

Production and Characterization of Aluminum Alloys used for Harvesting Energy from the Aluminum-Water Reaction

by

Shavinesh Sukesh

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

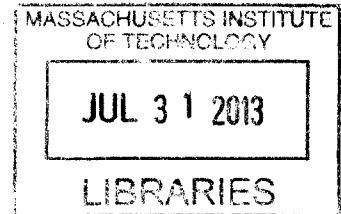
Bachelor of Science in Mechanical Engineering

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Abstract

Autonomous Underwater Vehicles(AUV) are heavily used by the military and in the industry for countless underwater tasks but currently have a limited mission time due to limitations in the energy density of their battery packs. Aluminum is an ideal energy source for AUVs because it exothermically reacts with water, producing hydrogen as one of its by-product, and it is two orders of magnitude more energy dense than lithium ion batteries. A method of using an aluminum-galinstan alloy was conceived to react with water where the presence of galinstan allows elemental aluminum to overcome the passivating aluminum oxide layer. The aluminum atoms reacts with water to produce heat and hydrogen at the grain boundaries with galinstan. This thesis attempts to develop a method of producing an aluminum-galinstan alloy. Several methods are explored to determine the most reliable method. Experiments were conducted to determine the percentage hydrogen yield to characterize the alloy.

Thesis Supervisor: Douglas P. Hart

Title: Professor of Mechanical Engineering

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Contents

1 Introduction	11
1.1 Autonomous Underwater Vehicles	11
1.2 Energy Forms	14
2 Aluminum as a Fuel Source	16
2.1 Background	16
2.2 Aluminum Depassivation	18
2.2.1 In-situ Interdiffusion Method	18
2.2.2 Percolating Gallium	19
2.2.3 Aluminum-Gallium Alloy	19
2.3 Thesis Summary	19
3 Alloy Production	20
3.1 Methods of Alloy Production	20
3.2 Tube Sealing	21
4 Experiments	33
4.1 Experimental Setup	33
4.2 Alloy Compositions	36
4.3 Reaction Results	38
4.4 Reaction Observations	39
4.5 SEM Imaging of Alloys	40
5 Discussion and Future Work	44
6 System Design	45

List of Figures

1.1.1 Artist impression of an AUV scanning the ocean floor	12
1.1.2 Current and desired range of AUVs	14
1.2.1 Volumetric energy densities of common fuel sources	15
3.2.1 Stock quartz tubing	21
3.2.2 Heating stock tubing on a hydrogen flame	22
3.2.3 Short tube with one end sealed	22
3.2.4 Inserting metals into tube	23
3.2.5 Necking the tube	24
3.2.6 Width of the necked region	25
3.2.7 Cross-section of tube neck	26
3.2.8 Tube under vacuum	27
3.2.9 Necked region being heated	28
3.2.10 Necked region stretching under gravity	29
3.2.11 Tube breaks away and seals	30
3.2.12 Sealed tube containing samples	31
4.1.1 High reaction rate experimental setup	35
4.1.2 Low reaction rate experimental setup	36
4.3.1 Hydrogen flow rate in time for 50-50 sample	38
4.4.1 Dark suspension during reaction	39
4.4.2 Hydrogen bubbles several hours after reaction started	40
4.5.1 SEM image of 50-50 sample	41
4.5.2 SEM image of 70-30 sample	42
4.5.3 SEM image of 90-10 sample	43
4.5.1 SEM image of 90-10 sample	44
6.1.1 Diagram of potential system layout	47

List of Tables

2.1.1 Thermodynamics of aluminum-water reaction	17
4.2.1 Purity of individual metals	37
4.2.2 Calculated mass of individual metals required	37
4.2.1 Measured mass of individual metals	37
4.3.1 Reaction completion rates	38

Chapter 1

Introduction

There has yet to be technology that generates power in underwater conditions where oxygen is unavailable. One possible solution is Aluminum. Aluminum reacts with water to produce 40 times more energy than lithium ion by volume and more than twice the energy density by volume of diesel burning in air. However, there has yet to be an underwater power source using aluminum as the fuel source. Developing a power system that uses aluminum as a fuel source could help make significant strides in underwater robotics and underwater power systems.

1.1 Autonomous Underwater Vehicles

The use of AUVs have greatly expanded within the maritime community in the past decade into search and rescue missions, ocean monitoring, sea floor mapping, oil rig constructions, military operations and many more. There are many features of an AUV that has made it very attractive for such applications such as the low cost of operation, designing and building, making it feasible for missions that would otherwise be too expensive. It also often has more capabilities than manned vehicles, and can even be used in hazardous environments with much lower safety risks.

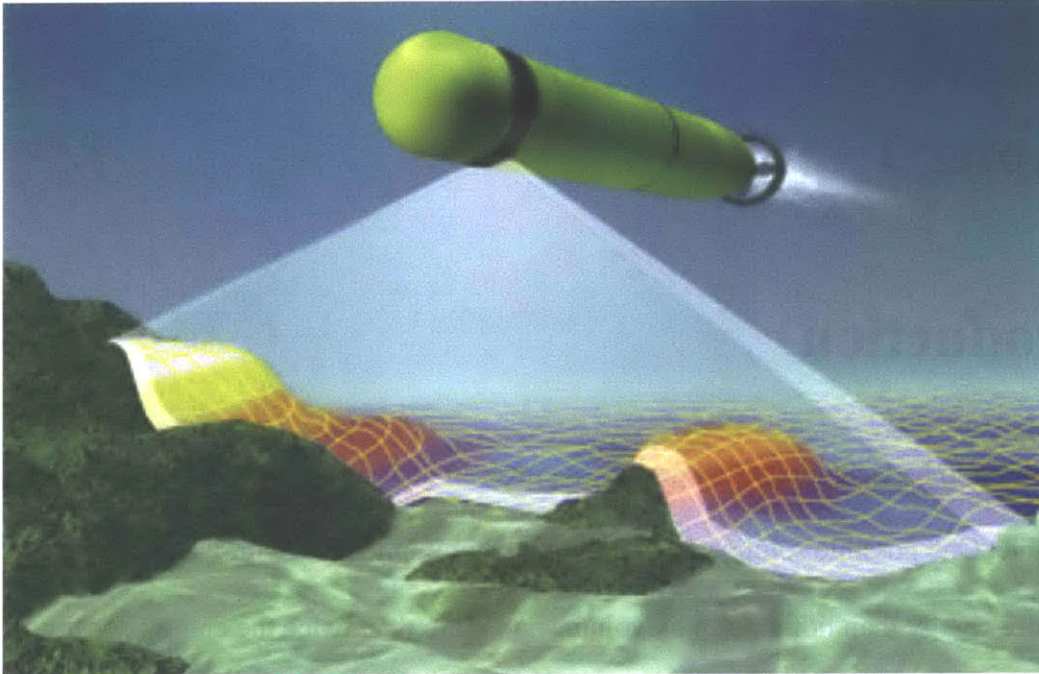


Figure 1.1.1: Artist impression of an AUV scanning the ocean floor

Batteries are currently the source of energy to power underwater vehicles. Two common propulsion systems used in AUVs are the buoyancy driven and propeller driven systems. A buoyancy driven system uses a hydrofoil that takes advantage of the vehicle's vertical motion to provide horizontal thrust. The vehicle relies on the ocean currents and can only move very slowly. Researchers at Rutgers University built such a glider that crossed the Atlantic Ocean going east in 221 days, averaging half a knot [1]. The entire mission consumed 28MJ of energy. Such slow speeds of gliders make it impractical for many applications, especially where ocean currents consistently exceed 0.5 knots. Propeller driven AUVs on the other hand, can travel much faster but are very inefficient.

The Hydroid REMUS 600 is single propeller driven and has a top speed of roughly 5 knots. It operates on a 15kWh(54MJ) lithium ion battery pack and has a standard endurance of

70 hours before the vehicle must recharge [2]. The REMUS 600 is used frequently for ocean exploration and Navy operations for its speed and sensor capabilities.

Due to the 70 hour endurance limit, a surface vessel needs to remain close to the AUV so that it can recharge its batteries at the end of the energy cycle. This then imposes a new problem, where the cost of operating these surface vessel are very high and quickly surpasses the cost of the AUV itself. Increasing the energy stored in the REMUS 600 by an order of magnitude would go a long way in increasing the feasibility of using the vehicle by extending its mission length up to a month before requiring refueling. The significance of the extended life is illustrated in Figure 1.1.2 below where the current range of the REMUS 600 is compared with the hypothetical range, centered at the United States Naval Base in the Pacific Ocean.



Figure 1.1.2: Current and desired range of AUVs. The smaller circle represents the current range and the larger circle represents the desired range. [3]

1.2 Energy Forms

There are many possible fuel sources that can be carried onboard an AUV and reacted to produce energy. Several common fuel sources are highlighted in Figure 1.2.1. Hydrocarbons have proven to be a good source of fuel for many applications. However, the oxygen depleted environment underwater poses a restriction on the vehicle operation. The AUV would have to burn the

hydrocarbon at the surface of the water using a snorkel to recharge its batteries each time the batteries run out of energy. Storing oxygen in the vehicle to allow a continuous burning of hydrocarbon underwater greatly reduces the energy density of the power system to the level of lithium-ion batteries. Therefore, it would be advantageous to use a fuel that reacts with water instead to allow the AUV to continuously produce energy at depth. This would also result in a lower output power required compared to a system that is only allowed to run intermittently. Aluminum reacts with water, has one of the highest energy densities and is very safe, as opposed to other hazardous materials that also react with water. For this reason, aluminum was chosen to be developed as an energy source for underwater vehicles.

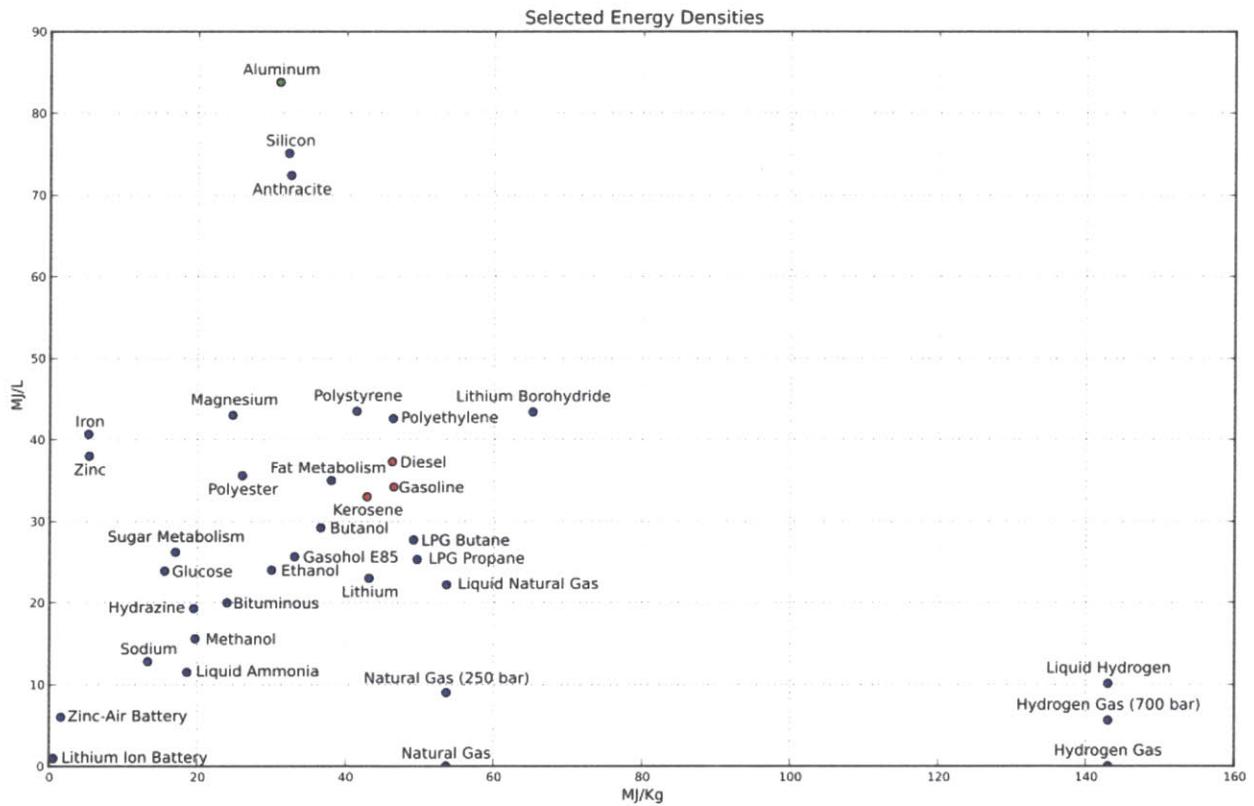


Figure 1.2.1: Volumetric energy densities of common fuel sources [3]

Chapter 2

Aluminum as a Fuel Source

It is known that pure aluminum reacts with water by oxidizing with the oxygen molecule from the H₂O molecule. However, aluminum that is exposed to standard atmospheric conditions naturally forms a protective passivating aluminum oxide layer (Al₂O₃) on the surface, preventing the aluminum molecules from reacting with water. This passivating layer needs to be overcome in order to allow the reaction to happen. In the Fall of 2013, several methods of depassivating the aluminum were explored by the MIT Rapid Development Group (MIT-RDG). The methods will be summarized below.

2.1 Background

Aluminum exothermically reacts with water to produce aluminum hydroxide and hydrogen.



The reaction is highly exothermic and can take place in many temperatures, as shown in Table 2.1.1. From the Gibb's free energy in Table 2.1.1, the reaction releases 15.89MJ/kg Al or

4.414kWh/kg Al of heat at a reaction temperature of 100°C. Taking the density of aluminum to be 2.7kg/L, that results in 42.9MJ/L or 11.92kWh/L of aluminum released in the form of heat energy, compared to the 38.5MJ of energy per liter of diesel released in the combustion of diesel fuel in air.

Temp (°C)	ΔH (kJ/mol H ₂)	ΔS (J/K)	ΔG (kJ/mol H ₂)
0	-277	26.2	-284
100	-284	3.29	-286
200	-291	-12.1	-285
300	-298	-25.1	-283
400	-306	-38.0	-280
500	-316	-51.8	-276
600	-328	-66.8	-270

Table 2.1.1: Thermodynamics of the aluminum-water reaction [4]

Additional energy can be obtained from this reaction if the hydrogen produced is stored and burned. The energy released in the combustion of hydrogen is described in the chemical reaction below.



This shows that the ratio of energy produced by burning the hydrogen and in the form of heat are equal. Harnessing the hydrogen produced along with the heat will now double the energy output.

This thesis focuses on the energy stored in the form of hydrogen.

2.2 Aluminum Depassivation

Professor Jerry Woodall from the Purdue University had conducted research on producing hydrogen using aluminum-gallium alloys. Here, the presence of gallium depassivates the aluminum and enables the reaction between aluminum and water to take place. Building off the research done by Prof. Woodall, the MIT-RDG explored three methods of developing a power source for AUVs utilizing the aluminum-water reaction using gallium to depassivate the aluminum. They were an in-situ interdiffusion method, a percolating gallium method, and using an aluminum-gallium alloy similar to the ones developed by Prof. Woodall [5].

2.2.1 In-situ Interdiffusion Method

This method involves a stationary pool of gallium with aluminum fed from one side, allowing it to diffuse through the gallium and eventually reacting with a stream of water flowing on the opposite side of the gallium pool [3]. This method allows the system to be compact and does not require the pumping gallium, which is highly viscous in its liquid form. However, this system is limited by the diffusion rate of aluminum in gallium and the MIT-RDG had several problems storing the gallium in an localized area without it wetting to any material or flow along with the water.

2.2.2 Percolating Gallium

In this method, gallium is used as a working fluid where it is pumped through a diffusion tank full of aluminum balls where it allows the balls to diffuse into the stream of gallium. The gallium then gets pumped into a reactor where water is brought into the system and allowed to react, before cycling through the diffusion tank again. In this method, the pumping of liquid gallium proved to be difficult as well as to keep the liquid gallium in the liquid form without freezing.

2.2.3 Aluminum-Gallium Alloy

In this method, an aluminum-gallium alloy was prepared beforehand and the alloy was reacted directly with water. Although this method has the simplest overall system, the production of hydrogen from the alloys were too low to be feasible.

2.3 Thesis Summary

This thesis explores an alternative method of producing alloys involving aluminum, gallium, indium and tin, as developed by the graduate student of Prof. Woodall, Jeffrey Ziebarth. The method of producing the alloy is discussed in chapter 3 and several experiments were done on the alloy samples in an attempt to characterize the alloys.

Chapter 3

Alloy Production

3.1 Methods of Alloy Production

Two different methods of producing aluminum rich alloy were explored. They were the vacuum sealed tube method and the purged furnace method. The purged furnace method involved heating the samples on a furnace boat in a tube furnace while purging the tube furnace with nitrogen. It was simpler to setup but it was not feasible to quench-cool the molten alloy as it was not possible to pull the sample out while at high temperatures. The vacuum sealed tube was the most ideal method as it allowed us to use a box furnace, where the tube will be easily accessible at high temperatures to quench cool the alloy in liquid nitrogen and it provides a much cleaner environment to prevent oxidation of the samples.

Several different forms were explored to determine a reliable method of vacuum sealing the tubes under an inert environment. After multiple attempts, a sample size of 3 grams was selected as it was large enough to weigh the individual metals but small enough so that reacting the entire sample would not produce an unreasonably large volume of hydrogen gas. The entire sample needed to be reacted in order to account for non-homogeneity of the alloy if only smaller samples were picked to react from a single batch of alloy. 19 mm outer diameter quartz tubes

were used to prepare the sample for its high resistance to thermal shock and its optimal diameter that is not too small that it spreads the individual metals too far apart, reducing homogeneity, and not too large that it becomes difficult and unreliable to seal under vacuum.

3.2 Tube Sealing

A stock 19 mm quartz tube was purchased from VWR and hydrogen torch was used for the sealing process. First, the tube torched approximately 12 inches from one end, gently rotating the tube as it heats up. The tube was heated until it melted and sealed to produce two separate sections with one end sealed.



Figure 3.2.1: A stock quartz tubing from the supplies

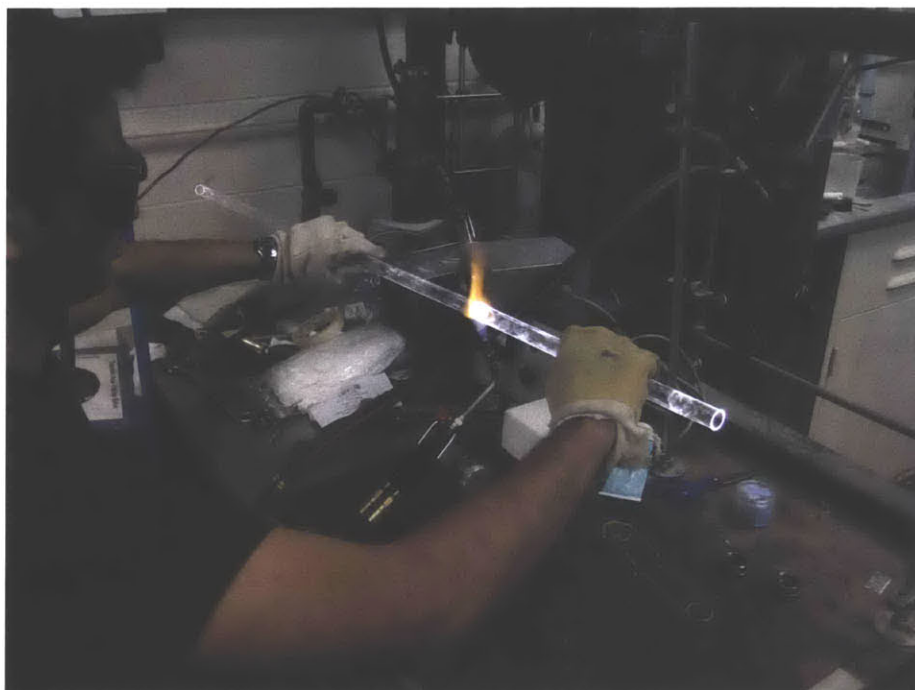


Figure 3.2.2: Heating the stock tube on the hydrogen flame, to produce a short piece of tube with one end sealed

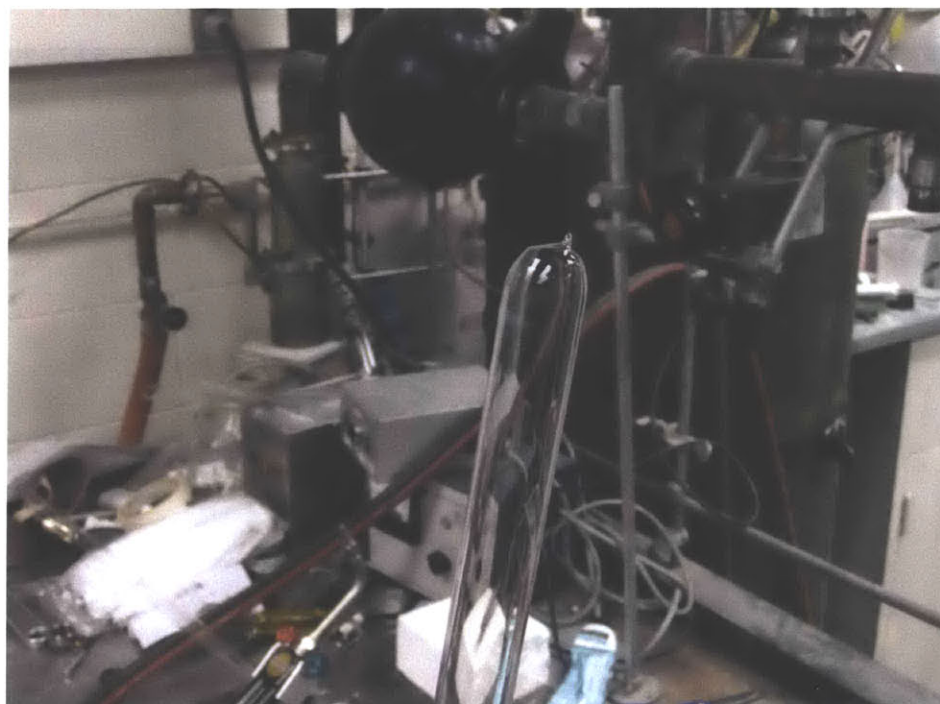


Figure 3.2.3: Image of a short tube with one end sealed

The tips of the sealed end is heated a little more to flame-polish the tip. The foot long section is then used for containing the samples. Each metal of the alloy is placed into the tube in its solid form.



Figure 3.2.4: Inserting the individuals into the tube in its solid form

The tube is then necked in the flame, approximately 3 inches from the open end. While in flame, the tube is constantly rotated to evenly distribute the heat all around the circumference of the tube and it is held at an angle to prevent the samples from falling out of the tube. Slight tension is also applied at both ends of the tube while in the flame so that as the tube gets hot and starts melting, it forms a neck and not folds on itself. The tension is applied very carefully to ensure a constant wall thickness is maintain and that it does not get separated without sealing. The tube is

necked such that the inner diameter of the necked region is between 2mm and 5mm. The length of the necked region will be approximately 4mm in length.



Figure 3.2.5: A tube being necked before vacuum sealing

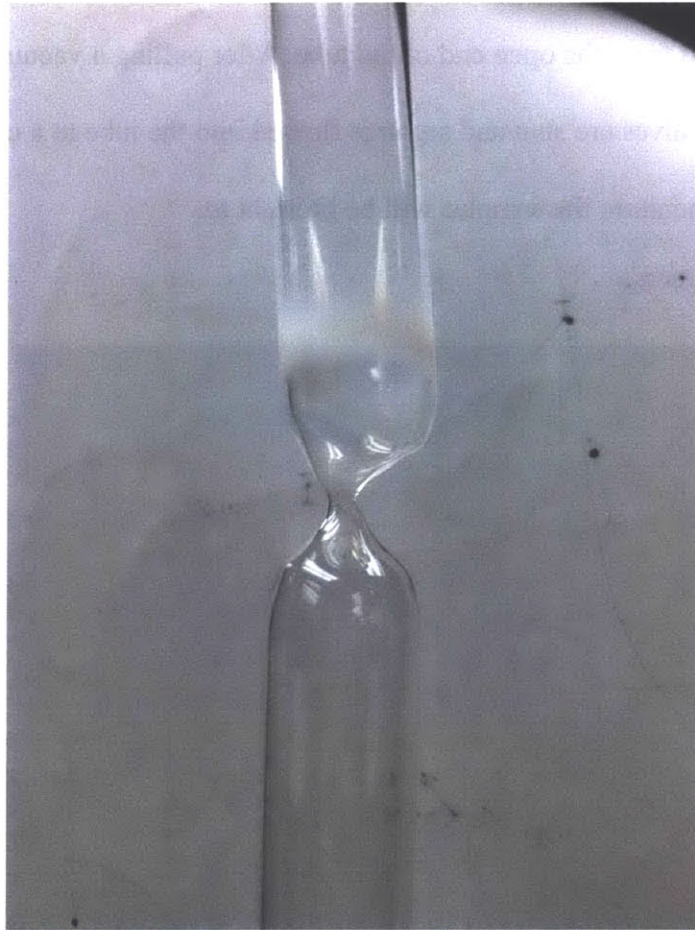


Figure 3.2.6: Approximate width of the necked region



Figure 3.2.7: Looking down into the neck

Next, vacuum is pulled from the open end of the tube. After pulling a vacuum for several minutes, the vacuum valves are shut and argon is flowed into the tube to a certain pressure based on the maximum temperature the samples will be brought to.

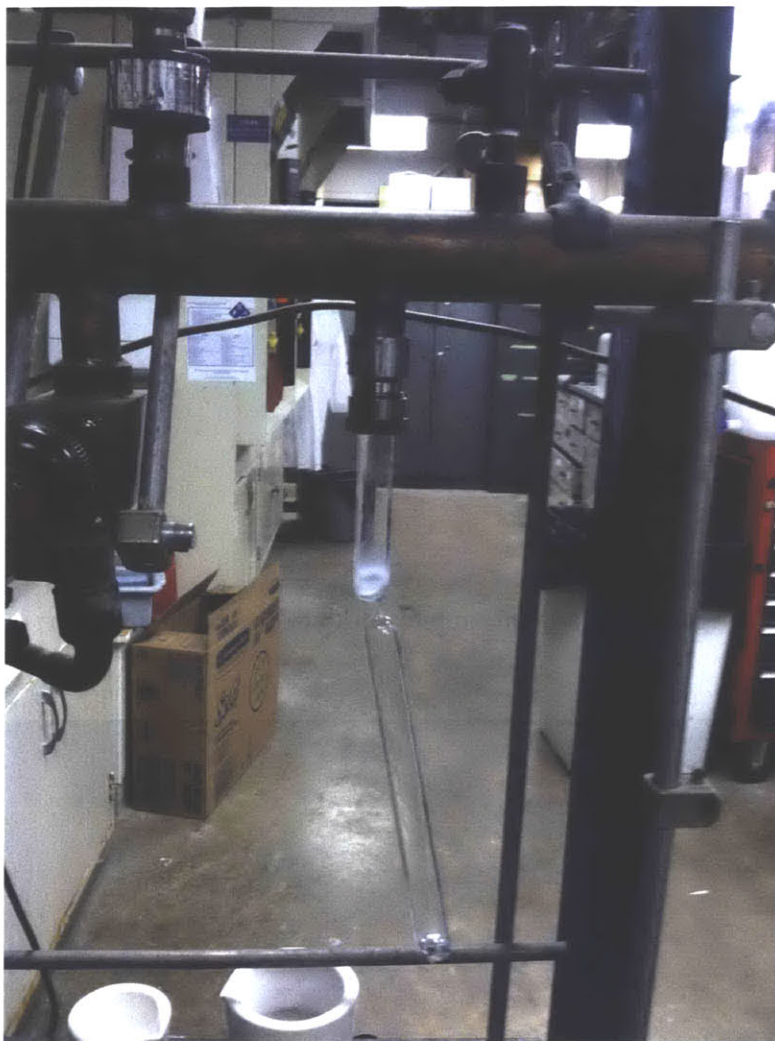


Figure 3.2.8: Image of the necked tube sitting under vacuum

Finally, the tube is heated at the necked region with a smaller flame and further away from the flame tip. The heat is spread around the neck of the tube as much as possible to avoid a heat

concentration. If a heat concentration is formed, the quartz tube starts getting soft in that region and due to the low pressure on the inside of the tube, a reverse bubble is formed at the heated spot where the tube starts to cave into itself, eventually popping to form a large hole, loosing vacuum and argon, which then cannot be repaired.

With the flame spread evenly, the necked region heats up and gently falls under gravity as it seals. Once the bottom end of the tube is separated away, the flame is maintained at the top of the separated tube for a few more seconds to flame-polish and thicken the seal.



Figure 3.2.9: Image of the necked region being heated

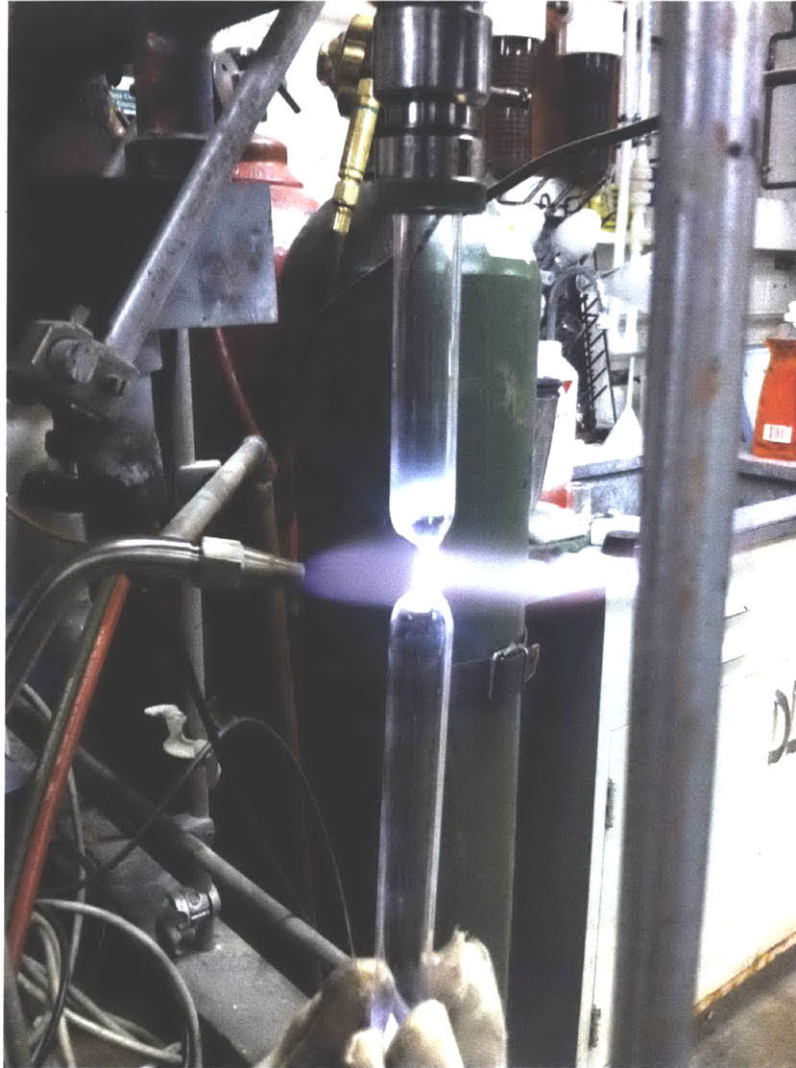


Figure 3.2.10: The necked area melts much quicker and is stretched under gravity

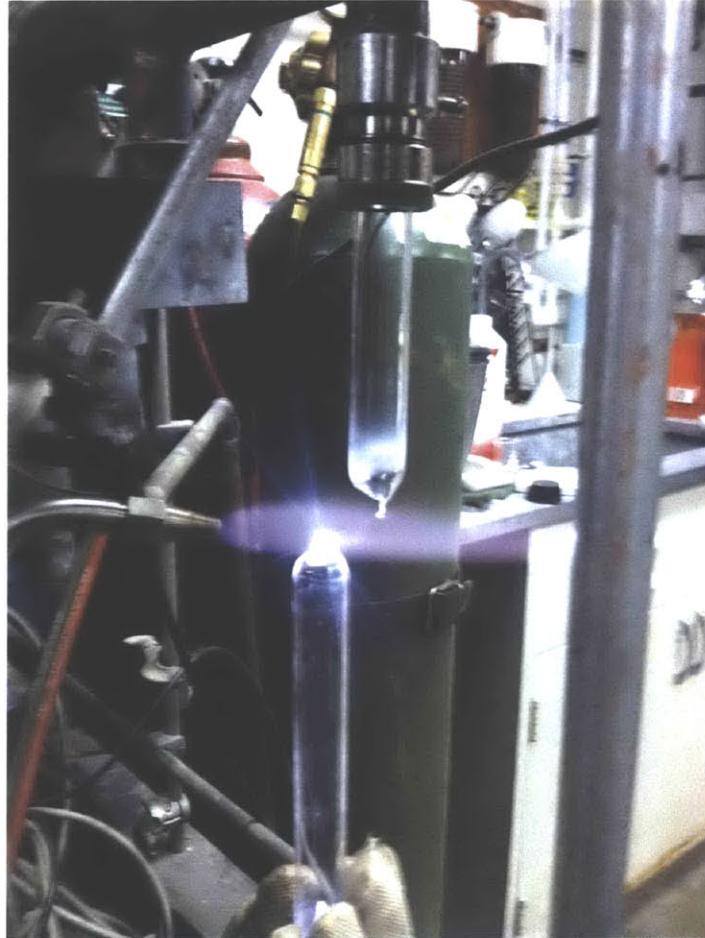


Figure 3.2.11: The bottom half of the tube containing the sample eventually breaks way as it is sealed off

The pressure gauge should be monitored as the tube separates out when sealing to ensure vacuum is not lost.

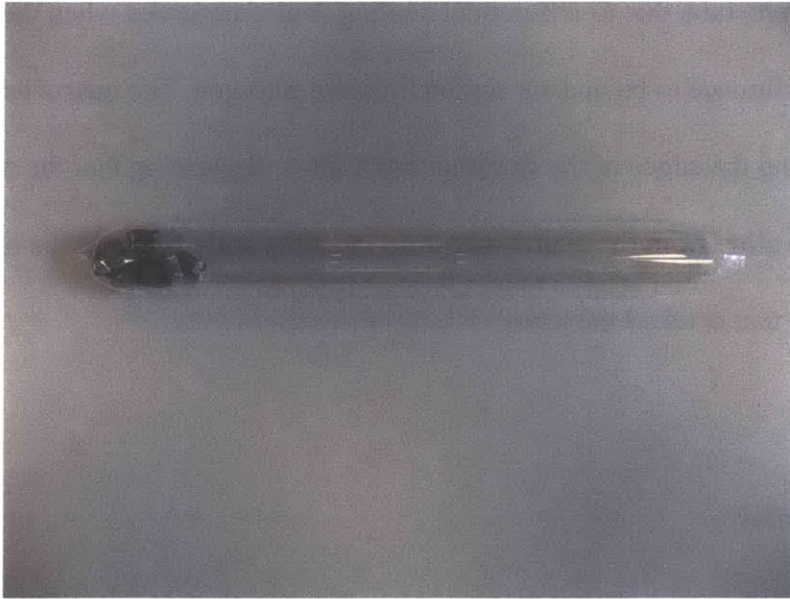


Figure 3.2.12: An image of a sealed tube containing samples

This method of sealing quartz tubes is a perfected method after several failed attempts with different sealing methods. Among the failed method was the the final vacuum sealing with only a very slight necking. With a necked area outer diameter of approximately 12mm, it was still too large and caused a the tube to pop under vacuum.

A different method that was also attempted but had only one successful attempt was to weld a 13mm tube into the open end of the 19mm tube where the neck would be. This 13mm tube was approximately 6in long and it was bent under heat at half distance. The free end of the 13mm tube was then connected to the vacuum system. The bent region was sufficiently necked and allowed greater room to spread the heat more evenly when vacuum sealing. However, every attempt but one of this method resulted in the tube cracking by itself just after the 13mm tube had been welded into the 19mm tube. This could be due to the differential cooling rate of both tubes, resulting in mechanical stresses applied onto both tubes, resulting in the crack. The

cracking of the quartz tube due to differential cooling is also observed when the samples were removed from the furnace to be quench cooled in liquid nitrogen. The quartz tube was often found to crack along the edges of the freezing liquid alloy, suggesting that the differential cooling rate of the alloy from the quartz tube with the alloy solidifying on the tube resulted in mechanical forces that cracked the tube.

3.3 Melting the Alloy

The vacuum sealed ampule containing the individual metals is placed in a box furnace programmed to ramp to a temperature of 800°C at a rate of 8°C/min and maintained at that temperature for between 6 to 10 hours. The ampule is then removed from the furnace while still at 800°C and quenched in liquid nitrogen. As the molten alloy solidifies and contracts, it pulls the wall of the ampule with it, causing the ampule to crack. The alloy is then retrieved and stored in a container with nitrogen vapor.

This method of protecting the alloy from exposure to air was found to still allow some amount of alloy oxidation. This was seen by observing the shiny silver color of the alloy when retrieving fade into a dark oxide that is present only on the outer surface of the alloy that starts forming as soon as it is placed in the container with nitrogen vapor. One possible solution would be to store the samples in a mini dewar that is able to hold liquid nitrogen.

Chapter 4

Experiments

Two main rubrics was selected to determine the quality of the alloys. They are the percentage hydrogen yield and the homogeneity of the alloys. Scanning Electron Microscope(SEM) images were taken of all the alloys produced to determine its homogeneity and a flowmeter was used to record hydrogen production when the alloys were reacted to determine the reaction completion.

4.1 Experimental Setup

Two different setups were used to determine the completion rate of the alloys. One setup was used for alloys with high reaction rates and the other for low reaction rates. The setup for high reaction rates is shown below in Figure 4.1.1. Due to the high hydrogen production rate, unlike most previous experiments done by the MIT RDG, reading measurements off a graduated cylinder was too inaccurate as it took too much time to take a reading by eye. Therefore, a flowmeter was used to record the hydrogen production rate. The gas produced was flowed through a tube sitting in a water bath to condense off any steam that might have been flowing through the tube. The flowmeter used was the Omega FMA-3709, which had a range of up to 5

liters per minute. The data produced was recorded via an Agilent Systems 34972A data acquisition system. The plot of hydrogen production rate was then integrated with respect to time on MATLAB to obtain the total amount of hydrogen produced.

The flowmeter could not be used to measure hydrogen production rate of alloys with low reaction rates as the hydrogen flow rate was too slow to be detected by the flowmeter. The hydrogen produced from the slow reaction was recorded by reading the volume measurements off a graduated cylinder by eye. However, because the expected yield is much higher than the total volume of the graduated cylinder itself, a separate syringe was connected to the gas bubble in the graduated cylinder to remove a known volume of hydrogen gas each time the graduated cylinder is nearly filled. The number total amount of hydrogen gas removed was recorded and added to the final reading on the graduated cylinder to obtain the total amount of hydrogen gas produced. When gas is syringed out, the tube connecting the syringe to the gas bubble in the graduated cylinder was pinched while the syringe was disconnected from the tube to release the gas that was pulled out. This was to ensure that the gas bubble in the measuring cylinder is maintained at a lower than atmospheric pressure so that the meniscus stays at the same place.

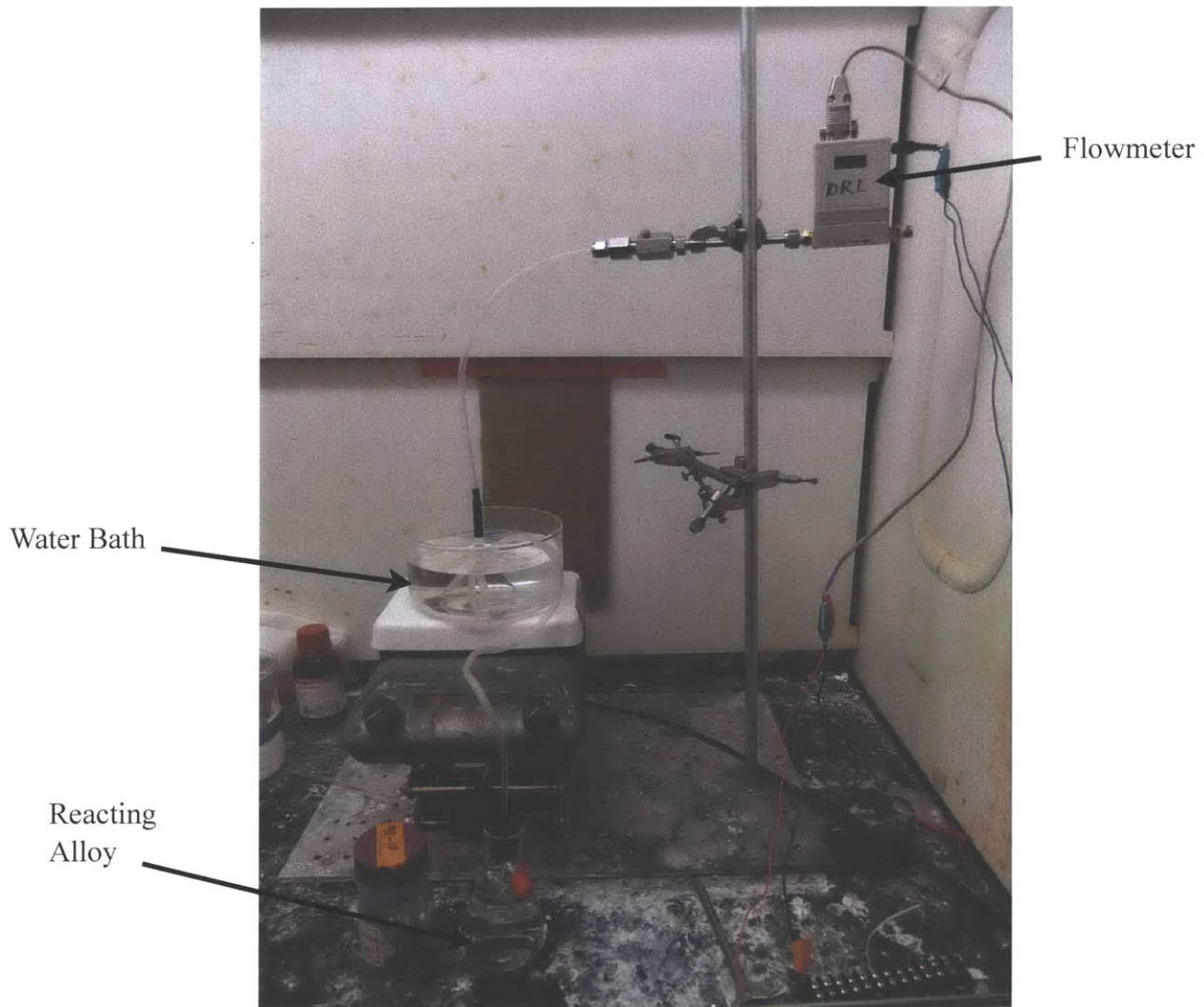


Figure 4.1.1: Experimental setup for high reaction rate alloys

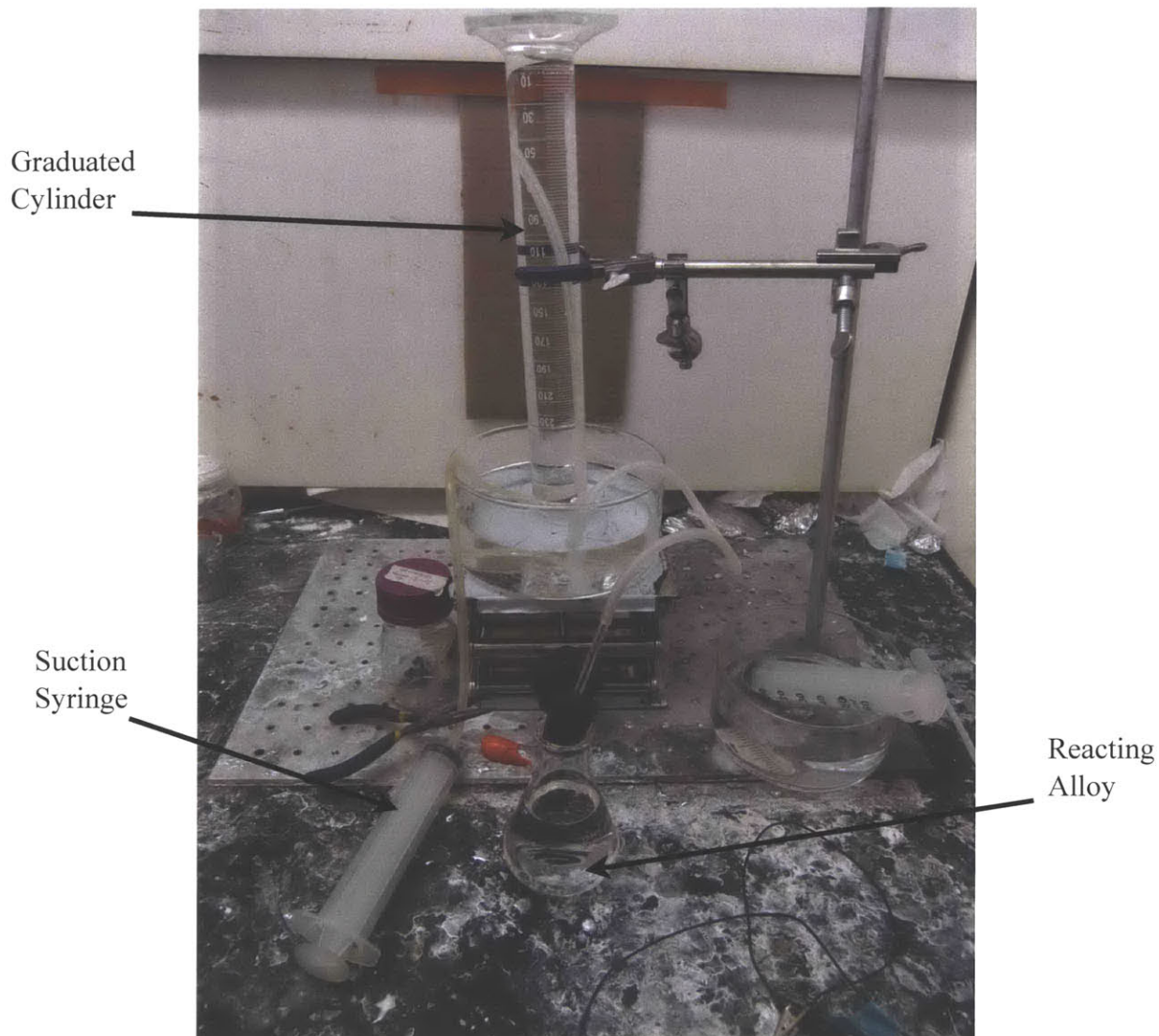


Figure 4.1.2: Experimental setup for low reaction rate alloys.

4.2 Alloy Compositions

The desired composition to be tested are as in the Table 4.2.2 below and the weighed sample of the desired alloys are in Table 4.2.3 below. The purity of each individual metals are as described in Table 4.2.1 below.

Metals	Source	Part Number	Purity
Aluminum	Sigma Aldrich	338788-50G	99.99%
Gallium	Gallium Source	12	99.99%
Indium	Sigma Aldrich	326607-10G	99.99%
Tin	Roto Metals	-	99.8%

Table 4.2.1: Purity of the individual metals used to make the alloy samples

% Aluminum	Mass Aluminum (g)	Mass Gallium (g)	Mass Indium (g)	Mass Tin (g)
50	1.5	1.02	0.33	0.15
70	2.1	0.612	0.198	0.09
90	2.7	0.204	0.066	0.03

Table 4.2.2: Calculated mass of individual metals needed

% Aluminum	Mass Aluminum (g)	Mass Gallium (g)	Mass Indium (g)	Mass Tin (g)
50	1.4944	1.0198	0.3304	0.1525
70	2.1058	0.6110	0.1960	0.0909
90	2.7315	0.2100	0.0675	0.0331

Table 4.2.3: Mass of the individual metals after weighing

4.3 Reaction Results

The 50-50 composition was reacted with the high power density setup and the flow rate measured using the flow meter was plot against time in Figure x.

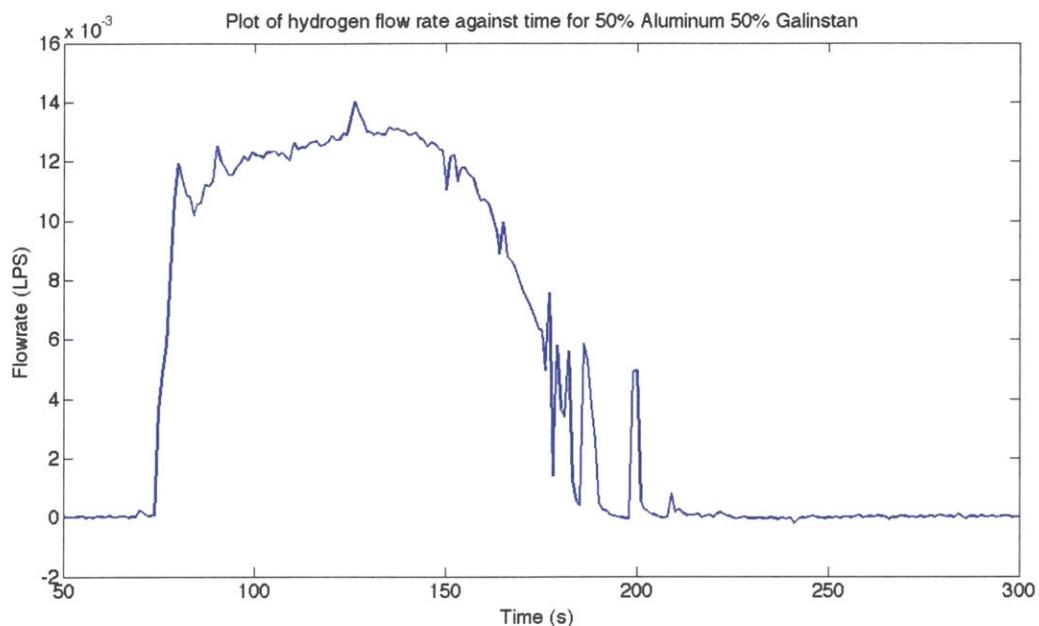


Figure 4.3.1: Hydrogen flow rate over time of the reaction of 50% aluminum 50% galinstan with water.

The volume of hydrogen gas produced from the remaining to samples were measured and all the hydrogen yields were compared with their theoretical yield value. The resulting reaction completion percentages are described in Table 4.3.1.

Composition	% Completion
50-50	66.25
70-30	69.65
90-10	70.85

Table 4.3.1: Reaction completion rates for different compositions

4.4 Reaction Observation

The reaction produced a dark grey suspension which is suspected to be unreacted aluminum that is released from the alloy as the water flows through the tiny veins to react with aluminum deeper in the alloy. An important observation made on the suspension was that as the reaction rate slowed down at the end, the reaction did not completely stop. The build up of suspended metal and aluminum hydroxide had trapped the hydrogen that was continuously being produced at the bottom. Occasionally, the build up of hydrogen pressure will push against the suspended metal and surface, causing a spike in hydrogen emission. This was allowed to continue in a separate beaker and left overnight. Eventually, the dark suspension had disappeared, leaving behind a white colored waste with a hint of grey, suggesting that the suspended aluminum had all eventually reacted with water to produce aluminum hydroxide, which is white in color.



Figure 4.4.1: Dark suspension during reaction

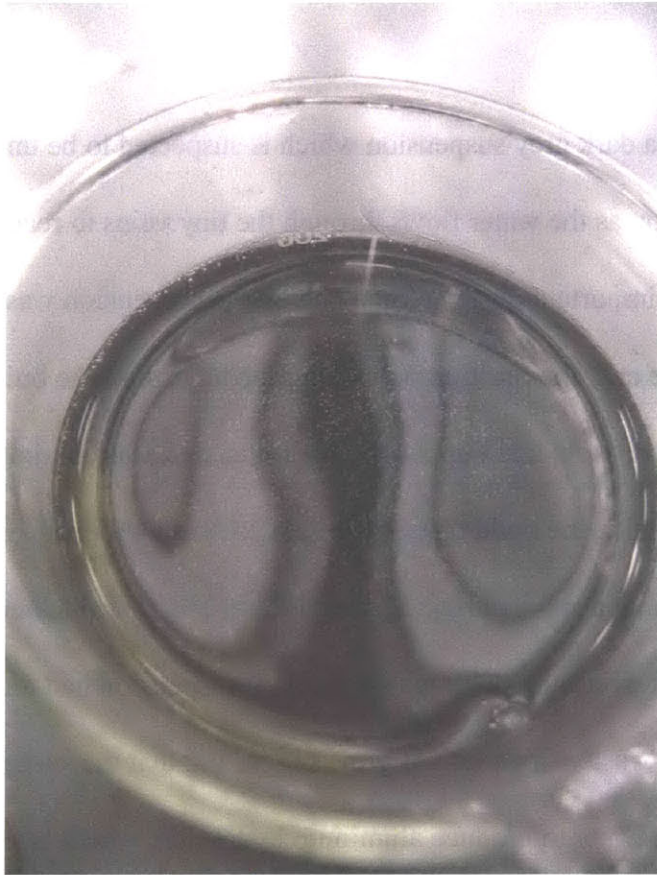


Figure 4.4.2: Tiny bubbles of hydrogen still being produced several hours after the reaction started

4.5 SEM Imaging of Alloys

The process of taking SEM images was when the alloys were first intentionally introduced to a non-inert environment. The alloys had to be removed from its container that was filled with nitrogen vapor to be placed on the SEM stage. In this time of approximately 30 to 45 seconds, the shiny silver faces of the alloy that was observed when the alloy was broken into pieces quickly started fading to a dusty grey color, showing the rapid oxidation of the alloy in air.

SEM images were taken of all the alloy compositions made and a select few images are attached in Figure 4.5.1, 4.5.2 and 4.5.3. It was found that the 50% aluminum 50% galinstan composition alloy had the best mixture of metals while the 90% aluminum 10% galinstan had the poorest mixture. The large pellets of aluminum was still very visible in the 90-10 composition, suggesting that the poor mixture could be what is resulting in a poor power density alloy.

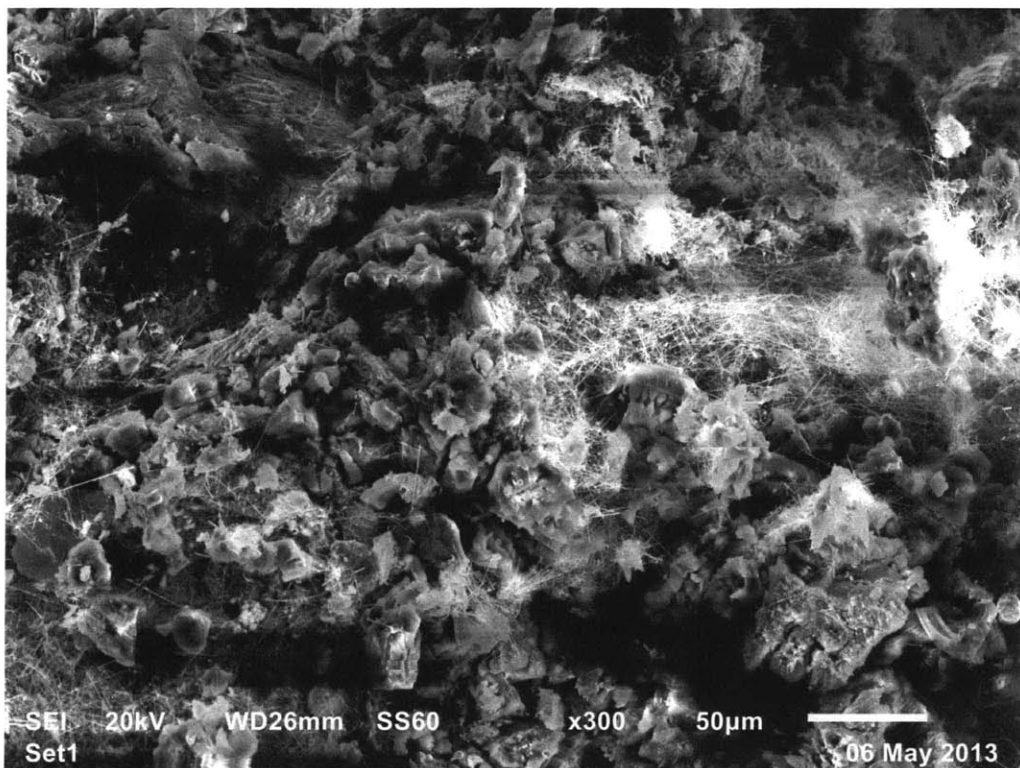


Figure 4.5.1: SEM image of a 50-50 composition

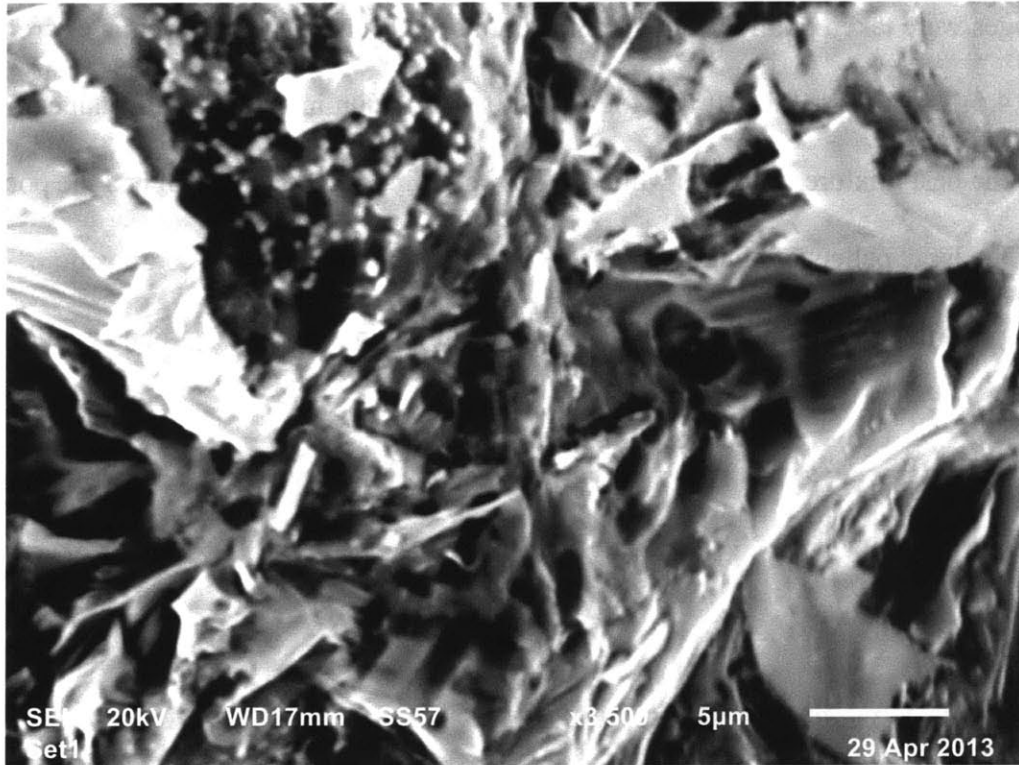


Figure 4.5.2: SEM image of a 70-30 composition

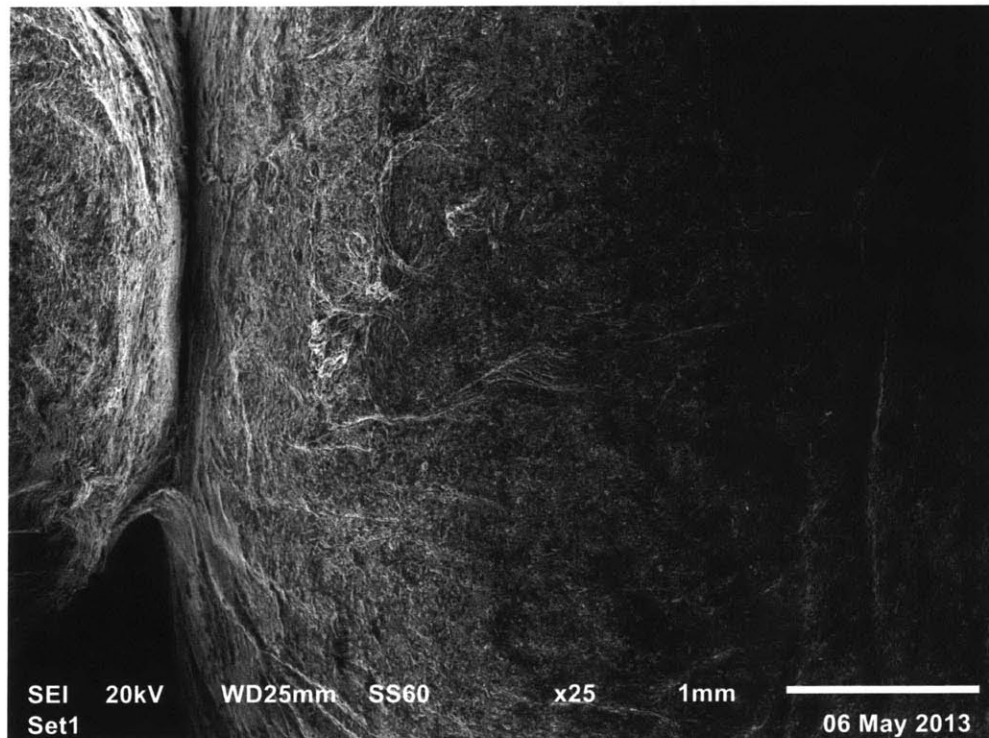


Figure 4.5.3: SEM image of a 90-10 composition showing distinct boundaries between the aluminum pellets

From Figure 4.5.3, it can be seen that the boundary of the aluminum pellets had not completely melted together in the furnace. The galinstan was spread around on the pellets without forming a clean homogenous alloy. Forming a higher quality alloy could require a much longer dwell period in the furnace at its peak temperature or even a furnace setup that could enable stirring of the alloy.

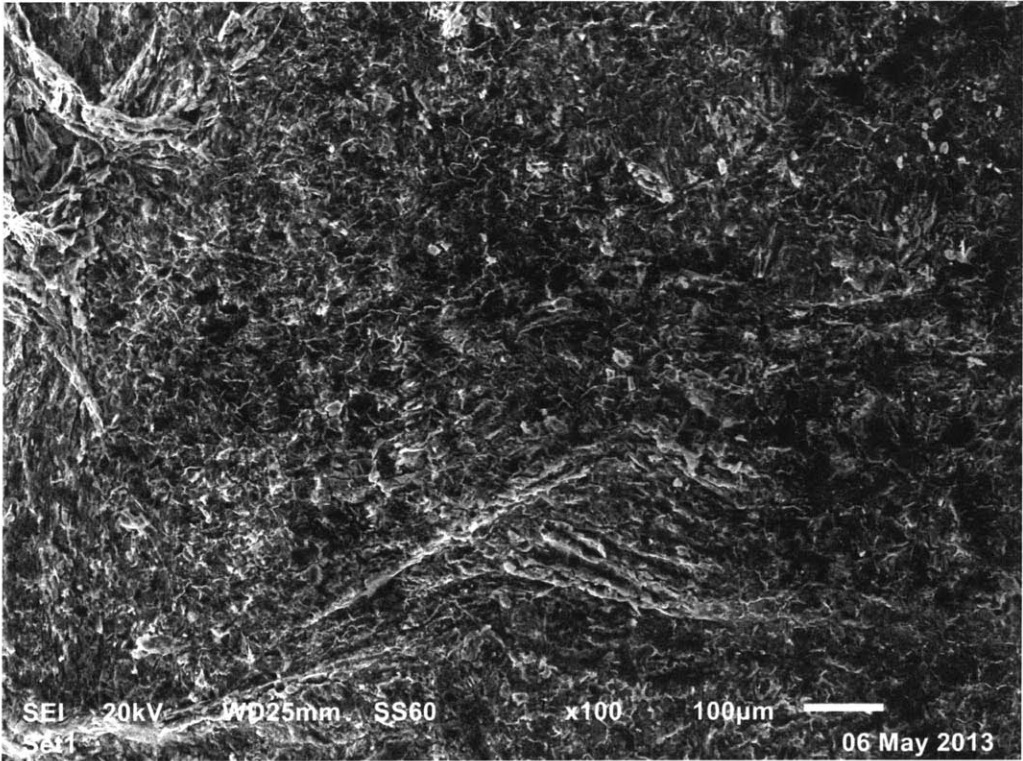


Figure 4.5.4: SEM image of a 90-10 composition. A closer look at the skin of the aluminum pellet covered in galinstan

Chapter 5

Discussion and Future Work

The reaction completion of the alloys of different compositions based on the quenching of sealed vacuum tubes were measured. It can be concluded that although alloys with higher aluminum composition have a high mass of aluminum to pellet volume ratio, they also have lower power densities. In order to extract a given amount of power, a higher reaction surface area is required for alloys with higher aluminum compositions. A system level design approach is needed, optimizing for total volume of system, to determine the composition of alloy that should be used and the number of alloy pellets needed to produce hydrogen at the desired rate. Further work should also be done to determine the consistency of the reaction completions as well as the power density of the alloys. In developing the system, special care should be given in the storage and handling of the alloys so as to minimize the oxidation of the alloys.

Chapter 6

System Design

The MIT Rapid Development Group (RDG) has developed a first prototype of a power system utilizing liquid gallium as a working fluid. In this system, gallium is percolated through a diffusion tank where it is enriched with aluminum before reacting with water in a separate reactor. The gallium is then pumped through the diffusion tank to get enriched with more aluminum. In this system, energy can be harnessed either through a fuel cell via a complimentary sodium chlorate dissociation tank or with a heat cycle from the heat produced in the aluminum-water reaction or utilizing both, the heat and the hydrogen in a fuel cell. While this system requires a limited amount of gallium, there are many complexities associated with this system. In its liquid form, gallium has a very high surface tension, making it a very difficult liquid to pump. The limits on the diffusion rate and reaction rate of aluminum in gallium also poses various geometric and volumetric constraints on the system, resulting in a large system. The high melting point of gallium, 30C, also causes freezing problems when the liquid gallium starts freezing in the lines, causing the system to get clogged.

A system that utilizes an aluminum alloy would eliminate many of these complications. There will be no limits imposed by the diffusion rates and the need for a diffusion tank will be

eliminated completely. Without the use of any working fluid, there will be no issues of fluids freezing in the lines, simplifying the plumbing of the system, and a pump for gallium will also be eliminated.

A first prototype of a full power system utilizing an alloy system was developed by the RDG in the Fall of 2012. The alloy system prototype involved dropping fuel into a steam of water, which would require each unit of alloy to be in a consistent form factor. This system helped reveal the main areas that require further work to produce a fully developed power system. Those areas are the alloy to be used in the system, fuel feed mechanism, control of system power output, as well as waste management. Further work have already been done in managing non-metallic waste by the RDG on the pumped gallium system that can be applied to the alloy power system and this thesis has advanced the knowledge required to produce an alloy composition with high hydrogen yield.

Based on the alloys presented in this thesis, an alternative alloy system prototype would be to feed water into a reservoir of alloy fuel. This poses a danger if the water feed starts leaking uncontrollably. The leaking water could produce a dangerously large volume of hydrogen. A solution to this problem would be to instead feed water from a water tank, where there is a limited amount of water stored. This tank can be replenished intermittently. Further safety precautions can be implemented using pressure release valves that will vent all excess hydrogen produced in the system.

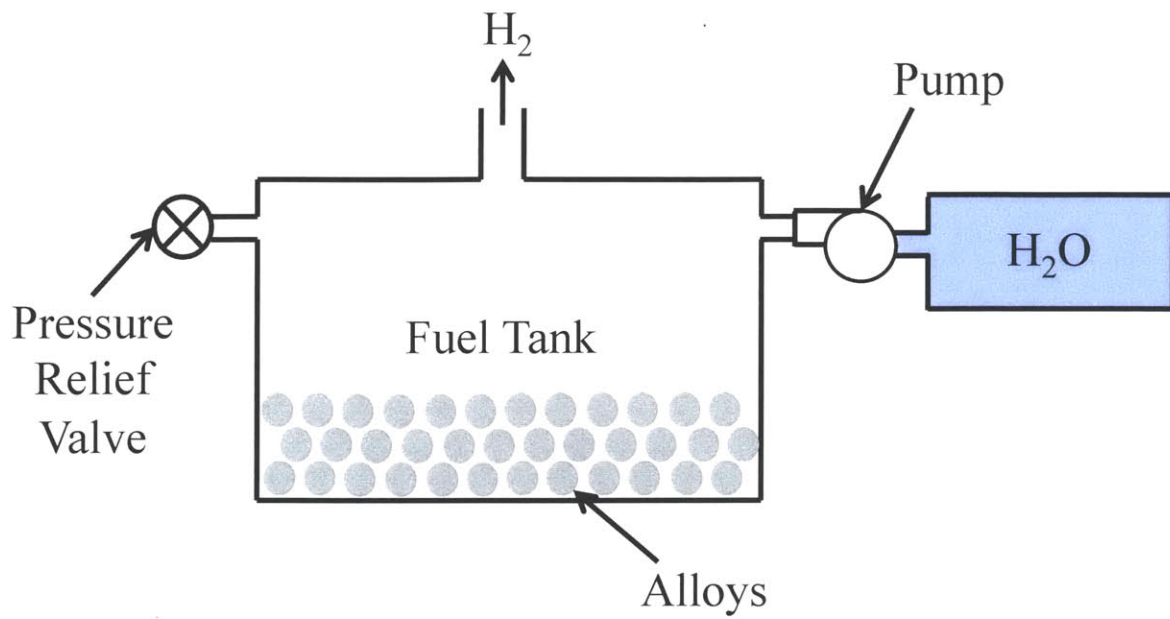


Figure 6.1.1: Diagram of a potential system layout

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