FROM LUNAR REGOLITH TO ALUMINUM ADDITIVE MANUFACTURING: BENCH-SCALE DEMONSTRATION OF METALLIC ALUMINUM PRODUCTION FROM A HIGHLAND SIMULANT AND ITS UTILIZATION AS FEEDSTOCK FOR 3D PRINTING ON THE MOON.

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Introduction: On the eve of humankind's return to the Moon, the possibility of sustaining a human presence has been raised. This will probably require the exploitation of lunar resources, which will reduce the transport required to support an extended human presence. In this project we are focusing on the selective extraction of Aluminum (Al), a plentiful, accessible, and commercially useful material on the Moon. Additive manufacturing (AM) has been booming as a highly-versatile manufacturing process. We suggest Al AM has the potential to be used on the Moon. In this work we're demonstrating different steps for this process: the selective extraction of alumina (Al_2O_3) and silica from a lunar simulant, the electrochemical transformation of Al_2O_3 into Al, and the processing of the Al into wire feedstock for a lunar 3D printer.

On Earth, aluminum extraction is mainly carried out by the Bayer and Hall-Heroult processes. Unfortunately, these processes 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, where 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.

Acid leaching pre-processing: To fill the gaps in the pre-processing of the samples, we to tried an alternative technique [1] based on hydrochloric acid (HCl) leaching (Figure 1). We started by beneficiating a lunar highland simulant (LHS-1) using a crude magnetic separation step. The beneficiated simulant was leached in a hot (94°C) concentrated HCl solution (38%). After a four-hour leach, the supernatant solution was separated from the remaining precipitate using a centrifuge. The solids were dried at 150°C for an hour and a sample was sent for analysis to analyze for the presence of silica. The supernatant solution was then sparged with pure HCl gas. This process forced the precipitation of dissolved aluminum chloride hexahydrate (ACH) crystals out of the solution. The precipitated ACH from the sparging step was separated from the remaining supernatant using a centrifuge. The ACH was processed with a two-step calcination process in a muffle furnace. The first calcination took place at a lower temperature (250°C) for two hours. The second calcination was performed at 800°C for two hours. The calcination of the ACH allows for the removal of any remnant water in the samples as well as decomposing the ACH into alumina. We were able to obtain silica with a purity of about 92.72% and Al_2O_3 with a purity of 85.66%.

Figure 1. Acid leaching of a lunar highlands simulant. The picture shows the three most important steps of the process. The picture on the left shows the magnetic beneficiation of the simulant. The picture on the center shows the resulting precipitate and supernatant solution after leaching. The picture on the right shows the sparging step to force the precipitation of ACH.

Electrochemical reduction: We designed and built a reactor to transform the produced alumina into metallic Al [2]. The electrochemical processing approach was based on the Fray Farthing Chen (FFC) Cambridge Process [3]. 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 Calcium Chloride ($CaCl_2$), the electrolyte. This process was selected due to is advantages for its implementation on the Moon [4]: First, calcium is widely available in lunar anorthite and $CaCl_2$ it's a byproduct of our pre-processing experiments. Second, the use of an inert anode facilitates the direct recovery of oxygen. Third, the process is highly versatile. It has been proven successful in the reduction of a wide variety of oxides.

The reactor that we built was comprised by the following elements (Figure 2): 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, a heat shield, a copper coil recirculation cooling system fot the lid, an alumina crucible to contain the electrolyte, the electrodes, and inlets and outlets for gas flow to sustain an inert atmosphere (Ar).

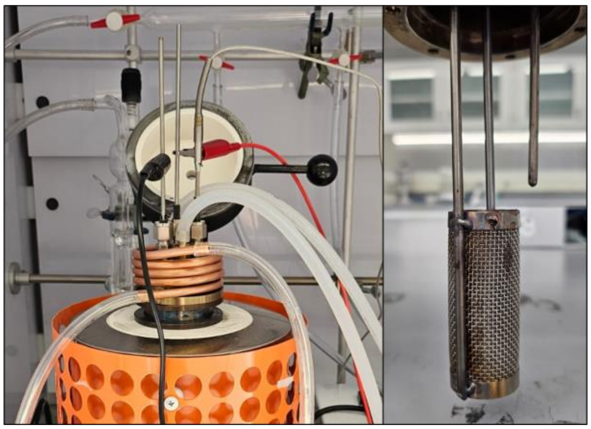

Figure 2. Electrochemistry bench-scale reactor. The left picture shows the exterior of the reactor. The crucible is placed inside the furnace (orange). The electrodes are connected to the power supply by alligator clips (red and black). Gases are going in and out of the system as shown by the opaque tubing. The cooling recirculation system has a coiled shape and is located around the lid. The refrigerant liquid is transported in and out of the coil by the transparent tubing. The right picture shows the elements introduced into the reactor: a basket cathode shown on the left, a rod anode located at the center, and a thermocouple located at the right.

The experiments were carried out by filling the crucible with pre-dried $CaCl_2$. An Al_2O_3 sample was placed into the cathode, which was subsequently placed in the lid along with the other components. The cell was sealed with the electrodes and thermocouple all sitting above the solid electrolyte line. Then, the temperature was ramped up to 950 °C. Once the temperature was reached, the cathode, anode, and thermocouple were lowered into the molten electrolyte. The current was then applied into the cell at 0.25A, ramping it up to 4A. Reduction took place for 12h. So far we have been able to electrochemically reduce Al_2O_3 into metallic aluminum. The samples are currently being analyzed by X-ray Diffraction (XRD) and Scanning Electron Microscopy (SEM) to determine the ratio of Al_2O_3 reduced into metallic Al.

Processing of metallic Al as feedstock for AM: The metallic Al produced by this process could be utilized on the Moon by elaborating a suite of lunar-manufacturable aluminum alloys. We are focusing our efforts on the processing of Al wire as feedstock for AM. The experiments were conducted by heating up a sample of Al at 900°C. Al went through a casting process by pouring the molten Al into a cylindrical mold. The solid Al is forged by sanding its surface and blowing it with a hammer. Once the diameter of the cylinder had decreased significantly it was rolled several times until the diameter was small enough to go through the wire drawing process. The tip of one of the aluminum cylinder ends was sanded and inserted into a tungsten carbide die. The tip was lubricated with mineral oil and was subsequently drawn with pliers using a mechanical system. The process was repeated several times using a smaller die each time trying to achieve the thinnest possible diameter for the wire. So far, we have been able to draw the following alloys: 1100, TAFA 01T, AlSi7, 4047, and 5356. We further plan testing the wire feedstock via electron beam (EB) additive manufacturing, a vacuum-based technology ideally suited for 3D printing on the Moon.

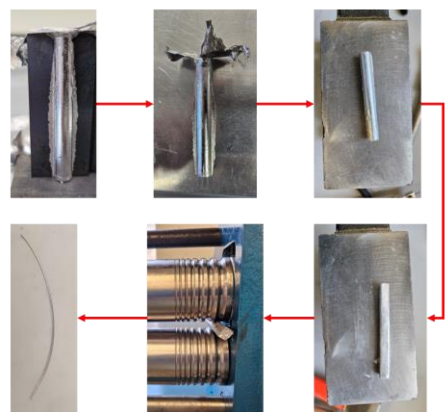

Figure 3. Metallic Al wire production. The different steps for the process are shown. First, Al was casted into a cylindrical mold. Then, the ingot was removed from the mold. Any excess material was removed by sanding and cutting. Then the material was forged using a hammer. The forged Al was wire rolled and finally drawn into a wire.

References:

[1] Thibodeau B., Ellery, A. and Walls, X. (2024) *ASCE E&S 2024,* 6962. [2] Walls, X., Ellery, A. Wanjara, P., Marczenko, K. (2024) *ASCE E&S 2024,* 7061. [3] Chen, G., Fray, D., Farthing, T. (2000) *Nature 407.* [4] Ellery, A., Mellor, I., Wanjara, P., Conti, M. (2022) *New Space 10* 224-238