concentrates on the additive manufacturing of thermally activated shape memory alloys, magnetic shape memory alloys, and magnetocaloric alloys. A brief overview of the state of the art in the additive manufacturing of these materials is presented in Table 2, based on the research results from [89–150]. An overview of the main additive manufacturing process categories (compared to the additive manufacturing processes in Table 2) for metal-based stimuli-responsive materials is presented below, following the definitions given in standard SFS-EN ISO/ASTM 52900:2017.

- **Material extrusion**—“An additive manufacturing process in which material is selectively dispensed through a nozzle or orifice”; an example process for metals is 3D ink printing, whereby metal powder is dispensed in a mixture with a bonding agent.
- **Powder bed fusion**—“An additive manufacturing process in which thermal energy selectively fuses regions of a powder bed”; the applied thermal energy can be either a laser (L-PBF) or an electron beam (E-PBF).
- **Binder jetting**—“An additive manufacturing process in which a liquid bonding agent is selectively deposited to join powder materials”.
- **Directed energy deposition**—“An additive manufacturing process in which focused thermal energy is used to fuse materials by melting as they are being deposited”; example processes include laser-based directed energy deposition of powder material (L-DED), plasma arc deposition (PAD), and wire and arc additive manufacturing (WAAM).

Table 2 A brief overview of state of the art in the additive manufacturing of metal-based stimuli-responsive materials

Group	Alloy	Process	Feedstock	Observations
SMA	Ni-Ti	L-DED	Pre-alloyed powder	Retention and finer grain sizes proved to have a higher hardness values due to a high temperature austenite phase [89, 90]
			Mechanically produced powder	Ni-rich Ni-Ti alloys required aging treatment [91]
				Solutionized and aged Ni-Ti had a better shape memory response [92]
			Gas atomized powder	Ni-rich Ni-Ti showed superelastic behavior [93]
				Shape memory effect recovery and unstable oriented/de-twinned martensite [94]
				NiTi50 showed excellent mechanical properties as compared to NiTi45 and NiTi55 [95]
	L-PBF			Using low scanning speed reduced the obtained shape memory effect [96]
				Superelastic behavior [96]
				Loss of nickel in the process [97]
				High relative density (>97%) and hardness reported [98, 99]
				Excellent compression fatigue resistance [100] with an irreversible strain behavior [99]
				Wider hatch distance decreased relative density [101]
				Superelastic response (95%) [102–108]
				Shape memory effect [94, 105, 109, 110]
				Recovery above 5.5% [106–108, 111]
				Desired stiffness was achieved by regulating the level of porosity and stiffness reduced from 69 GPa to 20.5 GPa for 58% porosity [112]
				Loss of nickel in the process [113, 114]
				Wider hatch spacing led to a highly irrecoverable strain [114]
				Microstructure influenced the shape memory response and mechanical behavior [109, 115]
				Martensite twins were formed easily after annealing process [116]
				Heat treatment above 400 °C decreased the shape recovery and transformation strain [117]
				Tensile strain recovery was 1.5 times larger than in compression [111]

	Plasma atomized	Reversible martensitic transformation. Processing parameters, Ni evaporation and oxygen content of the processing atmosphere (oxidation) impacted martensite transformation. Solution treatment resulted in sharpened transformation peaks and undid the influence of dissolvable Ni-Ti precipitates [118].
Micro L-PBF	Mechanically produced powder	Mechanical shape memory functionality was proven with a force range of 10–100 mN [119]
E-PBF	PREP atomized powder	Quality of deposited Ni-Ti was improved by increasing the scanning speed [120] No visible shape memory and pseudoplastic effect seen [121] The authors did not recommend E-PBF for Ni-Ti alloys. Preheating was required [97] E-PBF process resulted in better tensile properties than L-DED and L-PBF [97, 122]
PAD	Pre-alloyed powder	Linear superelasticity [123]
WAAM	Wire	Quasi-linear superelasticity with narrow hysteresis [123, 124]
Cu-Al-Ni	Elemental powders	High hardness and tensile strength [125]
Cu-Al-Ni-Mn	Gas atomized powder	High aluminum content led to dendrites and high hardness [126] High relative density (>92%) achieved [127–129] Reversible martensitic transformation with the formation of β_1' -martensite [127–129] Large strain recovery after unloading (up to 18%) [127] Strong distribution of pores produced by the L-PBF sample [127, 128] Additional re-melting led to smaller grain size and yielded a deformability of 14% [129] Higher strength and improved plasticity was observed for both samples (Cu-Al-Ni-Mn and Cu-Al-Ni-Mn-Zr) [128–130] For the Cu-Al-Ni-Mn-Zr sample, Zr-rich phase was found to precipitate at the grain boundaries during the annealing process [130]
Cu-Al-Ni-Mn-Zr		
Cu-Al-Ni-Ti		Copper alloy with Ti addition had a high hardness of about 280 HV due to the grain refinement. The relative density exceeded 99% [131]
Fe-Mn-Al-Ni		Reversible martensitic transformation and pseudo-elastic effect [132]

(continued)

Table 2 (continued)

<i>Group</i>	<i>Alloy</i>	<i>Process</i>	<i>Feedstock</i>	<i>Observations</i>
MSMA	Ni-Mn-Ga	3D ink printing Binder jetting	Ink with elemental powders Mechanically produced powder	Reversible martensitic transformation [133]
				Martensitic twins [134]
				Martensitic twins and reversible martensitic transformation after post-processing [135, 136]
				Binder jetting produced a complex-shaped porous Ni-Mn-Ga geometry with a reversible martensitic transformation [137–139]
				Sintering of Ni-Mn-Ga powder was shown to produce net-shaped porous structures [136, 138, 139]
		L-DED L-PBF	Gas atomized powder	Minor loss of nickel seen in sintered Ni-Mn-Ga in comparison to the initial powders [140]
				Narrow hysteresis and increase in saturation magnetization [141]
				Ni-Mn-Ga could be deposited on compositionally dissimilar materials [142, 143]
				Complex geometries [143, 144]
				High relative density of 98.3% [143]
Magnetocaloric	Ni-Co-Mn-Sn	L-DED	Mechanically produced powder	Martensitic twins and reversible martensitic transformation [143–146]
				Loss of Mn in process [143, 145, 146]
				Susceptibility to cracking [145]
				Reduction of Sn was observed at the subgrain boundaries [147]
				‘Properly built’ samples of Ni-Co-Mn-Sn exhibited better magnetic properties than ‘overbuilt’ samples [148]
	Ni-Mn-Cu-Ga	Binder jetting		Heat-treatment promoted martensite growth and increased twinning [149]
				Larger grain growth and Mn-O particles were observed at samples sintered at 1080 °C as compared 1050 °C and 1070 °C [150]

3 ADDITIVE MANUFACTURING OF SHAPE MEMORY ALLOYS

Shape memory alloys are alloys that can recover a limited applied strain of less than 10% either thermally or mechanically [168]. This property finds its origin in a thermoelastic martensitic transformation in some particular alloys. This transformation is characterized by its transformation temperatures (M_s , M_f during cooling reaching the martensitic phase, A_s , A_f during heating reaching the beta phase), exhibiting a relatively small hysteresis of about 10–40 K compared to the well-known martensitic transformation in many steels exhibiting a hysteresis of several 100 K. When a strain, limited to 10%, is applied in the martensitic state, this strain can be recovered by heating above the transformation temperature into the beta phase. This is called the thermal recovery or shape memory effect. When a strain of less than 10% is applied in the beta phase, above M_s , the strain is mechanically recovered upon releasing the applied stress, which is called superelasticity. Complete thermal or mechanical recovery can only be obtained in a limited temperature window around the martensitic transformation. The thermally activated shape memory effect occurs in some Cu-based alloys and Fe-based alloys, but it is mostly associated with Ni-Ti alloys. Ni-Ti is superior compared to other shape memory alloys for many reasons, including its high ductility, high strength, and very fine grain size. These properties enable the production of very thin devices (wires with a diameter down to 25 μm). Additionally, it is biocompatible, which is why more than 80% of the products made of Ni-Ti are medically related [169]. Besides their use in medical applications, shape memory alloys can convert heat into a high force or work output, which makes these alloys useful in the actuators of stress-creating components [23, 170].

From the perspective of conventional manufacturing processes, a major problem of Ni-Ti is its poor machinability, primarily due to the strong strain hardening effect. Thus, wire and tube drawing are the most common applied forming techniques used in the production of devices such as guise wires, stents, and actuators based on springs. This sets many limitations on the shape complexity of the manufactured devices. Therefore, additive manufacturing of Ni-Ti has gained the attention of designers of medical and other devices. As shown in Table 2, laser-based processes, especially L-PBF, are the most typical approach for the additive manufacturing of Ni-Ti. The same observation applies to Cu- and Fe-based alloys, although little scientific literature is available on the additive

manufacturing of these materials. In fact, the future for Cu-based SMAs does not look bright as the majority of research on additive manufacturing of SMAs concentrates on Ni-Ti. However, the additive manufacturing of Ni-Ti represents only a small fraction of the medical applications produced by metal-based additive manufacturing, and only a handful of studies on the additive manufacturing of Ni-Ti consider its stimuli-responsive properties, such as the very low stiffness (very low E-modulus), and its functional properties, such as superelasticity and the shape memory effect. However, a fair amount of research on the laser additive manufacturing (LAM) of Ni-Ti shape memory alloys has been conducted [23, 115, 171–174]. Therefore, we summarize here the most important observations of Ni-Ti deposited using L-PBF, as previously discussed by [173] and briefly overviewed in Table 2.

- Although Ni-Ti can be processed at a high density crack-free, the mechanical and functional properties of the processed material are on average inferior compared to the wrought material. However, using repetitive laser scanning in the process may allow improvement of the functional properties of deposited Ni-Ti.
- Controlling the transformation temperatures of the processed material is difficult, mainly due to the evaporation of Ni and precipitation based on impurities. Hence, the composition and transformation temperatures of the processed material are strongly dependent on the processing parameters and, therefore, the transformation temperatures of the final product are not necessarily the transformation temperatures of the initial powder.
- Additionally, the processing environment should be controlled to prevent oxygen and/or nitrogen pick-up that may lead to an increased density of impurities, which may influence the transformation temperatures and the mechanical properties of the processed material.
- The surface roughness of the final product should be considered in relation to potential wear or for the difficulties it causes in sterilization, which is required for biomedical applications.

4 ADDITIVE MANUFACTURING OF MAGNETIC SHAPE MEMORY ALLOYS

Besides the thermally activated shape memory effect, magnetic shape memory (MSM) alloys may also exhibit a straining phenomenon when the magnetic moments of the martensitic twin variants of the alloy align with the applied magnetic field [38, 175, 176]. This straining phenomenon is called the magnetic shape memory effect. The Ni-Mn-Ga system, which is the most studied class of MSM materials, has been shown to exhibit outstanding characteristics, such as magnetic-field-induced strains (MFIS) of 12% [176], which is a hundred times larger than the magnetically induced strains obtained in competing materials. In addition, the efficiency (mechanical work output / magnetic field energy) of the MSM effect can be over 95% and its fatigue life can exceed 2×10^9 cycles [177]. Characteristic of the MSM materials is that the strain remains unchanged after the magnetic field has been switched off (the strain can be recovered by applying a magnetic field in transversal direction or by force). This results in significant energy savings in many applications, especially on-off valves, because magnetic field energy is needed only during the brief time when the shape of the MSM element is changed. Additionally, Ni-Mn-Ga can exhibit high strain accelerations of 1.6×10^6 m/s² [178], which is assumed to be the highest acceleration of all actuator materials. These characteristics may be beneficial in several applications, such as in robotics, biomedical applications and optics. For instance, fast actuators/sensors [34, 176], micropumps [33], and vibration energy harvesters [35] have been identified as potential applications for MSM materials. However, commercial applications of MSM materials are still limited, possibly due to the relatively young age of the technology itself compared to competing piezo ceramics or giant magnetostrictive materials.

Typically, bulk polycrystalline Ni-Mn-Ga does not exhibit limited MFIS due to grain boundary constraints that effectively block twin boundary motion in the material. However, directionally solidified (textured) polycrystalline Ni-Mn-Ga has been shown to exhibit up to 1.0% strain [179], whereas polycrystalline Ni-Mn-Ga foam has been shown to exhibit up to 8.7% recoverable strain [180]. A smaller force output and brittleness are disadvantages of foamy polycrystalline compared to more conventional single-crystalline material. From a manufacturing perspective, the use of additive manufacturing offers better freedom of design, especially compared to typical single-crystalline material. Thus, the additive

manufacturing of MSM alloys aims at obtaining parts with controlled porosity while facilitating the possibility to manufacture complex geometries. Additionally, additive manufacturing places fewer limitations on the size of the manufactured object. Other advantages that may be potentially gained through additive manufacturing relate to the possibility to produce compositional gradients that allow for the tailoring of the properties for specific applications.

Compared to the additive manufacturing of Ni-Ti based thermally activated shape memory alloys, additive manufacturing of magnetic shape memory alloys is still in its infancy. All the scientific literature available at the time focuses on the additive manufacturing of Ni-Mn-Ga-based MSMAs. The most common approaches on additive manufacturing of Ni-Mn-Ga have concentrated on 3D ink printing [133, 134] and binder jetting [135–140]. However, also a few investigations into manufacturing of polycrystalline Ni-Mn-Ga using L-DED [141] and L-PBF [142–146] have recently been published. Each of the aforementioned processes have their own advantages and disadvantages concerning the manufacture of a material that exhibits MFIS. Nevertheless, a common aspect for all of the processes is the aim to obtain controlled composition, microstructure and porosity, which is essential for obtaining MFIS in polycrystalline Ni-Mn-Ga. Especially, assuring the chemical integrity of the manufactured material is also important because of the high susceptibility of the crystal structure of Ni-Mn-Ga to compositional variation and impurities.

In general, both 3D ink printing and binder jetting processes have been proven to be feasible for producing Ni-Mn-Ga with complex geometries. However, binder-based processes face a challenge regarding the control of the composition and microstructure because the consistent removal of binder elements post-processing is difficult and some oxidation and Mn evaporation may occur during the sintering process [139]. LAM processes base on melting the material, thus enabling the use of binders to be avoided. However, previous studies on the L-PBF of Ni-Mn-Ga show that some Mn is lost in the process and that this Mn loss is strongly influenced by the used process parameters [143–145]. In fact, loss of Mn during the L-PBF process is expected due to the high vapor pressure and low boiling temperature of Mn in comparison to the other elements in the alloy. Thus, control over the processing parameters and the thermal cycle that the processed material undergoes is critical for obtaining a controlled composition. This may also be an advantage, as the composition could be controlled through an adjustment of the process parameters, which could potentially

allow for the adjustment of the microstructure and stimuli-responsive properties of the processed material. However, excessive over-alloying of Mn into the used powder would be required for this approach to be feasible.

The control of the porosity in the additive manufacturing of Ni-Mn-Ga has typically based on manufacturing different types of lattice structures [133, 137, 142] or foam-like materials [134, 138]. Additionally, the sintering process used in binder-based additive manufacturing processes can also be adjusted to control the density of the processed material [140]. The processed material undergoes a repetitive cycle of heating and cooling in LAM processes as the heat from melting is conducted through the prior layers of deposited material. As a result, the processed material may exhibit regions with different thermal histories, which also affects the local microstructures. This has been observed as broad ferromagnetic hysteresis and wide phase transitions in as-deposited material [141, 143]. Additionally, Ni-Mn-Ga processed by L-PBF may exhibit cracking [145]. Post-process heat-treatment is required to retain the typical ferromagnetic behavior and material properties in the deposited material [141, 143, 144]. However, laser-based processes typically produce a microstructural texture [181], which is considered beneficial for obtaining MFIS.

Although additive manufacturing shows high potential for facilitating greater design freedom for MSM based devices, so far the functional properties of the additively manufactured material are inferior compared to the conventional oriented single crystals or textured polycrystalline material. By so far, Ni-Mn-Ga processed by binder jetting [136] and L-PBF [142] have been shown to develop a magnetically induced strains up to 0.01%, which are significantly lower than the 8.7% achieved in Ni-Mn-Ga based foams [180]. In conclusion, more research on additive manufacturing of MSM alloys is required for understanding relationships between the applied process parameters and the resulting functional properties.

5 ADDITIVE MANUFACTURING OF MAGNETOCALORIC MATERIALS

Some materials experience a change in entropy (Δs_T) when exposed to a magnetic field in an isothermal environment due to a phase change of either the first or second thermodynamic order [182–184]. When placed in an adiabatic environment instead, this magnetic-field-induced phase

change produces a temperature change (ΔT_{ad}) in the material, leading to the common designation of this phenomenon as the magnetocaloric effect [185]. The magnetocaloric effect can be observed in both first- and second-order materials, with the order parameter of magnetization. In a first-order material, the change in magnetization is discontinuous at the transformation, whereas the change in magnetization for a second-order material is gradual and continuous over the transformation. In the case of a second-order material, this magnetization change is caused by an alignment of magnetic moments around the Curie temperature (demagnetization temperature), reducing the magnetic entropy with increasing magnetic field and causing a corresponding increase in the thermal entropy. The entropy trade-off concept remains for first-order materials, but with the addition of a magnetostructural (or magnetoelastic) phase transformation that causes the direction of the entropy change with the addition of an applied field to be less straightforward. Near the transformation temperature, an applied magnetic field will stabilize the more magnetic phase, which could be either the high-temperature or the low-temperature phase. If the high-temperature phase is stabilized, the application of a magnetic field shifts the transition to lower temperatures and leads to a decrease in the temperature of the material—called the negative (or inverse) magnetocaloric effect. If the low-temperature phase is stabilized, the application of a magnetic field shifts the transition to higher temperatures and leads to an increase in the temperature of the material—the positive magnetocaloric effect.

Recently, the magnetocaloric effect has been researched for leverage in heat pumps, particularly for cooling in applications such as solid-state-based magnetic refrigeration requiring no harmful refrigerants and in localized hypothermia therapy to treat cancer [186, 187]. For the most common application of refrigeration, any magnetocaloric effect-exhibiting material that is to be considered a viable option as a heat exchanger within a heat pump must be formed with a high surface-to-volume ratio and must allow satisfactory fluid flow [188, 189]. Thus, the following two requirements are placed upon the heat exchanger [190]:

1. Maximize the volume fraction of the magnetocaloric effect material while maintaining a large surface area.
2. Minimize the pressure drop in the fluid across the heat exchanger.

Common methods for producing heat exchanger devices from magnetocaloric effect materials are [190]: packed powder beds [191–193], parallel plates [194–197], and microchannel systems [198]. Packed powder beds, though cheap and simple, have a high pressure drop across the device due to the presence of turbulent flow. Microchannels, though inducing only a low drop in fluid pressure, have a high manufacturing cost (if they can be currently manufactured at all for the given material). Parallel plate devices are a median between the two extremes, allowing for a fluid flow that is not as turbulent as in packed powder beds and a production that is not quite as expensive as with microchannels.

With an abundance of requirements on both the feedstock material and the final magnetocaloric-effect-based heat exchanger, fabrication complications are an inescapable challenge. For example, first-order phase transition materials tend to be brittle, which limits the ability to machine them into desired geometries [199]. Difficulties with fabrication can leave promising alloys showing only a modest magnetocaloric effect after device fabrication due to changes in microstructure or atomic ordering and defects [200, 201]. In addition, first-order phase transition magnetocaloric effect materials have narrow operating temperature windows [200, 202]. For ideally efficient operation, a heat exchanger using the first-order phase transition magnetocaloric effect must have stages or a gradient of material transformation temperatures [198]. With a transformation temperature gradient, the fluid will heat (or cool) as it passes through the series of materials, at each point existing within the operating temperature for the magnetocaloric effect material that it is currently in contact with. Second-order phase transition materials are less difficult to shape and have a wider operating temperature range, but the most promising material (Gd) is a ‘critical material’ as it is costly, has a high environmental impact, and its use in a large number of cooling applications would lead to demand far exceeding supply [190, 199, 203].

As a manufacturing method, additive manufacturing may allow for the inclusion of designed, multi-scale porosity; complicated geometries impossible with other methods; the processing of brittle materials that cannot be machined; and gradient or layered materials with gradient or staged material transformation temperatures. This combination of benefits can grant the ability to fulfill both heat exchanger requirements with no trade-offs: a minimal pressure drop across a material that has a high surface-to-volume ratio with a maximized volume of functional material present to produce a large temperature change across a wide temperature range.

Additive manufacturing for magnetocaloric materials is in its relative infancy, although it is increasingly being recognized as a potential production avenue for magnetocaloric effect materials. In 2013, [204] used selective laser melting to create heat exchangers from $\text{La(Fe, Co, Si)}_{13}$. Meanwhile, [137, 139, 147, 148, 150] conducted experiments with Ni-Mn-based Heusler alloys fabricated using L-DED and powder bed binder jet 3D printing. L-DED, with a laser as the energy source, required a heat treatment to homogenize the microstructure before promising properties were observed [147, 148]. Binder jet printing, since it requires no heat input that would change the feedstock powder's microstructure, showed a magnetocaloric response in the as-sintered state [150] [133]. [133] used inkjet printing to deposit a mixture containing elemental Ni, Mn, and Ga powders, then sintered them to create final lattice structures with 73–75% porosity in the micro-trusses. Published experimental studies are scarce compared to the literature on the additive manufacturing of structural metals. Nevertheless, as discussed here and in [190, 199], with the proper attention to tailoring the processing to maintain the functional properties and with measures taken to balance cost and effectiveness, additive manufacturing is a promising technology to address current manufacturing and design issues while at the same time improving the overall performance of magnetocaloric structures.

6 FUTURE ASPECTS OF ADDITIVE MANUFACTURING FOR NOVEL METALLIC MATERIALS

Besides enabling advances in freedom of design and the processing of stimuli-responsive alloys, additive manufacturing may allow the development and manufacturing of customized, application-specific materials and could thus enable the expansion of the exciting material box of different metal alloys. For example, recent developments have been made in the additive manufacturing of metal matrix composites and high-entropy alloys [8], which are favored for their outstanding mechanical properties. Additionally, significant progress has been made in engineering and manufacturing functionally graded materials, such as gradient materials or meta-materials [205–209]. A common additive manufacturing process for the fabrication of compositional gradient materials is DED, which offers unique capabilities, such as the deposition of more than one material simultaneously or the changing of the deposited material from layer to

layer. A second advantage of DED is that the build process itself is not limited, compared to PBF, where deposition is only possible in successive horizontal layers. This makes the DED process suitable for depositing material on 3D substrates, such as existing parts. In fact, repairing a worn part or tool represents a typical industrial application for this process. In principle, this type of additive manufacturing process allows a precise small-scale synthesis of materials during the manufacturing process itself, thus enabling the manufacturing of materials that are difficult to synthesize on a larger scale using conventional methods. Besides potentially allowing the creation of new alloys, this also enables the application-specific tailoring of the materials of the manufacturing process itself, which could be practical for on-demand manufacturing [210]. Additionally, LAM enables the composition and microstructures to be adjusted via the process parameters, which allows the integration of information within the processed material [211]. This could be used to enhance the traceability of the used materials or processes or of the ‘smart products’ themselves.

7 SUMMARY

In this chapter, we discussed how additive manufacturing could contribute to metal-based stimuli-responsive materials and material science in general. Although the future looks bright, substantial research is still required to extend the range of ‘printable’ materials and to achieve appropriate stimuli-responsive properties in additively manufactured metal-based materials. The complexity of the production and the material parameters create large challenges in producing dense, defect-free materials using the associated additive manufacturing processes. Indeed, specific processing conditions of metal additive manufacturing are challenging, and many material systems still suffer from cracks, unwanted porosity, high internal stresses, bad surface quality, and mechanical properties below the required levels. In many cases, this creates the need for post-processing, such as hot isostatic pressing (HIP), stress relieving, thermal treatments or polishing. However, additive manufacturing facilitates a great amount of design freedom for complex geometries and in some cases may enable the tailoring of compositional properties of the processed materials to an extent that is almost impossible to achieve using conventional manufacturing methods. Hence, additive manufacturing has a high potential for the development of novel types of stimuli-responsive devices.

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