FORMULATION OF A REACTIVE MATERIAL PASTE FOR ADDITIVE MANUFACTURING

By

Kelsay Neely

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Approved:

Alvin Strauss, Ph.D.

Kevin Galloway, Ph.D.

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CHAPTER I

INTRODUCTION

The term "reactive materials" describes a class of materials that combines two or more nonexplosive solids that release high quantities of chemical and kinetic energies upon ignition [1]. In this document, the reactive material discussed is thermite. The thermite reaction is a reduction-oxidation reaction, characterized by a high reaction temperature, low gas production, and production of molten slag. The reaction supplies its own source of oxygen and, therefore, does not require exposure to air. Once the mixture is ignited, it will burn until all fuel is used, even underwater. This characteristic allows it to be used in underwater and space applications.

$$Fe_2O_3 + 2Al \rightarrow 2Fe + Al_2O_3$$

A common example of a thermite reaction (seen above) involves iron oxide and elemental aluminum. The reaction releases a large quantity of heat and forms aluminum oxide and elemental iron. This specific reaction requires ignition temperatures of approximately 1650°C and reaches burning temperatures of over 2200°C. This is consistent with the characteristic properties of thermite reactions. Most thermite formulations are insensitive to the effects of corrosion, friction, spark, shock, contaminants, moisture, and variations in composition. Additionally, thermites have insensitive ignition properties, making them safer than many combustible mixtures [7]. Table 1 shows some common thermite mixtures, but there are many thermite reactions that can be tailored to various engineering applications.

Reactants		Adiabatic reaction Temperature (K)		State of products		Gas Production		Heat of Reaction	
Constituents	ρ_{tmd}, g/cm^3	w/o phase changes	w/phase changes	state of oxide	state of metal	moles gas per 100 g	g of gas per g	-Q, cal/g	-Q, cal/cm^3
2Al + Fe_2O_3	4.175	4382	3135	liquid	l-g	0.1404	0.0784	945.4	3947
8Al + $3Fe_3O_4$	4.264	4057	3135	liquid	l-g	0.0549	0.0307	878.8	3747
2Al + 3CuO	5.109	5718	2843	liquid	l-g	0.54	0.3431	974.1	4976
2Al + $3Cu_2O$	5.280	4132	2843	liquid	l-g	0.1221	0.0776	545.5	3039

Table 1 – Selected Thermite Reactions and their Properties [7]

Table 1, which contains only metallic thermite reactions, shows the constituents of the reaction and their theoretical maximum density. It also shows the maximum adiabatic reaction temperature, both with and without phase changes. The four listed reactions all contain aluminum as the oxidizing agent, with either an iron oxide or a copper oxide. These four were selected for their favorable materials properties. Aluminum forms a passivation layer, which allows for safe handling. Its low melting point and high boiling point lets the reaction propagate quickly and at high temperatures. Additionally, the formed aluminum oxide is low density, which causes the slag to float on the generated molten metal.

Constructive Applications

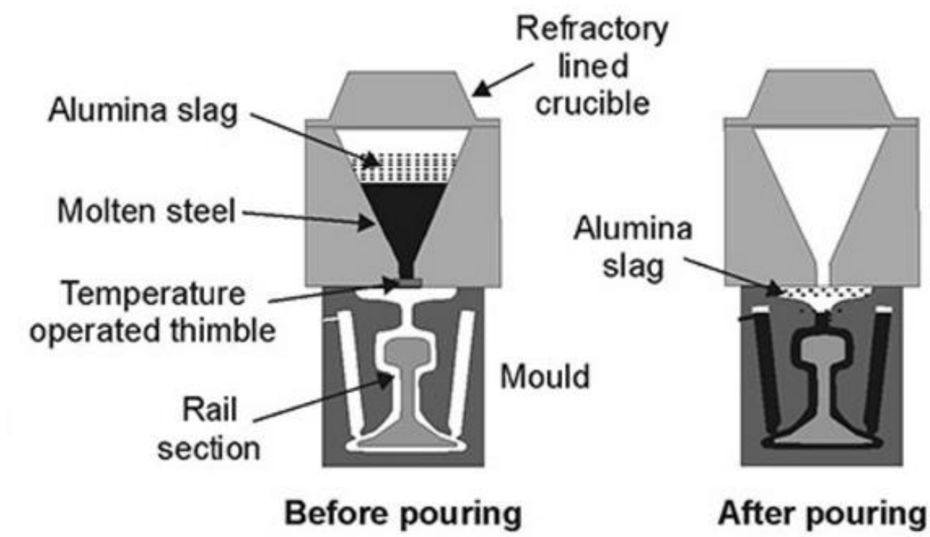

Figure 1 – Thermite Welding Diagram [15]

One engineering application is thermite welding, a metal joining process that uses the thermite reaction to join metals. It was first invented by Hans Goldschmidt in 1901, after his discovery of the thermite reaction [8]. The welding process uses simple forms to direct the reaction as depicted Figure 1. Bulk thermite powder is placed in a crucible above the joint to be welded. When thermite is ignited, and aluminum oxide slag floats to the top of the molten steel. The bottom of the crucible is then opened, allowing molten steel to flow around the rail section with minimal slag impurities. The metal then cools around the joint and the mold is removed.

Figure 2 – Thermite Welding on Railroad Tie [5]

Figure 3 – Completed Thermite Weld [3]

Figures 2 and 3 depict the thermite welding of a rail tie, with Figure 2 depicting the actual in-field process. Since the welding process is metallurgically similar to casting, many of the limitations of casting apply. The resulting joint has poor ductility, impact toughness, and possibly significant porosity issues, however, most of these problems can be mitigated by appropriate welding preparation conditions. Mold packing quality, air moisture, cooling time, rail gap, and rail movement during solidification also affect the quality of the weld. The most common use of rail thermite welding in industry today, is for defect repair. It is estimated that 42% of the 27,000 thermite welds made in 1996 were used to repair weld defects [12].

Thermite welding is also used to join large-diameter braided copper cables. Since the thermite formulation contains a copper oxide, the resulting joint is corrosion free and has a high current capacity. The cable welding process is nearly identical to the process used in rail welding, as seen in Figure 4.

Figure 4 –Thermite Weld of Copper Cables [6]

Destructive Applications

In addition to constructive uses, thermite has been used extensively for destructive applications. In 1980, a patent was issued for a thermite penetrator device. Thermite was placed in a conical crucible and then ignited. The flow of molten iron is controlled by the shape of the crucible, allowing for deep penetration of metallic targets [14]. This patent led to many advances in thermite incendiary devices, resulting in single- and multi-core burning incendiary devices[17][18].

The form of thermite now used by the United States Armed Forces is thermate. The primary reaction in thermate is still the aluminothermic reaction between powdered aluminum and a metal oxide. The composition by weight of Thermate-TH3 for military use is 68.7% thermite, 29.0% barium nitrate, 2.0% sulfur, and 0.3% binder. The addition of sulfur and barium nitrate increases the thermal effect, creates a burning flame, and significantly reduces the ignition temperature [21]. An example of thermate use is the AN-M14 TH3 incendiary hand grenade, as seen in Figure 5.

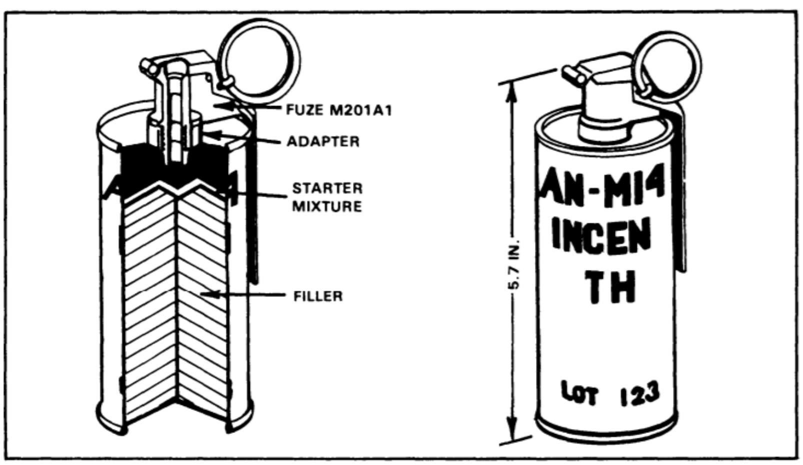

Figure 5 – Diagram of AN-M14 TH3 Incendiary Hand Grenade [21]

This incendiary contains approximately 26.5 ounces of thermate filler. The filler burns for 40 seconds and can burn through a 0.5 inch steel plate. Like all thermite mixtures, it produces its own oxygen and burns underwater. It will also fuse together any metallic parts that make contact [21].

Thermite is currently being used for a variety of constructive and destructive purposes, but uses are currently limited by the powder form of thermite. A powder-thermite reaction can be controlled only by changes in chemical composition, and containment, whether in the form of crucibles or canisters, is needed to focus the reaction. If the thermite could be formed into specific architectures, the reaction could be controlled by architecture rather than by compositional changes. A thermite paste, in conjunction with current additive manufacturing technology, would allow for reactive architectures that can be printed on surfaces and in structural materials, resulting in a more controllable form for both destructive and constructive applications.

CHAPTER II

LITERATURE REVIEW

Researchers at Lawrence Livermore National Lab (LLNL) have recently generated thin film reactive material architectures using a multi-step electrophoresis process. First, silver nanoparticle ink traces are printed into precise shapes. The shapes are annealed so that they become electrically conductive, and an aluminum/copper oxide thermite film is then deposited onto the surface using electrophoretic deposition. These films were classified as thin (26 μm) or thick (155 μm). Two different reactive geometries were explored, channels and hurdles, and were successfully tuned to control reaction propagation velocity[19]. However, the process itself is confined to the laboratory due the advanced equipment needed for electrophoretic deposition. Additionally, the scale of the experiment makes the method impractical for larger scale uses.

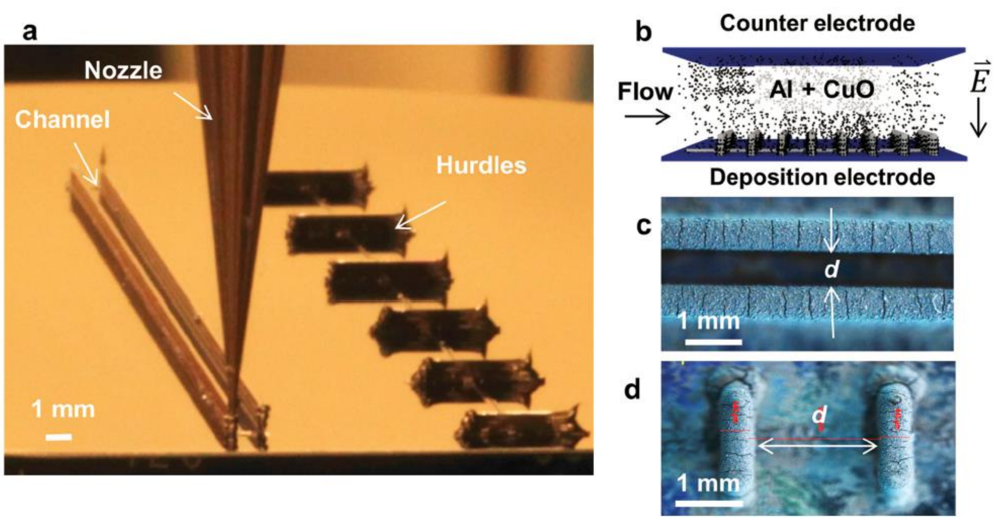

Figure 6 a) Optical image of 3D printing process for channels (left) and hurdles (right) composed of silver nanoparticle ink. The printed structures are thermally annealed to obtain conductive electrodes. B) Schematic illustration of the EPD process that is used to deposit composite Al/CuO mixtures onto the electrode surfaces. C) Optical images (top view) of a channel and d) hurdle architectures after deposition of the Al/CuO films with spacing, *d*, between the electrodes. [19]

Researchers at Los Alamos National Lab are also tuning explosives using additive manufacturing, specifically by controlling the voids present in the explosive mixture. Each layer of the material is configured to have a set number of voids arranged in an optimal hot-spot profile[10]. This allows the material to behave and ignite in a safer and more predictable manner. Current research has not moved beyond single material mixtures with air voids, but Los Alamos has the capability to work with mixed explosive materials to create more novel results.

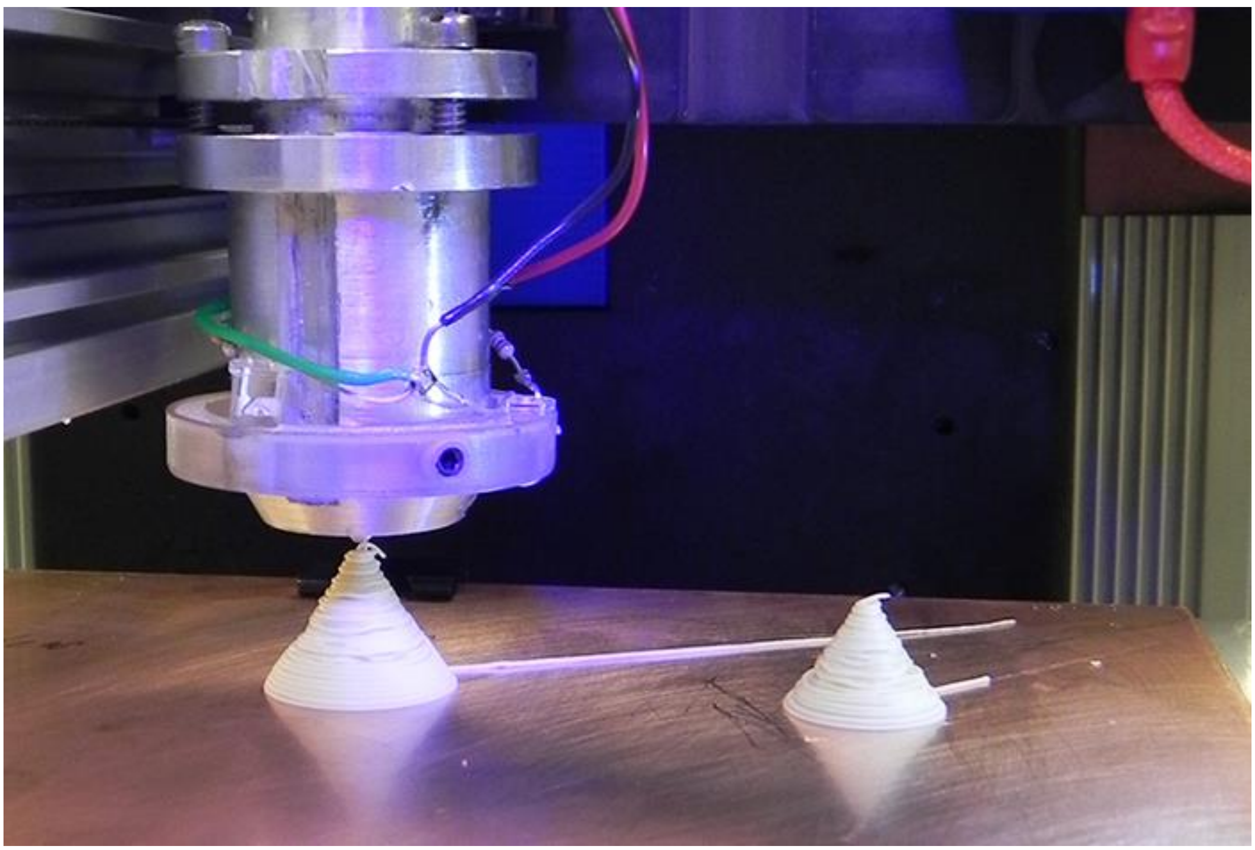

Figure 7– Los Alamos 3D Printed Explosive Cones [13]

There has also been success in depositing thermite on existing architecture using a process called cold gas dynamic spray (cold spray). During the cold spray process, solid powders are accelerated through a de Laval nozzle toward a substrate. If the impact velocity of the solids exceeds a certain threshold, plastic deformation of the particles occurs and the particles then stick to the surface. This process can create coatings up to several millimeters in thickness [16]. A diagram of the cold spraying process can be seen in Figure 8.

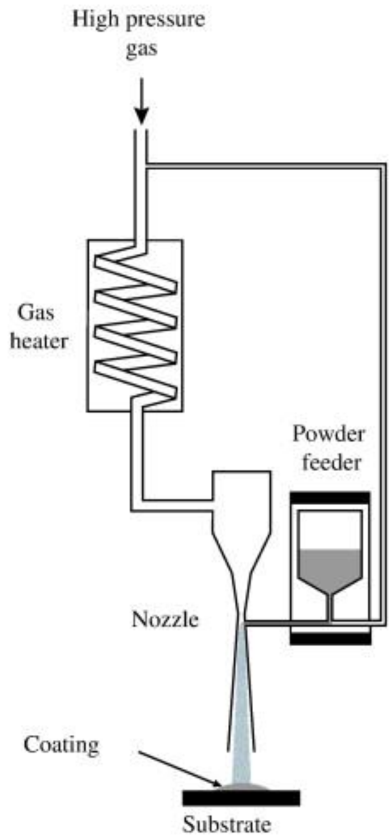

Figure 8 – Cold Spray Process Diagram [2]

An AL-CuO thermite mixture was successfully cold sprayed onto a substrate with a resulting thermite thickness of 10mm. The coatings approached 50% of the theoretical maximum density of thermite. This resulted in flame speeds approximately one order of magnitude lower than the flame speed of loose powder thermite [2].

Prior work has also been done in the realm of creating "cast" thermite, or thermite with an epoxy binder, but those efforts have been focused on structural properties and shear-induced ignition[4][20]. While suspending thermite into an ignitable, structural binder is a key goal, these molds are hard to ignite and not optimized for additive manufacturing. The mixing process of thermite into the epoxy binder is time consuming and requires additional specialized equipment.

Thermite geometries and architectures have been created at several different scales. Lawrence Livermore and Los Alamos national lab have generated reactive material architectures at the micro-scale, and work has been done on thin coatings at a large scale [19][10][2]. A void exists at the meso-scale, which is what this thesis addresses.

CHAPTER III

THERMITE PASTE DEVELOPMENT

For this work, we explored paste formulations composed of aluminum and ferric oxide (iron (III) oxide or Fe_2O_3). This formulation was selected due to its relative safety (due to its high ignition temperature) and commercially available components. Ferric oxide and aluminum was obtained in a powdered form from Alpha Chemicals, with a particle size of 30 µm (mesh 500).

Binder Selection

In order to formulate a viable paste, a number of different binders and thermite concentrations were tested. Four commercially available binding materials were selected. These materials are not typical pyrotechnic binders, so they were not selected to add fuel to the system. Rather, they were selected due to availability, viscosity, cure time, toxicity, and ability to ignite.

This first round of experiments all contained a stoichiometric ratio of aluminum to iron oxide, or an aluminum to iron oxide mass ratio of 2.7:8. The following table summarizes the first mixtures and their compositions.

	Binder	Thermite Mass	Binder Mass	Ratio of thermite to binder
1	Marine Build Epoxy	10g	10g	1:1
2	Marine Build Epoxy	5g	3g	5:3
3	Marine Build Epoxy	12g	6g	2:1
4	Wood Filler	10g	5g	2:1
5	DAP Patching Plaster	20g	10g	2:1

Table 2 – Binder Selection Summary

The mass of mixed thermite in this series was kept under 30g for safety. The amount of thermite mixed into each binder was primarily dictated by the saturation level of the binder. If more thermite could be easily mixed in, more thermite was added. None of these mixtures ignited, even if significant amounts of thermite powder was spread on top. Samples 1-3 were particularly resistant to ignition. Sample 1 was also placed directly under the flame of a butane torch for approximately two minutes, resulting in no ignition. For samples 2-3, thermite powder was placed on top, completely covering the paste sample, but successful ignition of the powder still did not result in successful ignition of the paste sample. These temperatures are far greater than the temperatures needed to ignite thermite, and in some cases even greater than the temperature produced by thermite. These mixtures, if even ignitable at all, would most likely not generate enough heat to propagate their own reaction and were therefore discarded. A successful thermite paste mixture was eventually formulated, and its properties are covered in the rest of this document.

Prior attempts to suspend thermite in a binder were likely hampered due to the complex chemical nature of commercially available binding pastes. Therefore, a binder was needed that was simple and, ideally, part of an existing thermite reaction. Aluminum paired with either calcium or sulfur is a thermitic reaction, so calcium sulfate was selected as a potential binder. Calcium sulfate is commercially available as calcium hemihydrate (gypsum plaster), which forms a moldable paste upon hydration. A ratio of $3Fe_2O_3$:2Al:$2CaSO_4$ was created. The first successful thermite paste ignition took the form of a thermite "puck" of 2 inches in diameter and approximately 0.75 inches in height.

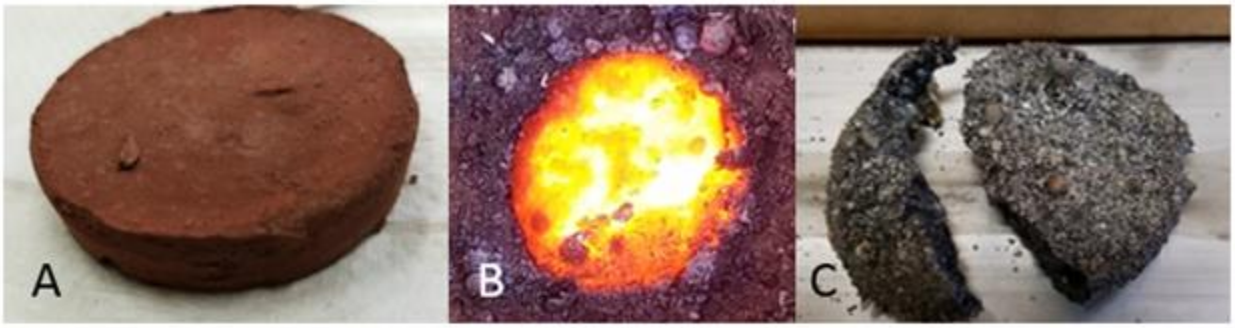

Figure 9 – A) Thermite with a plaster binder molded into a 2 inch diameter disk. B) Glowing slag immediately after the exothermic reaction of the thermite puck. C) Resulting slag and elemental iron

This mixture fully ignited, with the slag mixture glowing hot for minutes after the completion of the reaction. The resulting slag, as seen in Figure 9C, is slightly ferromagnetic due to the presence of elemental iron, and produces a sulfurous smell due to the presence of sulfur in the plaster binder. This reaction proceeded fairly quickly, but significantly slower than an equivalent mass of powdered thermite. The slag is also notably porous, which can be seen in Figure 10. These pores are likely caused by the energetic nature of the reaction and expanding gases.

Figure 10 – Slag Porosity

After the success of this initial sample, several studies were undertaken regarding the effects of cure time on completeness of reaction. A minimum of 24 hours at 22°C is needed to obtain a complete reaction of paste. If only partial curing is achieved, only a partial reaction is achieved. This is shown in Figure 11.

Figure 11 – Partial Ignition of Thermite Sample

This sample was formed in a trapezoidal mold and allowed to cure for approximately 4 hours. The ignition was initiated at the leftmost edge of the sample. The reaction then proceeded slowly and only propagated partially before stopping. This partial reaction is likely due to the excess moisture inhibiting the reaction. Despite the partial reaction, the slag still shows similar characteristics to the slag of the completely cured samples.

Plaster Content

While the initial successful ratio required equal parts aluminum powder and plaster powder, studies were undertaken to determine the minimum amount of plaster needed to form a structurally sound mixture. The following table shows the mixtures.

	Mass of iron oxide	Mass of aluminum	Mass of Plaster	Volume of water added	Mass ratio of dry components (Fe_2O_3:Al:$CaSO_4$)
1	21g	14g	14g	15ml	3:2:2
2	21g	14g	12g	15ml	3:2:1.7
3	21g	14g	10g	15ml	3:2:1.4
4	21g	14g	7g	15ml	3:2:1
5	21g	14g	4g	15ml	3:2:0.6

Table 3 – Plaster Composition Study Summary

All compositions resulted in a curing paste. All compositions ignited. However, sample 5 did not retain structural integrity when being removed from its mold. It flaked and crumbled significantly during removal. The ratio with the lowest usable composition is 3:2:1, but more investigation is necessary to fully determine its structural capabilities. A smaller amount of plaster is not recommended.

Work Time

The selected brand of gypsum plaster has a very short work life. The typical workable life is 6-10 minutes, with a setting time of approximately 30 minutes. Full cure will occur after 24 hours at room temperature. Work life can be extended by stirring the mixture less, using cold water, and by using more water. However, this does not extend work life significantly. A plaster retardant is needed to extend the work life for use in additive manufacturing. Tartaric acid can be a successful plaster retardant for gypsum plaster [11]. Tartaric acid was dissolved into water prior to mixing, and accounted for less than 0.1% (by weight) of the mixture. This significantly extended the work life. To quantify this effect, two mixtures of plaster were created. Each mixture had 40 grams of plaster and 15 ml of water. One mixture had tartaric acid dissolved into the water prior to mixing, and the other did not. Both mixtures were allowed to cure in the same room, and the work ability was tested. At the beginning, both mixtures had similar consistency, which can be visually approximated in Figure 12.

Figure 12 – A) Plaster without tartaric acid and B) Plaster with tartaric acid

The samples were checked every 5 minutes for changes in consistency. The samples processed through similar stages of consistency, but at different timescales. After 30 minutes, the sample with tartaric acid was still workable, while the sample without tartaric acid was fully solid. The sample without tartaric acid was able to be suspended upside down. The differences in consistency can be seen in Figure 13.

Figure 13 – A) Sample without tartaric acid, suspended upside down. B) Sample with tartaric acid, still in workable stage

Figure 13A shows the mostly cured sample. The stirring stick could not be easily removed. Figure 13B shows the sample with tartaric acid, which was still mostly workable and could still be stirred with minimal effort. This is a clear success in extending cure time.

Molding

After the formulation of a working paste, ignition and ignition results were formulated. Two silicone molds were created to mold the thermite paste into a regular brick. The resulting thermite bricks were 5 x 0.75 x 0.75 inches and 5 x 0.75 x 0.375 inches. The molds can be seen below in Figure 14, and an example of the thermite brick can be seen in Figure 15

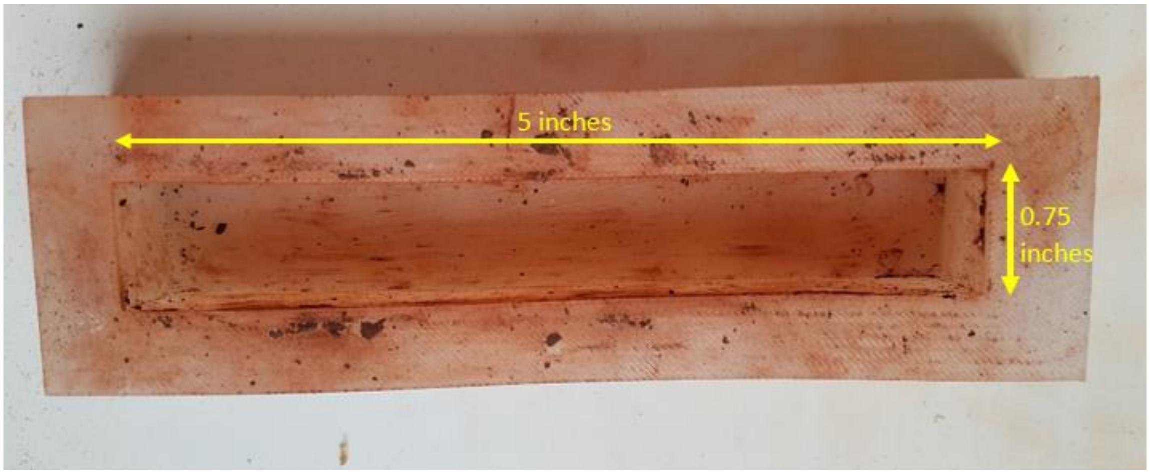

Figure 14 – Silicone Thermite Mold

Figure 15 – Standard Thermite Brick

Porosity

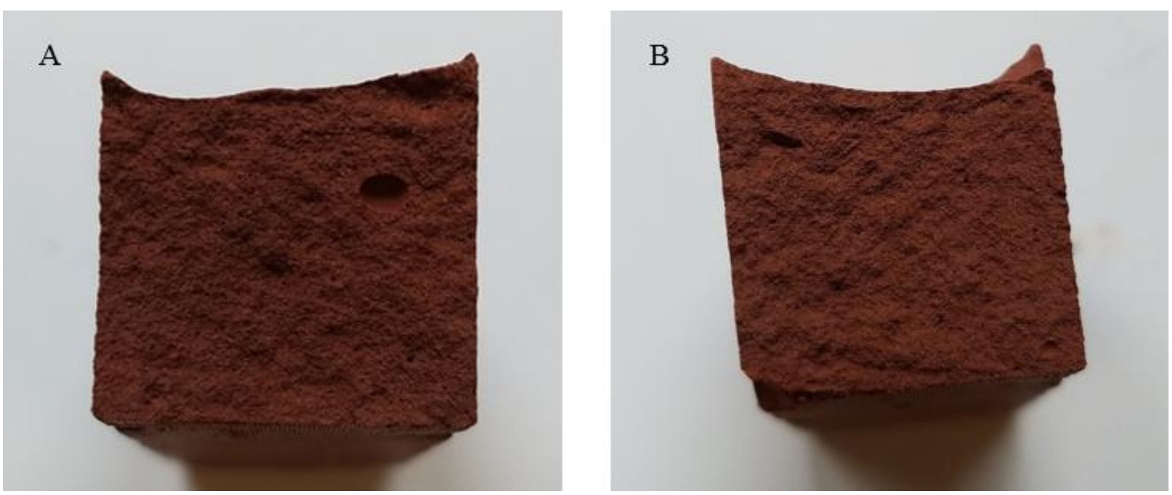

Figure 16 – A) Thermite cross section showing minimal porosity. B) Corresponding cross section

Porosity, if significant, will alter the rate of propagation. If porosity is significant enough, the reaction will likely not propagate completely. Figure 16 shows the cross section of a thermite brick. There are only two significant instances of porosity in this cross section. However, compared to the bulk cross section of the material, this porosity is insignificant. It is likely that any 3D printed structures will have similar porosity characteristics, which indicates that any 3D printed thermite will not be greatly affected by porosity.

Ignition

Figure 17 – Thermite Ignition Setup

Figure 17 shows the standardized ignition setup. A magnesium ribbon “fuse” leads into a pile of thermite and magnesium powder. The thermite is a high yield thermite sold by United Nuclear, not a custom mixture. To ignite the sample, the magnesium ribbon is ignited by a butane blowtorch. The magnesium ribbon is allowed to burn into the powder bed, which then ignites the thermite brick. This is a remarkably consistent ignition method. The temperatures obtained by a magnesium ribbon alone should be enough to ignite the thermite, but this does not result in consistent ignition. The magnesium ribbon does not even consistently ignite powdered thermite, failing approximately two out of every three times. This ignition combination consistently ignites thermite paste, and has never failed during many trials.

CHAPTER IV

IGNITION RESULTS

In order to determine a magnitude approximation of heat generated, several samples were ignited on thin aluminum sheets. All sheets are 0.0875 inches in thickness and of an unspecified aluminum alloy. Only particularly notable effects will be discussed here.

Figure 18 – A) Top side of ignition and slag. B) – Back side of ignition, with notable deformation

The ignition shown in Figure 18 contained approximately 50 grams of thermite and was in the 5 inch brick configuration. Standard ignition protocols were followed, and the overall reaction time was consistent with that size sample. The reaction melted the aluminum directly under the thermite brick, which can be seen clearly in Figure 18A. This is the only sample that resulted in a melted base plate.

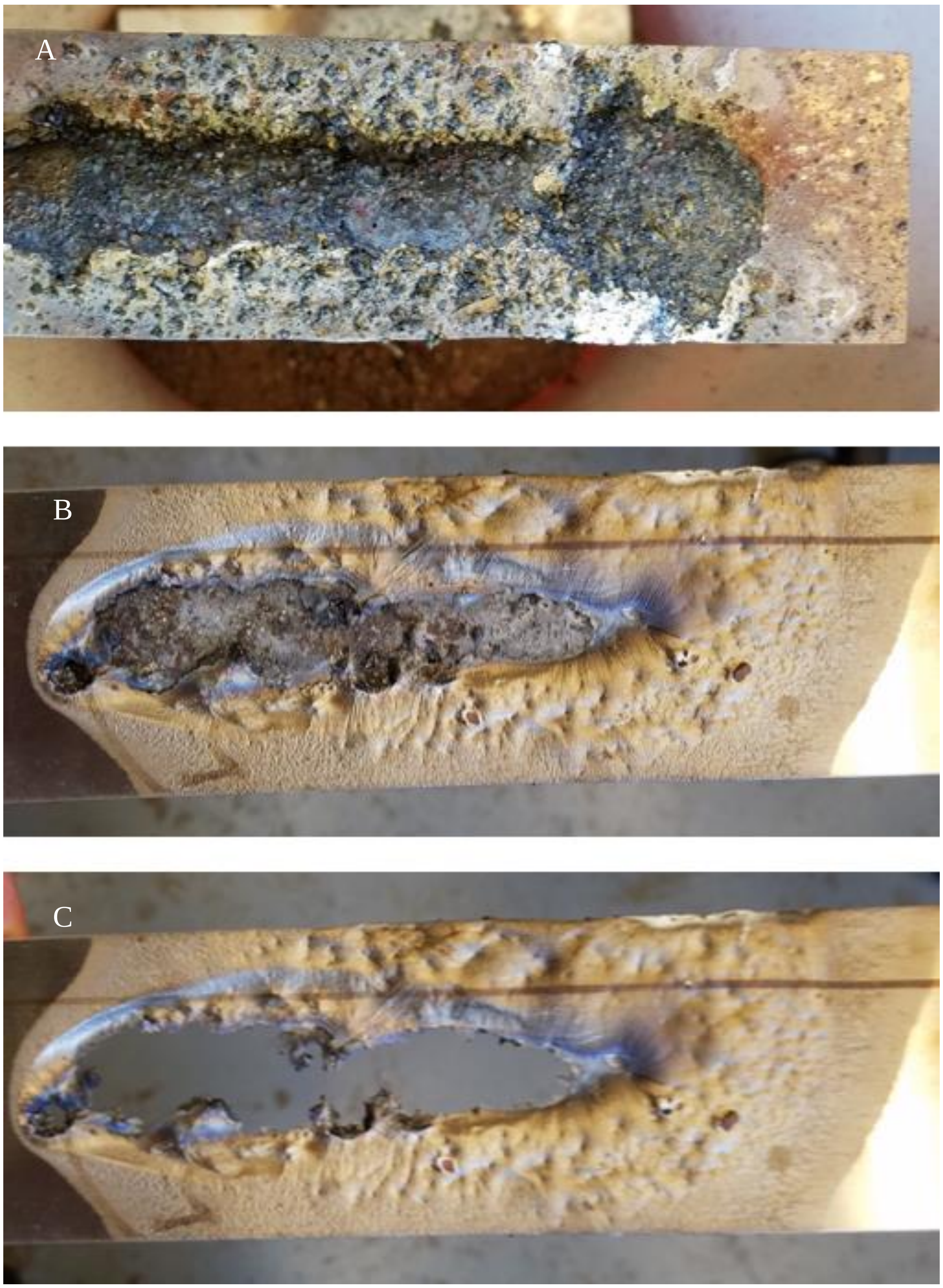

Figure 19 – A) Top side of ignition 2, with slag. B) Bottom side of ignition 2, with slag. C) Slag removed, revealing hole

Figure 19 shows a sample of thermite that burned through the aluminum test sheet. The sample was 5 inches long and weighed 52.2 grams. Figure 19B shows the hole, but the thermite is still fused to the opposite side. The slag was then gently pressed off, revealing the entirety of the hole.

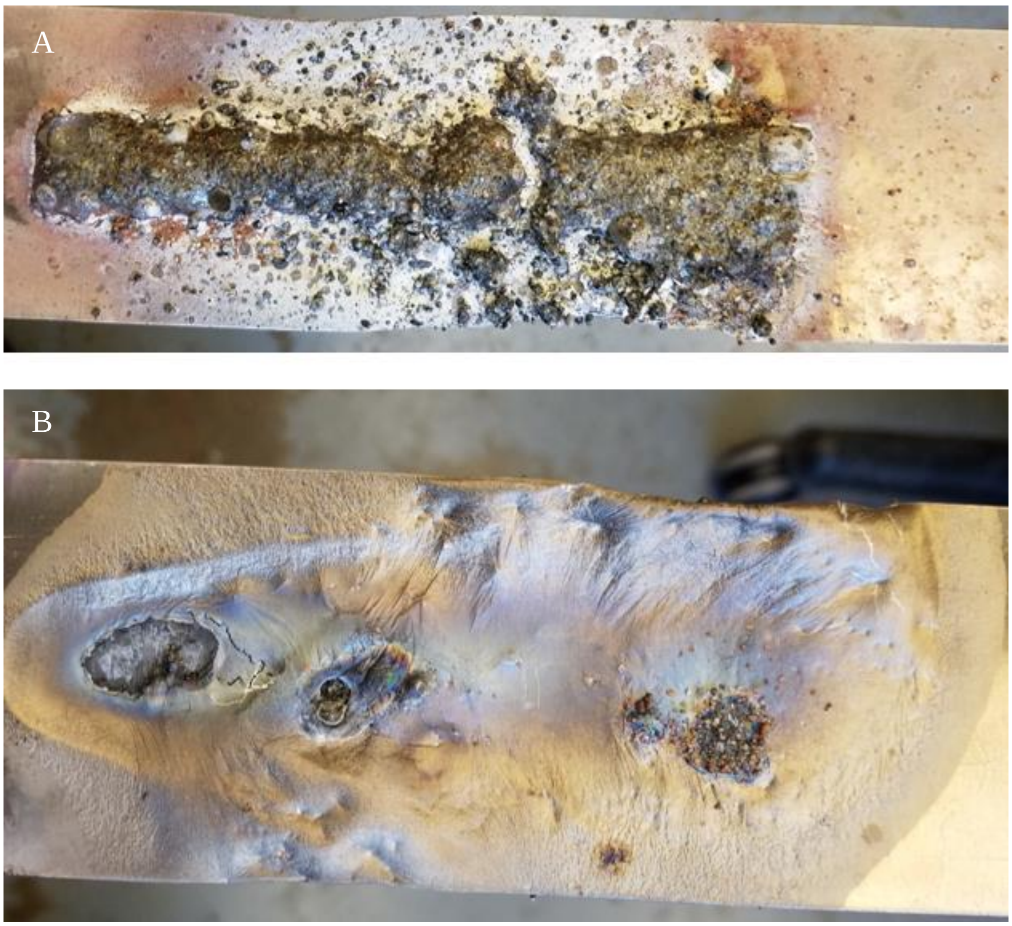

Figure 20 –A) Top side of ignition 3. B) Bottom side of ignition, showing small holes

A smaller thermite brick, containing approximately 66% of the mass of the largest brick, was ignited on an aluminum sample. The results of this ignition can be seen in Figure 20. This ignition did not result in a large burned hole, like in Figure 19, but instead a series of smaller holes. These holes can be seen in Figure 20B. The ignition still resulted in significant heat based deformation, even while resting on sand.

Speed of Ignition

An important characteristic of thermite paste architectures is the baseline speed of ignition. Four ignitions were performed, all with the same composition and molded size of 5x0.75x0.75 inches.

Sample	Ignition Speed
1	33.68 s
2	25.16 s
3	29.30 s
4	32.86 s
Mean	30.25 s
Std. Deviation	3.89 s

Table 4 – Ignition Speed

Table 4 shows an average burn time of 30.25 seconds, or approximately 0.165 in/s burn rate. The propagation is visually very steady and consistent. The burn rate is constant and does not have large localized variations in burn speed.

CHAPTER V

ADDITIVE MANUFACTURING

Equipment

Figure 21 – Ultimaker 2+ with Discov3ry Attachment

The 3D printer used to additively manufacture the paste is the Ultimaker 2+ with a Discov3ry paste attachment, which can be seen in Figure 21. The unmodified printer is capable of printing both PLA and ABS, and has a print volume of 23 x 22.5 x 20.5 cm. A full summary of the unmodified Ultimaker 2+ can be seen below in Table 5.

	Ultimaker 2+
Build Volume	23 x 22.5 x 20.5 cm
Layer Resolution	up to 20 microns
Print Speed	30 mm – 300 mm/s
Travel Speed	30 mm – 350 mm/s
Operating Nozzle Temperature	180°C – 260°C
Operating Heated Bed Temperature	50°C – 100°C

Table 5 – Ultimaker 2+ Summary

The Discov3ry paste system consists of a stepper motor attachment that interfaces directly with the Ultimaker 2+. The stepper motor actuates the plunger of a syringe, which is connected to an 18 inch long tube and nozzle system. The full set up can be seen below in Figure 22.

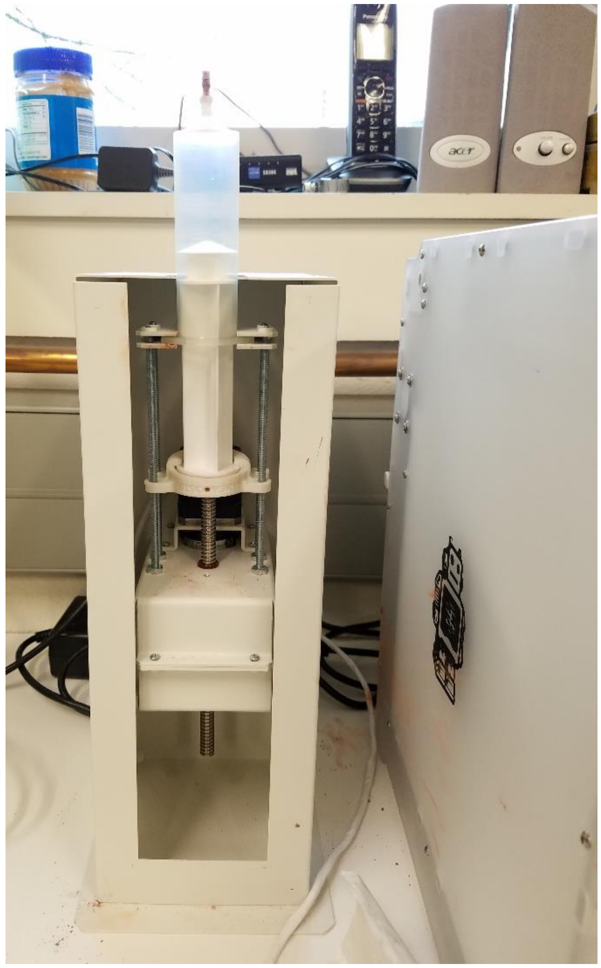

Figure 22 – Discov3ry Attachment

To print using the Discov3ry system, pressure must build up in the syringe and in the tubing in order to appropriately propagate the paste. Before beginning printing, a purge routine must be run. The purge routine just actuates the plunger of the syringe upward, moving the paste through the tube at a rate of approximately 0.03 inches/s. Once the paste has begun exiting the nozzle, the purge routine is done and printing can begin.

Slicing Software

Custom slice settings had to be chosen to successfully print the thermite paste. The chosen slicing software is Cura v15.04.6. The slicing settings are summarized below, and then discussed.

Basic Settings	
Quality	
Layer Height (mm)	0.3
Shell thickness (mm)	1.68
Fill	
Bottom/Top thickness (mm)	0.6
Fill Density (%)	100
Speed and Temperature	
Print Speed (mm/s)	15
Printing Temperature (°C)	0
Bed Temperature (°C)	0
Machine and Filament	
Filament Flow (%)	100
Nozzle size (mm)	0.84

Table 6 – Basic Cura Settings

Table 6 shows the affected Cura settings under the "basic" tab. The layer height is a function of the material and the nozzle size, and it is set to 0.3 mm. The shell thickness is purely a function of the nozzle diameter and should be twice the nozzle diameter. This slicing software creates an inner and outer shell for the print. When the shell thickness is twice the diameter size, there is no gap between shells because each shell is exactly one diameter in thickness. If the shell thickness is larger than the diameter size, a gap is left, which severely compromises the quality of the print and usually causes print failure. Under the fill section, the settings for top and bottom thickness were changed, as was the fill density. Once again, the thickness is a function of the nozzle size and material properties. The bottom/top thickness is twice the layer height (0.6 mm), which simply results in a top/bottom slicer path being run twice. The fill density is 100%. Whenever printing any paste, the fill density must be 100%. Otherwise, the slicing software attempts to generate a plastic style infill, which causes the paste print to fail. Under speed and temperature, the print speed is determined by the viscosity of the material and the speed at which it is coming out of the nozzle. The speed of 15 mm/s was selected by comparing the general viscosity characteristics of the thermite pastes to known paste parameters and selecting the speed. The printing temperature and bed temperature are set to 0°C, but those are not the actual printing temperatures. Setting the temperatures to 0°C just ensures that the heating elements for the bed and nozzle are not turned on. The filament flow is set to 100%, and the nozzle size is 0.84 mm.

Advanced Settings	
Retraction	
Speed (mm/s)	40.0
Distance (mm)	0
Dual extrusion switch amount (mm)	0
Quality	
Initial layer thickness (mm)	0.3
Initial layer line width (%)	100
Cut off object bottom (mm)	0.0
Dual extrusion overlap (mm)	0.15
Speed	
Travel speed (mm/s)	150
Bottom layer speed (mm/s)	10
Infill speed (mm/s)	0
Top/bottom speed (mm/s)	0
Outer shell speed (mm/s)	15
Inner shell speed (mm/s)	0
Cool	
Minimal layer time (sec)	5
Enable Cooling Fan	No

Table 7 – Advanced Cura Settings

Table 6 shows the Cura settings under the “Advanced” tab. Retraction occurs when the printer is done with a specified layer or feature and involves the quick lowering of the printer bed while the nozzle system is moved away. The retraction speed is set to 40 mm/s, which is higher than traditional plastic printing. This is to ensure a clean break of material from the current layer to the next layer. If a slower retraction speed was chosen, the material would continuing extruding during the movement, resulting in blobs or peaks at the spot of retraction. The advanced quality settings allow quality parameters to be changed for the initial layer. For this print, the initial layer settings are not different than the quality settings for the rest of the print. The advanced speed settings allow me to change speed settings for the top/bottom layer, infill, and inner and outer shells. The travel speed is the speed of the nozzle while not printing. The bottom layer speed is set to 10 mm/s to ensure the highest quality bottom layer possible, without moving too slowly for the material. Any setting set to 0 mm/s simply uses the default print speed setting of 15 mm/s. Minimal layer time dictates how long each layer must take. If a layer is completed in under 5 seconds, the machine waits until 5 seconds has passed until beginning the next layer. The cooling fan is not enabled.

G-Code

```
G21         ;metric values
G90         ;absolute positioning
M82         ;set extruder to absolute mode
M107        ;start with the fan off
T1         ;Set Paste Extrude #2
M302       ;Allow Cold Extrusion
M92 E2000 ;Set Extruder EEPROM for Paste (power setting)
G28 X0 Y0   ;move X/Y to min endstops
G28 Z0      ;move Z to min endstops
G1 Z15.0 F{travel_speed} ;move the platform down 15mm
G92 E0                    ;zero the extruded length
G1 F200 E3                ;extrude 3mm of feed stock
G92 E0                    ;zero the extruded length again
G1 F{travel_speed}
```

Figure 23 – Modified Start G-Code

In addition to custom slicing settings, manual changes were made to the start of the G-Code. Figure 23 shows the modified start G-Code, with boxes showing the important manual changes. The first box shows the M-Code command “M302,” which allows cold extrusion. Even with the custom slice settings allowing for cold extrusion, this extra command must be included to ensure that no heating occurs. The second box shows the command that controls motor steps per unit for that given axis. In this scenario, the command controls the speed of the stepper motor controlling the paste plunger.   Without this command, the Discov3ry system would attempt to operate at inappropriate motor settings, which would likely damage the system. The axis steps per unit setting is currently set to 2000 steps/mm, but can be altered according to the viscosity of the material.

Printing Results

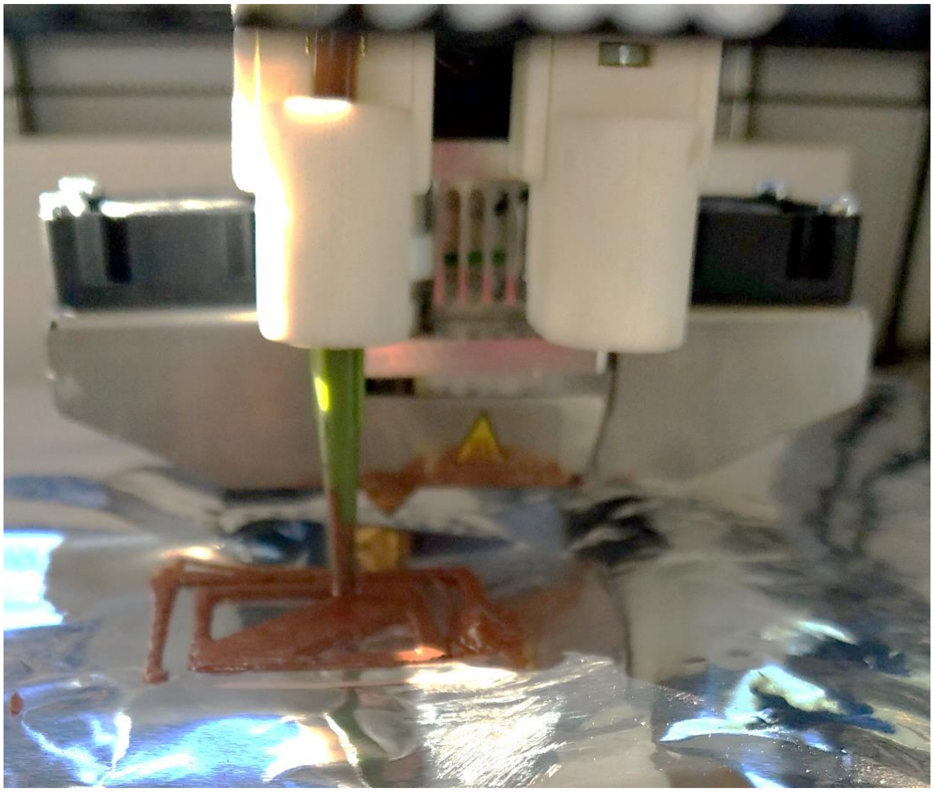

Figure 24 – Successful Printing of Thermite Paste

Figure 24 shows the successful printing of thermite paste, at an early stage in the print process. This image is taken very early in the printing process, before the first layer is even completed. Note the strange infill pattern, which is decided by Cura and not preprogrammed.

The infill starts in the middle, and then fills the two opposite corners. This will cause unevenness of the layer, but it is slight enough to not affect the overall print quality.

Figure 25 – Partial Print of Thermite

Figure 25 shows a partial print of the thermite, with several key features easily visible. The outermost ring of thermite is a printing feature called the "skirt." The skirt is simply the nozzle outlining the shape several times, which it does to eliminate any temporary movement or extrusion irregularities. An extrusion irregularity can be seen on the right side of the skirt. The large blob is simply a small over-extrusion of material in that localized region. It is caused by uneven movement of the syringe plunger, which is itself caused by friction and localized curing. The print region remains high quality, and shows part of the 100% infill. These lines are very clear, and contain no gaps. The line width and print speed are near or at the appropriate parameters due to the lack of gaps or thinning of the line. Overall, this is a successful print of thermite.

Figure 26 – Completed Thermite Print

A second print (with the same slicing settings) was allowed to progress further into the print process. This print is also successful, but has more variability in line and layer thickness. Because the syringe was not changed between printings, the variability is due to the increased viscosity of the thermite paste, which is beginning to set in both the tube and the syringe.

This second print was ignited to confirm success of both the 3D printing process and the paste. The sample weighed 2.9g and resulted in a fast and complete ignition. The slag produced can be seen in Figure 27.

Figure 27 – Slag of 3D Printed Sample

CHAPTER VI

CONCLUSIONS

Commercially available curing pastes and mixtures are not good thermite binders and result in mixtures that will not ignite. Most pastes with large quantities of thermite will not ignite at very high temperatures. Even with an ignitable binder, such as gypsum plaster, other binder-specific properties like curing time and moisture retention affect success of ignition. Therefore, binder selection is significantly more difficult than initially thought.

Additionally, significant conclusions can be drawn about the paste 3D printing process. While technology exists for paste printing, it is currently not as consistent or reliable as traditional plastic additive manufacturing. The printing process can still be successful, but it requires additional work to begin to approach the consistency of 3D printing thermoplastics.

This series of trials and experiments have confirmed the success in both the creation and printing of a thermite paste. The reaction propagates in a uniform manner with a consistent velocity. The paste ignites consistently and possesses a viscosity and work life that allows for 3D printing of structures.

CHAPTER VII

FUTURE WORK

Immediate next steps include further quantitative characterization of the paste, its heat generated, and its materials properties. Studies will be undertaken to determine the effect of geometry on reaction speed and time.

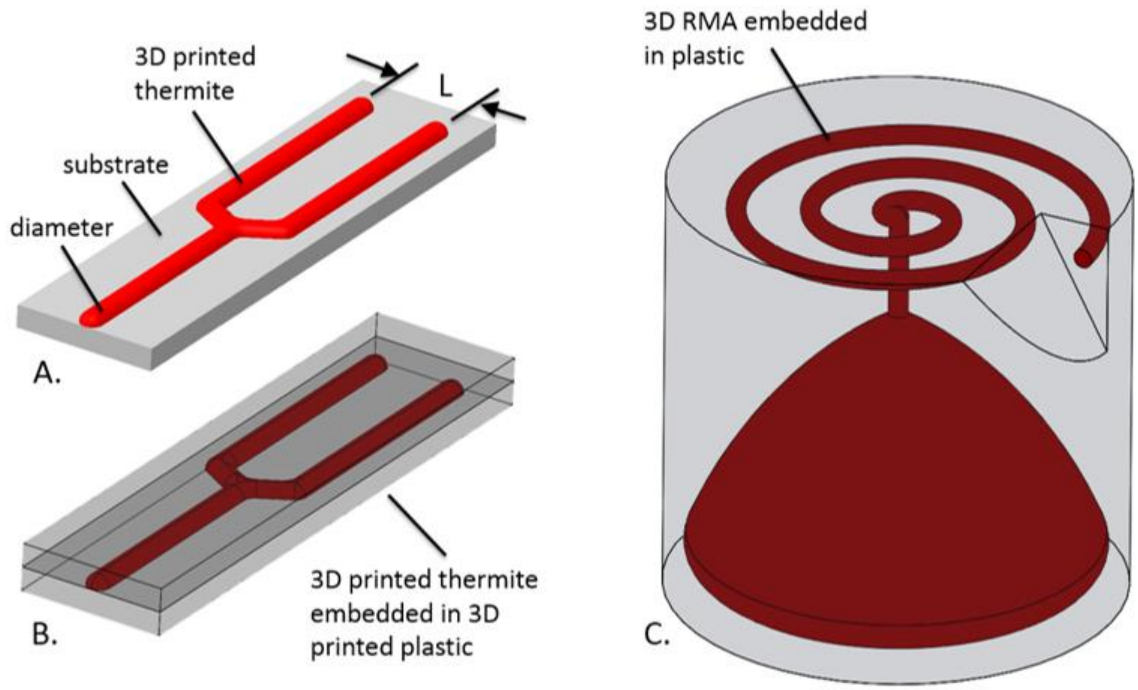

Figure 28 – A) Thermite 3D printed onto a flat substrate, where spacing and diameter can be varied to alter the reaction outcome. B) Thermite and plastic co-printed, resulting in a thermite structure embedded in plastic. C) A fully 3D shape co-printed and embedded in plastic

Figure 28 illustrates the three phases associated with the development of reactive material architectures. The first phase, shown in Figure 28A, is the natural extension of the work completed in this document. Thermite paste will be 3D printed in precise geometric configurations to determine quantitatively how geometry affects reaction properties. These architectures will be 2.5D, and printed on top of a flat substrate. The next step will involve coprinting thermite and a plastic, resulting in a thermite structure embedded in plastic. The final goal is a 3D structure completely embedded in plastic, as seen in Figure 28C.

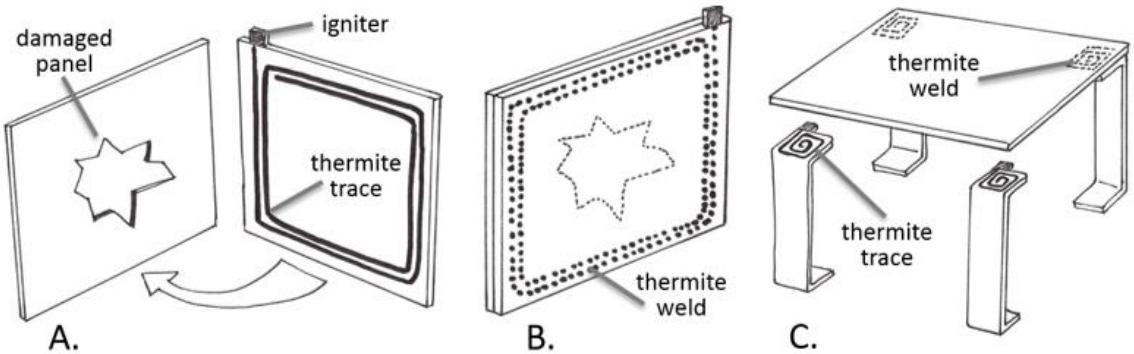

Figure 29 A) and B) A pre-printed thermite trace is used to rapidly repair a damaged panel. C) Pre-printed thermite traces assisting in rapid assembly.

Figure 29 shows constructive applications of 3D printed thermite. Figures 27A and B show potential repair applications. Figure 29C shows pre-printed thermite traces assisting in rapid assembly of a structure. More thermite paste applications will be explored as the paste and printing process is further developed.

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APPENDIX A: Ultimaker 2+ Data Sheet

Printer and printing properties

Technology	Fused Deposition Modeling (FDM)
Print head	Swappable nozzle
Build volume	223 x 223 x 205 mm
Filament diameter	2.85 mm
Layer resolution	0.25 mm nozzle: 150 to 60 micron 0.40 mm nozzle: 200 to 20 micron 0.60 mm nozzle: 400 to 20 micron 0.80 mm nozzle: 600 to 20 micron
XYZ accuracy	12.5, 12.5, 5 micron
Print head travel speed	30 to 300 mm/s
Build speed	0.25 mm nozzle: up to 8 mm^3/s 0.40 mm nozzle: up to 16 mm^3/s 0.60 mm nozzle: up to 23 mm^3/s 0.80 mm nozzle: up to 24 mm^3/s
Build plate	Heated glass build plate
Build plate temperature	50 to 100 °C
Build plate leveling	Assisted leveling process
Supported materials	PLA, ABS, CPE, CPE+, PC, Nylon, TPU 95A
Nozzle diameter	Included are 0.25, 0.4, 0.6 and 0.8 mm nozzles
Nozzle temperature	180 to 260 °C
Nozzle heat up time	~ 1 minute
Build plate heat up time	< 4 minutes
Operating sound	50 dBA
Connectivity	Standalone 3D printing from SD card (included)

Physical dimensions

Dimensions	342 x 357 x 388 mm
Dimensions (with bowden tube and spool holder)	342 x 493 x 588 mm
Nett weight	11,3 kg
Shipping weight	18,5 kg
Shipping box dimensions	390 x 400 x 565 mm

Power requirements

Input	100 - 240V 4A, 50-60Hz 221 W max.
Output	24 V DC, 9.2 A

Ambient conditions

Operating ambient temperature	15 - 32 °C See material specifications for optimal conditions
Nonoperating temperature	0 - 32 °C

Software

Supplied software	Cura, our free print preparation software
Supported OS	macOS, Windows and Linux
File types	STL, OBJ and AMF

APPENDIX B: Cube G-Code

```
;Sliced at: Wed 08-03-2017 11:47:50
;Basic settings: Layer height: 0.3 Walls: 1.68 Fill: 100
;Print time: 11 minutes
;Filament used: 0.289m 2.0g
;Filament cost: None
G21        ;metric values
G90        ;absolute positioning
M82        ;set extruder to absolute mode
M107       ;start with the fan off
T1                              ;Set Paste Extrude #2
M302               ;Allow Cold Extrusion
M92 E2000 ;Set Extruder EEPROM for Paste (power setting)
G28 X0 Y0  ;move X/Y to min endstops
G28 Z0     ;move Z to min endstops
G1 Z15.0 F9000 ;move the platform down 15mm
G92 E0                ;zero the extruded length
G1 F200 E3              ;extrude 3mm of feed stock
G92 E0                ;zero the extruded length again
G1 F9000
;Put printing message on LCD screen
M117 Printing...

;Layer count: 11
;LAYER:0
M107
G0 F9000 X85.541 Y85.541 Z0.300
;TYPE:SKIRT
G1 F600 X119.460 Y85.541 E1.11443
G1 X119.460 Y119.460 E2.22886
G1 X85.541 Y119.460 E3.34330
G1 X85.541 Y85.541 E4.45773
G0 F9000 X86.381 Y86.381
G1 F600 X118.620 Y86.381 E5.51696
G1 X118.620 Y118.620 E6.57620
G1 X86.381 Y118.620 E7.63543
G1 X86.381 Y86.381 E8.69467
G0 F9000 X91.061 Y91.061
;TYPE:WALL-INNER
G1 F600 X113.940 Y91.061 E9.44637
G1 X113.940 Y113.940 E10.19808
G1 X91.061 Y113.940 E10.94978
G1 X91.061 Y91.061 E11.70149
G0 F9000 X90.221 Y90.221
;TYPE:WALL-OUTER
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G1 X90.221 Y114.780 E14.12220
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G1 F600 X94.366 Y91.397 E18.78040
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G1 F600 X99.941 Y113.603 E29.20739
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G0 F9000 X91.396 Y112.185
G1 F600 X92.814 Y113.603 E30.43065
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G0 F9000 X97.565 Y113.603
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G1 F720 X105.058 Y91.397 E84.02698
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G1 F720 X113.603 Y98.754 E84.36882
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G1 F720 X113.603 Y96.378 E84.88691
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G0 F9000 X110.998 Y91.397
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G0 X113.523 Y91.396
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G1 F780 X93.691 Y113.603 E94.42879
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G0 F9000 X97.255 Y113.603
G1 F780 X113.603 Y97.256 E96.87315
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G0 F9000 X99.631 Y113.603
G1 F780 X113.603 Y99.632 E98.22672
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G1 F780 X111.147 Y91.396 E100.11728
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G0 F9000 X113.603 Y111.821
G1 F900 X93.178 Y91.397 E119.98342
G0 F9000 X94.366 Y91.397
G1 F900 X113.603 Y110.633 E120.87725
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G1 F900 X100.306 Y91.397 E124.51845
G0 F9000 X101.494 Y91.397
G1 F900 X113.603 Y103.506 E125.08109
G0 F9000 X113.603 Y102.318
G1 F900 X102.682 Y91.397 E125.58854
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G1 F900 X113.603 Y101.130 E126.04078
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G1 X113.940 Y91.061 E139.31594
G0 F9000 X114.780 Y90.221
;TYPE:WALL-OUTER
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G1 F900 X93.691 Y113.603 E145.47721
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G1 F900 X113.603 Y94.880 E146.34720
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G1 F900 X96.067 Y113.603 E147.16198
G0 F9000 X97.255 Y113.603
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G1 X114.780 Y114.780 E168.07438
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G1 F900 X93.178 Y91.397 E171.03185
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G1 F900 X97.930 Y91.397 E174.27598
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;TYPE:SKIN
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G1 F900 X113.603 Y103.506 E227.17794
G0 F9000 X113.603 Y102.318
G1 F900 X102.682 Y91.397 E227.68539
G0 F9000 X103.870 Y91.397
G1 F900 X113.603 Y101.130 E228.13763
G0 F9000 X111.821 Y113.603
G1 F900 X91.396 Y93.178 E229.08668
G0 F9000 X91.396 Y94.366
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G1 F900 X110.633 Y113.603 E229.98052
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G1 F900 X108.257 Y113.603 E231.60262
G0 F9000 X107.069 Y113.603
G1 F900 X91.396 Y97.930 E232.33086
G0 F9000 X91.396 Y99.117
G1 F900 X105.881 Y113.603 E233.00393
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G1 F900 X103.505 Y113.603 E234.18447
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G1 F900 X98.753 Y113.603 E235.88313
G0 F9000 X97.565 Y113.603
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G0 F9000 X91.396 Y108.621
G1 F900 X96.377 Y113.603 E236.40126
G0 F9000 X95.190 Y113.603
G1 F900 X91.396 Y109.809 E236.57755
G0 F9000 X91.396 Y110.997
G1 F900 X94.002 Y113.603 E236.69864
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G1 F900 X107.434 Y91.397 E237.80074
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G1 F900 X113.603 Y96.378 E238.03218
G0 F9000 X113.603 Y95.190
G1 F900 X109.810 Y91.397 E238.20842
G0 F9000 X110.998 Y91.397
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G1 F900 X113.940 Y113.940 E239.15768
G1 X91.061 Y113.940 E239.90938
G1 X91.061 Y91.061 E240.66109
G1 X113.940 Y91.061 E241.41279
G0 F9000 X114.780 Y90.221
;TYPE:WALL-OUTER
G1 F900 X114.780 Y114.780 E242.21969
G1 X90.221 Y114.780 E243.02660
G1 X90.221 Y90.221 E243.83350
G1 X114.780 Y90.221 E244.64040
G0 F9000 X114.260 Y90.651
G0 X113.523 Y91.396
;TYPE:SKIN
G1 F900 X91.397 Y113.522 E245.66849
G0 F9000 X92.503 Y113.603
G1 F900 X113.603 Y92.504 E246.64887
G0 F9000 X113.603 Y93.692
G1 F900 X93.691 Y113.603 E247.57406
G0 F9000 X94.879 Y113.603
G1 F900 X113.603 Y94.880 E248.44405
G0 F9000 X113.603 Y96.068
G1 F900 X96.067 Y113.603 E249.25883
G0 F9000 X97.255 Y113.603
G1 F900 X113.603 Y97.256 E250.01842
G0 F9000 X113.603 Y98.444
G1 F900 X98.443 Y113.603 E250.72281
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G1 F900 X113.603 Y99.632 E251.37199
G0 F9000 X112.335 Y91.396
G1 F900 X91.397 Y112.334 E252.34488
G0 F9000 X91.397 Y111.146
G1 F900 X111.147 Y91.396 E253.26256
G0 F9000 X109.959 Y91.396
G1 F900 X91.397 Y109.958 E254.12504
G0 F9000 X91.397 Y108.770
G1 F900 X108.771 Y91.396 E254.93232
G0 F9000 X107.583 Y91.396
G1 F900 X91.397 Y107.582 E255.68441
G0 F9000 X91.397 Y106.394
G1 F900 X106.395 Y91.396 E256.38129
G0 F9000 X105.207 Y91.396
G1 F900 X91.397 Y105.206 E257.02297
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G0 F9000 X100.455 Y91.396
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G0 F9000 X91.397 Y99.267
G1 F900 X99.267 Y91.396 E259.40346
G0 F9000 X98.079 Y91.396
G1 F900 X91.397 Y98.079 E259.71396
G0 F9000 X91.397 Y96.891
G1 F900 X96.892 Y91.396 E259.96929
G0 F9000 X95.704 Y91.396
G1 F900 X91.397 Y95.703 E260.16941
G0 F9000 X91.397 Y94.515
G1 F900 X94.516 Y91.396 E260.31434
G0 F9000 X93.328 Y91.396
G1 F900 X91.397 Y93.327 E260.40406
G0 F9000 X91.397 Y92.139
G1 F900 X92.140 Y91.396 E260.43858
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G0 F9000 X102.007 Y113.603
G1 F900 X113.603 Y102.007 E261.57140
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G0 F9000 X109.134 Y113.603
G1 F900 X113.603 Y109.135 E263.64511
G0 F9000 X113.603 Y110.323
G1 F900 X110.322 Y113.603 E263.79754
G0 F9000 X111.510 Y113.603
G1 F900 X113.603 Y111.511 E263.89476
G0 F9000 X113.603 Y112.699
G1 F900 X112.698 Y113.603 E263.93679
;LAYER:10
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;TYPE:WALL-INNER
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G0 F9000 X114.780 Y114.780
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G1 F900 X90.221 Y114.780 E267.75052
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G1 X114.780 Y114.780 E270.17123
G0 F9000 X114.260 Y114.350
G0 X113.009 Y113.603
;TYPE:SKIN
G1 F900 X91.396 Y91.990 E271.17547
G0 F9000 X91.991 Y91.397
G1 F900 X113.603 Y113.009 E272.17967
G0 F9000 X113.603 Y111.821
G1 F900 X93.178 Y91.397 E273.12870
G0 F9000 X94.366 Y91.397
G1 F900 X113.603 Y110.633 E274.02252
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G0 F9000 X96.742 Y91.397
G1 F900 X113.603 Y108.258 E275.64459
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G1 F900 X97.930 Y91.397 E276.37283
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G1 F900 X113.603 Y101.130 E279.18606
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G1 F900 X91.396 Y93.178 E280.13510
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G0 F9000 X108.622 Y91.397
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G1 F900 X109.810 Y91.397 E289.25685
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G1 F900 X113.603 Y94.002 E289.37789
G0 F9000 X113.603 Y92.814
G1 F900 X112.186 Y91.397 E289.44373
G0 F9000 X113.373 Y91.397
G1 F900 X113.603 Y91.626 E289.45439
G0 F9000 X113.603 Y91.626 Z8.200
;End GCode
M104 S0              ;extruder heater off
M140 S0              ;heated bed heater off (if you have it)
M92 E282                                  ; Reset Extruder EEPROM (to return to
plastic level)
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G91                           ;relative positioning
G1 E-1 F300                   ;retract the filament a bit before lifting the nozzle, to release some of the pressure
G1 Z+0.5 E-5 X-20 Y-20 F9000 ;move Z up a bit and retract filament even more
G28 X0 Y0                     ;move X/Y to min endstops, so the head is out of the way
M84                      ;steppers off
G90                      ;absolute positioning
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