Laboratory Demonstration of Aluminum Metal Extraction from a Lunar Highland Simulant using Electrochemistry

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ABSTRACT

The exploitation of lunar resources will significantly reduce the need for transporting essential supplies to support an extended lunar presence. This research project focuses on the extraction of metals, particularly aluminum, silicon and oxygen from lunar resources. We present the design and development process of a bench-scale reactor designed to selectively extract these elements from their respective oxides. So far, we have successfully designed and constructed a bench-scale reactor capable of electrochemically reducing alumina into aluminum. The reactor comprises several components, including a kiln-type furnace, a stainless-steel vessel, a lid with multiple inlets for electrochemical components, an alumina crucible, a set of electrodes, and inlets and outlets for gas flow. Our current efforts involve standardizing the reaction process and analyzing the resulting products using various analytical techniques. We further aim to assess the performance of a suite of lunar-manufacturable aluminum alloys through electron beam (EB) additive manufacturing, a vacuum-based technology that is ideally suited for 3D printing applications on the lunar surface.

INTRODUCTION

As missions from several countries to the Moon proceed apace, a strong representation will be forthcoming from the public and private sector. As such, there is a strong interest in developing business opportunities on the Moon to maintain a sustainable human presence there. This will require the exploitation of lunar resources, which will reduce the transport required to support an extended human presence. There are several resources on the Moon that are plentiful, accessible, and commercially useful. Although it is difficult to identify a single lunar resource that will be sufficiently valuable to drive a lunar resource extraction industry on its own, there are abundant raw materials that have a potential economic interest. The most promising lunar resources have been classified in the following categories (Crawford 2015):

1. Solar wind implanted volatiles
2. Rare earth elements
3. Water
4. Oxygen
5. Silicon
6. Metals

In this project we’re focusing on the latter three: metals, silicon, and oxygen.

SELECTION OF ELEMENTS FOR EXTRACTION

The lunar mineralogical composition varies across its surface. Two major areas have been defined based on the naked eye and instrumental observation of the Moon. Lunar Maria (darker to the naked eye) are lightly cratered areas formed from ancient volcanic eruptions on the Moon’s surface. Lunar Maria is primarily composed of basaltic rock containing minerals like pyroxene, anorthite-rich plagioclase, olivine, ilmenite, and glass. The Lunar Highlands (brighter to the naked eye) are a representative area of the Moon’s early crust, formed from the crystallization of a primitive magma ocean during the formation of the Moon. The Lunar Highlands have been shaped by meteoritic impacts making them heavily cratered areas that contain excavated soils from the Moon’s early crust. In contrast to the Lunar Maria, the Lunar Highlands are predominantly composed of anorthositic rocks, primarily anorthite, a calcium-rich plagioclase feldspar. While pyroxene and olivine can also be present in the Lunar Highlands, they are less common than in the Lunar Maria. Most of the lunar surface consists of Lunar Highlands and in total, considering both the Lunar Maria and Highlands, most of the lunar crust consists of 83% anorthositic rocks (Franknoi, et al. 2017). Studies have shown that lunar plagioclase feldespar is composed of roughly 95% anorthite ($CaAl_2Si_2O_8$) and 5% albite ($NaAlSi_3O_8$). As shown by the chemical formula, one of the most abundant minerals on the lunar crust, anorthite, presents itself as an abundant source of silicon and aluminum.

Aluminum has always been a key base element of many successful alloys/materials used in the aerospace engineering industry. It has a good weight to strength ratio, has a relatively low cost, is resistant to UV damage and has a high resistance to corrosion, making it widely useful for different aircraft parts. The development of techniques for its extraction will undoubtedly be essential for the future of a sustained presence on the moon, in which many metallic parts will be manufactured with this element. Silicon will also be useful in the future of space exploration. Silicone, a synthetic material made of polymers that has a chemical structure based on chains of alternate silicon and oxygen atoms has a broad history in the aerospace sector. It is a material that remains stable in extreme environments, whether hot or cold, and maintains its properties as temperatures fluctuate. This element could also be extremely useful for the manufacture of solar panels which would allow for a sustained human presence to obtain energy from the sun. Also, the mix of silicon and aluminum produces silumin alloys (having 3-25% Si). This material allows the production of structural materials with high heat resistance, low thermal expansion, good strength, and wear resistance for use on the Moon.

CHEMICAL PROCESSING ON THE MOON

On Earth, aluminum extraction is mainly carried out by pre-processing ores with high aluminum content (mainly Bauxite) followed by an electrochemical process. The pre-processing is known as the Bayer process and is carried out in 4 stages. The first stage consists of mixing the bauxite with sodium hydroxide (NaOH) to form a slurry. The slurry is then digested at high temperatures. The resulting mixture is clarified using flocculants and the solid products are separated by precipitation. Finally, the products are calcined to obtain alumina (Al_2O_3). The obtained alumina is electrochemically transformed into pure aluminum by the Hall-Héroult process. In this process, the electrolytic cell consists of a couple of carbon (coke) anodes and cryolite (Na_3AlF_6) as the electrolyte. The alumina is usually supplied from a hopper located on the top of the electrolytic cell. A couple of coke anodes are placed next to the alumina hopper and are submerged on the cryolite. The transformation occurs at the cathode, a large coke block located at the bottom of the reactor.

Unfortunately, these processes, so typical and effective on Earth, are not suitable for the Moon. The Bayer process has been optimized to use bauxite as feedstock, but aluminum on the Moon is available in the form of anorthite. The Bayer process involves a clarification and filtration step, and a continuous flocculant supply on the Moon would be challenging. Regarding the Hall-Héroult process, using cryolite would be unsuitable due to the scarcity of this mineral and several of its elements on the Moon. The development of electrodes for this process would be another issue to consider due to the absence of lunar carbon.

To fill the gaps in the pre-processing of the samples by the Bayer process, we decided to try an alternative technique based on the mining operations for the treatment of anorthosite at Qaqortorsuaq, Greenland. We were able to obtain silica with a purity of about 92.72% and alumina with a purity of 85.66%. The results from that work will be presented at this conference as well (Thibodeau, et al. 2024). In the present paper we are presenting the next step of this process, which consists of the electrochemical reduction of the silicon and aluminum oxides previously obtained.

THE SELECTION OF AN ELECTROCHEMICAL MODEL

We decided to try a different electrochemical processing approach based on the Fray Farthing Chen (FFC) Cambridge Process (Chen, et al. 2000) to solve the issues associated to the Hall-Héroult Process. Most of the research on the FFC technique has been conducted on rutile (TiO_2) reduction. In this technique, an anode made of either carbon (usually graphite), or an inert material (usually doped tin oxide) is used in conjunction with a cathode usually made with the metallic oxide to be reduced. The procedure is run in a temperature interval between 900 – 1100 °C and the electrodes are submerged in molten $CaCl_2$, the electrolyte. This process has great advantages (Ellery, et al. 2022) for its implementation on the Moon:

- The use of calcium chloride ($CaCl_2$) as an electrolyte makes more sense considering the wide availability of calcium in lunar anorthite. Calcium chloride is also a byproduct of the pre-processing technique we have proposed (Thibodeau, et al. 2024).
- The temperature interval used in this process is relatively low compared to other molten electrolysis techniques.
- The use of an inert anode facilitates the direct recovery of oxygen from the reduction of metallic oxides.
- This process has proven to be highly versatile and robust, as it has been proven successful in the reduction of a wide variety of metal oxides.

We will proceed to discuss all the technical aspects considered for the design and fabrication of a bench-scale reactor capable of performing this reaction on the Moon.

DESIGN OF A BENCH-SCALE REACTOR

Previous experiments have been conducted (Lomax, et al. 2020) where a lunar simulant (JSC-2A) was used. The products obtained were a mixture of metallic alloys and oxygen. They were able to demonstrate the removal of 96% of oxygen from the lunar simulant and the presence of three main alloys on the reduced product:

- An Al/Fe alloy, usually with Si inclusions
- A Fe/Si alloy, usually with Ti and Al inclusions
- A Ca/Si/Al alloy, usually with Mg inclusions

The complexity of the unbeneficiated lunar regolith simulant used in that work made it difficult to identify the minor phases present in the resulting product. We are interested in further studying the metals and alloys that can be obtained from lunar samples. The potential for metal/alloy separation and refining is exciting.

In this study we are presenting the design of a bench scale reactor capable of working with 4 different types of samples:

- A Lunar Highlands Simulant (LHS-1) unbeneficiated
- A Lunar Highlands Simulant (LHS-1) beneficiated by magnetization
- Powdered alumina
- Solid alumina blocks

Our intention is to perform the reaction with an unbeneficiated LHS-1 simulant to make a comparison with the previously obtained metallic products from the JSC-2A simulant. Carrying out this comparative is essential to understand the possible extractable alloys from lunar regolith, since the JSC-2A previously studied represents material from the lunar mare, while the LHS-1 we are using represents material from the lunar highlands. Testing on a beneficiated sample is also worthwhile. We have previously observed that the use of magnetic processing removes a significant fraction of iron from the samples, thereby favoring the production of aluminum alloys. Finally, we will perform the reaction on previously extracted alumina by means of a preprocessing step (Thibodeau, et al. 2024). This would allow us to demonstrate the selective extraction of metallic aluminum on the moon. We will now discuss the different components that we have designed and used to build a reactor capable of performing these reactions.

Heating device

The FFC process is carried out at relatively high temperatures. Therefore, it was essential to devise a heating unit. The main requirements that had to be met were:

- To be capable of reaching the process temperatures of 900 − 1100 °C.
- To be able to record the temperature in the unit.
- To be able to remain in operation for long periods of time.
- To have an access opening for the electrodes.
- To have a relatively compact size similar to a unit that could be tested on the Moon.

Most commercially available furnaces are usually square in shape with an opening in front of the unit. This type of shape is impractical for the desired experiments as a front opening complicates the insertion of the electrodes. Electrodes are traditionally placed at the top of electrochemical reactors, due to the usual elongated shape of the electrodes. Although it would be possible to use electrodes with a different form or to try to design a reactor with an alternative shape, we opted to adapt the furnace instead. Additionally, we opted for a type of furnace that could enclose the reactor. As a result, all the energy is applied into the reactor, avoiding the potential heat loss that sometimes takes place in the corners of conventional furnaces. The type of furnace that we selected was a kiln type furnace, as it met the previously mentioned characteristics.

The enclosure for the reactor is made with a heat dissipation ceramic surrounded with the heating element, comprised of a coil-shaped high-temperature resistant alloy. To record the temperature inside the device, the furnace is equipped with a type K thermocouple capable of recording temperatures in the range of 20 to 2000 °C. Figure 1 shows the elements of our furnace.

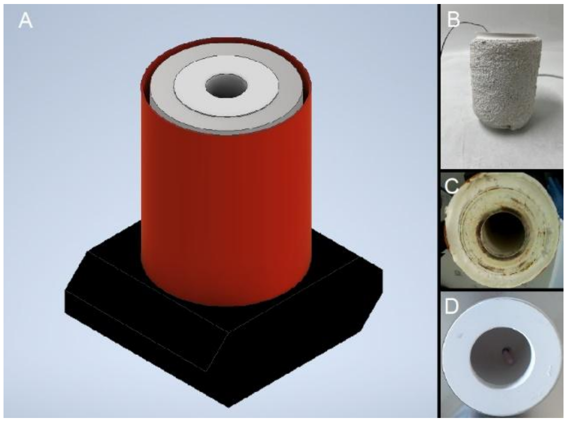

Figure 1. Furnace schematic and elements. A) shows a basic illustration of our furnace. Shown in black is the furnace base on which the power switch and temperature controller are located. A heat shield is shown in red. The metallic structure of the furnace can be seen in light gray. The heat dissipation chamber made of ceramic is shown in white. B, C and D) show the physical heat dissipation chamber from different perspectives. In B) the heating unit consisting of a ceramic cylinder surrounded by a high-temperature alloy wire in a coil shape is shown. The heating unit and wire were recoated to protect the other furnace elements from direct overheating. C) shows the heating chamber from above. It can be seen that the heating unit was wrapped with ceramic fiber. Finally, the interior of the chamber is shown in D). A small protrusion can be seen inside. This protrusion is the type K thermocouple tip.

Electrochemical reactor, reagents, and electrodes

Given the working temperatures, a container capable of withstanding 1100 °C is required. Graphite crucibles are usually used in kiln-type smelting furnaces. The shape of our reactor is inspired by one of these crucibles. However, our vessel is made from a more resistant material: (stainless steel grade 304). Stainless steel was chosen because graphite crucibles gradually flake off while being manipulated. We also wanted to prevent any oxidation and formation of CO/CO_2 at the working temperatures. It has also been found that graphite is not a suitable material to work with Si-Al melts (Hoseinpur & Safarian, 2020) because these elements are able to infiltrate the graphite forming aluminum carbide (Al_4C_3). The formation of this carbide causes expansion in graphite, which would cause a volume increase in certain sections of our container causing fractures in it. Figure 2 shows the stainless steel vessel for the reactor, as well as the different steps from design to the manufacturing process.

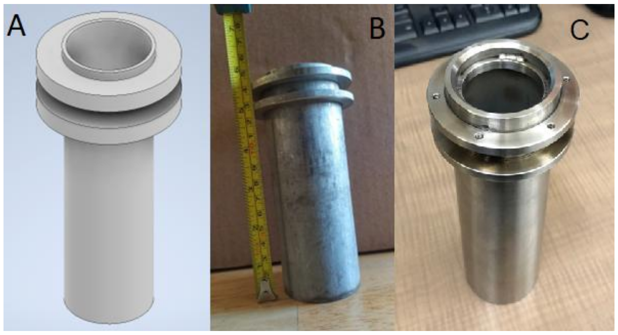

Figure 2. Stainless steel vessel for the electrochemical reactor. A) shows an image of the vessel design, B) shows the vessel being manufactured, and C) shows the final vessel after polishing, drilling, and welding. It is shown that the vessel is made of a stainless steel tube with a uniform diameter surrounded by two larger diameter stainless steel flanges on the top. There is a gap between the two flanges, which is designed to allow vessel removal (using tongs) from the furnace if necessary.

To preserve the integrity of the reactor during the electrolysis, a second crucible is placed inside the stainless steel vessel. This crucible (Figure 3) is made of >99% alumina, which was selected for its high-temperature resistance. The alumina crucible dimensions were selected with the intention of covering as much of the stainless steel vessel as possible but leaving space for handling the electrodes. This would allow the electrodes to be placed inside the reactor while the electrolyte is in a solid state. Once the temperature has reached the electrolyte melting temperature, the electrodes can be lowered and immersed in the electrolyte for the reaction to take place.

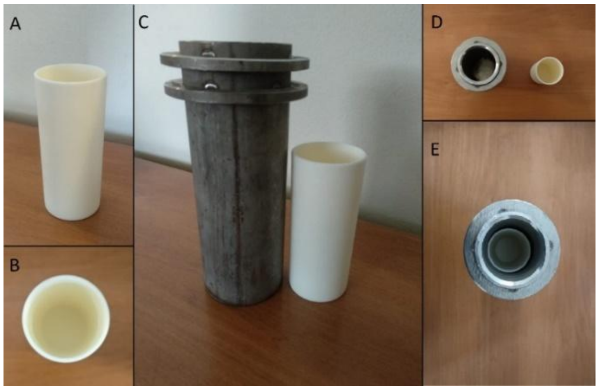

Figure 3. Alumina crucible for the reaction. A & B) show the crucible from different angles. A) shows it from the front while B shows the crucible viewed from above. C) shows a size comparison between the alumina crucible and the stainless steel vessel. It is clearly visible that a free space is left at the top of the alumina crucible where the electrodes can be placed while the electrolyte is in solid state. D & E) show a comparison between the diameter of the alumina crucible and the stainless steel vessel. D) shows a top view of them where it is shown that the diameter of the alumina crucible is slightly smaller than the stainless steel vessel. E) shows the alumina crucible introduced inside the stainless steel vessel. It can be seen there is just a small gap between them.

The electrolyte is really important for the FFC process reaction. Two electrolytes have been proposed for this process: barium chloride ($BaCl_2$) and $CaCl_2$. We decided to use the latter as the versatility of calcium to oxidize and reduce itself during the FFC process has been demonstrated and it is fundamental for the FFC process. Calcium oxide (CaO) and elemental calcium (Ca) are produced during the reaction, it is also important that the electrolyte can dissolve both substances, otherwise they could solidify around the electrodes interrupting the reaction. $CaCl_2$ also satisfies this requirement since both substances have good solubility in it. As $CaCl_2$ meets all the previous conditions, it is ideal for the reaction (Mohandas & Fray, 2004). Calcium chloride is highly hygroscopic and is commercially available in various hydration states: anhydrous, dihydrate, tetrahydrate and hexahydrate. It could be best to use the anhydrous option, however it is very costly to acquire. We opted for a cost-effective option by selecting calcium chloride dihydrate ($CaCl_2 \cdot 2H_2O$), a slightly hydrated calcium chloride. Fray and Chen, creators of the FFC process, also preferred $CaCl_2 \cdot 2H_2O$ over the anhydrous option, as it led them to obtain higher purity. We also decided to follow their heating procedure (Chen & Fray, 2002), which consisted of increasing the temperature from 20 to 300 °C at a rate of 300 °C/h, holding at 300 °C for 6 h or longer, then heating it under argon to 700°C, and maintaining the temperature for a couple of hours before the reaction.

The electrodes were carefully selected for the reaction. For the initial tests we're using a rod-shaped graphite anode due to its low cost and good efficiency. Once the process approaches standardization, we intend to improve the experiments using an inert electrode that will allow the recovery of oxygen, instead of carbon oxides. As for the cathode material, we are using molybdenum (Mo). This material was selected because some studies have shown its effectiveness as an electrode due to the low capability of Mo to form alloys with Al (Kadowaki, et al. 2018). Different shaped electrodes (Figure 4) are being utilized and their use depends mostly on the physical properties of the reagent. Rod-type electrodes have proven to be more useful for reagents in the shape of a compact solid block, while the use of basket-type electrodes has proven to be more useful for reagents in a powdered state. It is worth mentioning that the dimensions of the rod electrode were designed to allow the alumina pellets to be introduced into the electrode through a hole on the center of the pellets. On the other hand, for the basket electrode, a mesh size smaller than the reagent powder sieve was chosen.

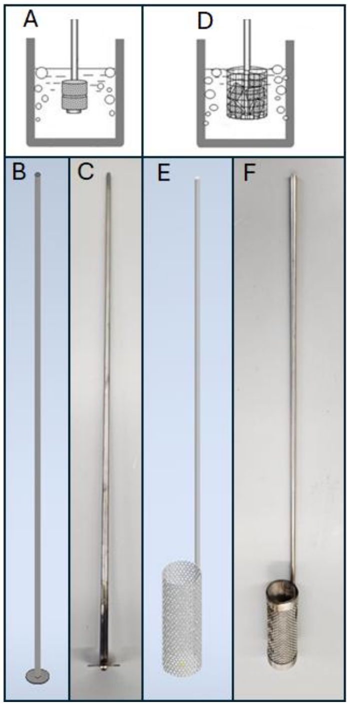

Figure 4 Different types of electrodes for the electrochemistry process. Figures A) and D) are modified theoretical representations of two electrode shapes proposed by (Chen, 2013) for the electrochemical process. A) shows a rod-type electrode where the reagents are solid pellets with a hole in their center which allows them to be inserted into the electrode. D) shows a basket-type electrode where the reagents are pellets placed on the inside of the basket. A), B), and C) represent our manufacturing process for the rod-type electrode. The process starts from the theoretical representation A), going through the design phase B), finishing with the manufactured electrode C). The dark area shown on the finished rod-type electrode C) was the product of welding the rod and the disk where the reagent is placed. D), E), and F) represent our manufacturing process for the basket-type electrode. The process starts from the theoretical representation D), going through the design phase E), finishing with the manufactured electrode F). It is shown that the manufactured version F), presents two solid sections in its basket, one located at the top and the other at the bottom. This allowed the basket to be properly welded to the rod without damaging the basket mesh.

A stainless steel lid (Figure 5) was designed for the reactor and made on site with five feedthroughs for the electrochemical cell. Each feedthrough has a fitting depending on the desired insertion. Two of the feedthroughs are used as gas inlet and outlet ports. Two of the feedthroughs are used for electrodes. The last feedthrough is used for a type K thermocouple to track the temperature inside the reactor. A high-temperature and electrical insulating seal is used on each of the fittings. A high-temperature O-ring type gasket is used at the juncture between the lid and the stainless steel vessel to prevent gas leakage. To prevent damage to the electrodes, the lid has room to accommodate a cooling system made by copper tubing and a recirculator.

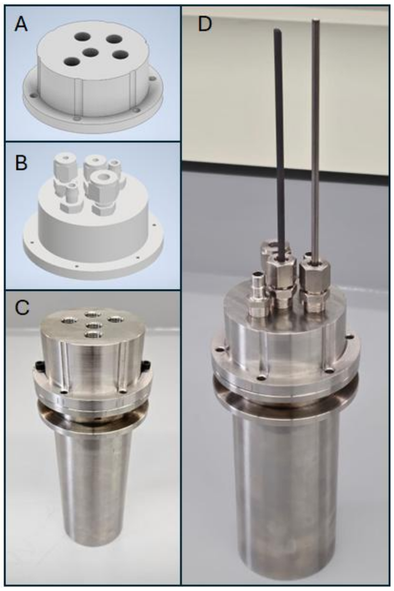

Figure 5. Stainless steel lid for the reactor. A) Shows an illustration of the stainless steel lid design. B) Shows a design where the fittings have been placed into the lid feedthroughs. C) Shows the feedthrough-exposed lid on top of the finished reactor. D) Shows the finished reactor (fittings included). The rods shown on D) are the electrodes already placed inside of the reactor.

Experimental procedure

The experiments are carried out by placing the alumina crucible inside the stainless steel vessel. The alumina crucible is filled with 150 g of pre-dried $CaCl_2$ into the crucible. Roughly, a 5 g sample is placed into the cathode, which is then placed in the stainless steel lid along with the other components. The cell is sealed with the electrodes and thermocouple all sitting above the solid electrolyte line. Then, the temperature is ramped up to 900 °C. Once this temperature is reached, the cathode, anode, and thermocouple are then lowered into the molten electrolyte. The current is then applied into the cell at 0.25 A, ramping it up until a current of 4 A is achieved. Reduction is allowed to take place for a period of approximately 12 h. Following electrolysis, the cathode with the product is removed from the electrolyte and is left to cool to room temperature under an Ar atmosphere. Once the sample reaches room temperature, it is washed with distilled water to dissolve the salt and separate the product from the electrode. Once the product is separated from the electrode, it is placed in a funnel with a paper filter and washed with distilled water until all the remaining salt is removed (a conductivity meter is used to verify this). The washed product is then dried for 2 hours at 250 °C.

Considerations on the physical properties of the reagents

Even though Chen and Frey were able to demonstrate the electrochemical reduction of different metals using the FFC Process (Chen, et al. 2000), most of their efforts were focused into elaborating solid-state electrodes that could remain in a solid state before, during, and after the reaction. As a result, they could easily demonstrate the industrial scale extraction of elements such as titanium and tantalum. Both their oxides and their metallic form have extremely high melting points (TiO_2 = 1843 °C, Ta_2O_5 = 1872 °C), (Ti = 1668 °C, Ta = 3017 °C). The same principle can be applied to other elements extracted by means of this process such as zirconium and chromium (ZrO_2 = 2715 °C, Cr_2O_3 = 2435 °C), (Zr = 1852 °C, Cr = 1907 °C). Most of the oxides will remain solid in the temperature range used for the process (900 − 1100 °C). Processing oxides from elements such as boron, palladium, silver, cadmium, osmium, platinum, gold, thallium and lead is however more difficult if they are used on their own for the FFC process as they would melt as soon as an electrode made from any of these sole materials is immersed in the molten electrolyte at 900 − 1100 °C. A similar problem would exist for some elements after their electrochemical transformation into metals such as magnesium, calcium, gallium, cadmium, indium, tin, barium, thallium, lead, and aluminum (the object of our study) where the electrode would melt as soon as the oxides are transformed into their corresponding metal. Chen and Frey ingeniously solved both issues by making electrodes with powder containing a mixture of different oxides preventing thereby the melting of either the oxides or the metals by facilitating the formation of alloys between different substances (Chen & Fray, "Electro-deoxidation of metal oxides", 2001).

In this study we aimed to develop a device capable of processing both, a lunar simulant and alumina. The processing of the first case is straightforward as it has been widely studied through the FFC process. On the other hand, not much work has been done on cases where the material melts at the end of the reaction. The best example found for aluminum processing (Kadowaki, Katasho, Yasuda, & Nohira, 2018) concluded that a Mo box-type electrode allowed the chemical transformation of alumina to aluminum. Unfortunately, the electrode in question was not able to contain the metallic aluminum, which was poured to the bottom of the crucible, making it difficult to retrieve the metal produced.

In this project, we are taking into account that the electrode containing alumina (Al_2O_3 melting point = 2072 °C) would constantly lose mass and melt continuously according to the rate at which the oxides are transformed into their corresponding metal (Al melting point = 660 °C), causing the metal to either precipitate to the bottom of the reaction crucible or remain suspended in the electrolyte, depending on the ratio between the densities of the metal and the electrolyte. In this case the density of liquid aluminum is 2.3 g/cm^3 at temperatures between 950 and 1000 °C while the density of liquid $CaCl_2$ in the same temperature range is 2.0 g/cm^3. This temperature difference allows molten aluminum to separate properly from the electrolyte and settle at the bottom of the electrolysis cell. According to those considerations we are proposing the design of a cell (Figure 6) that would allow an adequate separation of the produced aluminum.

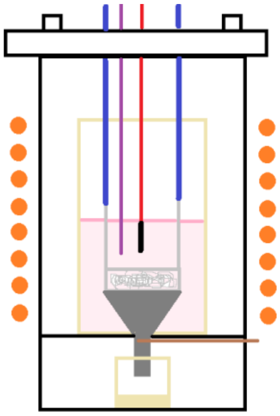

Figure 6. Proposed electrolysis cell. The image depicts a suggested design for an electrolysis cell that allows a proper separation of alumina. The heating mechanism is shown in orange. The reaction vessel with the lid for the electrodes is shown in black. The thermocouple is

shown in purple. The graphite anode is shown in red. The cathode is shown in blue, and it's made with a Mo basket containing a powdered sample (light gray). Two alumina crucibles are shown in yellow. A separation funnel (dark gray) with an actuation mechanism is shown in brown. That actuation mechanism would activate as soon as the temperature of the reaction reaches 775 °C, i.e., the temperature at which the calcium chloride would solidify, while the aluminum remains in a liquid state (melting point = 660 °C).

CONCLUSION

We have successfully designed and built a bench-scale reactor for the selective extraction of aluminum from lunar regolith. The designed reactor is composed of a kiln-type smelting furnace capable of reaching the necessary temperatures for the reaction (900 – 1100 °C), a stainless steel vessel, a stainless steel lid with several inlets for the electrochemical components, an aluminum crucible to contain the electrolyte ($CaCl_2$), a carbon (graphite) anode, a molybdenum cathode, and inlets and outlets for gas flow to sustain an inert atmosphere (Ar). We have successfully produced metallic aluminum using our reactor. We are currently in the process of standardizing the reaction and analyzing the reaction products through different analytical techniques. We further aim to test the performance of a suite of lunar-manufacturable aluminum alloys via electron beam (EB) additive manufacturing, a vacuum-based technology ideally suited for 3D printing on the Moon.

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