The Electrochemical Production of Oxygen and Metal via the FFC-Cambridge Process.
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Introduction: The most significant component in any bi-propellant rocket is the oxygen required for fuel combustion. This may account for up to 85%wt of the rocket propulsion reactants. Therefore, it is vital that locally produced oxygen is available from off-world sources to enable more economically viable space exploration to more distant regions of solar systems, and beyond.

Our Moon in particular is a desirable location for the refuelling of rockets. Its relative close proximity to the Earth (~380,000km), and it being the only off-world site so far successfully visited by man, makes it the obvious first candidate when considering the location of a spacecraft refuelling station. Furthermore, elemental analysis of lunar material from Apollo and Luna landings, has yielded that the composition of the lunar surface material is approximately 44%wt oxygen, locked in the form of metal oxides (Figure 1)1. Through the novel FFC-Cambridge Process, it may be possible to extract the oxygen component from the lunar regolith to ultimately fuel spacecraft, and produce a potentially useful metallic side-product.

The FFC-Cambridge Process: The novel FFC (Fray-Farthing-Chen)-Cambridge Process was discovered in 1997, by three scientists, Derek J. Fray, Tom W. Farthing and George Z. Chen, at the Materials Science and Metallurgy Department, University of Cambridge, UK. They found that metal could be directly produced from its oxide by negatively charging it in a molten salt electrolytic cell2.

A significant amount of work has since been concentrated on the electrolytic synthesis of numerous metals and alloys directly from their cathodic metal-oxides. This has involved the reduction of primary lunar regolith constituent metal-oxides, such as: silicon oxide (SiO2); aluminum oxide (Al2O3); and iron oxide (Fe2O3).

The FFC-Cambridge Process is a relatively simple process, with low labour and energy requirements. It operates by placing a metal-oxide (MO) into a molten calcium chloride salt bath (CaCl2), and making it a cathode. Consequently, the oxygen from the metal-oxide becomes ionised through the half reaction presented in [1], and leaves the negatively polarised electrode.

\[ \text{MO} \rightarrow \text{M} + \text{O}_2^- \quad [1] \]

The inventors of the FFC-Cambridge Process have termed the ionisation of oxygen at the metal-oxide, as electrodeoxidation. Over a period of time, a low-oxygen containing metal (M) is produced at the cathode. The oxygen ion (O2-) moves through the molten salt bath through migration, diffusion, and convection processes to arrive at the positively charged anode. Conventionally-used graphite materials react with these oxygen ions to form carbon monoxide or carbon dioxide gas at the anode.

**Graphite Anode:**

\[ \text{nO}_2^- + \text{nC} \rightarrow \text{nCO} + 2\text{ne}^- \quad [2] \]

However, by replacing an inert anode for a graphite anode it is possible to liberate and generate the desired oxygen gas.

**Inert Anode:**

\[ \text{nO}_2^- + \text{n/2 C} \rightarrow \text{n/2 CO}_2 + 2\text{ne}^- \quad [3] \]

The basis of this work is therefore essentially to find a suitable non-reactive anode material that is capable of generating oxygen gas from the metal-oxide cathode, and consequently producing a metallic by-product.

**Thermodynamic Considerations:** The standard reduction potentials for the anodic reactions [2], and [3] have been calculated from a thermodynamic database, at 1173K, using a Ca2+/Ca reference point of zero, and by assuming that all reacting or produced species were at unit activity or unit partial pressure. It was determined that an anodic potential greater than 1.116V and 1.025V would be required to synthesise oxygen on an inert anode, with reference to CO and
CO₂ synthesis on a consumable graphite anode. This energetical demand cannot be avoided as oxygen and not carbon oxides are the desired product. However, it should be noted that oxygen liberation is thermodynamically more favourable than undesired chlorine synthesis (from the decomposition of the calcium chloride salt) by 0.557V. This may ensure that high purity oxygen gas is ultimately obtained.

**Experimental Work:** Candidate anode materials were selected for testing using the a two-electrode set-up presented in Figure 2. A 4.0g titanium-oxide pellet was made the cathode, with a constant 3.0V potential applied between the electrodes, when immersed into a molten CaCl₂ / CaO salt mixture, at 900°C. The oxygen composition of the exhaust gas stream was measured using a stablised zirconia analyzer.

Figure 2. [Schematic diagram of the FFC-Cambridge Process electrolytic cell.]

Once the most suitable candidate anode material had been established, further experimental work was conducted in which JSC-1 (a lunar regolith simulant material obtained from NASA) was made the cathode to establish whether it could be metallised.

**Anode Results:** It was found that a tin-oxide based material was the most suitable anode for the FFC-Cambridge Process after testing of numerous metal, cermet (metal dispersed within a ceramic matrix), ceramic, and also carbon-based anodes. This is since it remained relatively inert during electrolysis (Figure 3).

Figure 3. [Tin-oxide anode before (left) and after 24hour electrolysis (right).]

Furthermore, it was found that the oxygen detected during electrolysis in the exhaust gas stream using the tin-oxide anode was significantly above its background level. This result indicates that oxygen may indeed be produced from metal-oxides, such as those constituting the lunar surface, using the FFC-Cambridge Process.

**Cathode Results:** JSC-1 pellets of similar composition to lunar regolith were reduced using a tin-oxide anode. It was found that the material became metalised through the application of the FFC-Cambridge Process. Loose, as-received JSC-1 material was also found to be successfully reduced.

Figure 4. [Oxygen composition and current-to-time profile for the tested tin-oxide anode.]

Figure 5. [JSC-1 pellets sintered in argon (left), and metallic pellets obtained after electrolysis (right).]

**Conclusion:** The FFC-Cambridge Process was found to be suitable for the production of oxygen from metal-oxides through initial bench-scale testing using a tin-oxide anode. A metallic product was also directly produced from JSC-1 (a lunar regolith simulant material) using the FFC-Cambridge process. This work may be beneficial with regard to producing oxygen and metal on the lunar surface. The oxygen may ultimately be used to fuel spacecraft, and enable more economically viable space-exploration.

**References:**