Review

A Review on Advanced Manufacturing for Hydrogen Storage Applications

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Abstract: Hydrogen is a notoriously difficult substance to store yet has endless energy applications. Thus, the study of long-term hydrogen storage, and high-pressure bulk hydrogen storage have been the subject of much research in the last several years. To create a research path forward, it is important to know what research has already been done, and what is already known about hydrogen storage. In this review, several approaches to hydrogen storage are addressed, including high-pressure storage, cryogenic liquid hydrogen storage, and metal hydride absorption. Challenges and advantages are offered based on reported research findings. Since the project looks closely at advanced manufacturing, techniques for the same are outlined as well. There are seven main categories into which most rapid prototyping styles fall. Each is briefly explained and illustrated as well as some generally accepted advantages and drawbacks to each style. An overview of hydrogen adsorption on metal hydrides, carbon fibers, and carbon nanotubes are presented. The hydrogen storage capacities of these materials are discussed as well as the differing conditions in which the adsorption was performed under. Concepts regarding storage shape and materials accompanied by smaller-scale advanced manufacturing options for hydrogen storage are also presented.

Keywords: bulk hydrogen storage; advanced manufacturing; 3D printing; carbon; metal hydrides

1. Introduction

With the growing demand for clean energy, a tremendous amount of research and resources are being poured into the development of efficient energy carriers: methods used to take the energy in its supplied form (wind [1], wave [2], solar [3] hydro [4], nuclear [5,6], etc. [7,8]) and deliver that energy how and when it is needed [9]. For example, a city uses more power in the day, but a hydroelectric dam produces power at a constant rate. An energy carrier, such as hydrogen, can be used to store excess power produced during the night hours and released when needed in the day, or a future day [10,11].

Hydrogen as an energy carrier has been of growing interest in a time in which natural resources are depleting through fast consumption of fossil fuels [12]. Hydrogen could be used as a viable energy carrier as it produces zero emissions and is reproducible through the use of natural gasses [13]. To manage the storage of hydrogen, which is a gas notoriously difficult to manage, advanced manufacturing techniques are being investigated in hopes of solving the problem [14,15]. It is hoped that 3D printing and other advanced manufacturing technologies will enable the rapid prototyping of different pressure vessels, varied in geometry and material composition [16].

Hydrogen storage is one of the most challenging issues in today's energy sector due to its storage needs [17,18]. Hydrogen can be stored in three forms, via compressed gas, as a cryogenic liquid, and by solid state storage [13]. When stored as compressed gas, hydrogen

needs to be in a highly pressurized tank which makes it not ideal for hydrogen storage as a fuel source [19]. Storage via cryogenic liquid requires high funding costs and issues of boiling over time [20]. With that in mind, solid state storage is where current research on hydrogen storage is focused on, allowing for adsorption (physisorption/chemisorption) and/or absorption onto the surface of metal hydride and or carbon nanostructured materials [13,21,22]. Through the use of advanced manufacturing, metal and carbon materials can efficiently be produced [23,24].

Advanced manufacturing generally implies the use of 3D printing and other additive technologies [25–29]. Traditionally, 3D printing is done by extruding plastic layer by layer until a part is formed. In this case of hydrogen storage, the materials required are metals of high strength and corrosion resistance. Technology for the 3D printing of metals and ceramics has been explored and a summary of many of those manufacturing techniques is given in this review. Through the use of 3D printing, metal hydride and carbon nanostructured materials can be made which have been found to store hydrogen efficiently through physisorption of hydrogen onto the surfaces of the material [30]. This review can be helpful to provide an insight towards achieving Net-Zero via different pathways for example CO_2 capture, storage and utilization [31–33].

2. Advanced Manufacturing Options

2.1. Advanced Manufacturing (AM) Techniques

To understand how to apply advanced manufacturing to the research of hydrogen storage, some basics have to be discussed first, beginning with how a 3D part is formed from two-dimensional layers to three-three dimensional parts [34–36]. Many strategies have been employed to accomplish this, and they each have different uses and applications. Seven categories have been identified which include almost all advanced manufacturing technologies that have been discovered [37]. These categories are: binder jetting, material jetting, material extrusion, vat photopolymerization, powder bed fusion, energy deposition and sheet lamination [38–40]. An explanation and illustration of these processes is given below.

(a) Binder Jetting

In binder jetting (BJT), a thin layer of particles (maybe 2 layers of particles) is spread (through various methods) across the print area [41]. Then, a glue or solvent called the "binder," is drizzled over the print as shown in Figure 1. This binder holds the individual particles together. It then either evaporates away (in the case of a solvent binder) or is removed in post-processing in an oven for example, leaving only the desired print material. Thermal post-processing is vital in accomplishing enhanced functional properties to obtaining the maximum output from the part. This manufacturing technology can print a variety of materials that are obtainable in powder form, with materials such as polymers, ceramics, and even metals [42].
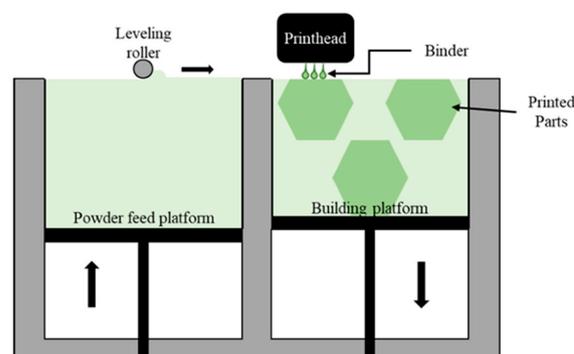

Figure 1. Schematic representation of binder jetting working mechanism additive manufacturing printing process. Reprinted with permission from Ref. [43]. Copyright 2020 by the authors and licensee MDPI, Basel, Switzerland.

BJT was not established to be effective in printing fully dense ceramic parts. However, it has been proven that material systems that integrate functional ceramics draw advantage from designed porosity, in addition to the improved surface area contributed by lower-density structures. Thus, binder jetting is a potential candidate for the manufacturing of efficient material systems where functional ceramics are used.

(b) Material Jetting

Material jetting (MJ) does what the name implies; shoots or drops a stream of the material to be printed down onto the print bed or previous layer [44]. This material is often cured with UV light. Though the design of MJ printer differs slightly from manufacturer to manufacturer, a universal schematic depiction of MJ can be seen in Figure 2. The material jetting is a potential additive manufacturing technique in the polymer industries owing to its easy operation and compatibility in contrast to other manufacturing techniques. Different ink materials such as polylactic acid, polyamide acrylonitrile butadiene styrene, and their blends can be shared in a single ink jetting, called the multi-material jetting approach [43]. The multi-material jetting approach can be employed to fabricate composite parts for specific uses [45]. Finally, MJ printers come with an enclosed ambient environment which avoids undesirable effects of draught or dirt and can be used outside of clean labs. However, industrial applications of materials jetting method still require further research. Particularly, in the aerospace industries, MJ was used only in printing small prototypes for wing related applications. Its potential for energy storage purposes where mock-ups are required for proper installation is worth investigating.
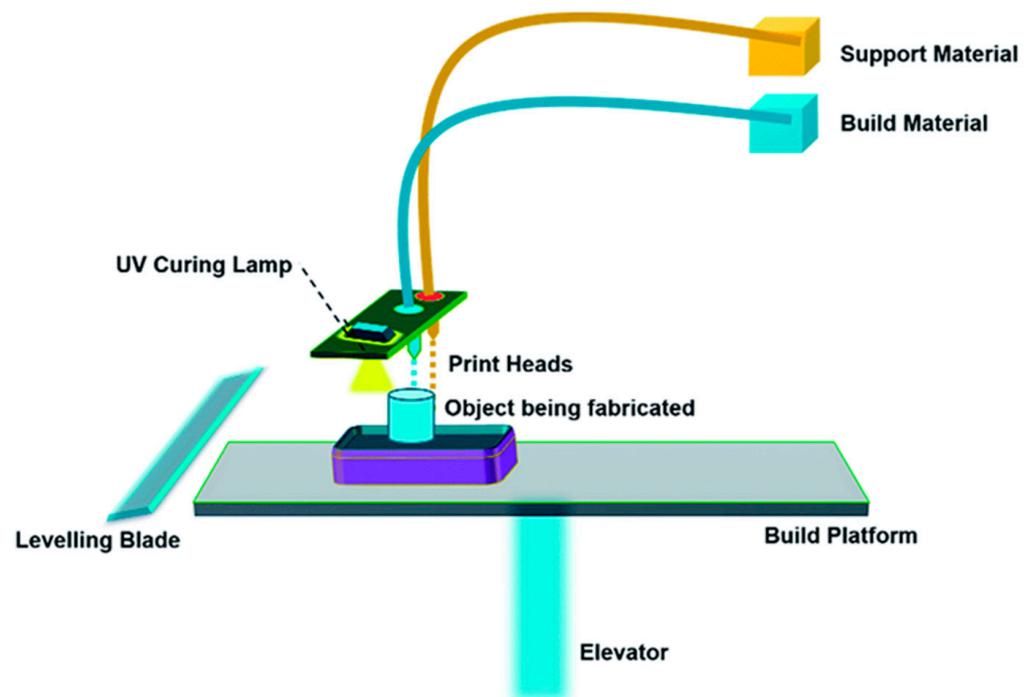

Figure 2. Schematic representation of the material jetting process. Reproduced from Ref. [46]. with permission from the Royal Society of Chemistry.

(c) Material Extrusion

Material extrusion is what is generally implied when 3D printing is discussed [47]. Figure 3 shows the printing process using the material extrusion (ME) process. Usually, a plastic filament is fed through a heated nozzle to a liquid or near liquid state and is squeezed in tiny layers to form a part. This clearly has limitations; metal parts would be very difficult to melt without damaging the nozzle, print bed, and surrounding parts [48]. The melting point is simply too high and it would also be very energy inefficient. Ceramics

can be extruded, but usually in a soft form, such as unfired clay, and rely on post-processing (such as being fired in a kiln) to finish the part. Post processing is both expensive and affects the dimensions of the final part in a way that is difficult to control. Main advantages of this AM process contain use of readily available acrylonitrile-butadlene-styrene (ABS plastic, a thermoplastic) that can yield models with good structural properties, nearly resemble a concluding production model.
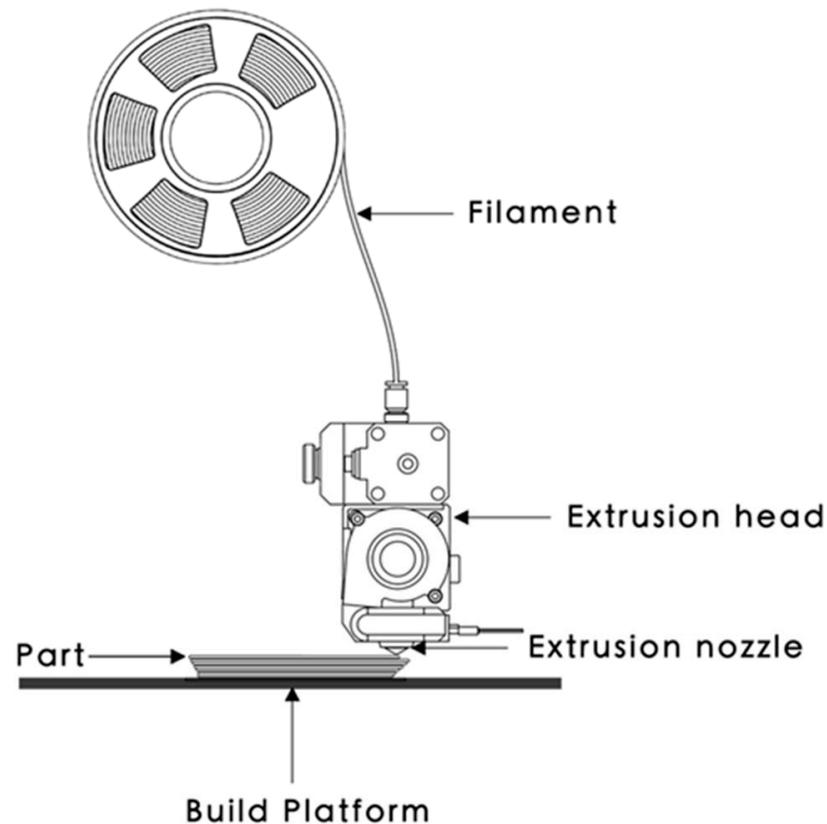

Figure 3. The schematic shows material extrusion (ME) process. Reprinted with permission from Ref [49]. Copyright 2020 by the authors and licensee MDPI, Basel, Switzerland.

(d) Vat Photopolymerization

In vat photopolymerization, the bed is often inverted so it is systematically raised out of a vat of liquid [50]. A ray of ultraviolet light is used to cure the liquid polymer layer by layer onto the part, as shown in Figure 4. This is most frequently used to print with resin, but there is research on ceramics or ceramic-plastic composites being produced using this or similar methods. Digital light projection (DLP) printing [51] and Stereolithography (SL) [52] are the two VAT polymerization methods. SL has turned out to be an excellent industrial additive manufacturing choice for rapid prototyping. Multiple material stereolithography is also accessible, which alters the materials used for subsequent layers. Industries including chemical engineering, packaging, entertainment, sporting goods, biomedical automotive, aerospace, and energy apply SL.
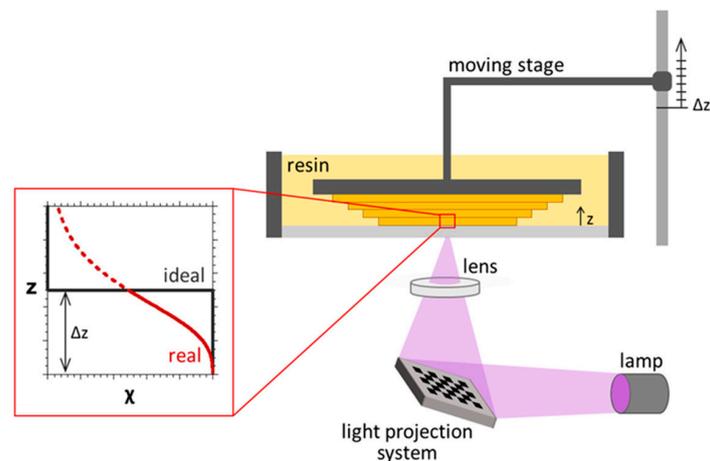
Figure 4. Schematic showing of a bottom-up vat photopolymerization 3D printer. Each printed layer, with a thickness of Δz, was characterized by a non-constant monomer-to-polymer conversion χ, which showing a sigmoidal profile (red), and varies from the ideal square profile (black). Reprinted with permission from Ref. [53]. Copyright 2016 by the authors and licensee MDPI, Basel, Switzerland.

The main disadvantage of Vat Photopolymerization is that the process is relatively expensive. Time consuming post processing steps and removal of printed parts from resin are also problematic. Limited material availability of photo-resins is also a drawback and this AM process often requires support structures and post curing steps for parts to be strong enough for structural use.

Digital light processing technique is an imminent 3D printing method that utilizes illumination of polymers and resins. This advanced manufacturing process is like SL, but the cutting-edge DLP technique is equipped with a digital mirror, an array of millions of self-rotated mirrors, and by projecting the 2D pixel pattern whole layers can be formed in a single step which decreases the build time drastically.

(e)   Powder Bed Fusion

Powder bed fusion is perhaps the most well-known process for the printing of metal parts. In this process (shown in Figure 5), the print bed is coated in a layer of fine powder of the material desired [54,55]. A laser then draws the 2-dimensional layer slice [56]. The powder is fused together and to the bed floor or previous layer to form a solid metal part. Another layer of powder is evenly distributed, and the next layer is traced with the high-power laser, and so on and so forth. This technique requires careful storage, as steel, aluminum, and many other metals are highly reactive in a powdered state due to extremely high surface area exposure.
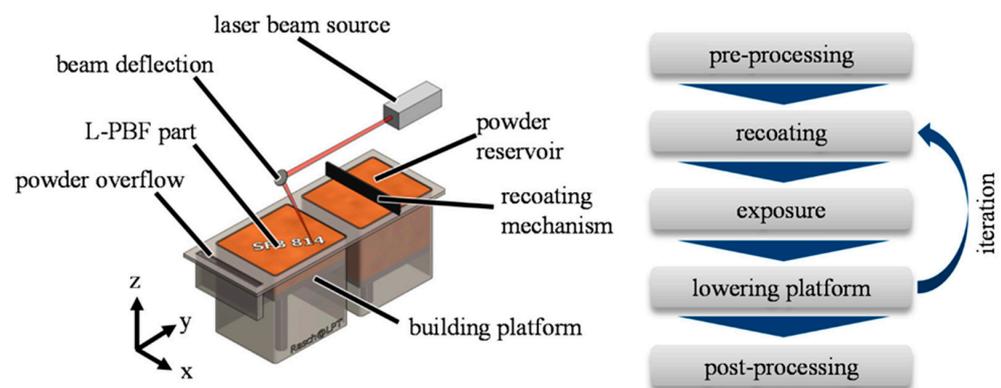

Figure 5. Schematic of the laser powder bed fusion process. Reprinted with permission from Ref. [57]. Copyright 2018 by the authors and licensee MDPI, Basel, Switzerland.

(f)   Direct Energy Deposition

Direct Energy Deposition (DED), an additive manufacturing method to print metals, alloys and functional components, is a layer-by-layer (LBL) AM process [58,59]. Theoretically, in distinction to the powder bed fusion methods where a powder bed is selectively melted, direct energy deposition is operated using concurrently supplied powder feeding of wire and a focused energy source such as such as Nd: YAG or CO_2, or other energy sources. In this printing, a melt pool is designed on the substrate surface or previous coated layer by using a relatively high-energy laser, and powder is propelled through the laser and into the melt pool. All layers are produced track by track via a user-defined tool path. Figure 6 displays a diagram of the DED technique for the molten pool, where the substrate and powder are melted. This technique generates less waste, allows for production of bigger parts, modification/repair of existing parts, and enhaces speed of manufacturing of new parts. However, it also means that no support is possible, so there are more stringent restrictions on design.
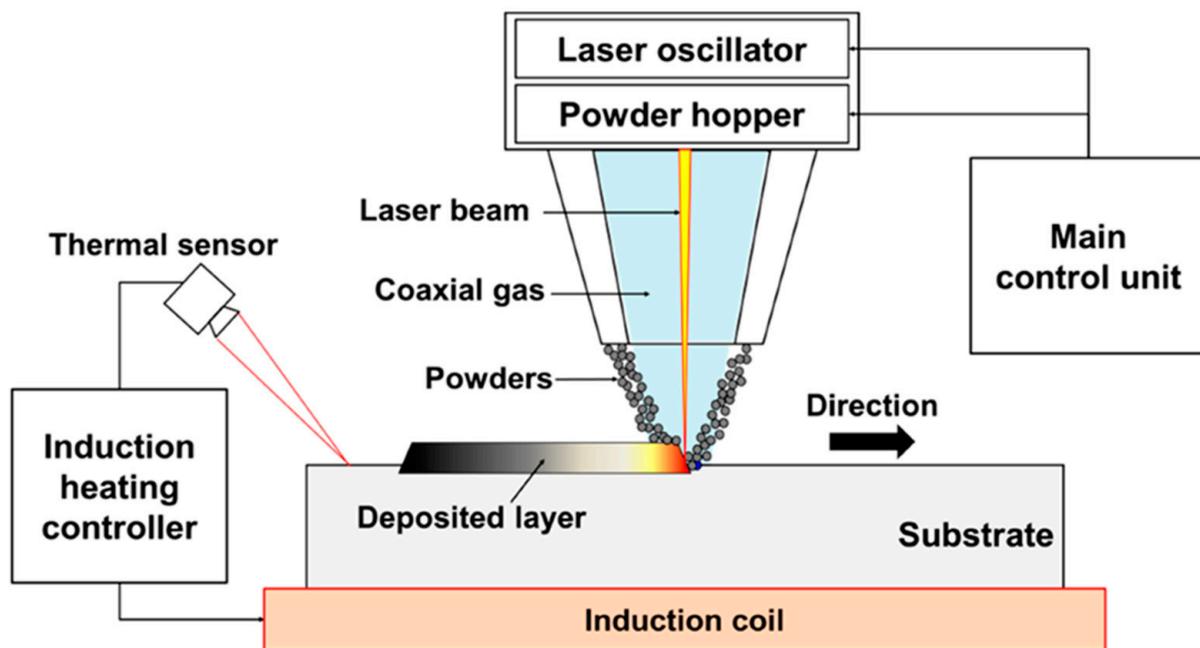

Figure 6. Schematic of the laser powder bed fusion process. Reprinted with permission from Ref. [60]. Copyright 2019 by the authors and licensee MDPI, Basel, Switzerland.

(g)   Sheet Lamination

Sheet lamination (SL) process, also known as Laminated Object Modelling (LOM) manufactures items and prototypes by cutting, successively laminating, and bonding [61–63]. In this process, the printer lays layers of foil down and are bonded with some combination of an adhesive or ultrasonic blast to induce bonds between layers with mechanical pressure. LOM operates with an ultrasonic welding and a laser cutter. A diagram for the representation of the sheet lamination process is shown in Figure 7. The LOM process involves solid state bonding and thus additional adhesives are used since it is not required for the material to reach its melting point for bonding to occur. A wide range of materials can be manufactured using the LOM method which comprises metals such as aluminum, stainless steel, copper and titanium, plastics, fabrics, paper, ceramics, synthetic materials, and composites [46].
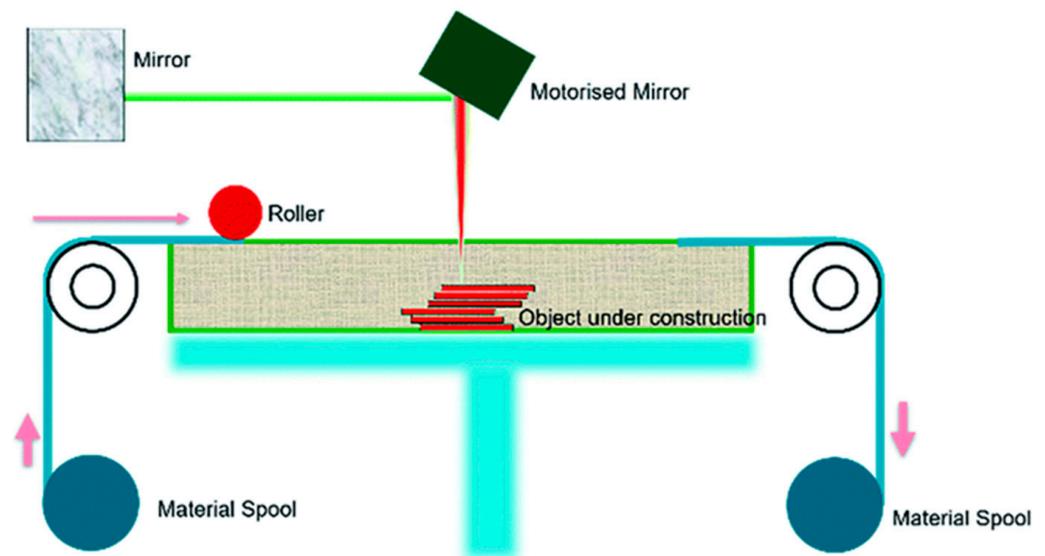
Figure 7. Schematic representation showing sheet lamination process. Reproduced from Ref. [46]. with permission from the Royal Society of Chemistry.

A convenient means of summarizing the various AM process spectra, and categorizing process candidates for applications is according to their material compatibility along with their advantages and disadvantages which is displayed in Table 1 [64–67].

Table 1. Different AM printing methods comparison.

AM Technique	Material	Advantage	Disadvantage
Fused Deposition Modeling (FDM)	Thermoplastics such as carbon nanofiber/AB S, PLA, PA Nylon, Graphene/AB S	Low cost, high speed, deposition of various materials, printed parts that are multi-functional.	Thermoplastic polymer as the only working material, material should be in filament form, absence of homogeneity in disperse material.
Stereolithography (SLA)	Photocurable TiO2/epoxy acrylate, CNT/acrylic ester BST/epoxy	Best for making of concept prototypes, quick manufacturing times and decent surface appearance and geometrical accuracy	Restricted to procedure non-functional materials, for example resins or plastics, Materials are costly and inadequate in accessibility, unable to process functional materials such as metals, and needs support structures.
Binder Jetting (BJ)	Aluminum oxide (Al_2O_3) and alumina-silica powders, PCL, PLA polymers and binder materials, amorphous or colloidal silicon carbide (SiC)	Decent printing resolution, soft materials with multi color ability, and low cost processing compared to SLM, SLA printing techniques	Mechanical strength of printed parts is not decent, high surface roughness, existence of large porous microstructures in manufactured parts.
Sheet Lamination (SL)	Any sheet material foil such as paper, metals, plastics, fibers glass, composite	Appropriate for processing of medium and large sized constituents, such as dies or metal making tools and extensive choice of obtainable materials in the form of sheet form, no obligation of pre-designed support structure, it is a faster technique.	Poor layer bonding carries the risk of de-lamination, Strength of the produced components in the perpendicular path to the layers is considerablly less than in additional directions and several post processing methods are required, post processing is a must, manufacturing time increases as no. of layers grows

Table 1. *Cont.*

AM Technique	Material	Advantage	Disadvantage
Direct Energy Deposition (DED)	HSS, Tool steel, nickel—based alloys titanium aluminum	Layer can be made-up in any alignment, range of materials in the form of powder can be handled, large parts can be fabricated and improved deposition rates are conceivable	Geometrical accuracy is inferior, stair-stepping consequence can constrain geometric precision and post-processing might be needed
Selective laser sintering (SLS)	PCL and polyimide powder, carbon black/nylon-12, Al_2O_3/polystyrene, silica/nylon	Materials which can be managed include plastics, ceramics, sands and some metals, parts shaped are appropriate for functional testing and no support structures are needed	Accessibility of metallic materials is limited; a surrounded chamber is essential and metal sintering brings porosity and mechanically weak components produced.

2.2. Application and Trends of 3D Printing Technologies

Lately, there has been a lot of publicity about the potential of what can be accomplished by adopting 3D printing as one of the key additive manufacturing technologies [68–70]. 3D printing technologies have developed significantly in recent years and can now execute essential roles in many applications, such as manufacturing [71], automobile [72], aerospace [73], defense [74], medicine [75], energy [76], architecture [77], construction [78], food [79], cosmetic [80], fashion [81], art [82,83], supply chain [84], prosthetics [85] and even fighting against coronavirus disease in 2019 (COVID-19) [86] (Figure 8). Over a long period of time, the main issue with 3D printing techniques was that it entailed high access costs, which prevents its lucrative employment in mass-manufacturing [87]. However, as the commercial market for 3D printed materials and parts has shown some of the fastest growth within the manufacturing sector in recent times, it has been discovered that this issue is finally going away [88].
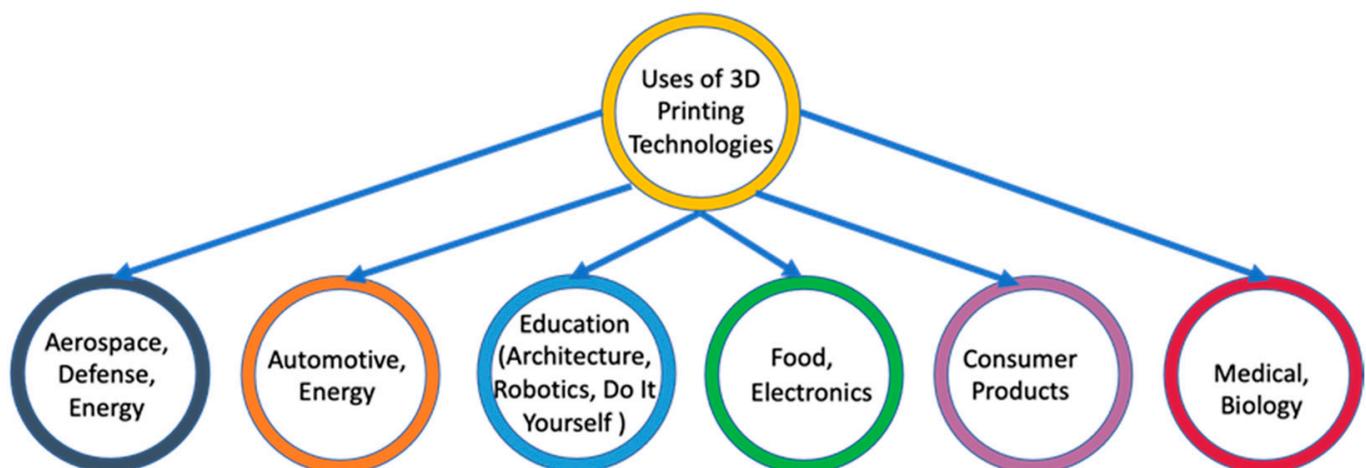

Figure 8. Application of 3D printing technologies.

AM is already well known in the biomedical field for making surgical guides and custom-made health care products [89]. There are still not adequate manufacturing choices in 3D printing techniques and materials in the medicine, food, electronics, and energy markets, compared to the range of materials used for other manufacturing techniques (e.g., injection molding). It signifies that new developments are much needed to fill the gaps in printing options and materials, pouring a new cohort of 3D printing applications. It is expected that further options for high-temperature polymers, composite materials, and

3D printers will be emerging in the market soon. With the growth of 3D printings, the evolution of software for 3D printings will grow significantly.

Concerning energy storage, specifically electrochemical energy storage devices such as supercapacitors, and batteries, 3D printing allows for complementary form factors to be considered based on the need of the end user [90]. 3D printed energy storage devices need active materials and composites that are printable, owing to their basic electrochemistry requirements [91]. Traditional electrochemical energy storage device (EESD) fabrication contains electrode fabrication, electrolyte preparation and device assembly, and it has been reported that 3D printing has led to improved performance in batteries and supercapacitors [92].

In a recent study, Liu et al. [93], described a low temperature direct writing (LTDW)-assisted 3D printing technique to fabricate 3D LiFePO4 (LFP) electrodes. This study shows that a LFP ink was printed into a low temperature compartment (−40 °C) and frozen as the ink was deposited layer by layer. The frozen electrode was then subsequently freeze-dried to attain a 3D electrode with porous morphology. The schematic of the LTDW process is exhibited in Figure 9 [93]. Fan et al. [94] demonstrated (shown in Figure 10 (a–j)) that 3D-printed sodium-ion hybrid capacitor (SIC) supercapacitor devices containing both high areal energy and power densities established an activated carbon (AC) cathode and nitrogen-doped porous Ti3C2Tx (N-Ti3C2Tx) anode.

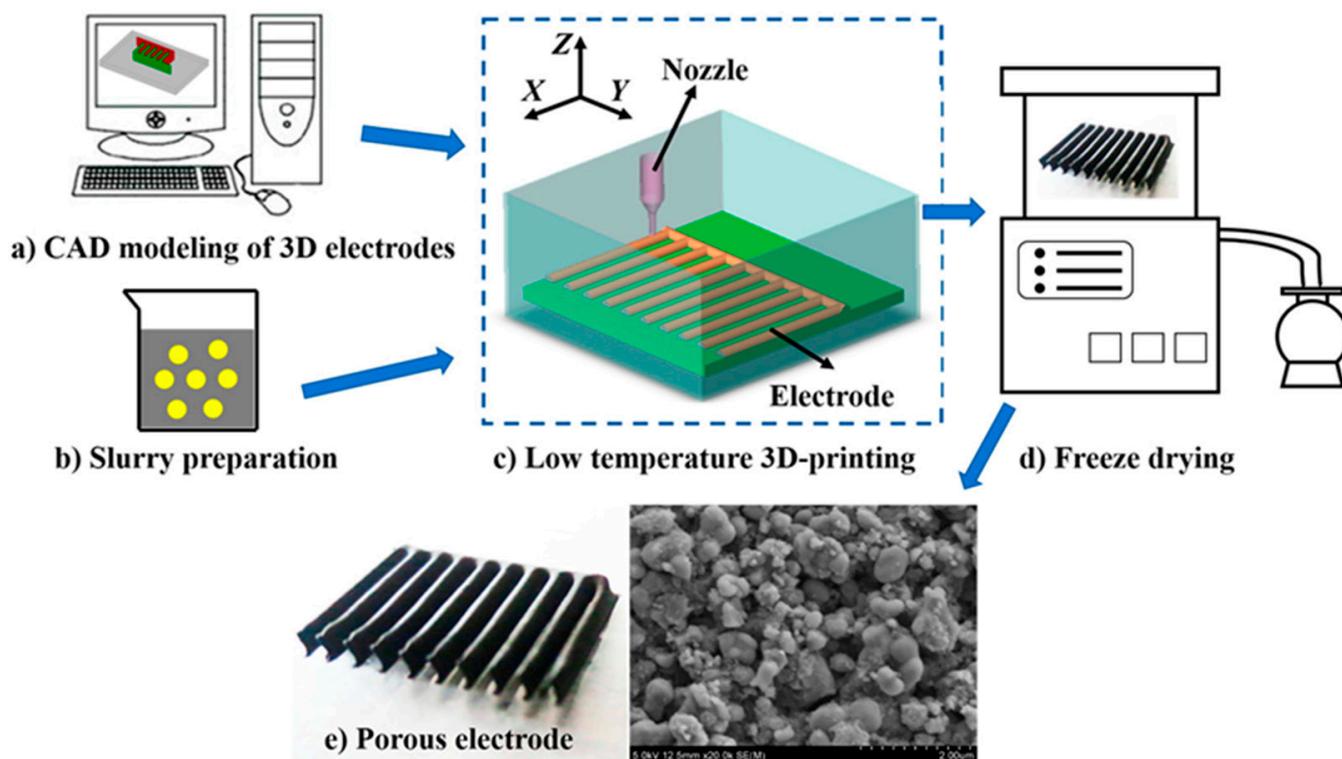

Figure 9. Manufacture of LiFePO4 (LFP) electrode by low temperature direct writing (LTDW). (**a**) Computer-added design (CAD) modeling of 3D electrodes, (**b**) slurry making, (**c**) low temperature direct writing, (**d**) freeze-drying step, (**e**) obtained porous electrodes. Reprinted with permission from Ref. [93], Copyright 2017 by the authors and licensee MDPI, Basel, Switzerland.
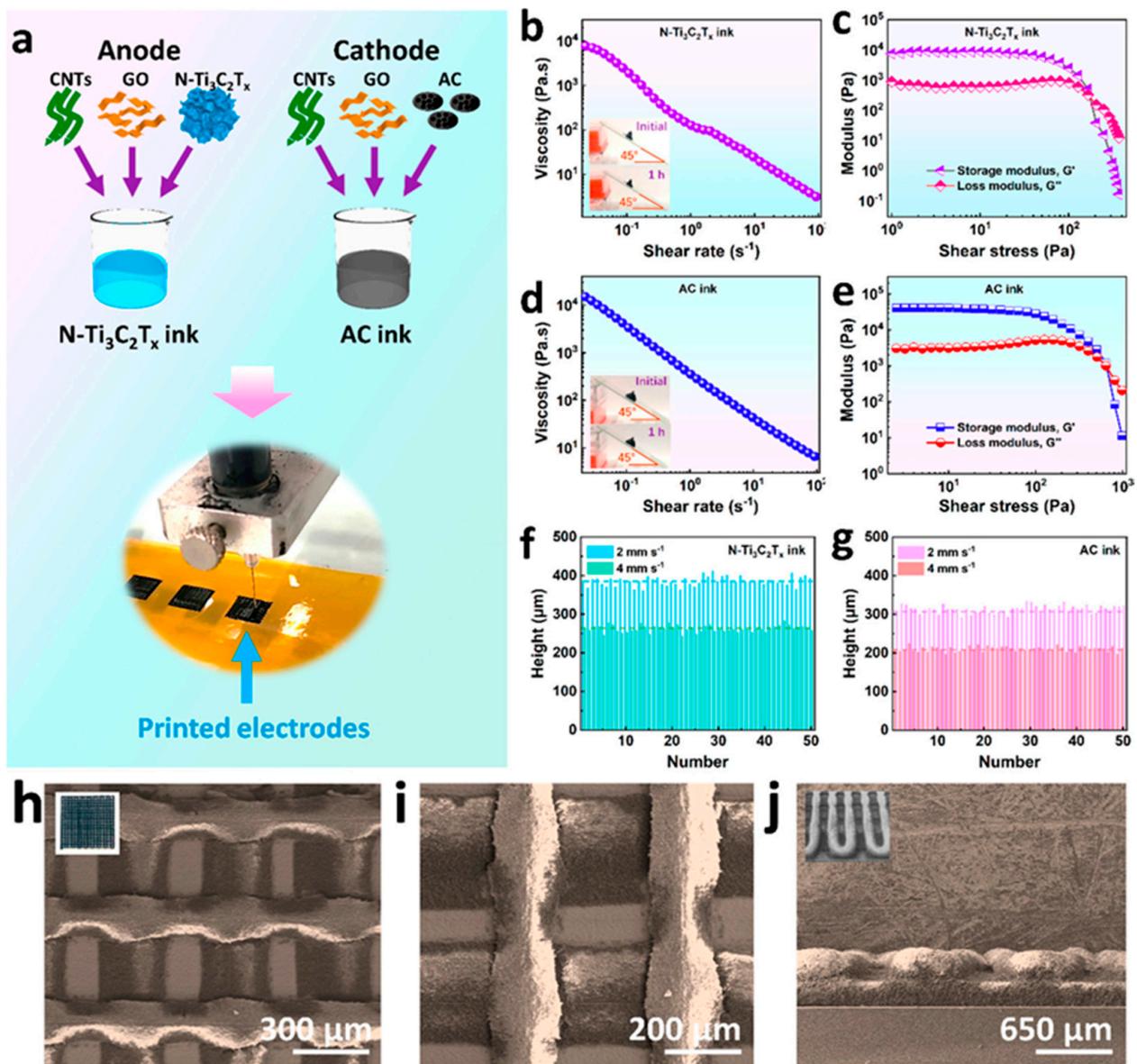
Figure 10. Description of a 3D printing method, rheological properties of inks, and microstructure of the woodpile-shaped electrodes. (**a**) Illustration of the printing process of the 3D-printed cathode and anode. (**b**) Apparent viscosity as a function of shear rate for the N-Ti3C2Tx ink. (**c**) Storage modulus (G′) and loss modulus (G″) as a function of the shear stress for the N-Ti3C2Tx ink. (**d**) Apparent viscosity as a function of shear rate for the AC ink. (**e**) Storage modulus (G′) and loss modulus (G″) as a function of the shear stress for the AC ink. (**f,g**) Height distribution of the 3D-printed (**f**) N-Ti3C2Tx (M8T1) and (**g**) AC lines with changed speeds. (**h–j**) SEM micrographs of the (**h,i**) top analysis and (**j**) side analysis of the 3D-printed N-Ti3C2Tx electrode. The inset in (**h**) displays a real photograph of the woodpile electrode. The inset in (**j**) shows a low-magnification SEM image of the woodpile electrode. Reprinted with permission from Ref. [94], Copyright 2020 by the American Chemical Society.

Generation, handling, transportation, and storage of hydrogen is a challenging issue of modern times [95]. By 3D printing materials and composites, it is hoped that hydrogen storage materials issues will be addressed [16]. Printed metal–organic framework materials (MOFs) have been the focus of much research given their significantly large internal surface areas [96]. MOFs are usually synthesized in powder form [97,98]. For hydrogen storage applications, MOFs need to be shaped into monoliths or structural forms that can be easily handled. Lim et al. [99] demonstrated a 3D-printing of pure MOF monoliths where

accessible porosity and surface area was retained well after shaping. Figure 11 shows the 3D printing of MOFs and the related rheological properties.
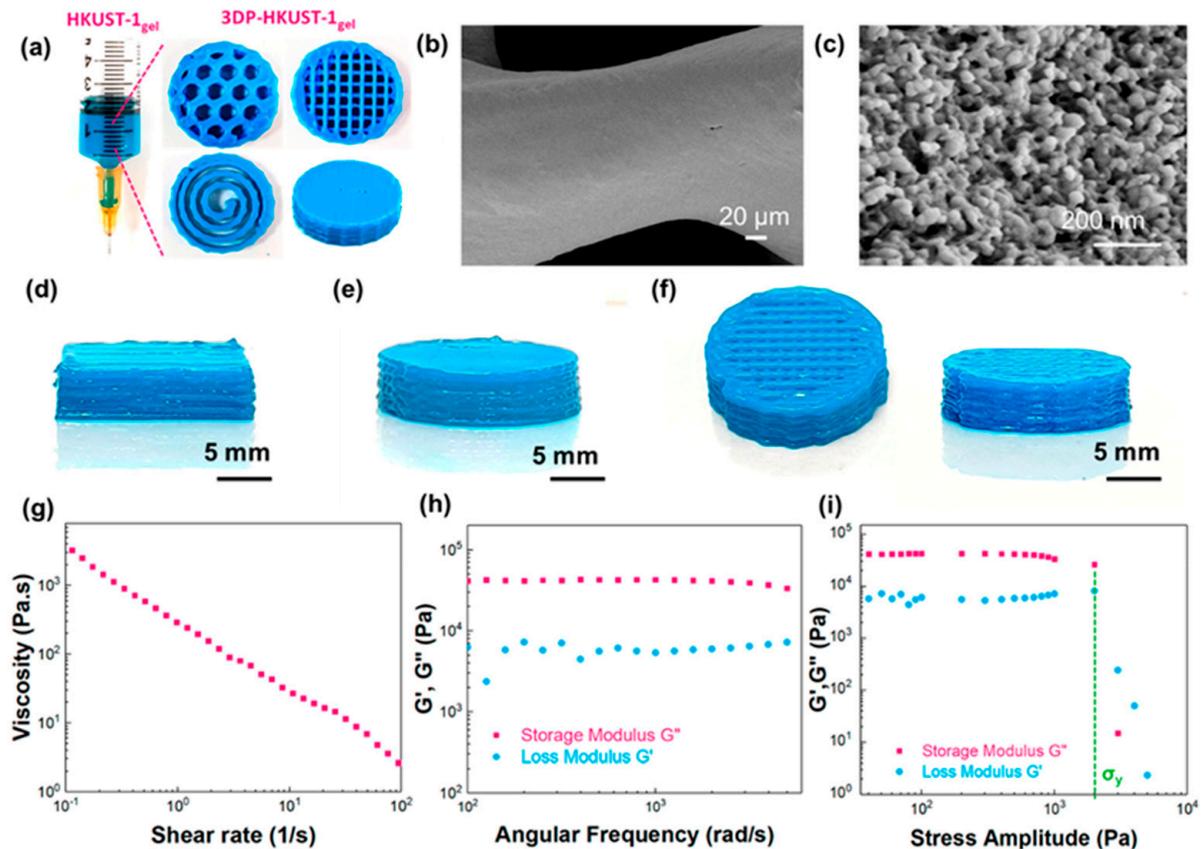

Figure 11. (**a**) Colloidal gels comprising ethanol and Cu3(BTC)2 (BTC = 1,3,5-benzenetricarboxylate) (HKUST-1) gel filled into a syringe and distinctive 3D printed assemblies. (**b**) SEM micrograph of a distinct filament from a dried 3DP-HKUST-1gel monolith. (**c**) SEM image of HKUST-1 nanoparticles within the dried 3DP-HKUST-1gel monolith. (**d**,**e**) Side vision of high-profile 3D-printed monoliths: (**d**) square-shaped monolith with ten number of layers and (**e**) showing a circular pellet monolith with ten layers. (**f**) Top vision and side vision of a high-profile mesh-like monolith. (**g**–**i**) Rheological characteristics of HKUST-1gel presentation that the gel is solid-like at rest and displays shear-thinning performance with growing shear force: (**g**) Apparent viscosity as a function of shear rate. (**h**,**i**) Small amplitude oscillatory shear outcomes. (**h**) The storage modulus G' and the loss modulus G'' attained against the angular frequency. (**i**) G' and G'' noted against the shear stress-amplitude at a constant frequency of 6.283 rad/s. Reprinted with permission from Ref. [99], Copyright 2020 by the American Chemical Society.

3D printed proton exchange membranes, which are a critical component of hydrogen generation, are already in the market [100]. Advancements in 3D printing technologies have facilitated the integration of the traditionally external heat exchanger into the liquid hydrogen storage tank structure itself, thus decreasing mass and volume while delivering vapor cooling for the stored liquid hydrogen given that liquid hydrogen storage systems employ various insulation approaches that have direct bearing on the mass and overall volume of the tank. Recently, Kong et al. [101] demonstrated 3D printing of 316 L stainless steel in a proton exchange membrane fuel cell environment and found that the printed part showed superior resistance to hydrogen damage. Compressed gas is the most well-known hydrogen storage technology. Lastly, a steam storage tank was reported using 3D printing which could be useful for the hydrogen storage application [102].

3. Hydrogen Storage Options

3.1. Mechanical Hydrogen Storage Options

There are several methods of hydrogen storage available in solid and liquid form. These methods are, (i) H_2 storage in high-pressure gas cylinders (fit for 800 bar), (ii) liquid hydrogen in cryogenic tanks (maintained at $-252\ °C$), (iii) adsorption of hydrogen on materials with a large specific surface area (at $T < -173\ °C$), (iv) absorption of H_2 on interstitial sites in a host metals at ambient environmental conditions, (v) chemically bonded in covalent and ionic compounds at an ambient pressure, and (vi) via oxidation of reactive metals, e.g., Li, Na, Mg, Al, Zn with water [19,103,104].

To proceed along the research path, it is important to understand the different parallel paths taken to achieve the same goal. In this case, hydrogen as an energy carrier is the ultimate goal. There are several approaches to achieve this goal, including cryogenic freezing, high pressure tank storage including underground salt caverns, and use of metal hydrides for absorption. Though our research path will look specifically at man-made vessel high pressure storage considerations, it is noted that literature review on other research methods was performed.

Cryogenic freezing of hydrogen is an option for storing pure hydrogen [105]. It is a rather involved process, using a standard method used to liquify other types of gases, and then to cool the hydrogen in the last phase, it is throttled, turning a percentage of the gas to a liquid. It is very challenging to store liquid hydrogen in a closed system because any increase in temperature can increase the pressure in the system dramatically. This means there are losses of hydrogen from the system.

High pressure vessels for hydrogen storage have their own difficulties. Hydrogen is very high in energy density per unit weight (3 times more than gasoline), but very low in energy density per unit volume (four times less than gasoline) [106]. Hydrogen must be stored at a much higher pressure than other types of natural gas such as propane to produce the same energy outputs. More pressure means more energy input, which implies higher cost. These pressure constraints also encompass the materials used as vessels. Therefore, geometry as well as materials are considerations in research. For example, the conventional cylindrical tanks used for pressure vessels are in fact less efficient at withstanding pressure than a perfect sphere [106]. Cylinders are used because spheres are complicated geometries to manufacture [106]. That is one advantage of advanced manufacturing; geometrical constraints are significantly less stringent than conventional manufacturing.

New materials are also being sought for storing hydrogen, not only because of the need for resistance to high pressures, but the resistance to hydrogen embrittlement. Hydrogen is considered non-corrosive [107], but it will bore its way through many materials, including steel and most other metals, forming bubbles under the surface. These bubbles build pressure and burst, leaving tell-tale "blisters" and cold work the metal, hardening and embrittling it. This could lead to unexpected material failure [108]. This has led to a search for materials that resist this "corrosion". Polymers are thought to resist this blistering better than metals, but polymers are terrible in mechanical strength. Ceramics can be very porous, though some would hold the gas. However, ceramics are already very brittle. The topic of materials for this task is a complicated one. Another approach to hydrogen storage, meriting its own section, is chemical absorption with the use of metal hydrides.

3.2. Chemical Storage Options

a.   Metal Hydrides

Metal hydrides are a current area of study in the future of large-scale hydrogen storage as metal hydrides have the ability to accommodate high densities of hydrogen [109]. The use of metal hydrides for hydrogen storage is a form of solid-state hydrogen storage as hydrogen can either be adsorbed or absorbed to allow for higher densities of hydrogen at moderate temperature and pressure levels [110]. Within solid-state hydrogen storage, hydrogen can be stored via physisorption, binding via van der Waals forces. Or chemisorption, binding via chemical bonds, which pertains to metal hydrides and complex metal hydrides.

Storage of hydrogen within metal hydrides occurs in the gas phase allowing hydrogen to react with the metals, specifically light metals, to create a metal hydride [30]. Metals often involved in the hydrogen storage process are light metals including that but not limited to, lithium, sodium, magnesium, boron, tin, and aluminum [109]. Figure 12 depicts a zirconium metal hydride and although zirconium is not commonly used in the formation of metal hydrides for hydrogen storage purposes, the formation of the cubic structure depicts a similar formation to that of metal hydrides formed to create hydrogen storage.
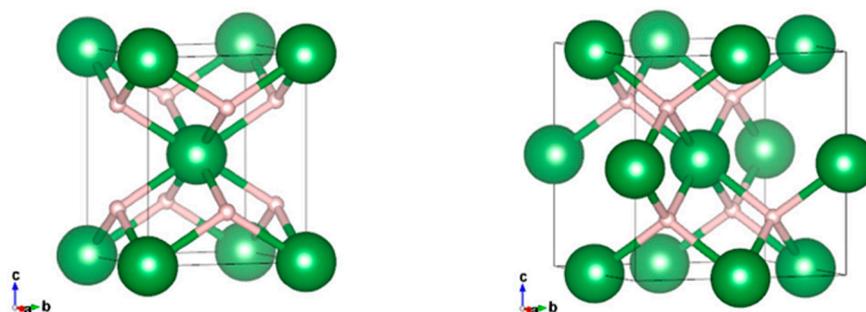

Figure 12. Simulation image of a crystal unit cell for ϵ-ZrH2 (**left**) and δ-ZrH1.5 (**right**). Zirconium atoms are colored green (large), and hydrogen atoms are rose (small). Reprinted with permission from Ref. [111]. Copyright 2021 by the authors and licensee MDPI, Basel, Switzerland.

The ability for metals to absorb and desorb hydrogen via the bonding and breaking of chemical bonds makes the use of metal hydrides one of the most efficient forms of hydrogen gas per volume compared to liquid and other solid phase storage techniques of hydrogen. Developments in the use of metal hydrides for hydrogen storage focus on improving the thermodynamic reaction involved in the absorption and desorption of hydrogen.

Based on research conducted on the creation of metal hydrides for the storage of hydrogen, commonalities arose regarding heat issues that were a result of creating the metal hydrides. According to Graetz et al. [110], there was a large hydrogenation enthalpy and this production of high enthalpy is meant to make up for the large change in entropy during the hydrogenation process. This creation of heat impacts the amount of time taken to fill the storage tank and this time constraint becomes a problem as current research in hydrogen storage is geared towards the automotive industry, using hydrogen as a fuel source. The time taken to fill up a hydrogen fuel tank is an important factor if hydrogen fueled cars are to be made available to the masses. In terms of the release of hydrogen from the storage tank, researchers found difficulties due to the production of a high enthalpy and a slow kinetic rate of the reaction leading to high temperatures needed to allow the release of hydrogen from the tank [112]. According to Kim et al., based on thermodynamic calculations associated with reversible hydrogen storage in light metal hydrides, 359 crystalline structures were used to create thermodynamically viable reactions for hydrogen storage and ultimately two reactions were determined to be the most thermodynamically favorable, those being the metal hydrides including a mixture of $MgH_2/Mg(NH_2)_2$, and that of $LiNH_2/LiH/KBH_4$ [112]. Reactions including $MgH_2/Mg(NH_2)_2$, were widely studied pertaining to hydrogen storage and although there are still issues regarding their thermodynamics as the hydrogen adsorbed is released at high temperatures, $MgH_2/Mg(NH_2)_2$ can release at least 6.0% wt of hydrogen at the completion of the reversible reaction [9,100]. With that, the addition of other hydride forming metals, or the formation of a quaternary structure can improve the reversibility of the reaction and MgH_2 alone is not reversible in nature [30]. Forms of Mg and Li compounds used for metal hydride hydrogen storage are of the most researched as those elements are of the lightest and most reactive hydride forming elements in the periodic table. Through continued research on the thermodynamics of metal hydride formation for hydrogen storage tanks, an efficient metal hydride could be produced without the production of high temperatures due to enthalpy values when absorbing and desorbing hydrogen.

b.   Carbon nanostructures

Hydrogen storage within carbon materials is an attractive form of storage as carbon has a low specific weight and high specific surface area, allowing for high gravimetric storage of hydrogen [113]. Carbon in its activated form is also widely available and inexpensive to manufacture [114–122]. For hydrogen storage there are several forms of carbon materials used: carbon nanofiber (CNF), single walled carbon nanotubes (SWNT), and multiwalled carbon nanotubes (MWNT) [108]. Carbon materials have high storage capacity of hydrogen roughly ranging between 3.0–7.0 wt% [123] under ambient temperatures with variance in wt% based on the form of carbon material. Hydrogen gas is commonly adsorbed onto the surface of carbon materials which is the physisorption of hydrogen and the size of the pores is also a factor that can impact the wt% of hydrogen adsorbed. Pore sizes range between 2 and 50 nm with 3 size categories, micropores, mesopores, and macropores. There are also ultra-micropores which are under 1 nm in size but are not as commonly used in hydrogen storage applications despite the fact that ultra-micropores are predicted to be best for hydrogen storage [113].

Under a study conducted by Mu et al., under ambient conditions using micro and mesopore CNFs, the wt% of hydrogen adsorbed was at low levels. Through those results, it was inferred that the pore size and surface area were more significant contributors to the wt% of hydrogen adsorbed, and as the micro and mesoporous carbon materials used were calculated to adsorb and desorb nitrogen, those sizes were not sufficient to adsorb hydrogen. It was predicted that an ultra-micropore size, under 1 nm, would better aid hydrogen adsorption [113].

Single walled carbon nanotubes (SWNT) often undergo a deposition process to lay a layer of metal nanoparticles onto the surface of the nanotubes. The deposition of a metal acts as a catalyst in the reaction, metals often used include Fe, Co, Mg, Ag, Pt, Li and Pd [22]. As depicted in Figure 13, the structure and formation of SWNT and MWNT are shown [124]. The study in which the wt% of hydrogen adsorption into a SWNT was measured with and without the deposition of Pt. The conditions of the research were 77 K and 25 bar. Ultimately the adsorption of hydrogen using SWNT alone had a 0.55–0.65 wt%, while under the same conditions, the SWNT with the deposited Pt layer had a wt% between 65–70 [22]. Although the deposition of the Pt aided in the adsorption of hydrogen, the conditions on the reaction and the wt% threshold are both too low for Department of Energy (DOE) standards for hydrogen storage, which is refueling at ambient conditions with a 6.0 wt% [123]. This makes CNF a better carbon material for hydrogen storage applications [125]. The results of the data discussed contradicts that of other data collected on SWNT hydrogen storage adsorption with wt% between 3.0–7.0% and that is due to the difference in conditions.
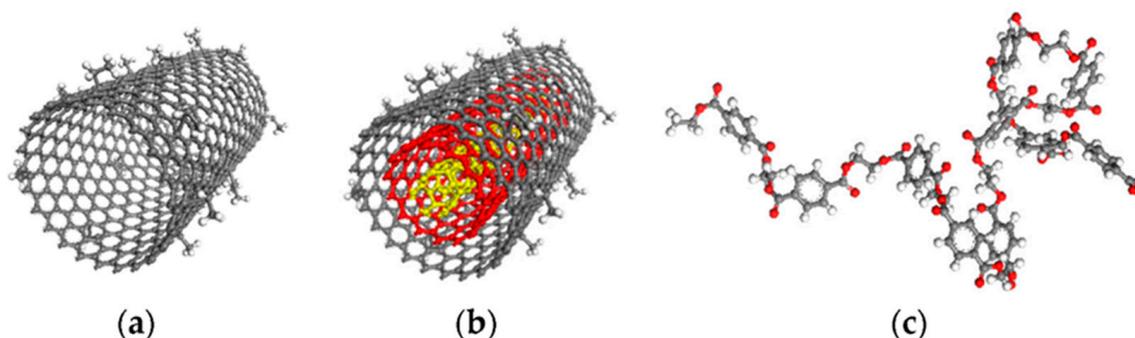

Figure 13. Molecular models of (**a**) (23, 0) single-walled carbon nanotubes (SWNT), (**b**) (23, 0), (14, 0), (5, 0) multi-walled carbon nanotubes (MWNT), and (**c**) polyethylene terephthalate (PET) chain. Reprinted with permission from Ref. [124]. Copyright 2021 by the authors and licensee MDPI, Basel, Switzerland.

SWNT and MWNT are made of rolled sheets of graphene and formed via chemical vapor deposition (CVD) [37]. Similar to data collected on SWNT, MWNT also has conflict-

ing hydrogen wt% information based on various research studies. Although differences in conditions of the pressure and temperature are a significant reason why the wt% of hydrogen has been found to be varied, metal doping as well as differences in the amount of moisture measured in the hydrogen gas is also a contributing factor to the differences seen of the wt% of adsorbed hydrogen [37].

4. Conclusions and Recommendation

Hydrogen storage is a complex subject with many areas of study. There is still much research needed to bring hydrogen out of theory and into practice as an efficient energy carrier. There are many challenges to address. For example, high pressure storage in tanks yields lower energy capacity unless stored at very high pressures and imposes other obstacles such as hydrogen embrittlement. Additionally, low temperature liquid storage works well for storing hydrogen but in general is used for small scale quantities and is rather inefficient. In terms of chemical storage, Mg and Li metal hydride materials produce the highest wt% of hydrogen adsorbed compared to other commonly used metals for hydrogen storage, like that of Al and Na. Based on studies reviewed, of the various forms of carbon materials used for hydrogen storage, CNF have the highest wt% of hydrogen adsorption under ambient conditions, yet more research on SWNT and MWNT materials needs to be conducted under ambient conditions to better compare the wt% to the DOE goal of 6.0 wt%. Further development in advanced manufacturing technologies [126,127] including various 3D and 4D printing approaches should also be geared towards solving the mechanical and chemical hydrogen storage option.

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