Development of a 3D Printer for Oxide-free Aluminum Transportation

By

Henry R. Smith

Submitted to the Department of Mechanical Engineering
in partial fulfillment of the requirements for the degree of

BACHELOR OF SCIENCE IN MECHANICAL ENGINEERING
at the
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Abstract

Hydrogen as an energy carrier is abundant, has a high caloric value and only produces water when combusted. A challenge is directly transporting hydrogen incurs high costs due to its low density. The low flammability limit also makes it dangerous to transport. Aluminum has been proposed as an alternative energy carrier for its high density and ability to be stored at ambient conditions, allowing for cheaper transportation. Hydrogen can be produced on-site by reacting the aluminum fuel with water. However, when exposed to air aluminum forms an inert oxide layer on its surface, preventing reaction. High reaction temperatures are required to overcome the oxide layer leading to a high energy penalty.

This thesis proposes a novel concept of aluminum encapsulation with water-soluble polymer. A 3d printer was designed and fabricated which creates aluminum-polymer structures that do not oxidize during storage and can achieve a wide range of reaction rates with water by varying the structure surface area. This new approach provides several benefits, by removing the oxide layer before the reaction happens, the aluminum is in an “activated” state and can react at room temperature. This reduces the energy required for reaction. Additionally, by having control over the reaction rate, ideal production rates can be achieved, reducing waste products and meeting consumption demands. The unique manufacturing flexibility of 3d printers enables the fabrication of structures with wide ranges of surface area to volume ratios. By shipping activated aluminum in the polymer structures, hydrogen can be produced locally and the need for expensive hydrogen transport can be eliminated.

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Contents

List of Figures

List of Tables

1. Introduction and Background

1.1 Hydrogen as an Energy Carrier

Global production of energy requires substantial use of processes that emit greenhouse gases and contribute to global warming [1]. The energy crisis requires sustainable technologies for energy sources, transfer and storage to meet global energy needs and reduce the impact of climate change. Energy carriers are used, as the name implies, to carry energy overtime (storage) or distance (transportation). Common energy carriers are used as fuels to generate electricity, power vehicles and run industrial processes. Hydrogen has grown popular as an energy carrier due to its high abundance and heating value [2]. Hydrogen is naturally found in air and water and has a heating value 3x that of natural gas and diesel [3]. Additionally, only water is produced when hydrogen is combusted (see eq. 1), making it a carbon-free energy carrier.

$$2H_2 + O_2 \rightarrow 2H_2O + \text{Energy}$$

Equation. 1**:** Combustion of hydrogen in pure air.

A major challenge with hydrogen is storage and transportation. Hydrogen is the smallest element and is not very dense compared to other fuels. Hydrogen in liquid form has a density of 70.9 kg/m^3 compared to gasoline density of 765 kg/m^3[4][5]. To be cost-effective, Hydrogen needs to be transported and stored as a liquid at cryogenic temperatures or at very high pressures. Additionally, a unique issue with hydrogen storage is embrittlement. Embrittlement occurs when the hydrogen permeates into its surrounding material, binding to the microstructure and lowering the stress required for crack growth [6]. This is especially concerning structures under stress, such as highly pressurized vessels which are used in hydrogen storage. Direct transportation of hydrogen has significant drawbacks, but by using an alternative energy carrier, costs and hazards can be reduced.

1.2 Aluminum as Energy Carrier

Aluminum has been proposed as a clean alternative energy carrier to hydrogen. Aluminum as a fuel is non-toxic, has about 4.5x lower caloric value than hydrogen but 38x the density, making it suitable for cost-effective transportation (see Figure 1) [2].

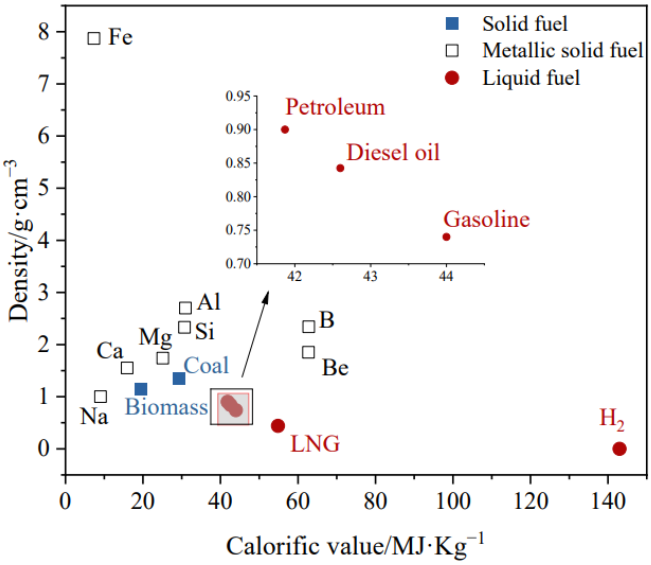


Figure 1: Energy density and gravimetric density comparison of different fuels. Figure adopted from [2].

The advantage of using aluminum as an energy carrier is it can be transported globally then reacted with water locally to produce hydrogen and heat. This eliminates the need for expensive storage and transportation of hydrogen.

1.3 Aluminum Water Reactions

Aluminum water reactions have been widely studied. When oxide-free form of aluminum reacts with water, the aluminum is oxidized, and the water is reduced. The reaction is exothermic and produces hydrogen, alumina and heat as seen in Table 1. Aluminum has an energy density of 31 MJ/kg, during oxidation approximately 15.5 MJ/kg of aluminum turns into heat and 15.5 MJ/kg of aluminum transfer into the reduced hydrogen [3]. Depending on the heat of reaction, as shown in fig. 1, the aluminum product is either aluminum hydroxide, aluminum (I) hydroxide or aluminum oxide.

Each product has a different process required to recycle back to aluminum. The Bayer process refines aluminum hydroxide into aluminum oxide and the Hall-Héroult process smelts aluminum from aluminum oxide [2]. The metal is not without its challenges. Aluminum requires specific conditions to be suitable fuel.

Temperature Ranges	Aluminum-Water Reaction Equation	Reaction Product Conversion
<200-300 °C	$2Al + 6H_2O \rightarrow 2Al(OH)_3 + 3H_2$	$Al(OH)_3 \rightarrow 2AlO(OH) + H_2O$
~200-570 °C	$2Al + 4H_2O \rightarrow 2AlO(OH) + 3H_2$	$2AlO(OH) \rightarrow Al_2O_3 + H_2O$
>570 °C	$2Al + 3H_2O \rightarrow Al_2O_3 + 3H_2$	$Al(OH)_3 \rightarrow Al_2O_3 + 3H_2O$

Table 1: The reaction products for aluminum water reaction at increasing temperature ranges. At greater temperatures, a greater fraction of hydrogen forms hydrogen gas instead of bonding to aluminum. Adopted from [2].

1.4 Aluminum Oxide Layer

Exposure to air forms an aluminum-oxide layer on the surface of the material, rendering the metal inert and unable to combust. In this paper, the aluminum in considered "activated" when the oxide layer is absent on the material. Methods for overcoming the oxide-layer have included disrupting the layer by mixing it in an indium-gallium solution [9], reacting at high temperatures to decompose the oxide layer, corroding the layer in an alkaline solution or pulverizing the aluminum via ball milling [2]. The existing techniques incur a substantial energy penalty and aren't viable for storing or transporting aluminum because the oxide-free aluminum would form an oxide-layer the moment it contacts air again. For long-term storage and transportation, it's desirable to have a method of protecting the oxide free aluminum so that it may remain reactive.

The solution proposed in Godart & Hart (2020) solves this issue by storing the aluminum in a noble gas, but the use of precious metals increases the cost. A low-cost solution is to use a water-soluble polymer to encapsulate the activated aluminum during transport. The layer will then dissolve during the reaction with water.

1.5 Scope of Work

The goal of this thesis is the development of a novel 3d printer to create aluminum-polymer structures that do not oxidize and can achieve a wide range of reaction rates with water by varying the structure surface area. A water-soluble polymer is used to coat activated aluminum, enabling the metal to be stored and transported without oxidation at a low cost. The unique manufacturing flexibility of 3d printers enables the fabrication of structures with wide ranges of surface area to volume ratios. It is desirable to control the reaction rate to achieve a target production rate of hydrogen. By varying the surface area exposed to water, the reaction rate can be predicted. Shipping activated aluminum in the polymer structures allows hydrogen to be produced locally and the need for expensive hydrogen transport can be eliminated. The qualitative requirements shown in Table 2 outline what the printer needs to achieve to be successful.

Name	Requirement
Thermals	Polymer solid at room temp, must have heater to melt
Material compatibility	Protect solution from degrading Not react with aluminum printing material
Syringe Pressure	Extrude solution sufficiently accounting for viscosity at various temperatures
Structure stability	Structure is stable and resistant to vibration
Component precision	Accurately produce target printed structures
Output quality	Final printed structures are stable and able to dissolve in water
Printer Size	Can't use more than 1g of Al nanoparticles (NPs) at a time and must fit on a lab bench
Vibrations	Structures must be stiff/supported so as not to vibrate and damage or loosen components during operation
Accessibility	Printing components must be easily accessible for general use and maintenance.

Table 2: List of requirements of printer. These are the primary functions for the final printer. Each design decision was made because of one of these requirements.

2. Printer design

To achieve the goal of rigid aluminum-polymer structures, we identified several key design requirements. The first is the thermal requirement for both the environment the printer lives in as well as the operating conditions for the heater. This will drive material and component selection of the heater assembly. Second, material compatibility of the syringe and printing bed must ensure the aluminum-printer structures don't react or degrade. Syringe pressure refers to the force from the motor on the syringe and into the polymer mixture. Too low and the syringe doesn't extrude, too high and excessive polymer is deposited. Structure stability and vibrations are related. Both are important to ensure final structure quality. If the printer structure is compliant, the nozzle will move during operation relative to the bed and the final dimensions will not be accurate. The final requirement is accessibility. When parts are replaced, or the syringe is exchanged, the accessibility of the assembly greatly contributes to the speed of maintenance. Figure 2 shows the assembled printer with the bed able to move in X and Y directions.

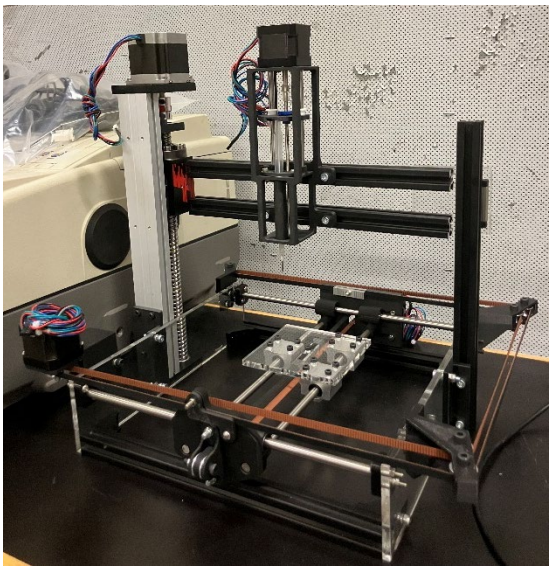


Figure 2: 3D printer fully assembled with all systems.

2.1 Design Overview and Process

The printer design consists of a 3 mL piston syringe pump with a 10cm x 10cm bed. The pump moves in the Z direction while the bed moves in both X and Y. A resistive heater attaches to the syringe to melt the polymer mixture for extrusion. The pump and bed axes are controlled by an external Arduino Uno R3 and CNC shield extension board. The heater is controlled by a temperature control board and thermal sensor.

The 3D printer developed in this work is intended to be used by other lab members for multiple years. A systematic design process was followed to ensure a high-quality device that is user friendly, requires minimal maintenance and meets user needs. The design process (see Figure 3) began with design requirements for the printer. High level sketches were done based on requirements to determine initial packaging, mechanism design and component selection. Initial sketches allow for quick ideation and elimination of options instead of spending design time modeling each idea. “Space fill” CAD was completed based on final sketch concepts to further specify the component sizing and interfaces. Space fill is a CAD practice referring to modeling the maximum space parts will take up in an assembly. Individual parts were then further refined and modeled.

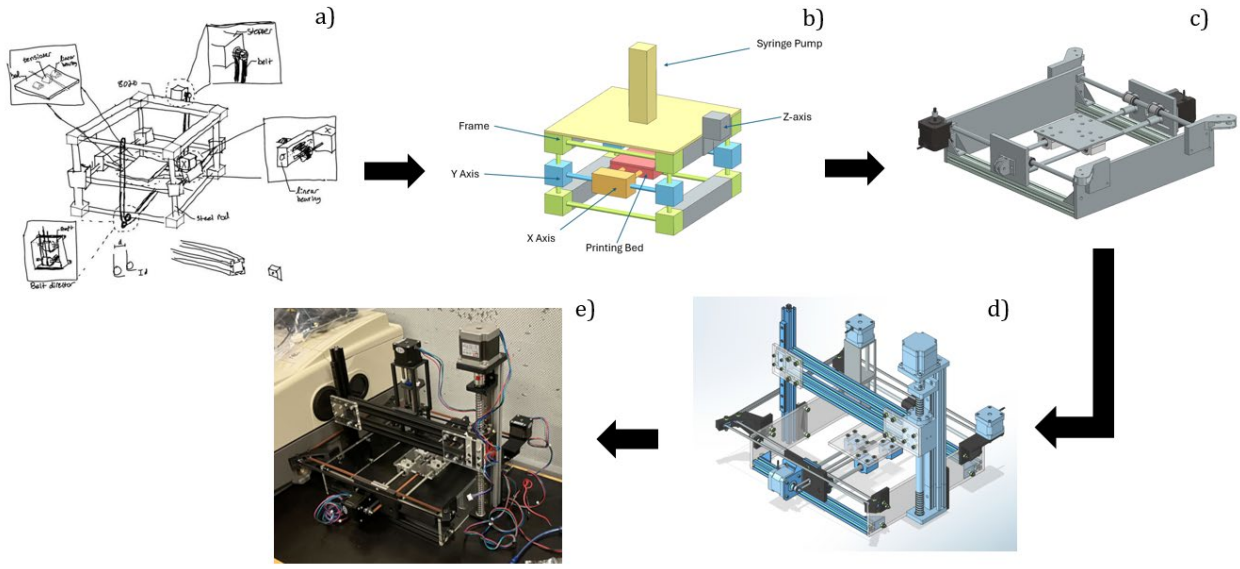


Figure 3: Design workflow of 3d printer from a) initial sketches, b) space fill CAD c) component selection, d) detailing and refinement, e) assembled printer.

2.2 Architecture Selection

There are many types of printing processes: fusion deposition modeling (FDM), stereolithography (SLA), powder bed fusion, binder jet, ink jet and concrete extrusion. Each one has a unique method to extrude, bind or cure the material. Many of these are commercially available. The challenge is that each requires pre-made base materials that are compatible with the printer. Additionally, commercially available printers for this application are expensive (>$1000) [7]. To accommodate the polymer aluminum mixture, it would have to be formed into a compatible material for commercially available printers, requiring addition process complexity. It was decided to design a custom printer to synthesize the polymer cell structures.

2.3 Material Selection

The polymer to encapsulate the aluminum was decided to be polyethylene glycol 1500 (PEG 1500). PEG is water soluble, so it will be able to dissolve during the aluminum-water reaction allowing the water to contact the aluminum particles. PEG 1500 has a melting point of 49 C. The melting point is crucial for manufacturing, storage and transportation. It must be solid at room temperature to react at room temperature. Additionally, if the melting point were too high, expensive manufacturing processes would be required to form it into the desired structures. The cost of PEG 1500 is ($40-60/kg) [8]. Compared to Indium (>$700/kg) [9] which has been used in aluminum reaction processes, PEG is relatively cheap making it a viable material when scaled.

2.4 Movement

Removing a lateral translation from the extruder is uncommon in commercial printers. Packaging two axes of movement on the bed is difficult to be designed compactly

because the bed is larger than the extruder and requires a greater footprint to move completely in two directions. If a 100 cm x 100 cm print area was required, the 100x100 bed needs twice that area to travel. Since the extruder is smaller, if it travels along one of the lateral axes, the required total footprint of the bed can be reduced. Two lateral axes of movement on the bed were necessary, however, to avoid excessive vibrations from the movement of the extruder in the X or Y direction.

2.5 Syringe Pump Extruder

The syringe pump was chosen for its flexibility in material and precision control. A syringe will extrude any mixture of material so long as it fits through the nozzle. Additionally, the structures being made for the proof of concept are not large (<1g of aluminum will be used for safety) and therefore it was determined <10mL of polymer would be required. A disadvantage of a syringe is the volume required to package it since the plunger needs twice its length to work. Another disadvantage is adjustability, the entire extruder including the pump and heater assemblies are designed around one size of syringe and need to replace the syringe after each use. A disadvantage of using a liquid medium for printing is a lagging effect from the fluid. When the syringe extrudes material, it builds up pressure in the syringe, forcing out the liquid. When the syringe stops, there remains pressure in the fluid, causing the fluid to be forced out at the end of a print, leaving behind excess material on the print bed. This error can be fixed in software by adjusting the syringe turn-off time before the bed stops moving.

2.6 Heater

PEG 1500 was selected as a material for its ability to dissolve in water and its melting temperature of 49 C. Since the polymer is solid at room temperature, a heater is required to liquify it for 3D printing. The primary requirement for the heater is to maintain the liquid polymer state while printing. A calculation was done to determine the heating power requirement. The melting temperature of PEG is 49 C and the decomposition temperature is 180 C [10]. Increasing the heater temperature above the melt temperature will decrease the time required to melt the polymer. Additionally, the entire syringe volume must be heated or local areas of the polymer will solidify due to thermal gradients, leading to clogging in the syringe. To decrease melt time, account for thermal gradients and prevent decomposition of PEG, 80 C was chosen as an initial heating temperature.

A 3 mL syringe was used in this printer and a heating temperature of 80 C was selected. To heat 3 mL of PEG 1500 to 80 C requires a 9.65 W heater at minimum (see Fig. A-1.1 in appendix for calculation).

The accessibility requirement influenced the design of the interface between the heater and the syringe. After each use, the syringe must be replaced. If the heater is attached directly to the syringe, it would be troublesome to replace the heater in addition to the syringe. To work around this, thermally conductive housing was chosen for the syringe. The heater attaches directly to the outer housing surface. Steel was chosen as the housing material due to its thermal conductivity and material availability. Because air is significantly less thermally conductive than steel, 0.026 W/(m-K) vs. 45 W/(m-K) respectively, it's important to minimize the air gap between the housing and the syringe to

maximize heat transfer from the heater [11][12]. To account for this, the inner diameter of the housing is than the outer diameter of the syringe. The disadvantage of this design is that it's designed around one size of syringe in both length and width. To change to a larger or smaller syringe would require a redesign of the heater and extrusion sub assembly.

A resistive heater was chosen for its size flexibility and large heating area. Resistive heaters use the joule effect by generating heat when current faces resistance in a wire. Commercial resistive heaters have a variety of sizes and powers densities to choose from. Another option considered was a thermal block. A heating element is installed inside the thermal block as well as the desired target to be heated. A custom heating block would need to be made to fit the syringe. Heating blocks have an advantage because they can be precisely designed to an application and are generally more efficient. The disadvantage is the complexity of designing the heating block and the additional space it would take up in the design.

The temperature controller is a crucial part of the heater design. To accurately melt the polymer in the syringe, it's necessary to integrate a feedback loop measuring the temperature of the syringe and changing the power of the heater accordingly to reach a target steady state temperature. At this stage in the heater design the only requirement is to melt the PEG for printing so control system parameters such as overshoot, settling time and rise time are not important to control. Future research will study the effect of the polymer temperature on cooling time. If the temperature requires more complex control schemes in the future to achieve optimal process control, the controller may be modified. For now, an off-the-shelf controller is used which incorporates an on/off controller based on a target temperature state point.

3. Manufacturing and Assembly

To achieve a low-cost device, all components on the printer are 3d printed, acrylic or off-the-shelf parts. Additionally, where it was possible, components were designed for easy accessibility and maintenance. All acrylic parts only had 2D geometry and were cut on a laser cutter. All 3D printed parts were made from PLA.

3.1 Structure

The structure of the printer is made using acrylic plate, 8020 bars and 3D printed components. The 8020 bars were bought to length as were the brackets mounting them to the acrylic plates. The Z axis structure is an assemblage of acrylic plate, and 8020 bars bolted together. The vertical bars are mounted to acrylic using 3d printed brackets.

No modeling of the structure was done in advance of the design. This led to compliance in the printing structure. The 1/4" acrylic plate flexes under horizontal loading. However, there was no requirement set on accuracy or precision so the impact of this has not been quantified, only compared to other printers on the market. The initial tests came out well, however if more precise designs are required, the acrylic plates will require additional members to add stiffness.

3.2 Carriage

The carriage is made from off-the-shelf steel rods and bearings, 3D printed parts and acrylic plate. When the bearings were first pressed in the 3D printed housing, the PLA cracked because it was too stiff. The housing was redesigned with additional compliance to prevent cracking. A similar issue arose on the clamps for the carriage rods. During assembly the rods are pressed into the clamps then bolted down. In the first iteration, the clamps cracked due to the PLA expanding too much. Additional clearance was given to prevent this issue in the next iteration.

4. Results

Initial experiments have validated the basic functions that the printer will perform. No aluminum particles were included in this phase of the testing so that the printing parameters could be dialed in without wasting material. Only PEG 1500 was used. A series of line tests were conducted on glass slides over a length of 1.8”/45.8 mm to observe the performance of the printer. The following was performed before testing

1. The PEG was bought in solid form and liquified on a hot plate at 100 C.
2. PEG was extracted into a syringe and solidified.
3. The syringe filled with PEG was mounted onto the extruder.
4. Heater temperature was set, and 5 minutes elapsed before printing to ensure all PEG was melted in syringe.
5. The following print parameters were set in code: length of print, bed speed and extrusion speed.
6. The bed was manually moved in X, Y and Z directions to the starting location of line.
7. Code was uploaded to controller (see appendix Fig. A-1.2 for detailed upload procedure).
8. Print began.
9. Steps 5-8 were repeated if multiple trials occurred.

4.1 PEG Melt Test

For 3D printing, the PEG melting temperature affects many printing parameters: extruder warmup time, extruder temperature, extruder operating conditions and PEG cooling time. The resistive heater does not equally heat the full volume of the PEG in the syringe, leading to thermal gradients. In the case of the heater temperature being exactly the melting temperature of PEG, if there are any thermal gradients then there must be PEG in the syringe that remain solidified. Solid PEG leads to clogging in the syringe nozzle. Clogging in the syringe is a high concern due to the nozzle not being heated (shown in Figure 4) further increasing the thermal gradient and required heater temperature. The PEG in the nozzle is designed to be melted by conduction through the bulk PEG in the syringe.

The initial heater temperature was 80 C as discussed in section 2.6. There were no issues with clogging when a new syringe was being used. After three printing trials, the

syringe was left sitting at 80 C. While sitting, the PEG in the both the 0.049” syringe nozzle and the primary syringe nozzle solidified, impeding printing function.

The heater temperature increased to 100 C. As seen with the 80 C trial, there were no issues clogging with a new nozzle. When the syringe was left to sit at 100 C, the PEG in the 0.049” nozzle solidified, but the PEG in the primary nozzle remained melted. This performance is acceptable because 0.049” nozzles are easy to replace during printing operation, but the primary nozzle cannot. A heating temperature of 100 C is ideal without additional heating on the nozzle.

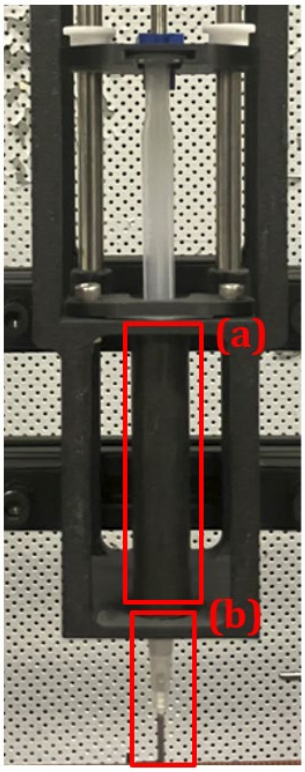


Figure 4: a) Main body of syringe where heater attaches. b) 0.049” syringe nozzle that is not directly heated.

4.2 Printing Test

The primary performance of the printer is to form structures. The quality of the print is dependent on multiple parameters: print speed, nozzle diameter, nozzle height, cooling time, material temperature. The impact of nozzle diameter and height on print quality was qualitatively measured to understand initial printing performance.

An initial test was conducted using the primary nozzle of the syringe with diameter 0.089”/2.26 mm. It was seen that additional polymer bled out at the end of the printing line due to residual pressure in the syringe (see Figure 5). The extrusion was also delayed from

the bed because the pressure took time to build up in the syringe. Initial results did not have qualitatively constant line thickness or height.

Figure 5: a) Initial test of printing capabilities. b) Trial to measure printer's ability to extrude a straight line.

Two nozzle diameters were used to determine the effect of nozzle size on line thickness and printing precision. The results are showing in Figure 6. Though not measured, the lines from the 0.049" nozzle were consistently thinner than those from the wider 0.089" nozzle. Further testing is required to determine the minimum nozzle size for PEG. This will drive the precision achievable by the printer.

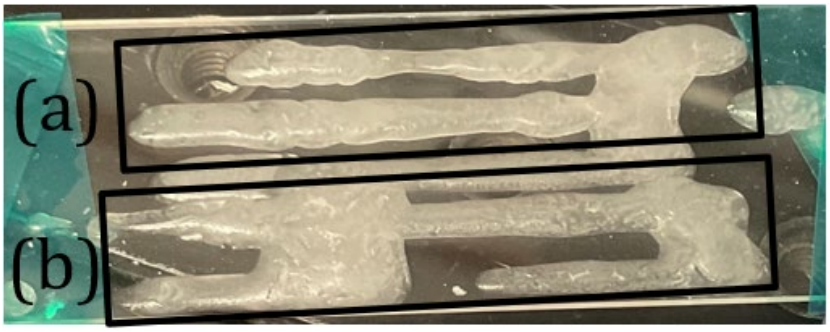


Figure 6: a) Line test using the syringe with no tip. Nozzle diameter of 0.089"/2.26 mm. b) Line print test using a nozzle size of .049"/1.25 mm. The overlap between lines is where the line finished printing and additional PEG was extruded due to residual fluid pressure in the syringe.

The nozzle height began at 0.039"/1 mm off the bed and decreased to 0.020"/0.5 mm. The results are shown in Figure 7. An even layer height is desired for layer adhesion and part accuracy. The 0.020"/0.5 mm layer height had the most consistent layer height. Further testing is required to determine the ideal nozzle height for different diameter nozzles.

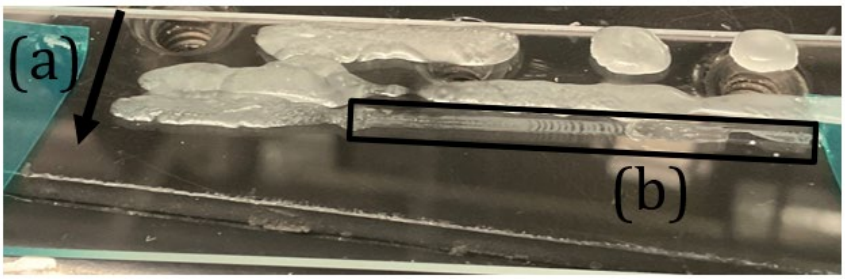


Figure 7: Tests in image were conducted using 0.049”/1.25 mm nozzle diameter. a) Direction of decreasing Z height. b) Located when syringe ran out of PEG. The residual marks show the real dimension of the nozzle, which is thinner when compared to the line tests demonstrating how much the PEG expands when extruded.

5. Conclusion

The 3d printer developed in this work enables the production of aluminum polymer structures to lower costs of hydrogen transportation. The printer is novel by designing a syringe pump and heating system for use in additive manufacturing. Additionally, this printer reduces the vibrations on the syringe pump by designing the bed to move in both lateral directions. Challenges came up with nozzle clogging, delay between bed movement and syringe extrusion. Increasing heater temperature solved the nozzle clogging and further tuning in the printing software will occur to eliminate the extrusion delay. An initial nozzle height of 0.020”/0.5 mm and nozzle diameter of 0.049”/1.25 mm was determined to for best print quality. Further testing is required to validate the inclusion of aluminum in the polymer printed structures. Additionally, future research will analyze the viability of using an aluminum for transportation at a system level.

Only initial testing and tuning of printer performance with PEG was completed in this work. Further experimentation is required to tune nozzle diameter, nozzle height, print speed, layer adhesion and syringe extrusion delay.

When looking at the viability of an energy process on the global scale, it’s important to understand the whole system. To understand if all the contributing energy and financial costs. The Levelized Cost of Energy, or LCOE, is a crucial metric for comparing different energy sources and carriers. This value refers to the total lifecycle of the product, from mining to processing, to the consumer and through any recycling processes. If the LCOE of a product is too high, it won’t be competitive on the global market. This is a major challenge for hydrogen right now. There have been few studies looking into a system view of aluminum as an energy carrier but due to its limited technology readiness, many assumptions had to be made about the required financial costs [13]. A full techno-economic assessment of aluminum as an energy carrier is future work for this research once the basic process is understood and scaling has started.

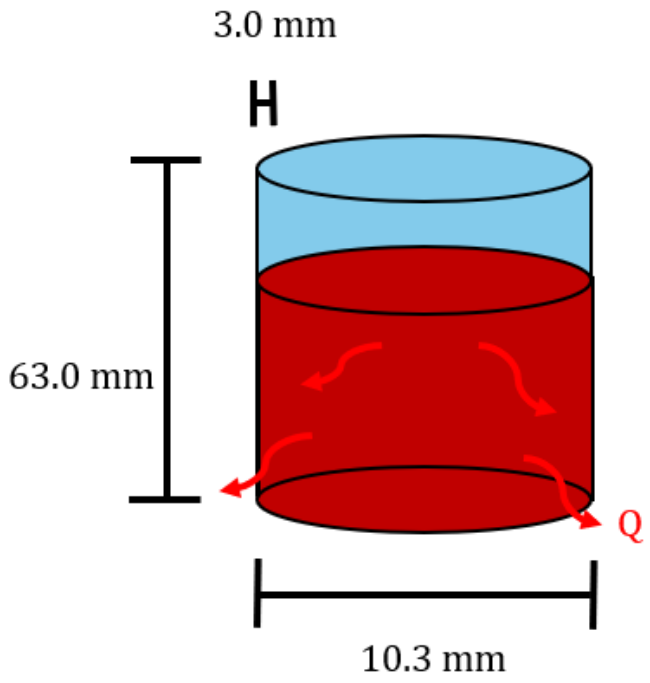
The proposed process doesn’t have a method to activate the aluminum before coating. This additional step incurs an additional energy cost which must be considered to determine viability of the whole energy carrier cycle. Further research into the cheapest

method for this is required to build an entire sustainable system and take advantage of the energy and cost savings proposed by using the water-soluble polymer coating.

To form a complete cycle, the aluminum oxide produced from the water reaction will be recycled to turn it back into aluminum. The Hall–Héroult process is a well-established method to smelt aluminum from alumina using electrolysis. The energy penalty from this process must also be considered when calculating system efficiencies. The process additionally uses carbon to reduce the aluminum oxide which produces carbon dioxide. Further research must be carried out to determine the sustainability of the aluminum recycling system.

Appendix

Figure. A-1.1: Thermal calculations for sizing the syringe heater. The model assumed the syringe was heated to steady state then calculated the heat dissipation and therefore the heating power required to keep the PEG in a liquid form.



Syringe properties:

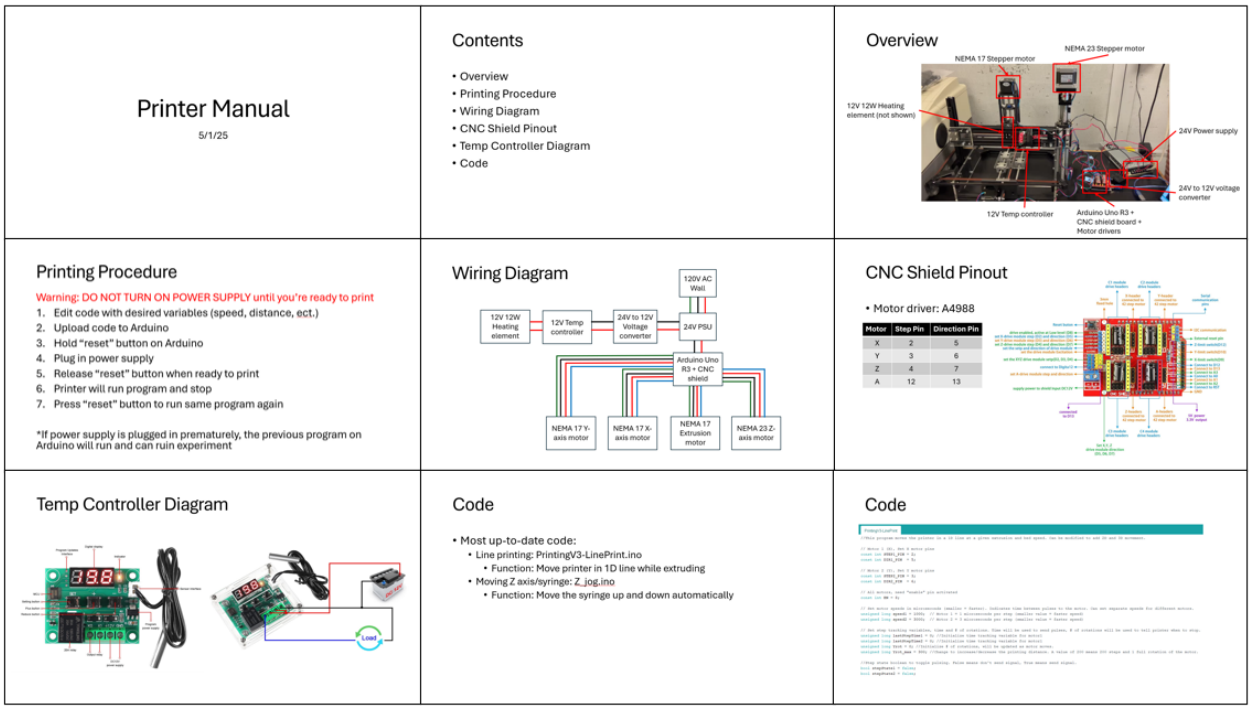
$$Material = PET\ Plastic$$
$$K_{PET} = 0.29 \frac{W}{m * K}$$
$$T_{amb} = 30\ C$$
$$T_{syringe} = 80\ C$$
$$t_{syringe} = 3\ mm$$

$$\dot{Q} = \frac{K_{mat} * A * \Delta T}{t} \quad (1)$$

$$\dot{Q} = \frac{0.29 \frac{W}{m*k} * 0.08m * \pi * 0.063m * 50K}{0.003m} \quad (2)$$

$$\dot{Q} = 9.85W \quad (3)$$

Figure. A-1.2: The designed printer was made for use in lab by multiple users and flexible applications. A printing manual was created to easily learn how to run the printer.



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