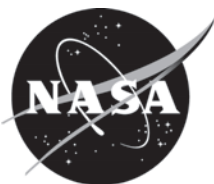NASA/TM—2006–214600

Process Demonstration For Lunar In Situ Resource Utilization— Molten Oxide Electrolysis

(MSFC Independent Research and Development Project No. 5–81)

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August 2006

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LIST OF ACRONYMS

BUNDLE   Bridgman Unidirectional Dendrites in Liquid Experiment

EDAX   energy dispersive x-ray analysis

FTE   full-time equivalent

IRAD   Independent Research and Development

ISRU   in situ resource utilization

JSC   Johnson Space Center

MOE   molten oxide electrolysis

MSFC   Marshall Space Flight Center

SEM   scanning electron microscopy

TM   Technical Memorandum

NOMENCLATURE

F	Faraday constant
I	current
M	molecular weight
n	valence
t	time

TECHNICAL MEMORANDUM

**PROCESS DEMONSTRATION FOR LUNAR IN SITU RESOURCE UTILIZATION—
MOLTEN OXIDE ELECTROLYSIS
(MSFC INDEPENDENT RESEARCH AND DEVELOPMENT PROJECT NO. 5–81)**

1. INTRODUCTION

The purpose of this Focus Area Independent Research and Development (IRAD) project was to conduct, at Marshall Space Flight Center (MSFC), an experimental demonstration of the processing of simulated lunar resources by the molten oxide electrolysis (MOE) process to produce oxygen and metal. The duration of the project was March 2005 through March 2006.

2. BACKGROUND

2.1 Production of Materials and Energy From Lunar Resources

In essence, the vision of the IRAD project was to develop two key technologies (fig. 1)—the first to produce materials (oxygen, metals, and silicon) from lunar resources and the second, to produce energy by photocell production on the Moon using these materials. Together, these two technologies have the potential to greatly reduce the costs and risks of NASA's human exploration program. Further, it is believed that these technologies are the key first step toward harvesting abundant materials and energy independence from Earth's resources.
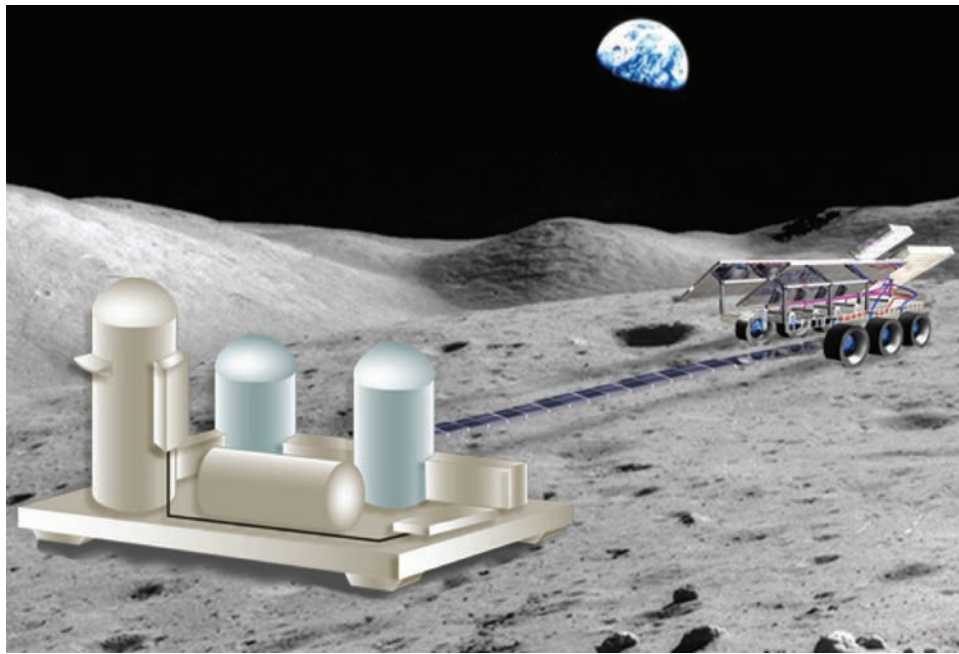
Figure 1. Artistic rendering of MOE unit (front) and photovoltaic cell-producing rover on the lunar surface.

The lunar surface soil (regolith) possesses the elemental components of Si solar cells and the Moon's vacuum environment (on the order of 10^{-10} torr) allows for direct vacuum deposition of thin-film materials.[1] Producing thin-film solar cells on the Moon requires individual elemental components to form a solar cell structure and a stable underlying substrate—the most massive part of the cell structure. The high cost of transporting material from Earth to the Moon's surface ($\approx$\$100,000/lb) drives the need for using lunar resources to make all components.[2] These space resources can be used for the fabrication of thin-film solar cells directly on the lunar surface using materials extracted from lunar regolith.[3]

The goal of this research effort was to advance the MOE process for the extraction of oxygen for life support and propellant, and silicon and metallic elements for use in fabrication of thin-film solar cells. The Moon is rich in mineral resources, but it is almost devoid of chemical reducing agents; therefore, MOE is chosen for extraction, since the electron is the only practical reducing agent.

2.2 Basics of the Molten Oxide Electrolysis Process

The Moon is rich in mineral resources capable of sustaining the production of Si as well as a variety of metals, e.g., Fe, Al, and Ti. However, the extraction of these elements will require the use of rather different processes from those used on Earth. For example, mineral beneficiation has been an enduring paradigm in terrestrial extractive metallurgy for economic reasons, but the reliance on such unit operations as froth flotation with its attendant consumption of huge quantities of water summarily disqualifies beneficiation from consideration in the lunar setting. To eliminate the need for beneficiation prior to processing and to minimize the import of consumable reagents from Earth, the advancement of the known technology of MOE to space operations for the production of electronic grade Si and metallurgical grade Fe, Al, Ti, and Si using lunar regolith as feedstock that has not been subjected to any form of pretreatment (fig. 2). In addition to providing the materials for photocell production, the metals and oxygen produced will serve as feedstock for lunar in-space manufacturing, life support, and propulsion needs.
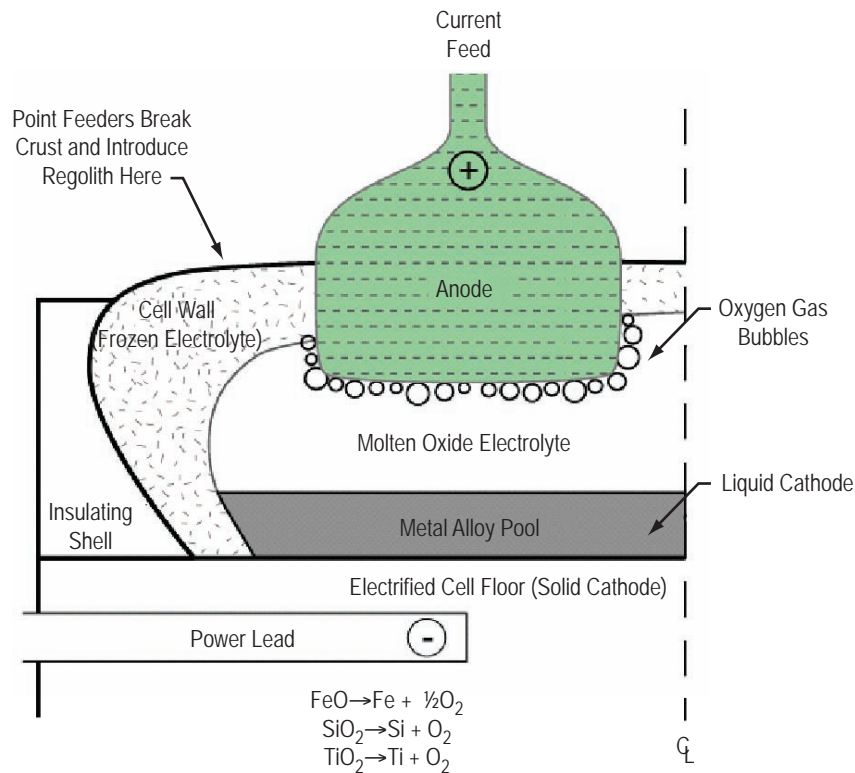
Figure 2. Schematic of MOE cell.

MOE was chosen for several reasons. First, electrolytic processing offers uncommon versatility in its insensitivity to feedstock composition. Secondly, oxide melts have the twin key attributes of highest solubilizing capacity for regolith and lowest volatility of any candidate electrolytes. The former is critical in ensuring high productivity since cell current is limited by reactant solubility, while the latter simplifies cell design by obviating the need for a gas-tight reactor to contain evaporation losses as would be the case with a gas or liquid phase fluoride reagent operating at such high temperatures.

Alternatively, MOE requires no import of consumable reagents (e.g., fluorine and carbon) as other processes do and does not rely on interfacing multiple processes to obtain refined products. Electrolytic processing has the advantage of selectivity of reaction in the presence of a multicomponent feed. Products from lunar regolith can be extracted in sequence according to the stabilities of their oxides as expressed by the values of the free energy of oxide formation (e.g., Cr, Mn, Fe, Si, Ti, Al, Mg, and Ca).

The presence of Fe can result in undesirably high levels of electronic conductivity with attendant loss of faradaic efficiency. Sadoway et al. have shown that it is possible to repress electronic conduction and thereby achieve high levels of efficiency by tailoring the chemistry of the supporting electrolyte.[4,5]

Previous work has demonstrated the viability of producing Fe and oxygen from oxide mixtures similar in composition to lunar regolith by MOE (electrowinning), also called magma electrolysis,[6–11] as well as the electrolytic extraction of Si from regolith simulant.[12]

3. APPROACH

3.1 High-Temperature Molten Oxide Electrolysis Process Enabling Lunar Oxygen and Solar Cell Production

Depending on regolith composition at lunar locations, operating temperatures are expected to be between 1,300–1,500 °C at a power consumption rate of 500 W_e for the target yield of ≈1 kg, enough to supply solar cell paver operations for more than 14 days. Once introduced in the reactor, the regolith becomes molten electrolyte from which three products are separated: gaseous oxygen, an alloy of Fe-Si-Al-Ti, and the expended electrolyte, rich in magnesium oxide. The metal alloy is then transferred to a refining cell to extract Si and metals. Figure 3 gives the process flow diagram for the MOE-based extraction and refining process enabling lunar solar cell production.
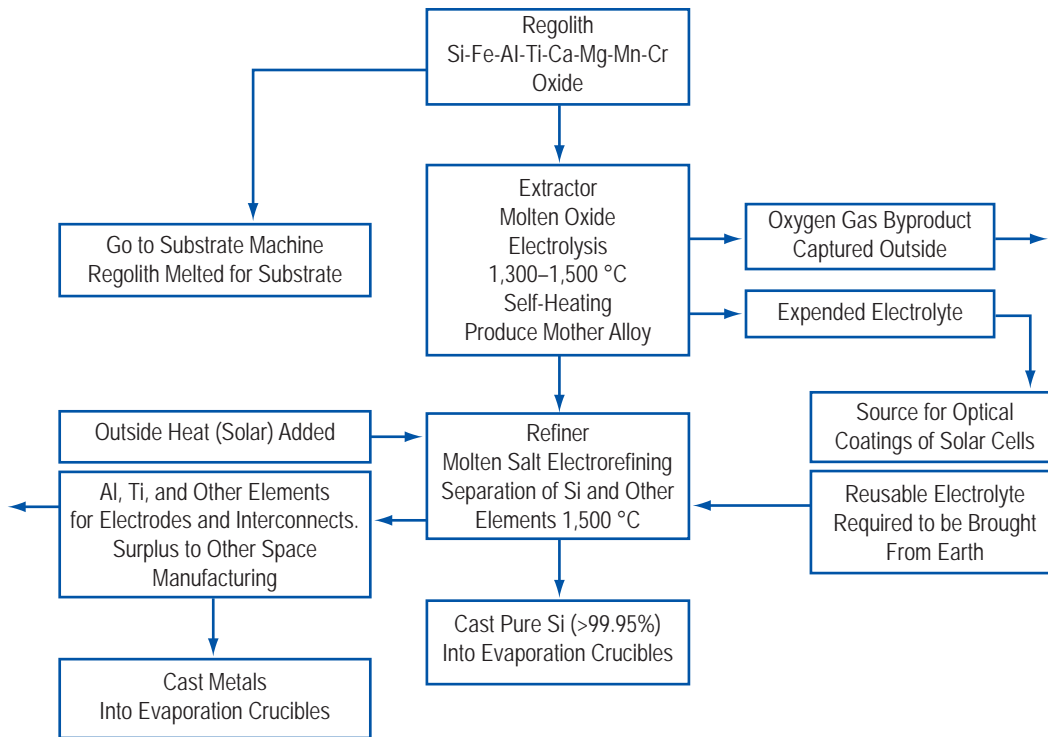
Figure 3. Process flow diagram for the MOE-based extraction and refining process.

Since the Focus IRAD was only funded for civil servant FTEs, it was not possible to complete the high-temperature (1,500 ºC) MOE experiments as originally proposed. Instead, we prioritized the lower temperature experiments (sec. 3.2) and proceeded with the higher temperature apparatus on a best effort basis. Presently, we have designed and fabricated crucibles and electrodes and refurbished and tested a suitable high-temperature furnace.

3.2 Low-Temperature Supporting Electrolyte Molten Oxide Electrolysis Process Enabling Lunar Oxygen and Iron Production

There is an experimentally verified approach for substantially lowering the oxide melt temperature needed for electrolysis if a quantity of supporting electrolyte were to be transported to the Moon. Sadoway, MIT,[13] has developed a low-temperature supporting electrolyte, SiO_2-B_2O_3-Na_2O (0.72:1:1) that allows oxygen-producing electrolysis of JSC–1 Mars simulant at 850 °C. The low-temperature supporting electrolyte is capable of solvating JSC–1 Mars simulant. MOE experiments with the supporting electrolyte and JSC–1 Mars simulant verified the production of reduced iron at the cathode and gaseous oxygen at the anode. The properties of the supporting electrolyte are given in table 1.

Table 1. Properties of low-temperature supporting electrolyte.

Property	Value
Homogeneous melting temperature	700 °C
Conductivity at 850 °C	0.036 S/cm
Diffusion coefficient of Fe^{2+}	5×10^{-10} cm^2/s
Current efficiency (ionic/electronic conductivity)	95%

This approach, while not optimum for providing materials for lunar photovoltaic cell production, can be utilized to reduce the iron oxide portion of lunar regolith to yield oxygen for propellant and life support. The lower temperature also makes the process experimentally more accessible by allowing the use of conventional crucible materials.

4. ACCOMPLISHMENTS

4.1 Preparation of the Low-Temperature Supporting Electrolyte

The low melting temperature electrolyte was modeled after Trapa et al.[13] The average molecular composition of the base flux is SiO_2-B_2O_3-Na_2O (0.72:1:1). Laboratory chemicals were used to synthesize the flux. Table 2 shows the formula for a 100-g batch of the flux.

Table 2. Low-temperature supporting electrolyte preparation.

Compound	Molar Conc. (%)	Reagent	Formula	Source	Weight (g)
B_2O_3	36.8	Boron oxide	B_2O_3	Alfa Aesar	39.81
SiO_2	26.5	Silicic acid	SiO_2-$4H_2O$	Fisher Scientific	16.61
Na_2O + rest of SiO_2	36.8	Sodium orthosilicate	Na_2O-SiO_2	Alfa Aesar	52.64

One hundred gram batches of the flux were weighed, mixed, and charged into 100-ml "high form" alumina crucibles (CoorsTek™ 65505). They were held at 850 °C for 2 hr to remove adsorbed water, CO_2, to homogenize the melt, and to remove bubbles. The melt was quickly quenched into water to make frit. The frit was quickly dried by microwave heating to remove the adsorbed water and stored in a dessicator with drierite.

4.2 Preparation of Iron Oxide and JSC–1 Lunar Simulant Mixes for Electrolysis

The frit was used along with iron containing lunar soil simulant to prepare glass for use with the electrolytic reduction process. The first iron oxide containing lunar simulant was Fe_3O_4, an effective 1:2 mixture of Fe+2 and Fe+3 (FeO-Fe_2O_3). A 25 wt. % iron oxide in flux was used in the first experiment.

One hundred gram batches were prepared by separately weighing 25 g of Fe_3O_4 and 75 g of electrolyte frit. The two components were hand mixed prior to charging into a 100-ml alumina crucible. The material was calcined to remove any residual water, by slow heating to 850 °C. The mixture was held at temperature for ≈4 hr to homogenize the melt. The melt was stirred in the crucible prior to casting onto graphite, squeezed into thin cross section, broken into manageable pieces and stored in a dessicator. The resulting glasses are shown in figure 4. For the experimental electrochemical reduction runs, glass pieces were then placed into the electrochemical cell crucible.

Figure 4. Low-temperature supporting electrolyte (left), with 25 wt. % iron oxide (center), and with 23 wt. % JSC–1 lunar simulant (right).

4.3 Electrochemical Analysis

To reduce the level of empiricism associated with the design of efficient reactors for extraction and refining, a study was done of the relevant electrochemistry and electrolysis testing in laboratory scale cells. To determine the critical voltages at which each element is deposited in the extractor and the refiner, sweep voltammetry was performed in melts of similar composition to what will be encountered in the full-scale process.

To enable the control and measurement of the electrolysis process a Zahner IM6ex electrochemical workstation was used. Before attempting MOE at elevated temperatures, the apparatus was tested using a room temperature, aqueous nickel sulfamate plating cell (fig. 5). During electrolysis, oxygen evolution at the platinum anode was observed. After electrolysis, nickel plating was observed on the carbon cathode. Cyclic voltammetry (fig. 6) revealed a nickel-plating peak at about –2 V.
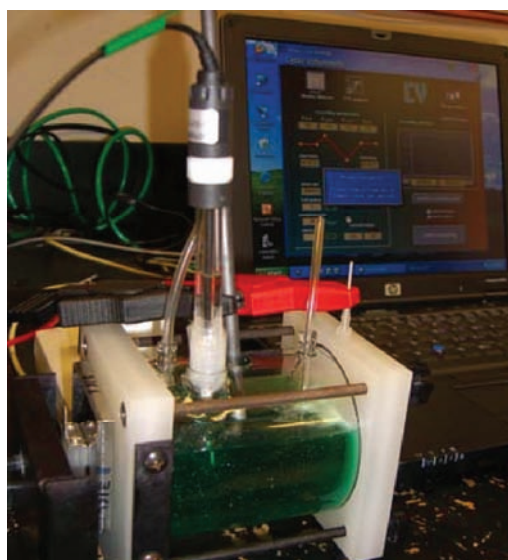

Figure 5. Aqueous nickel sulfamate test cell.
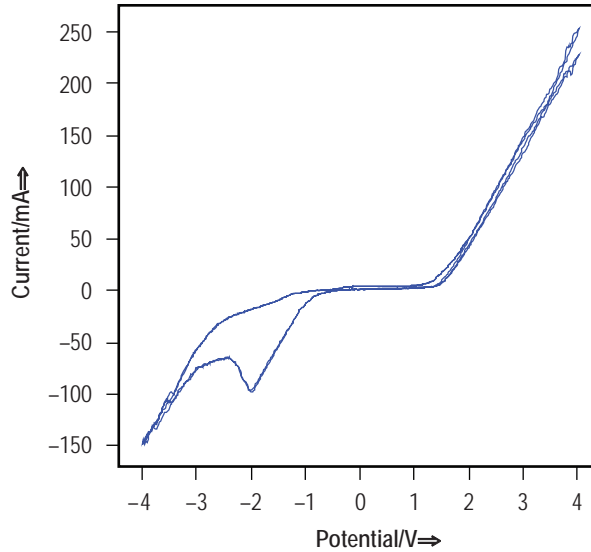
Figure 6. Cyclic voltammetry curve showing the nickel plating peak at about –2 V.

4.4 Two Electrode Molten Oxide Electrolysis Experiments

For simplicity and ease of observation the initial MOE experiments were run at atmospheric conditions, using a small bench top (Thermcraft, Inc.) furnace with a 3-in diameter by 6-in depth bore (fig. 7). The furnace controller (Omega CN7600) was set at 1,000 °C, which yielded a crucible internal side wall temperature of 850 °C.

All MOE experiments reported here were made with a two electrode system. Thus, the potentials reported and the potentials used for experimental control are the full cell potential—the potential between the anode and the cathode across the melt. A second set of experiments is planned using a reference electrode that will allow determination of the absolute potentials and of the potentials near the anode or cathode.[13]

All the experiments utilized platinum 40-percent rhodium wire as the anode (the positive electrode where oxygen gas is evolved). Three materials were tested for the cathode (the negative electrode where iron is plated), graphite, platinum 40-percent rhodium, and nickel-plated platinum rhodium. Only the experiments with platinum rhodium cathodes are discussed in this Technical Memorandum (TM).

4.5 Electrochemical Stability of the Supporting Electrolyte

Ideally all the iron oxide in the lunar soil or (simulated lunar soil) dissolved in the melt should be reduced without reducing the oxides in the supporting electrolyte. The relative stability of various oxides can be deduced from oxidative decomposition potentials. Table 3 lists the oxidative decomposition potentials, $-E°$, given in reference 14 relative to the JSC–1 simulant and lunar soil compositions given in reference 15.
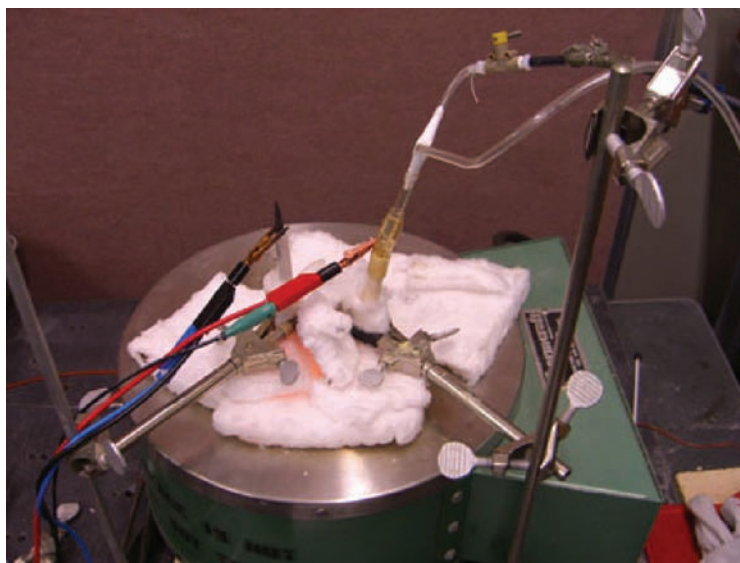
Figure 7. Bench-top furnace set with a graphite cathode (black clip) and platinum anode (red clip) with oxygen collection tubing.

Table 3. Oxidative decomposition potentials at 1,300 K versus wt. % JSC–1 and lunar soil major elements.[14,15]

Oxide	$-E°$ (V)	JSC–1 Conc. (wt. %)	Lunar Soil Conc. (wt. %)
K_2O	0.748	0.82	0.6
Fe_2O_3	0.842	3.44	0.0
FeO	0.986	7.35	10.5
Na_2O	1.117	2.7	0.7
Cr_2O_3	1.363	0.04	0.2
MnO	1.486	0.18	0.1
SiO_2	1.757	47.7	47.3
TiO_2	1.822	1.59	1.6
Al_2O_3	2.179	15.02	17.8
MgO	2.376	0.18	0.1
CaO	2.59	0.04	0.2

After noting that the $-E°$ given in reference 14 for B_2O_3 is 1.638 and recalling that the other constituents of the supporting electrolyte are SiO_2 and Na_2O, one would expect the supporting electrolyte to be electrochemically stable for electrolysis voltages from 0 to the $-E°$ for Na_2O (1.117 V).

Figure 8 gives the cyclic voltammetry curve for the supporting electrolyte using platinum 40-percent rhodium anode and cathode. The run was made for three cycles from –2 to +2 V at a 50 mV/s slew rate.
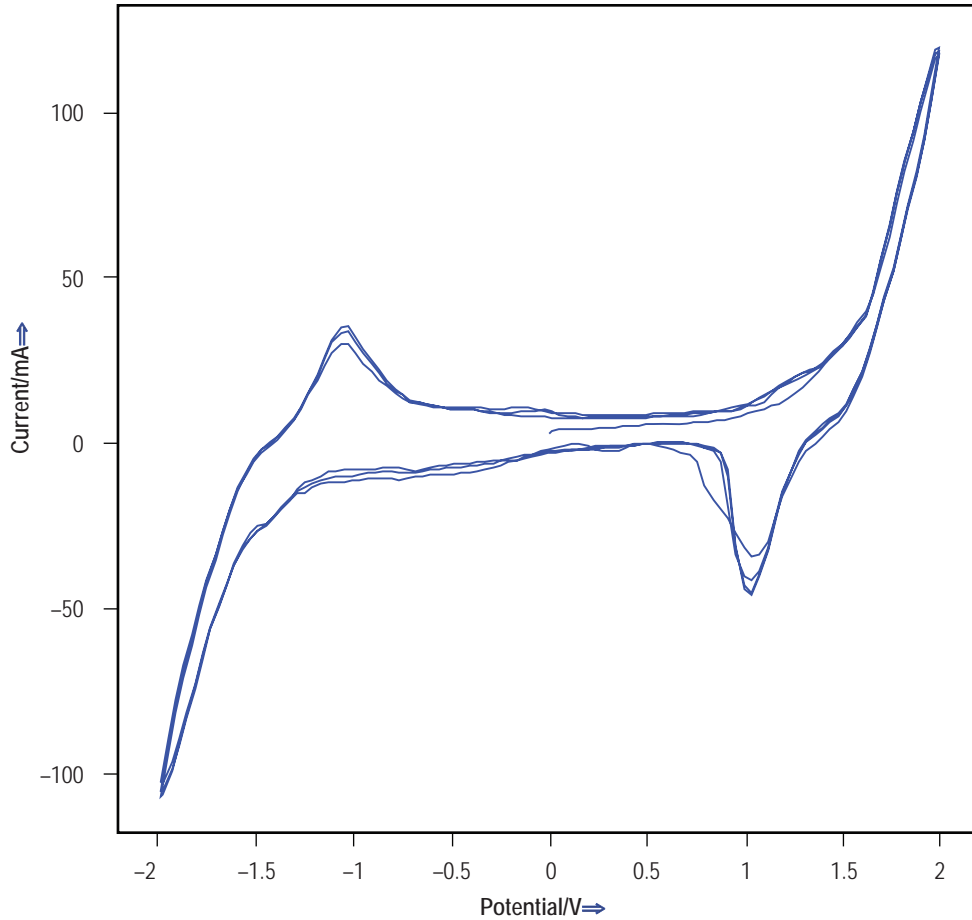
Figure 8. Cyclic voltammetry curve for B_2O_3, SiO_2, Na_2O supporting electrolyte.

The cyclic voltammetry sweep shows that the supporting electrolyte experiences a peak current flow as the voltage sweeps from 0 to just above plus or minus 1 volt. This is consistent with the oxidative decomposition potential value of 1.1 for Na_2O (table 3). It suggests that with care, iron oxide may be reduced without reducing the supporting electrolyte. Figure 9 simulates such a careful run, sweeping the voltage only from 0 to –0.85 V.

Note that figure 9 has a current scale of only 2 mA compared to figure 8 that spans 100 mA. Thus, it is evident from figure 9 that very little current flows in the supporting electrolyte if the voltage is kept below –0.85. In this voltage range, the supporting electrolyte can be considered stable.

4.6 Molten Oxide Electrolysis Supporting Electrolyte With 25 wt. % Iron Oxide

A series of metal oxide electrolysis experiments were performed on the supporting electrolyte with 25 wt. % addition of iron oxide. The goal was to understand the electrochemical characteristics of iron reduction before attempting the more complex JSC–1 lunar simulant. During these experiments, the influence of electrode area, the production of oxygen gas at the anode, and the production of reduced iron at the cathode were studied.
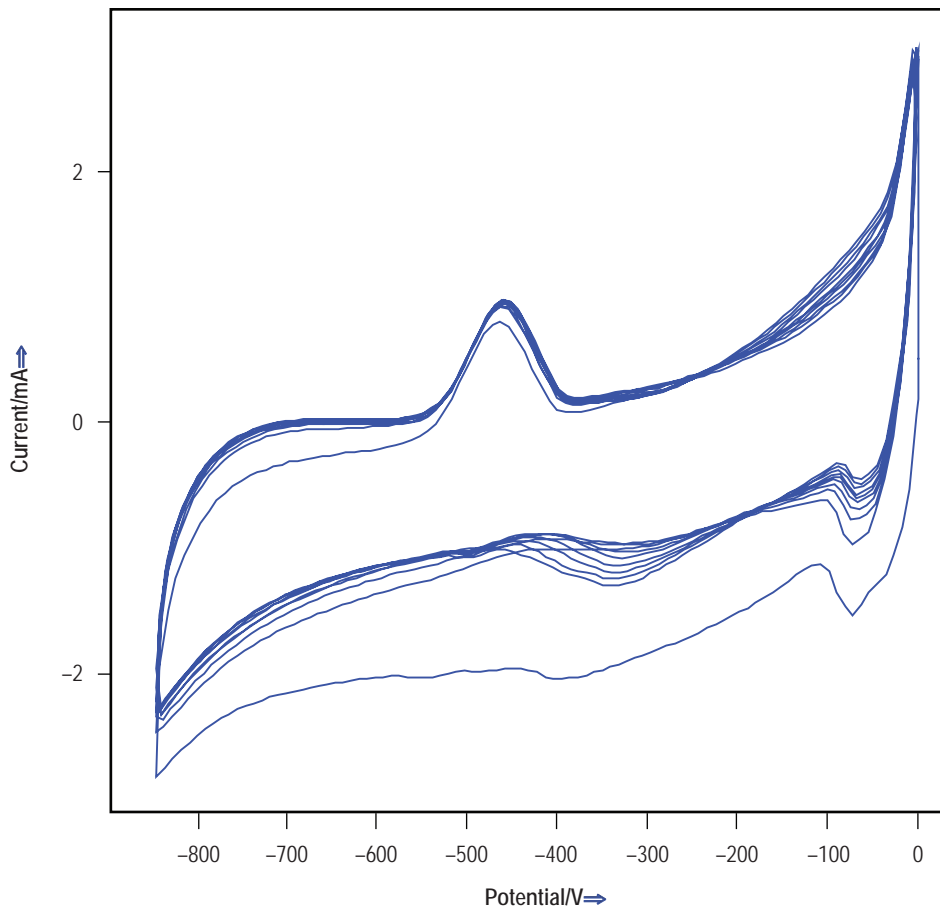
Figure 9. Cyclic voltammetry of supporting electrolyte from 0 to –0.85 V.

4.7 Effect of Electrode Area

It is expected that the electrolysis current and thus the oxide reduction rate will vary linearly with the electrode area. To test this relation, two platinum rhodium electrode geometries (fig. 10), straight and coiled, were used and the electrolysis currents at similar potentials compared. The result was that the coiled electrodes (fig. 11) had about three times higher cell current than the straight electrodes (fig. 12) at the same voltage.

4.8 Visualization of Oxygen Gas Production at the Anode

During the electrolysis run shown in figure 11 (supporting electrolyte and 25 wt. % iron oxide) an attempt was made to visually observe the evolution of oxygen gas at the anode. At –0.8 V no gas bubbles were visible to the naked eye. At –1.5 V gas bubbling at the anode was obvious and at –2.0 V gas bubbling was vigorous. Figure 13 shows the furnace with gas bubbling at the anode (right electrode).

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To enable quantitative measurement of the oxygen evolution flow and composition we have designed and built an inverted bell anode. We purchased and have begun testing an environmental oxygen measuring system that measures composition and flow rate.

Figure 10. Platinum rhodium electrodes: (a) Low surface area straight and (b) increased surface area coiled.

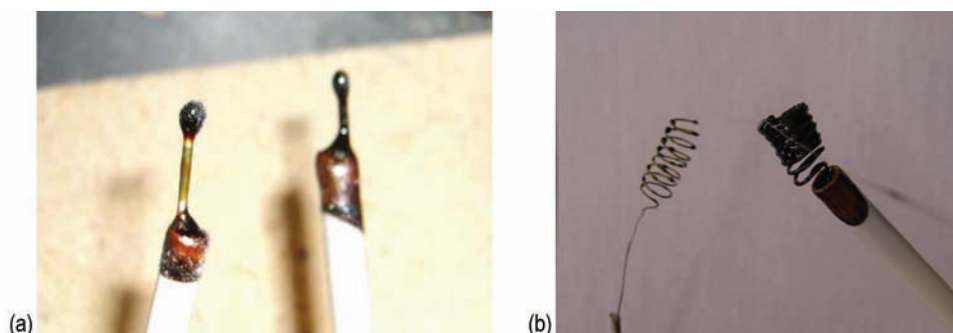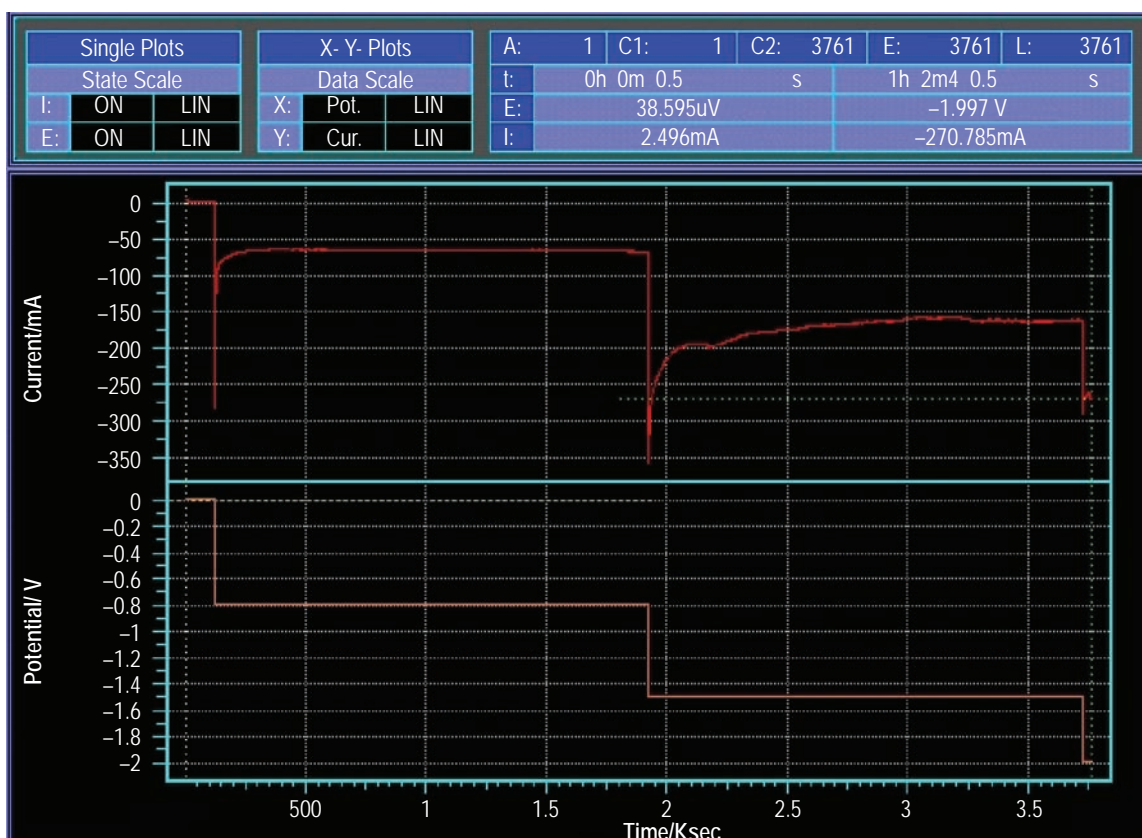Figure 11. Current versus voltage for electrolysis using coiled electrodes.
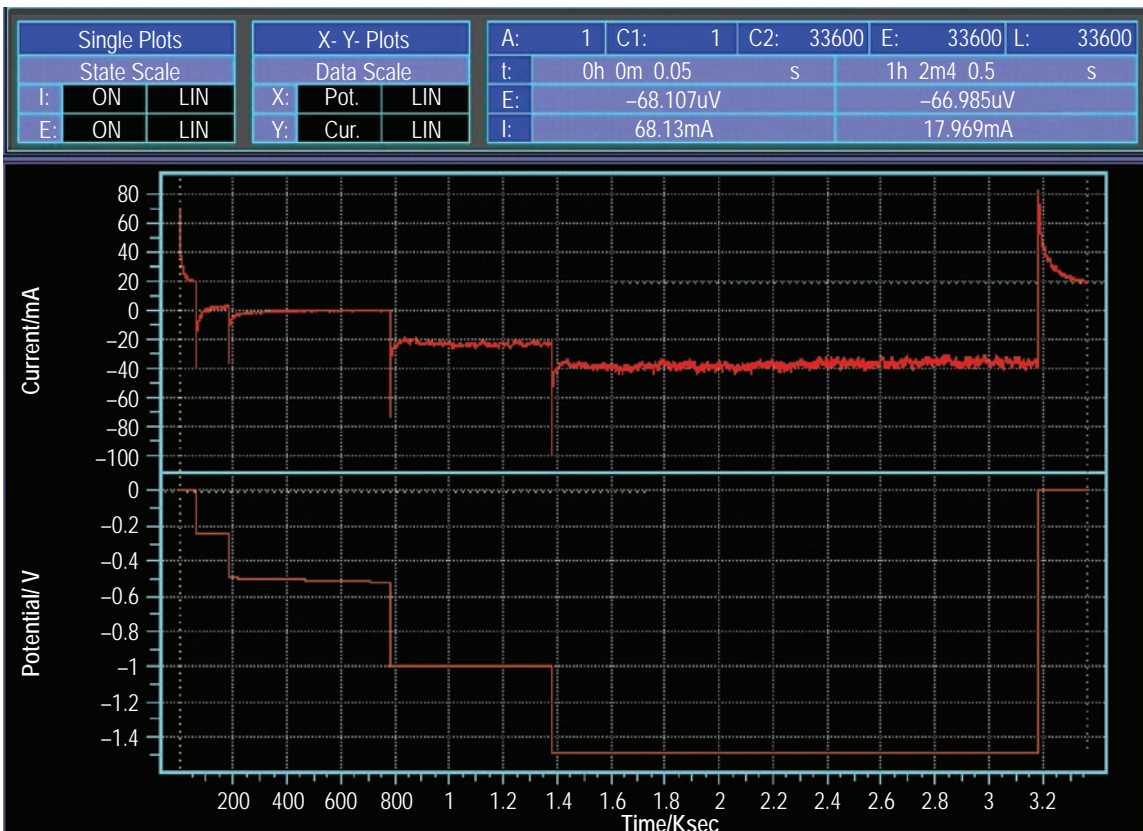
Figure 12. Current versus voltage for electrolysis using straight electrodes.
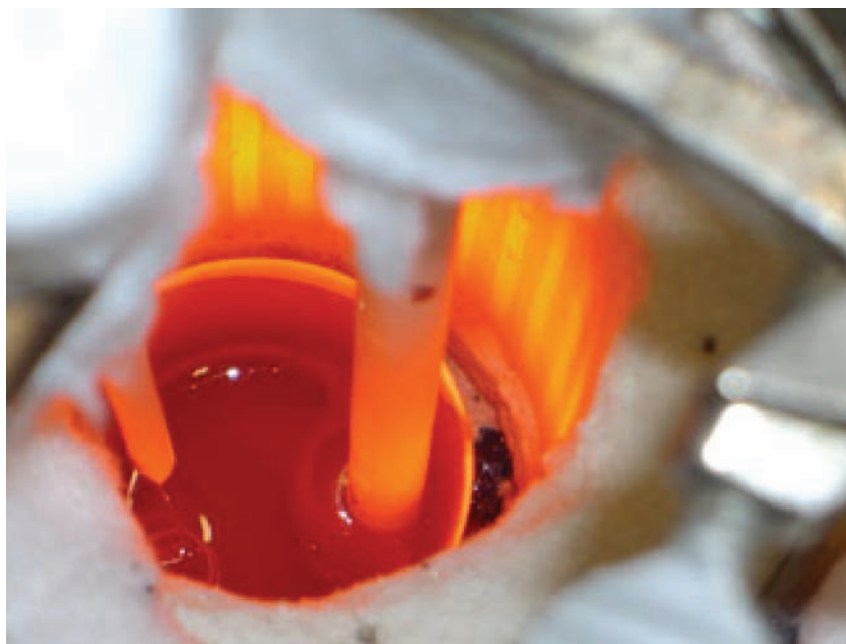
Figure 13. Furnace during electrolysis run in figure 11 showing gas evolution at the anode (right).

4.9 Verification of Iron Oxide Reduction

To verify iron reduction, at the cathode a transverse section of the straight electrodes shown in figure 10 was made after the electrolysis run in figure 11. These sections were examined using scanning electron microscopy (SEM) and energy dispersive x-ray analysis (EDAX). Figure 14 shows an EDAX iron element mapping of the frozen electrolyte around the platinum rhodium anode (top white). The light needles are a dendritic iron-rich phase. The iron-rich phase was absent from the electrolyte surrounding the section of the anode.
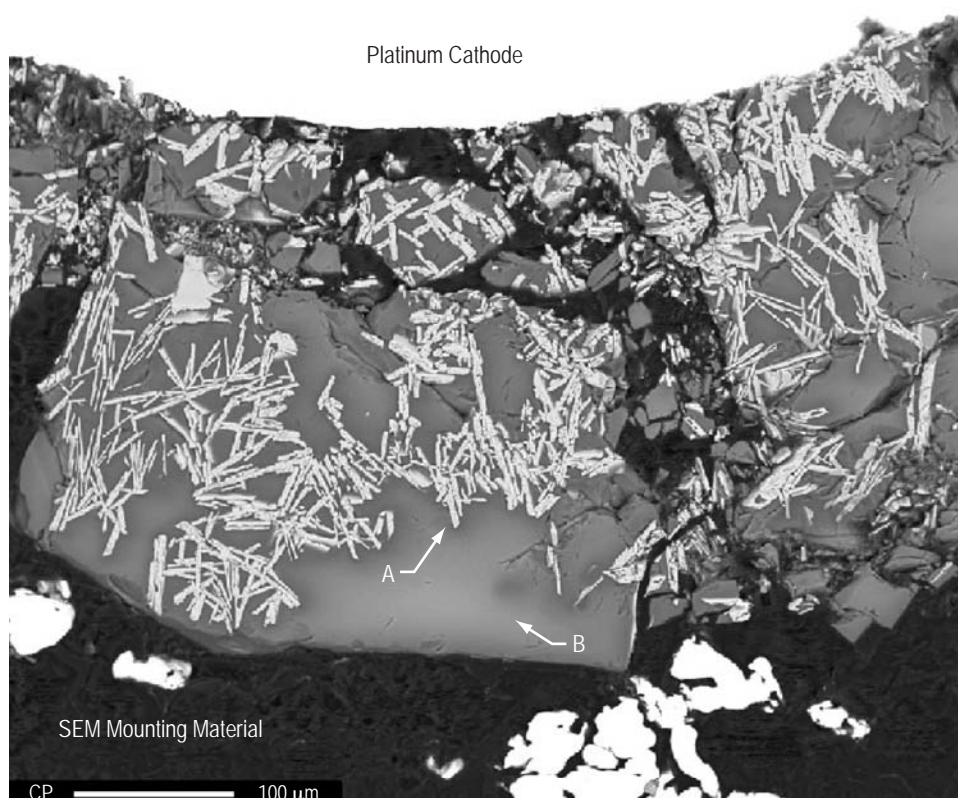
Figure 14. SEM image showing iron dendrites in the solidified electrolyte around the platinum rhodium cathode (top white).

4.10 Molten Oxide Electrolysis of JSC–1 Lunar Simulant

To complete this preliminary series of experiments, cyclic voltammetry and electrolysis was performed on the supporting electrolyte with 23 wt. % JSC–1 lunar simulant. In figure 15 the cyclic voltammetry curves for the supporting electrolyte with and without 23 wt. % JSC–1 lunar simulant are plotted on the same graph for comparison. The cell current for the supporting electrolyte alone between 0 and 0.75 V is negligible while the current for the electrolyte with JSC–1 shows significant current and a peak at –0.5 V thus indicating reductive reaction in the simulant.

16
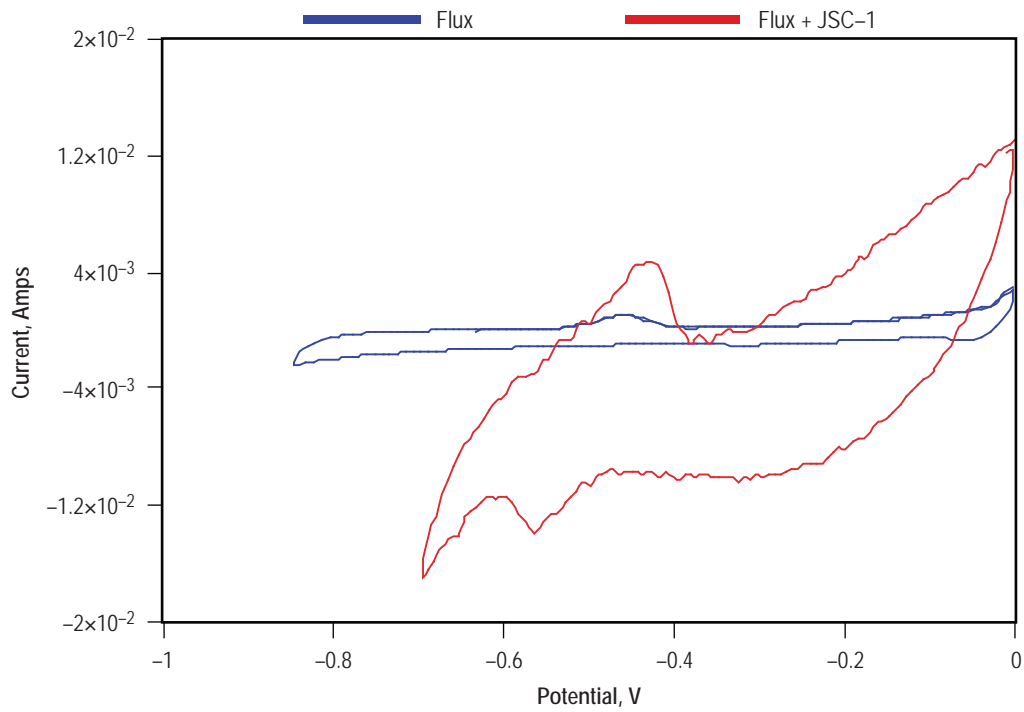
Figure 15. Cyclic voltammetry of supporting electrolyte (flux) with
and without 23 wt. % JSC–1 lunar simulant.

5. PLANNED FUTURE WORK

5.1 Potential Candidate for Robotic Lunar Exploration Lander Oxygen Production Experiment

One of the objectives of the robotic lunar exploration landers will be to demonstrate the production of gaseous oxygen from lunar soil. To quote NASA Administrator, Michael Griffin,[16]

Possibly, one of the most useful (resources) we will get from the Moon is liquid oxygen. It can be extracted fairly easily from the lunar soil. If shipped from the Moon to other storage depots, it will have very high value because it is half of the propellant needed for any exploration or any other rocketry activity over the next few decades.

When the various approaches for oxygen production were compared, molten oxide (or magma) electrolysis was found to have the most favorable power consumption and the second most favorable mass throughput.[17] Also MOE is the most direct approach toward meeting the materials requirements for solar cell production from lunar materials.[18] This allows the realization of the second part of the NASA Administrator's statement about the Moon's resources.

It is also reasonable to think about manufacturing solar arrays on the Moon and then beaming that power around the Earth/Moon system so that every spacecraft we build doesn't have to carry its own power system.

Lunar robotic lander experiments are expected to be limited to ≈100 W and under ≈10 kg. Can a meaningful MOE lunar oxygen production experiment be done under these constraints?

To estimate the MOE flight furnace's weight and power requirements one can consider the MSFC developed Bridgman Unidirectional Dendrites in Liquid Experiment (BUNDLE) hardware. The heating element consists of a pyrolytic boron nitride tube on which a pattern of graphite is deposited. The core, in this case, was ≈25 cm in length and 2.5 cm in diameter. To promote high temperatures and low-power usage the heating element was subsequently surrounded by layers of refractory foil.

The overall furnace is 40 cm in length, 9 cm in diameter, and has a 30-cm hot zone and must be run in a vacuum environment. Power usage is on the order of 25 W at 600 °C, 60 W at 850 °C, and 100 W at 1,100 °C. The assembly weighs 2.4 kg and was designed for a maximum operating temperature of 1,200 °C.

One can predict the expected results using the model of the BUNDLE furnace. The furnace has a core diameter of 2.5 cm (≈1 in) and length of 25 cm (≈10 in). To accommodate a crucible one can assume an internal crucible diameter of 1.9 cm (¾ in) and molten zone length of 39.2 cm (≈6 in). This can accommodate a volume of molten material of 111.3 cc. The density of molten regolith at a solidus temperature of 1,200 ºC is ≈2.69 g/cc. Therefore the mass of molten regolith that will be processed is

$111.3 \times 2.69 = 299.3$ g. Approximately 42% (by weight) of the regolith is O_2. If one completely extracts all the O_2 from the molten regolith one will be extracting $299.3 \times 0.42 = 125.7$ g of O_2. For simplicity, this example ignores any O_2 that may come from any fluxes that would have to be accounted for in the actual experiment.

If the furnace is operated ≈ 850 °C it will need about 60 W of power. That leaves ≈ 40 W for electrolysis. If the electrolysis cell runs at 2 A, 5 V to extract 125.7 g of O_2 according to the anodic reaction:

$$O^{-2} \rightarrow 2e^- + \frac{1}{2}O_2 \ . \tag{1}$$

Time, t (seconds) required to extract 125.7 g of O_2 under these conditions is given by:

$$W_{O_2} = \frac{MIt}{nF} \ , \tag{2}$$

where M is the molecular weight, I is current, n is the valence, and F is the Faraday constant. Thus ≈ 105 hr would be required to extract all of the oxygen. In an experiment with low-temperature electrolyte, about one-third of the volume would be regolith and only FeO would be reduced. This would yield ≈ 1 g of O_2 in 4 hr (at 1 V).

Thus, we believe that MOE is the most attractive technology for an early lunar robotic lander demonstration of oxygen production from lunar soil. Assuming the availability of lunar robotic experiment resources (ISRU Bridging Funds), we will build a flight definition test-bed in anticipation of lunar robotic experiment flight opportunities.

5.2 Beyond Oxygen Production

Further development of the MOE process could lead to extraction of other elements on the Moon and beyond including, Fe, Al, Si, Ti, Mg, Ca, etc. These elements (fig. 16) could eventually enable in situ fabrication and repair and production of power from in situ fabrication photovoltaic cells.
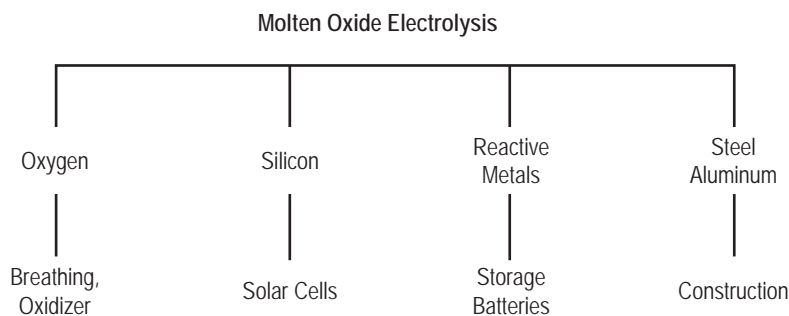

Figure 16. Future space resources from the development of MOE.

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The purpose of this Focus Area Independent Research and Development project was to conduct, at Marshall Space Flight Center, an experimental demonstration of the processing of simulated lunar resources by the molten oxide electrolysis process to produce oxygen and metal.

In essence, the vision was to develop two key technologies—the first to produce materials (oxygen, metals, and silicon) from lunar resources and the second to produce energy by photocell production on the Moon using these materials. Together, these two technologies have the potential to greatly reduce the costs and risks of NASA's human exploration program. Further, it is believed that these technologies are the key first step toward harvesting abundant materials and energy independent of Earth's resources.

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space resources, molten oxide electrolysis, lunar oxygen production, lunar photovoltaic power production, in situ resource utilization

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