Molten Regolith Electrolysis: Core Sample Analysis

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Introduction: *In-situ* production from lunar resources including oxygen (O_2) for human life support and propellant supply, as well as metals for construction materials are required for a sustainable long-term presence on the moon. One of the processes being considered for lunar extraction of oxygen from regolith is Molten Regolith Electrolysis (MRE)[1], [2]. This is an electrochemical process by which regolith is melted from its granular state and then electrolyzed to produce molecular oxygen. Reactor temperatures must exceed 1700°C, in addition to a constant draw of power of several kW to sustain the electrochemical separation on the melt. Once oxygen is extracted from the regolith, the once abundant metal oxides, should now reduce to metals, in the form of molten slag. This slag can be removed and cooled for materials processing and follow on use.

The Space Technology Mission Directorate (STMD) Game Changing Development (GCD) program funded a collaboration between the Kennedy Space Center (KSC) MRE team and Lunar Resources, Inc. (LUNAR) to develop a reactor for testing in the KSC Atmospherically Sealed Simulator for In-situ System Testing (ASSIST) chamber. LUNAR was funded via an SBIR Phase III contract (Contract #80NSSC22C0001). The GCD MRE project goals and objectives culminated into an integrated demonstration with a LUNAR developed MRE reactor that was delivered to KSC and tested in the ASSIST vacuum chamber.

Materials and Methods: The regolith selected for this test was ICN-LHT-1G (also known as CSM-LHT-1G) [3]. During electrolysis, evolved gases were monitored with the NASA Volatile Monitoring and Oxygen Measurement system (VMOMS) to measure oxygen recovery during the MRE process of Lunar regolith simulant in a vacuum environment. After completion of electrolysis, the reactor cooled to ambient conditions in the ASSIST chamber. Once the reactor was cooled and removed from the ASSIST chamber, a core sample was removed from the hardened lunar regolith that solidified in the reactor as displayed in Figure 1.

Figure 1. Core sample removed from reactor after MRE testing complete.

A computed tomography (CT) scan was performed on the core sample, with a North Star Imaging unit with its Microfocus Digital Radiography and Computed Tomography System (NSI X5000 DR/CT). X-ray photoelectron spectroscopy was performed with a Thermo Scientific NEXSA G2 Surface Analysis instrument. Scanning electron microscopy (SEM) of the main core and additional pieces (cathode, anode, and 'foam') was performed using a TESCAN SEM with an energy dispersive spectrometer (EDS), which allowed the entire main core piece to be analyzed. Sampling was performed along the entire vertical depth of the core (as considered from its original geometry in the reactor) in 1.2 mm increments; this allowed for a continual analysis from the cathode to the anode. ESD data were collected as elemental maps as well as a sum spectrum which was used to determine the elemental composition over the imaged region. SEM and EDS were also performed with a JEOL JSM-IT800. X-ray diffraction measurements were taken with a Panalytical Empyrean. X-ray fluorescence (XRF) measurements were also collected to provide insights on composition.

Discussion: The LUNAR Reactor successfully completed a batch test for MRE, resulting in oxygen evolved during the electrolysis phase of the experiment. The core sample data analysis can provide insight into metal removal and metal processing, as well as behavior of the anode/cathode presence in the regolith during electrolysis. The resulting mass of the extracted main core region was 631.0 g and a total volume of 9.714 cm^3. The CT scan measured a complete 3-dimensional data of the core and full cross-sectional data (Figure 2). This information was used to calculate the total volume of the main core piece. Along with mass, a calculation of the macro-density of the main core piece and macro-pore volume was performed, to obtain an estimated regolith mass loss and compare with oxygen production calculations.

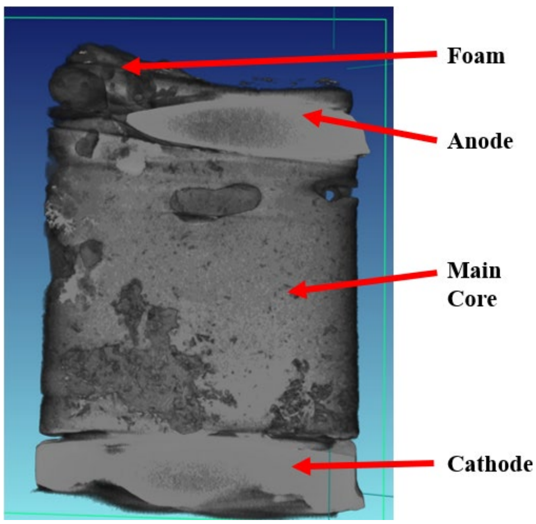

Figure 2. Cross Section CT Image of core sample pieces.

SEM analysis provided insight on morphology and cooling effects of the molten material, as well as elemental composition of the cross-sectional core sample. The core sample consisted of metals primarily associated with the lunar regolith, such as Si, Al, Fe, Ca, Na, Mg, Mn, Si and Zn. Additional materials of the anode and cathode were present throughout the main core, implying that circulation of these anode and cathode materials are producing a 'lava lamp' type of effect in the melt during electrolysis. SEM imaging revealed crystal formation was predominant in the pore regions of the melt.

Recommendations: The MRE technology has shown promise for producing O_2 and metals at a scale that could meet NASA's needs. A fully functioning MRE system would require a slag removal system as well as oxygen recovery and cleanup system to deliver a complete capability that transforms raw regolith into metals and oxygen to provide prolonged human presence on the Moon. The composition of this slag, based on repeated testing is required to develop a robust system that is compatible with processing materials in a lunar environment. Mixed metal removal is a challenging task in the lunar environment, and the insights on material composition, morphology, or oxide state can inform future resource recovery efforts.

References: [1] Standish (2010) *Design of a Molten Materials Handling Device for Support of Molten Regolith Electrolysis*. Thesis, Ohio State University. [2] S. S. Schreiner, (2015) *Molten Regolith Electrolysis reactor modeling and optimization of in-situ resource utilization systems*. Thesis, Massachusetts Institute of Technology, 2015. [3] A. Slabic et al., (2024) *Lunar Regolith Simulant User's Guide Revision A*.