

3D printing of carbon-based materials: a review

Supplementary Information

P. Blyweert, V. Nicolas^{*}, V. Fierro, A. Celzard^{*}

Université de Lorraine, CNRS, IJL, F-88000 Epinal, France

A survey of 3D printing technologies

In this section, we review the main processes used in additive manufacturing, highlighting requirements for printing materials, speed, and print resolution.

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1. Powder bed fusion

Powder bed fusion is an additive manufacturing process using a laser or an electron beam as an energy source to sinter or melt powdered materials at points in space defined by a 3D model to create a solid structure (**Figure S1**). Depending on the phase of the powder under

laser exposure, partially or completely melted, the process is referred to as selective laser sintering (SLS) or selective laser melting (SLM) [1].

Each layer made of powder with a typical particle size between 10 and 100 μm is created by selective laser scanning on cross-sections sliced from a 3D model. As the build platform is lowered, a further powder layer is spread on top of the previously processed layer. The scanning process is repeated, simultaneously creating and bonding a new layer to the previous one, and so on until the final structure is achieved.

Due to its versatility, powder bed fusion can be applied to a wide range of materials. The structural characteristics (dimensional accuracy, possible distortions, ...) are strongly dependent on the properties of the powder particles (grain size and morphology, thermal properties, etc. [2] but SLM/SLS processes can print complex geometries suitable for various applications, ranging from biomedical engineering to the automotive sector [3,4].

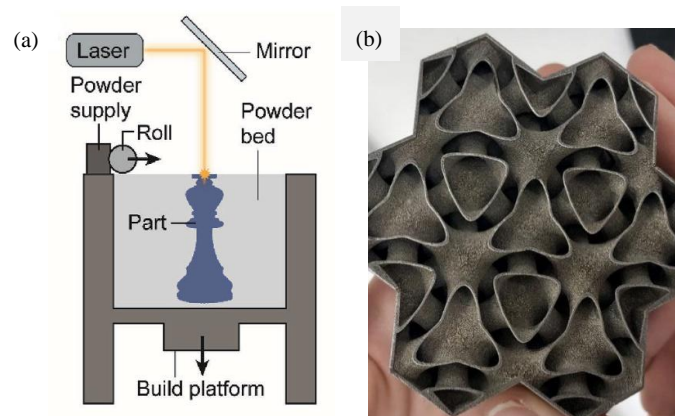


Figure S1. (a) Scheme of the powder-bed fusion process. Reproduced with permission from [5]. Copyright 2020, Wiley-VCH; (b) Lung-inspired heat exchanger printed by SLS. Credit 2019 – GE Research.

2. Material extrusion

In this method, materials are extruded through a print head and selectively deposited in a predetermined path layer by layer. This 3D printing technique refers to the technologies known as “fused deposition modelling” and “direct ink writing”.

2.1 Fused deposition modelling

Fused deposition modeling (FDM), also called “free form fabrication” or “fused filament fabrication” (FFF), is the most common and least expensive type of additive manufacturing technology. In this technique, a thermoplastic filament (e.g. Polylactic acid (PLA), Acrylonitrile butadiene styrene (ABS), Polypropylene (PP), etc.) is heated at a nozzle to a fluid state, is laid down, and cooled layer by layer on the build platform until the required object is formed (**Figure S2**) [6].

This process allows structures to be built quickly as basic proof-of-concept models or simple prototypes. However, poor mechanical properties, low dimensional accuracy and poor surface quality, such as layer-by-layer appearance, are the main disadvantages of FDM [7].

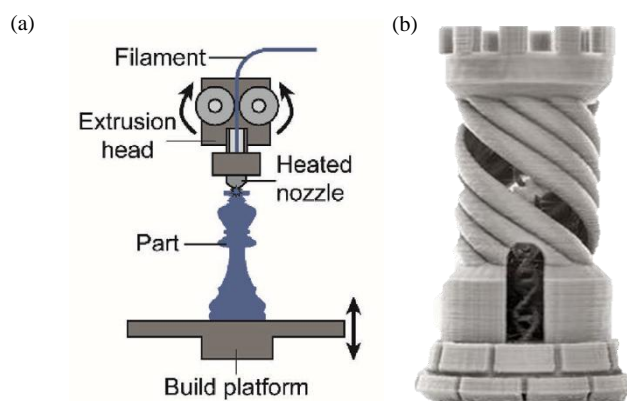


Figure S2. (a) Scheme of the FDM method. Reproduced with permission from [5] Copyright 2020, Wiley-VCH; (b) Chess tower produced by FDM. Copyright 2019, Formlabs.

2.2 Direct ink writing (DIW)

Direct ink writing (DIW) has been developed to print viscoelastic inks (**Figure S3**). This method allows complex structures to be printed using materials (or multi-materials) with controlled yield stress and shear-thinning behavior, including many polymers as well as their related composite inks [8].

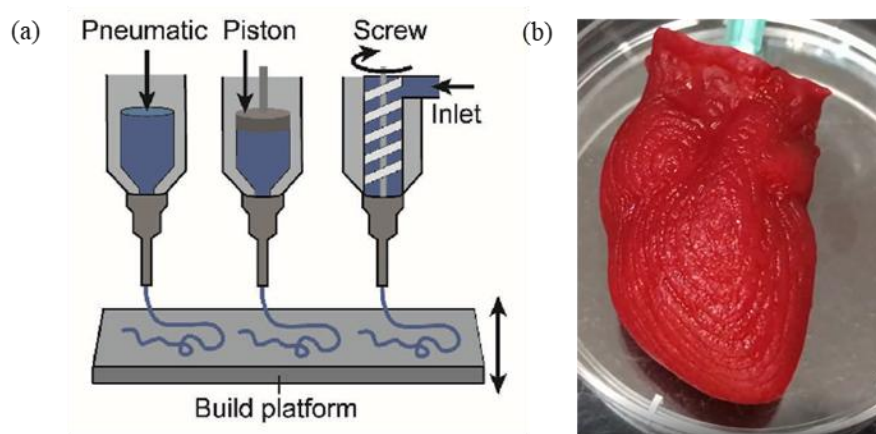


Figure S3. (a) Scheme of the DIW method with different print heads. Reproduced with permission from [5]. Copyright 2020, Wiley-VCH; (b) Biomimetic soft robotic heart printed by DIW. Reprinted with permission from [9]. Copyright 2019, American Chemical Society.

Direct ink writing (or printing) is based on a transient shear process: to induce flow through the nozzle, the applied shear stress exceeds the yield stress of the ink. Once the ink leaves the nozzle, its viscosity increases significantly to a gel state (storage modulus $G' >$ loss modulus G'') in order to retain the shape of the structure on the build platform or on previously printed layers. Thus, DIW is highly dependent on the composition of the ink, especially the amount and nature of the filler. When the layers are entirely printed, UV or thermal post-curing is often required to solidify the 3D structure.

This method offers a high degree of freedom in structure design, but this robust multi-material process has a low build speed and fixed resolution (mainly due to the size of the nozzle), although, under optimum conditions, the printing resolution can reach a few micrometers.

3. Material jetting

Material jetting is a technique in which droplets of build material are selectively deposited to form the desired structure [10].

3.1 Ink jetting

Ink jetting, a process adapted from 2D printing, is subdivided into two methods: continuous inkjet printing (CIJ) and drop-on-demand inkjet printing (DOD) (**Figure S4**).

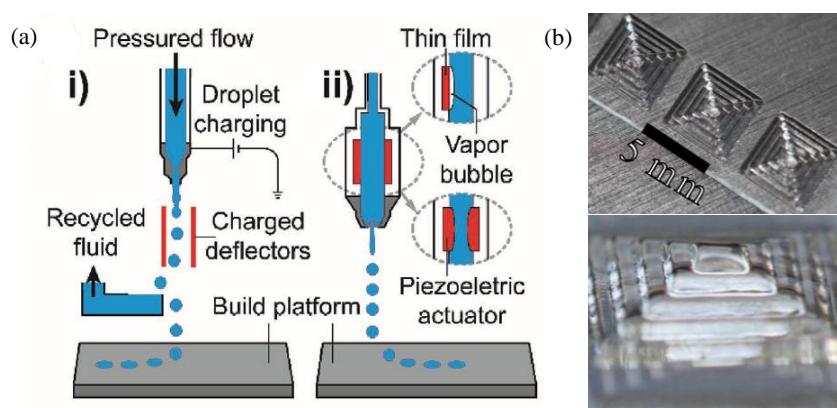


Figure S4. (a) Scheme of inkjet printing: i) CIJ mode; ii) DOD mode. Reproduced with permission from [5]. Copyright 2020, Wiley-VCH; (b) Pyramids with a 5 mm square base printed by ink-jet technology with reactive polydimethylsiloxane. Reproduced under terms of the CC-BY license from [13]. Copyright 2017, published by the Royal Society of Chemistry.

In the CIJ method, droplets are continuously pushed through the nozzle and deflected to the desired position, on the surface under construction, by means of an electrostatic field. In DOD, droplets are produced on demand by thermal ejection, piezoelectric effect, electrostatic or acoustic ejection, and then deposited on the platform. As the material is still liquid when deposited (viscosity less than 0.02 Pa s), the building material is usually ejected together with a dissolvable support material that will be removed during post-processing or, in the case of a photocurable ink, that must be cured directly [11,12].

Ink jetting is a versatile process allowing easy multi-material printing when several printing heads are used. However, the printed parts are structurally weak, which limits their use.

3.2 Aerosol jetting

Aerosol jetting or “aerosol jet printing” (AJP) is a hybrid microfabrication process. First introduced around 2000 [14], this method has opened up new possibilities for direct print using a directed aerosol stream that ensures a uniform deposition (**Figure S5**). This relatively new approach facilitates the patterning of complex surfaces that may not be achieved by conventional nozzles.

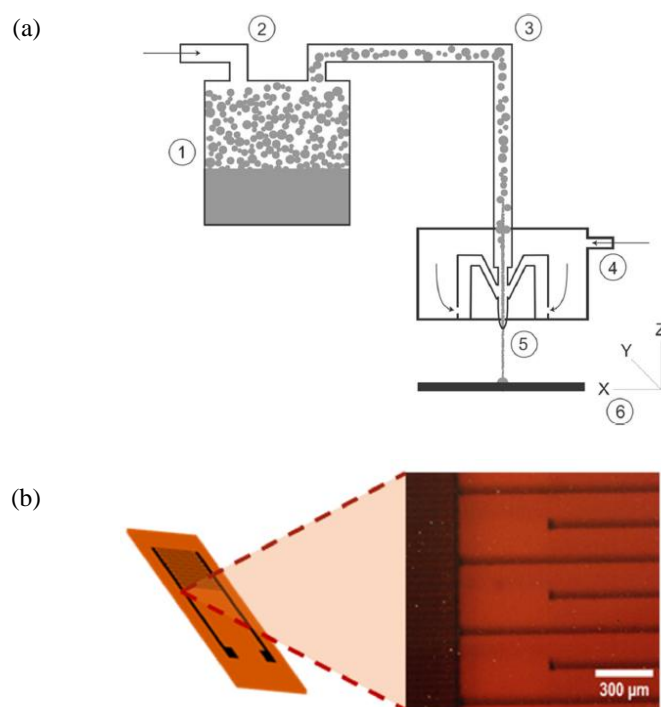


Figure S5. (a) Scheme of AJP: (1) aerosol generation using either ultrasonic or pneumatic atomizer, (2) introduction of a carrier gas to transport the aerosol, (3) transportation and refinement, (4) focusing, (5) deposition, and (6) computer-controlled translation of the substrate. Reproduced under terms of the CC-BY license from [16]. Copyright 2019, published by Springer Nature; (b) Optical micrograph of a graphene immunosensor printed by aerosol jetting. Reprinted in part with permission from [17]. Copyright 2020, American Chemical Society.

Ink aerosols (viscosity tolerance from 10^{-3} to 2.5 Pa s [15], higher than for ink jetting) are generated by ultrasonic or pneumatic atomization. The ultrasonic atomizer generates a highly monodispersed aerosol but is limited to low viscosities (below 0.01 Pa s), whereas the pneumatic method produces less uniform aerosols that require extra flow refinement steps before deposition but allows the use of higher viscosity materials. Any material that can be suspended in an aerosol is compatible with AJP but will present challenges in process development due to the interplay between the material properties (viscosity, volatility, density) and the aerosolization method [16].

4. Binder jetting

Binder jetting is a process in which a liquid binding agent is selectively deposited to join powdered materials together [1]. The individual layers are bonded to each other, resulting in a powder box with a binder arranged in the desired 3D geometry (**Figure S6**).

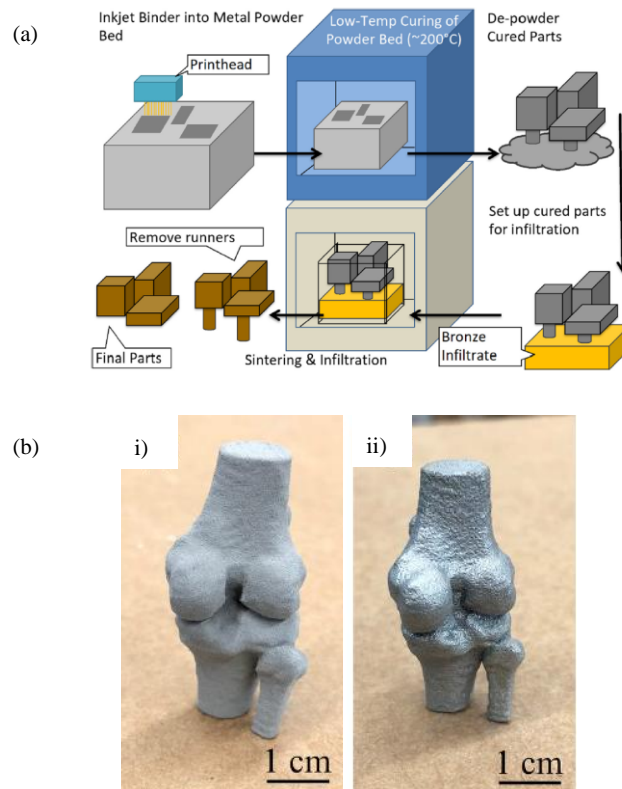


Figure S6. (a) Illustration of binder-jetted 3D parts followed by curing, depowdering, and densification by infiltration steps. Reproduced under terms of the CC-BY License from [19]. Copyright 2020, published by Elsevier; (b) Photograph of a prototype knee joint sample printed in 3D by binder jetting: i) before, and ii) after sintering. Reproduced in parts with permission from [20]. Copyright 2019, Elsevier.

This box can be cured and the structures obtained are considered green and therefore require post-treatment (such as sintering and metal infiltration) to achieve the required mechanical properties [18]. The typical layer height depends on the material and ranges from 50 μm for metal parts to 400 μm for sand-casting mold materials. This multi-step process, taking place at room temperature, is generally compared to powder bed fusion processes. Binder-jetting printed parts usually have lower mechanical properties and less accurate detail definition than structures prepared by SLS or SLM. Indeed, significant distortion of geometry can occur due to the low relative density of the printed parts, which currently limits the potential of binder jetting [19].

5. Vat photopolymerization

Developed in the early 1980s, vat photopolymerization is a specific process that uses light to selectively cure a liquid polymer to form the desired structure. Various vat photopolymerization processes (including stereolithography, digital light processing, continuous light interface production, two-photon polymerization, computed axial lithography, and direct laser writing) have been developed to meet the requirements of printing resolution and speed. The most common vat photopolymerization processes (with the exception of computed axial lithography) are following the same setup and printing process. During printing, a light source emits photons to cure the liquid photopolymer (or photoresin). By moving the light source or using a mask, the photopolymer forms a thin layer of patterned solid. The build platform moves by one layer thickness (upwards or downwards, depending on the vat construction), and a new layer of liquid photopolymer flows into the printing area, allowing the process to be repeated until completion [21,22]. In vat photopolymerization technologies, the photosensitive resin must have low viscosity or good fluidity. Resins with low viscosity are usually of low molecular weight, which induces a brittle photocured material, while high molecular weight monomers with high mechanical performance are often too viscous. Thus, the perfect balance between low and high molecular weight resins must be addressed to fabricate a low viscosity, high performance resin.

5.1 Stereolithography (SLA)

The laser of the most commonly used vat process points at two mirror galvanometers that direct light in the x and y axes to cure the layers (**Figure S7**). Depending on the laser wavelength, a cationic photoresin (355 nm), a free radical system (365 or 405 nm), or a hybrid dual-cure system (405 nm) is used. This robust and technologically important process allows

large models to be printed, but the larger size of the model, the longer the printing time. In addition, the printing resolution strongly depends on the size of the laser beam, and therefore, compared to other photocuring techniques, SLA has a rather low resolution [21,23].

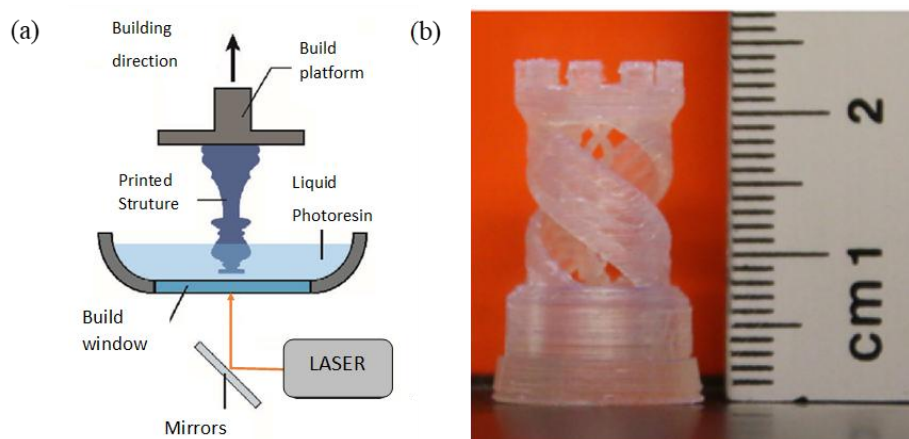


Figure S7. (a) Scheme of a bottom-up SLA apparatus. Adapted from [5]. Copyright 2020, Wiley-VCH; (b) Chess tower printed by SLA. Reprinted with permission [24]. Copyright 2018, American Chemical Society.

5.2 Digital light processing (DLP)

In the DLP technique, illumination is achieved by a dynamic mask (DMD) consisting of thousands of micromirrors, which can rapidly orientate and reflect light, or by dynamic liquid-crystal masks (LCD), which can directly form a mask pattern (**Figure S8a**).

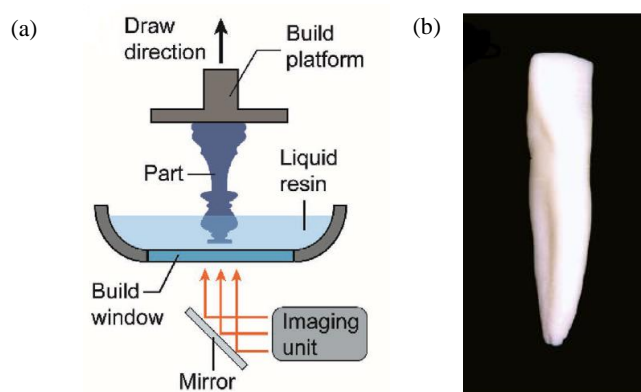


Figure S8. (a) Scheme of a DLP apparatus. Reproduced with permission from [5]. Copyright 2020, Wiley-VCH; (b) Zirconia dental implant printed by DLP. Reproduced with permission from [26]. Copyright 2017, Wiley-VCH.

In contrast to other processes that merely decompose the 3D model into layers, the DLP technique decomposes the image of each layer of voxels in 3D space (known as voxel effects), allowing high printing accuracy. However, the exposure area is still limited, which leads to a maximum printable size currently between 100×60 mm and 190×200 mm [21,25].

5.3 Continuous light interface production (CLIP)

In the CLIP method, a dead zone is created in the container between an oxygen-permeable window and the surface of the curing part, where photopolymerization is inhibited (**Figure S9**). In this way, the object to be cured is continuously pulled out of the resin bath at a production rate of 25 to 100 times higher than that of the DLP process. Nevertheless, this technique requires low-viscosity resin and hollow models to promote accurate photopolymerization. Besides, the oxygen-permeable membrane, which has to be changed regularly, is expensive [21].

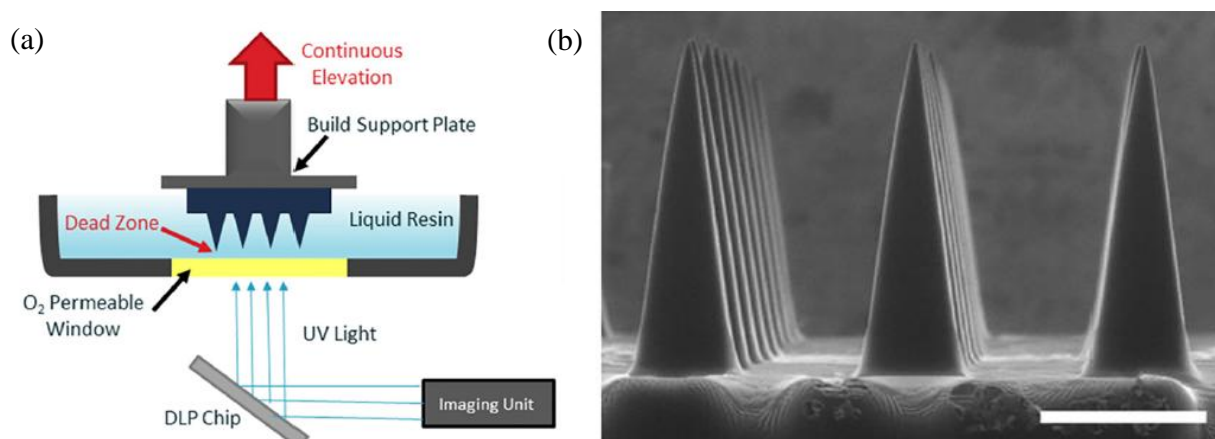


Figure S9. (a) Scheme of a CLIP printer where the object, here a microneedle patch, is continuously produced by simultaneously elevating the build support plate while changing the 2D cross-sectional UV images from the imaging unit. The oxygen-permeable window creates a dead zone (persistent liquid interface) between the elevating part and the window; (b) ESEM image of polyethylene glycol microneedles measuring approximately $1000 \mu\text{m}$ in

height and 333 μm in width. The scale bar is 500 μm . Reprinted in part under terms of the CC-BY license from [27]. Copyright 2016, Published by PLOS One.

5.4 Two-photon photopolymerization (TPP or 2PP)

TPP is a photochemical process based on two-photon absorption (TPA or 2PA) in which the photoinitiator simultaneously absorbs two photons to change from the ground state to the excited state and lead to subsequent resin polymerization. Optical systems for TPP can vary depending on the application, but are typically based on a near-infrared femtosecond laser beam (usually a Ti:Sapphire laser, $\lambda = 780\text{-}800\text{ nm}$, repetition frequency 80 MHz) focused on a volume of photocurable resin. The spatial resolution is mainly controlled by the laser power and the exposure time (**Figure S10a**). Thus, the TPP method offers an extremely high printing resolution of less than 100 nm. This process is also referred to as multi-photon polymerization (MPP, also based on TPA) [28] or even direct laser writing (DLW) [29,30].

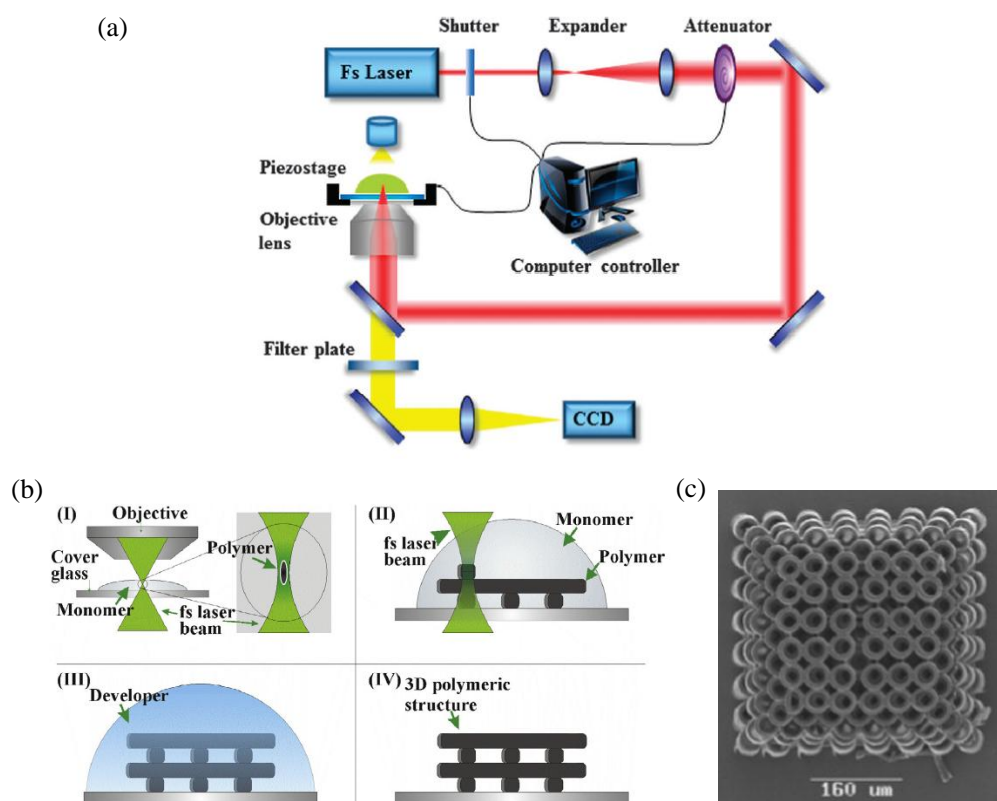


Figure S10. (a) A typical experimental setup for TPP manufacturing. Reproduced with permission from [29]. Copyright 2015, the Royal society of Chemistry; (b) TPP/DLW experimental procedure, showing: (I) beam focusing, (II) laser writing, (III) development, (IV) completed structure; (c) Scaffolds produced by DLW using PEG as photopolymer. Reproduced with permission from [31]. Copyright 2015, Elsevier.

5.5 Computed axial lithography (CAL)

Recently developed, this process allows a rotating volume of photosensitive material to be illuminated with a dynamically changing light pattern. Unlike conventional vat photopolymerization, in CAL the light energy is delivered to the volume of material in the form of a series of 2D images, and this superimposition causes the entire object to polymerize at once (**Figure S11**) [32].

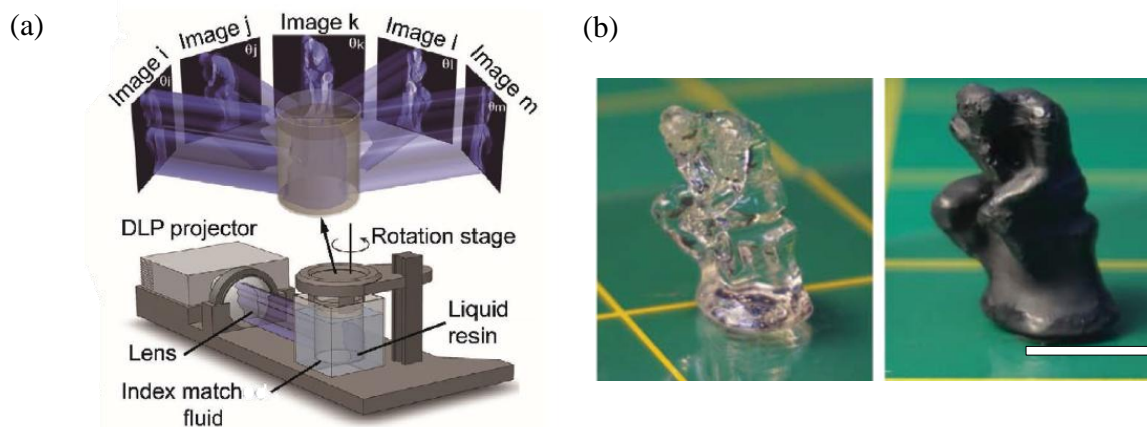


Figure S11. (a) Scheme of a CAL system; (b) Corresponding 3D-printed object before and after painting. The scale bar is 10 mm. Reprinted with permission from [32]. Copyright 2019, exclusive licensee American association for the Advancement of Science.

6. Sheet lamination

6.1 Laminated object manufacturing (LOM)

Laminated object manufacturing is based on layer-by-layer cutting and lamination of sheets of materials (“form-then-bond” or “bond-then-form” methods). The bonding process is performed on the bottom layer by heating and pressurizing it with a hot plate or hot laminating roller (**Figure S12**).

To date, specific studies have been published that focus on LOM for the fabrication of paper, metal laminate, polymer matrix composites and ceramics. The LOM process is a good alternative for the manufacture of functional parts, especially for 3D metal printing. Depending on the material used, this method offers one of the largest operating temperature windows. However, this process has limited 3D geometries, and the strength of the printed structures is highly dependent on the laminates used. In addition, LOM faces many significant challenges due to misalignment of the layers, and the efficiency of bonding is reduced by the thermal energy given off by laser cutting, which requires further improvement [33].

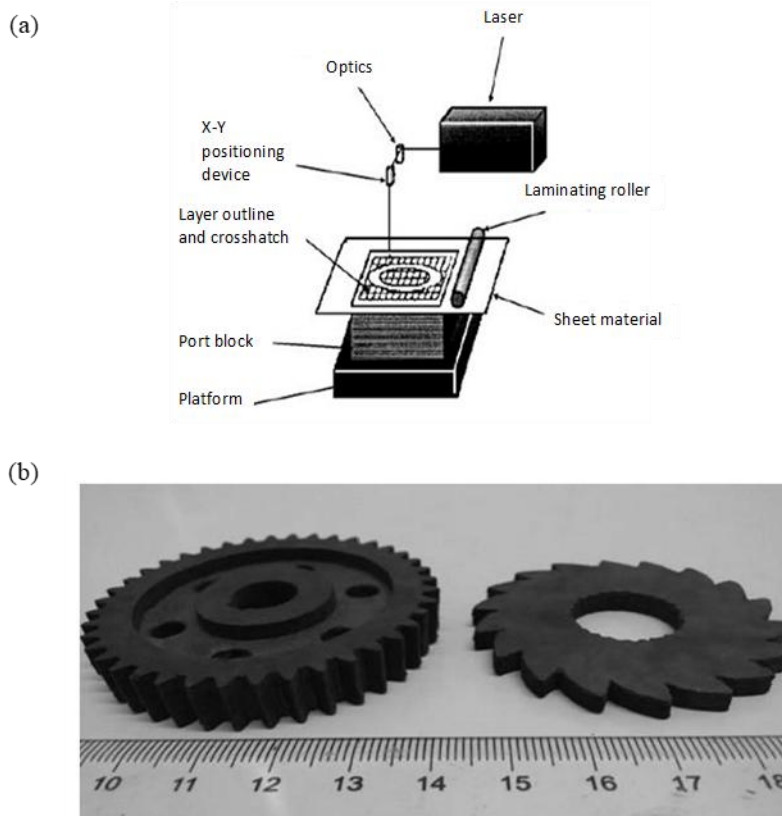


Figure S12. (a) Scheme of a LOM apparatus; (b) 3D-printed gear wheels using the LOM process with SiC tapes and after thermal treatment. Reproduced from [34] under terms of the CC-BY license. Copyright 2015, Published by Göller Verlag.

6.2 Ultrasonic additive manufacturing (UAM)

UAM is a new subclass of the LOM process that sequentially bonds metal foils together by ultrasonic welding, disrupting the oxide layers between metal foils by high-frequency plastic deformation. UAM also integrates computer numerical control (CNC) to remove undesired material to create a specific geometry during the additive build-up process (**Figure S13a**) [35]. UAM is the only additive manufacturing method capable of mechanically bonding dissimilar materials together in the solid state (as no melting occurs), incorporating heat-sensitive or functional components between the foil layers, and bonding the structure into a dense matrix. This process, still limited to metal foils, does not reliably produce a finely

detailed structure that cannot tolerate the load applied throughout the process and requires a stronger supporting material than the inclusion to withstand [36,37].

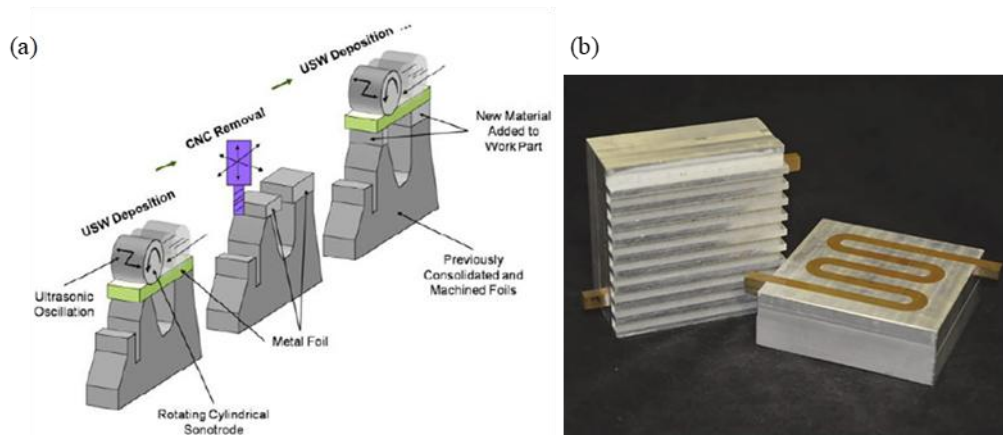


Figure S13. (a) Scheme of the UAM Process. Reproduced from [35] under terms of the CC BY- NC- ND license. Copyright 2013, Published by Elsevier; (b) Copper-aluminum heat-exchanger produced by UAM. Credits 2015 – Fabrisonic.

7. Directed energy deposition (DED)

Directed energy deposition (DED) has been used for the manufacture of high-performance super-alloys or, marginally, polymer matrix coatings. This method, also known as “laser engineered net shaping” (LENS), “laser solid forming” (LSF), “directed light fabrication” (DLF), or “direct metal deposition”, uses an energy source (laser or electron beam) that is directly focused on a small area of the substrate to melt it simultaneously with a feedstock material to be fused (**Figure S14**). With lower accuracy and surface quality than power-bed fusion processes, DED is commonly used for large, low-complexity parts or their repair [38,39].

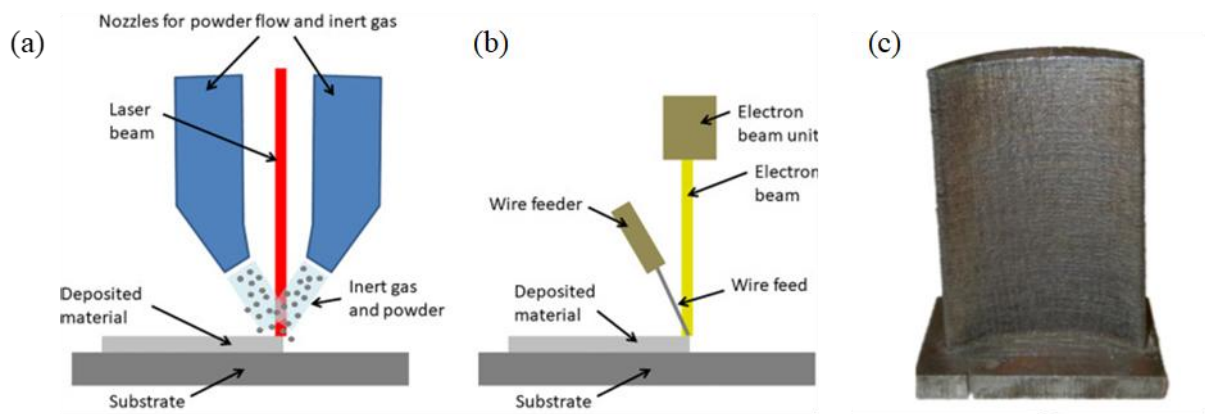


Figure S14. Scheme of two DED systems, using: (a) a laser with a powder feedstock, or (b) an electron beam and a wire feedstock. Reproduced with permission from [40]. Copyright 2020, Elsevier; (c) Turbine blade printed by the DED method. Reproduced with permission from [41]. Copyright 2014, Elsevier.

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