White Paper

Mini-ROXY: The next step towards an efficient method for oxygen extraction from regolith

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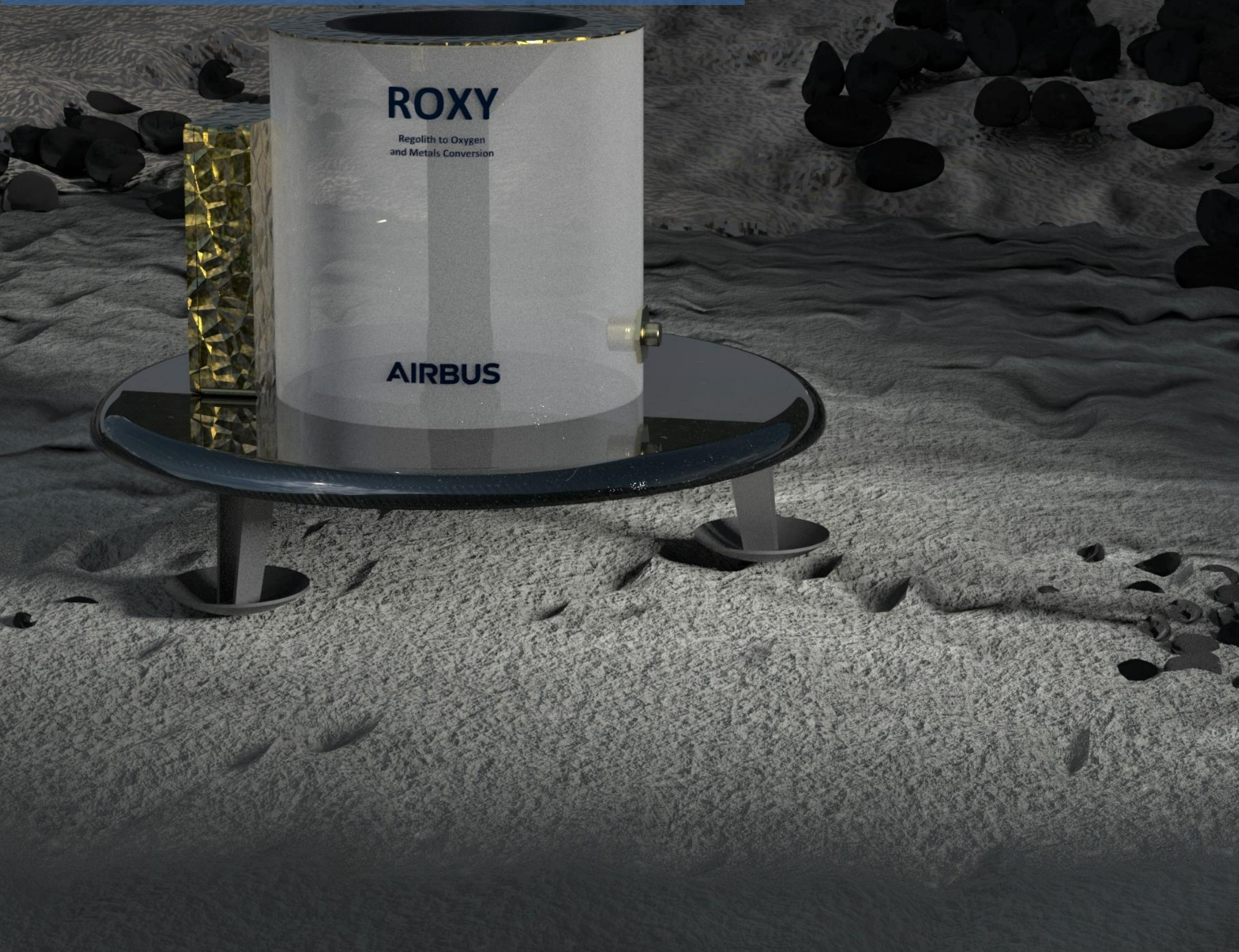

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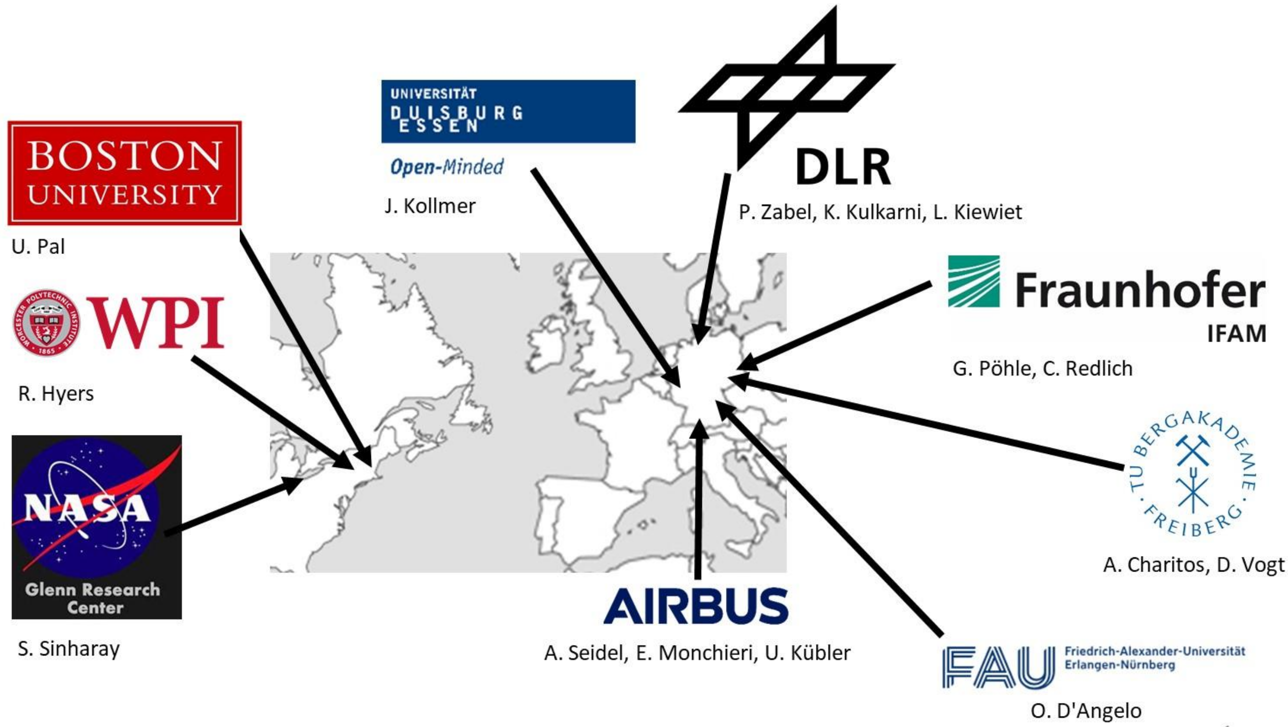

Summary
Space exploration will be enabled by ISRU provided that ISRU processes meet the following viability criteria: the mass of resources produced must exceed the mass of ISRU plant that produces them, and the mass of resources produced per unit time must significantly exceed the mass of consumables or spare parts per unit time that are required to maintain the production process.

Core resources to support a lunar economy are oxygen and metals. Many processes have been proposed to extract oxygen and metal from lunar regolith. Most of them do not meet all of the viability criteria for an efficient lunar process, due to low conversion efficiency, high processing temperatures, use of fluids and other consumables, etc.. With this in mind the ROXY (Regolith to Oxygen and Metals Conversion) process has been invented by Airbus and meets all of the above viability criteria. ROXY could therefore be the core of a large-scale lunar oxygen and metal product value chain, as shown in Figure 1.

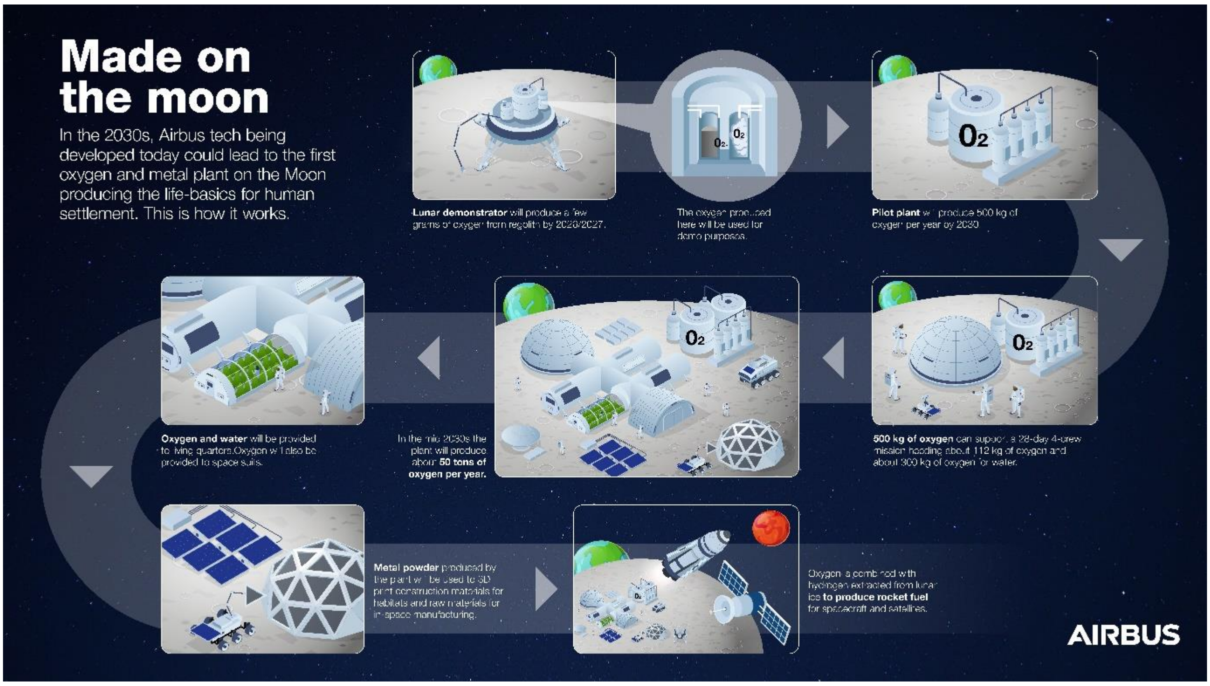

Figure 1: ROXY technology as the core of a large-scale lunar oxygen and metal product value chain

The ROXY process has also huge potential in terrestrial application, in particular in the area of sustainable, clean metal and energy production.

It is expected that the ISRU market value will grow exponentially over the next decades. Oxygen production will be the major source of growth. Accordingly, ISRU processes will need to be developed and successively up-scaled to meet the demand for resources. This requires a long-term and multi-step development program, starting with ground demonstrators, followed by lunar demonstrations, and then successively larger lunar production facilities.

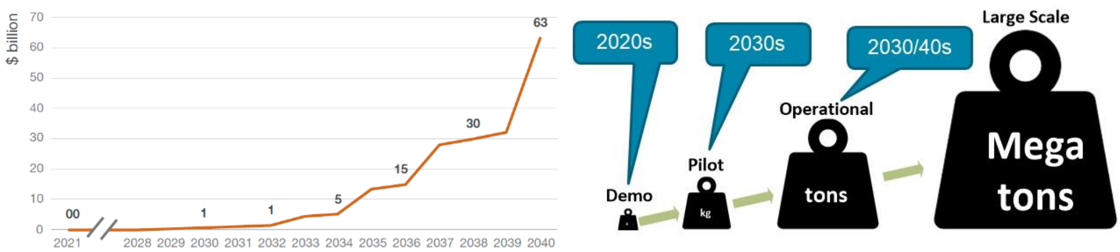

Figure 2: Left: Evolution of the cumulative SRU market size (source: pwc), right: ISRU scaling approach

A first major step will be the demonstration that ISRU processes will work in the lunar environment, under reduced gravity and with true lunar regolith as feedstock. The essential goal of such a demonstration is the characterization of the process for oxygen extraction using state of the art process diagnostics. Meeting this objective does not require that a large amount of oxygen is produced. This opens up the possibility to design a cost-optimized lunar demonstration mission with a simple, compact and low-mass demonstration facility, provided that a miniaturized process can be designed. The Mini-ROXY process has been invented, conceived, and validated by Airbus in collaboration with Boston University and Fraunhofer IFAM to meet those objectives. It uses a unique feature of ROXY that is not possible with other processes to achieve a miniaturized cell design. With this design it will be possible to achieve the demonstration objectives in the simplest possible way with a low-mass demonstration facility that can be accommodated on a variety of lunar landers as a secondary payload.

Currently, there are still significant gaps in our knowledge of how best to extract and process lunar regolith into usable metal products and oxygen. In particular, there are significant uncertainties on the processability of lunar regolith and the gravity dependency of the processes involved. A detailed understanding of these processes when applied to true lunar regolith under lunar environmental conditions such as reduced gravity is indispensable for future developments in this field. This leads to a number of scientific objectives that will need to be addressed by a multi-disciplinary science team in parallel to the development of the demonstrator flight hardware. This science team includes universities and research centers both in the US and Germany with a wide experience in related aspects including electrochemistry, metallurgy, regolith handling, and space hardware development for materials science applications. The science objectives, and the scientific rationale for a Mini-ROXY mission, the configuration and key technical data of a lunar Mini-ROXY demonstrator, and the development status of the hardware are summarized in this document. With the work performed to date the concept has been proven and key technical issues of a Mini-ROXY reactor have been demonstrated in a ground test campaign. As a next development step the design and manufacturing of an end-to-end Mini-ROXY ground model has been initiated, which will serve also as a development model for a future lunar demonstrator. In parallel, a lunar mission is under preparation by defining mission and lunar model requirements, designing a lunar model, and performing accommodation studies on available lunar landers and rovers.

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1 Introduction: ISRU and Viability

Space exploration is an evolving mega-trend with plans to colonize the Moon and eventually Mars in the coming decades. Space exploration is resource-intensive. The costs to transport resources from Earth to the Moon or even Mars are prohibitive and will remain so even if future technical advances are considered. Therefore, the use of local resources – ISRU (in-situ resource utilization) – will be an enabler for space exploration. However, for ISRU to fulfill its promise, it will need to produce resources from local materials very efficiently such that the mass of the produced resources exceeds the mass of any needed consumables or spares to sustain the process. Further, the mass of ISRU facilities will need to be minimized, since an ISRU facility will need to produce more that its own mass of resources in order to achieve break-even.

From these considerations a figure of merit for any ISRU facility can be defined as the ratio of the mass of the produced local resource and the mass of the items needed to be uploaded to produce the resource. Maximizing this figure of merit is a key requirement for any ISRU process considered for large scale implementation on the Moon.

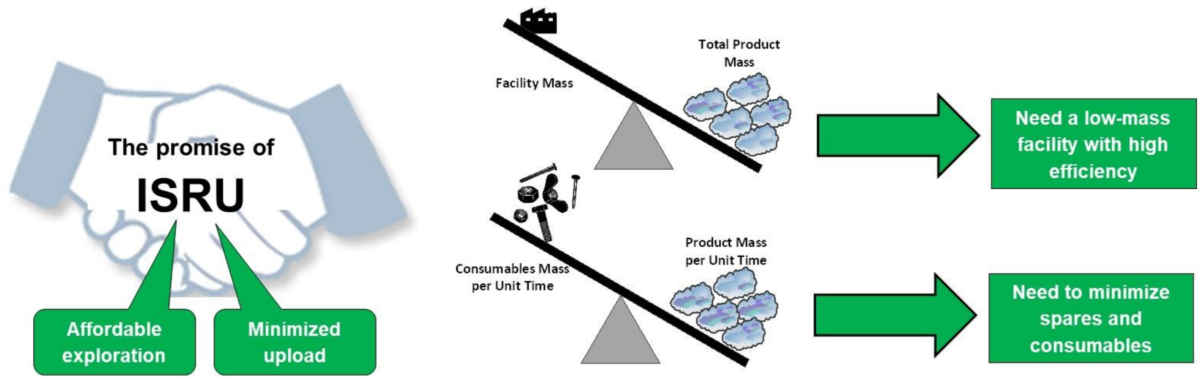


Figure 3: ISRU viability requirements

For the specific case of oxygen and metals extraction from regolith that is considered herein, the optimum process would therefore have the following desirable characteristics:

- it should work on almost anything that can be scooped up, without excessive requirements for mineral beneficiation
- it should produce large amounts of oxygen; that is, it should reduce the majority mineral constituents of lunar regolith and have a high regolith to oxygen conversion yield
- it should recycle all reactants or use only reactants produced from lunar material
- it should occur at a reasonable temperature, minimizing the need for exotic/high temperature resistant materials for components, the system power demands and heat losses to its surroundings.
- it should produce a reduced byproduct either in easily-separable elemental form, or in a state that may be easily refined for use in other manufacturing processes

2 ROXY and Mini-ROXY – Optimized for Lunar ISRU

Many processes have been proposed for the reduction of lunar regolith to produce oxygen. Most of them do not meet all of the above viability criteria. Chemical reduction of lunar regolith using hydrogen or methane is characterized by the need for process gases and low conversion efficiency. Molten oxide electrolysis (MOE) operates at high temperatures (>1500°C, depending on the oxide composition and the metal to be reduced), which presents significant technological challenges related to containment and anode degradation. Regolith reduction with ionic liquids provides a pathway for electrochemical production of metals and oxygen at low process temperatures, but challenges exist due to the high stability of the oxides, which makes dissolution of minerals, e.g., olivines and anorthites, difficult. Regolith reduction by molten salt electrolysis is very promising, but most applications of the process are problematic due to a number of issues, including the need for an inert gas, difficult separation of oxygen, difficult recycling of the salt, corrosion of the reactor, oxygen losses due to corrosion, contamination of the metallic product by oxygen, parasitic reactions of oxygen, reduced power efficiency, anode lifetime and more.

2.1 ROXY for Lunar ISRU Viability

The ROXY (Regolith to Oxygen and Metals Conversion) molten salt electrolysis process has been specifically conceived for oxygen and metal extraction from lunar regolith. It is based on a combination of key technologies and features, specifically a molten salt reactor, an optimized fluoride salt electrolyte, a highly selective oxygen ion pump anode, a porous metal cathode cup, that are integrated to mitigate drawbacks of other processes and provide a novel solution. Invented by Airbus, the ROXY process is based on a long heritage of the SOM (Solid Oxide Membrane) process developed by Boston University. ROXY meets all of the ISRU viability criteria. The principle is shown in Figure 4.

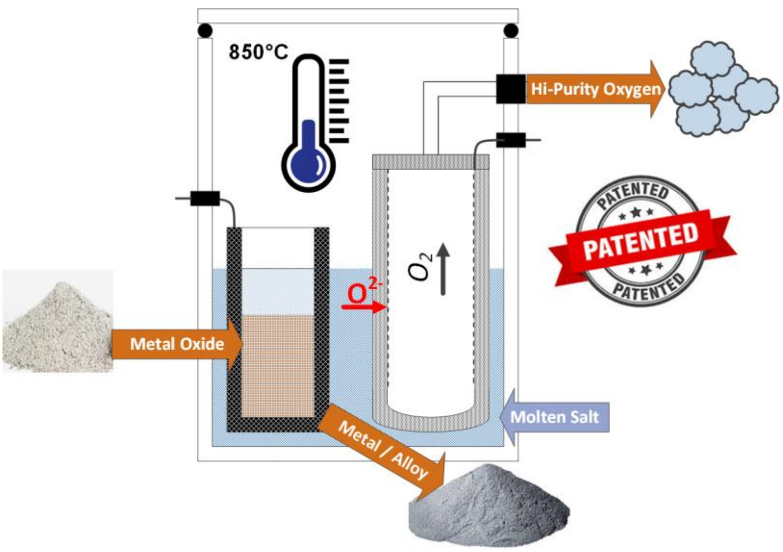


Figure 4: Principle of the ROXY Process

2.1.1 Main Features of the ROXY process

The main features of the ROXY process that make the process attractive for regolith reduction are the solid oxide membrane and the use of an optimized fluoride salt electrolyte, as discussed in the following sections.

2.1.1.1 Solid oxide membrane (SOM)

Yttria-stabilized zirconia (YSZ) is one of a number of ceramic materials that can conduct electrical current via oxygen ion transport. In contrast to the concentration gradient-driven separation employed by traditional membrane separators, separation of oxygen in an electrolytic cell is an electrochemical process, driven by electrical energy supplied by a direct current power source. This fact gives solid electrolyte membrane separators several advantages over traditional membrane-based oxygen separation schemes including *infinite selectivity* and the ability to deliver oxygen at elevated pressures (pumping).

The fluorite crystal lattice of yttria-stabilized zirconia contains oxygen ion vacancies. When an electric field is applied to such crystal oxygen ions migrate between vacancies, creating an appreciable oxygen ion conductivity. An oxygen separation cell is formed when a membrane of yttria-stabilized zirconia (YSZ) is sandwiched between two gas-permeable electrically conductive electrodes. Once in the crystal lattice, oxygen ions move under the influence of the electric field provided by the power supply. The transport of ions between vacancies is moderated by the presence of an activation energy barrier resulting in an effective ionic resistivity, see also Figure 5.

Because the vacancies responsible for oxygen ion conduction in materials like YSZ accept only oxygen ions, membrane selectivity is infinite, regardless of the other gases present in the feedstock. This results in *100% pure product oxygen* in a single separation stage. For example, the production of 99.99% oxygen has been demonstrated with an oxygen generator designed using thick–wall (0.5–0.6 mm) solid electrolyte tubes based on zirconium dioxide manufactured according to the conventional ceramic technology and Pt electrodes and current leads.

In terms of oxygen production rate, various authors report on current densities of 1.0 A/cm^2 and more that are used with YSZ membranes. Other membrane materials allow even larger current densities such that membrane-based oxygen extraction facilities can be used on a macroscopic, industrial scale, see also examples below.

<u>Technology Heritage</u>
Electrochemical oxygen <u>pumps</u> have been extensively covered in the literature for many years. The process has been found to be a suitable source of pure oxygen, including high pressure oxygen, or an oxygen free envionment, i.e. oxygen can be electrochemically pumped and compressed to high pressures in a single stage.
For example, it is possible to remove oxygen which exists in the percent or parts per million range from nitrogen or a noble gas supply, such that purification of inert gases to an oxygen content of about 1 ppm is readily achievable. An important element of the pump design is the choice of the membrane material, the most preferred being zirconia. The key property of the membrane material is that it has a high thermodynamic decomposition potential, i.e. the membrane must be stable under the influence of high voltages at elevated temperatures. Yttrium-stabilized zirconia (YSZ) has such stability.
With respect to the longevity and scalability potential of the technology, it has been reported that the process exhibits exceptionally high rates of oxygen flux, is capable of separating industrial fluid streams, and exhibits excellent long-term stability of the material in air at elevated temperatures, and the high oxygen flux.
The process is therefore well suited for producing pure high-pressure oxygen,
Its main benefits include:

- Simple: Eliminates pumps and rotary compressors and does not require compression sealing
- Reliable: Contains no moving parts, increasing reliability and requiring minimum maintenance
- Compact: Reduces device mass and volume compared to current oxygen-generation devices
- Stable: Avoids large pressure changes across individual cells
- Product quality: directly produces highly pure "medical grade" oxygen.

YSZ elements are widely used as <u>oxygen sensors</u> in industrial applications, e.g., by Metrotec (Figure 5), or SST Sensing. YSZ sensors are compact, robust, reliable, and offer measurement ranges over several orders of magnitude. For example, the Metrotec product has a measurement range of 100% to 10^{-31} bar oxygen, and an uncertainty of log(pO2/bar) <0.02. An oxygen control system with an YSZ oxygen pump and an YSZ oxygen sensor from Airbus (Figure 5) has confirmed this figure over 17 orders of magnitude of pressure.

Figure 5: Left: Principle of oxygen ion conduction in yttria-stabilized zirconia, center: commercial oxygen sensor (Metrotec), right: combined oxygen pumping and sensing element for EML OCS on ISS (Airbus).

<u>Large-scale production</u> of high-purity oxygen with ceramic membrane technologies is an area of active research. Fraunhofer IKTS has built the largest membrane plant worldwide for high-purity oxygen production from air, based on MIEC (Mixed Ionic Electronic Conductor) membranes. At high temperatures, these are permeable only for pure O_2. Since 2009, several demonstration plants using these membranes have been realized.

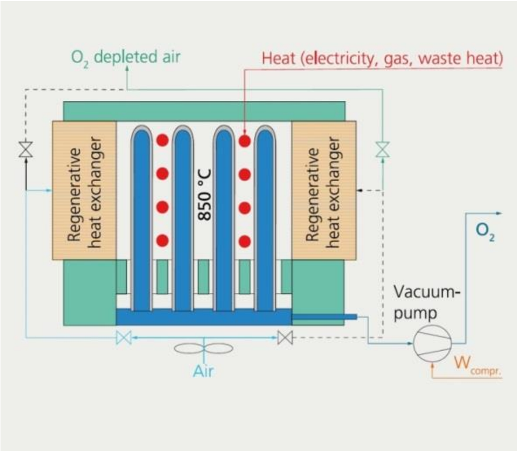


Figure 6: Largest membrane plant worldwide for the production of 14 kg O_2/h. © Fraunhofer IKTS

<u>Applications in Electrochemical Reduction of Metal Oxides</u>

The use of solid oxide membranes for electrochemical reduction of metals oxides offers a number of advantages due to the features discussed above, see also Figure 7. The SOM membrane allows oxygen ions to pass through but essentially blocks the transport of anything else, including other gases, salt vapor, and electrons.

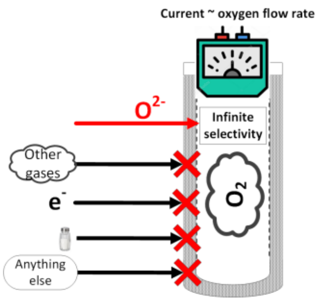


Figure 7: Solid oxide membrane as part of an anode assembly in an electrochemical cell

The SOM process therefore is particularly interesting for the direct production of oxygen since it uses a zirconia oxygen ion pump as the anode in combination with a fluoride salt electrolyte. As a result, higher voltages and current densities are possible and the energy efficiency of the reaction increases. To the best of our knowledge, no other anode technology can achieve these results because all other anodes produce gases in the electrolyte that react with the metals produced and the electrodes, or corrode the reaction chamber.

The SOM process has been applied widely over the last decades, and has been applied to reduce various metal oxides or oxide compounds to the respective metals or alloys, including Mg, Al, Ti, Ca, Fe, Cu, Ta, Cr, Nb, Yb, Nd, Pr, Dy, Si, CeNi5, $La_xCe_{1-x}Ni$, Ti–Fe alloy, Ti–Si intermetallics, etc. See also Figure 8. It is therefore ideally suited to reduce regolith.

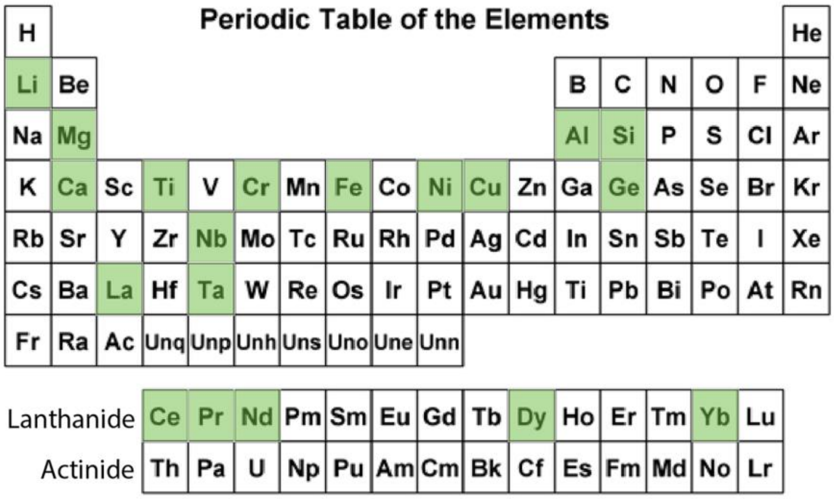


Figure 8: Metals produced to date by the SOM electrolysis process (highlighted in green in the chemical periodic table)

Summary of YSZ SOM benefits

- YSZ anodes have been widely used in molten salt electrolysis processes for improved process performance
- The electrical conductivity of the YSZ membrane is high and well suitable for molten salt electrolysis.
- The achievable current density of YSZ membranes is above 1 A/cm^2, which is far above what is needed for a lunar demonstrator or even a scaled-up lunar pilot plant
- The lifetime of YSZ membranes in contact with molten fluoride salts is suitable for a lunar demonstration mission, and can be further enhanced by fine-tuning the composition of the salt electrolyte
- With YSZ anodes the current efficiency of the process can be increased to up to 100% since the YSZ membrane blocks the electronic current path to the anode. This is a big advantage since it allows increasing the energy efficiency of the reaction.
- The maximum cell voltage can be increased when using YSZ membranes since the membrane remove the constraint due to the decomposition potential of the salt. As a result, much higher cell voltages are achievable, resulting in much higher reaction rates and oxygen production rates.
- Advanced membrane materials (scandia- and ceria-based) are available for future optimizations of the process, but are not required for a lunar demonstration

2.1.1.2 Fluoride salt electrolyte

There are two type of molten salts that are commonly used in the electrolysis of metal oxides: chloride and fluoride salts. A wide range of salt mixtures is available to tailor to the needs of a specific process. In particular, the process temperature can be established by using a dedicated salt mixture. Low process temperatures can be achieved by using eutectic salt mixtures.

Fluoride salts are preferred over chloride salts due to their superior properties for molten salt electrolysis:

- Their current efficiency is higher, leading to a higher energy efficiency of the process,
- Their decomposition potential is higher, which allows operating the process at higher potentials thus increasing the oxygen production rate
- Their volatility is lower which is an advantage since it allows running the process for a long time without major changes occurring in the composition of the salt electrolyte, and it is more compatible with the lunar environment, in particular vacuum conditions.

Fluoride salts are less aggressive than chloride salts and are compatible with YSZ anodes in terms of corrosion when properly selected. Fluoride salts have been used to reduce a wide range of metal oxides on a lab scale, industrial applications are under development. Operational and safety procedures related to the handling of fluoride salts are available from molten salt electrolysis applications and nuclear applications where fluorides are used as coolants in molten salt reactors.

The operating temperature of the ROXY process can be tailored to the application in a certain range, from 800 °C to 1100 °C. The temperature depends mainly on the composition of the salt mixture and design details of the anode. For ROXY, the standard process temperature is as low as 850 °C.

2.1.2 Main Benefits of the ROXY process

The ROXY process has a number of benefits both for the electrochemical process, the design of an overall system, and the development of a lunar payload. Compared to other molten salt electrolysis processes, the implementation of the concept will result in a drastic simplification of the reactor and downstream gas management system, and will result in a robust, small, low mass, simple and compact overall system design with very low requirements for maintenance and/or replenishment of consumables, as shown in Figure 9.

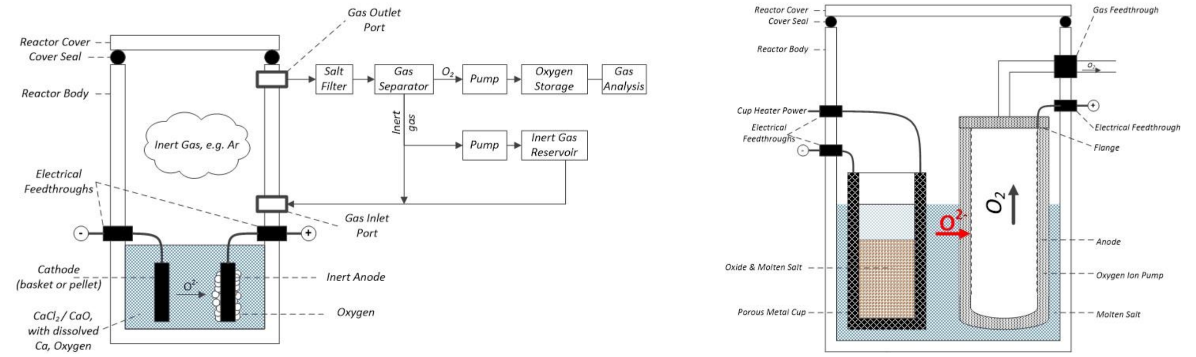


Figure 9: Benefits of the ROXY Process: left: Electrochemical process without SOM anodes with ancillary equipment shown in boxes, right: ROXY process which does not need such ancillary equipment.

This is due to features that are unique to the ROXY process and not achievable with other molten salt electrolysis processes, such as:

- Direct one-step production of oxygen
- Largely consumable-free process
- No corrosion issues with reactor, cathode, and reduced regolith, due to extraction of oxygen from the reactor with the SOM-type anode
- Near 100% current efficiency

leading to:

- A much simpler, smaller reactor
- Lower complexity and mass
- Higher energy efficiency

The unique features that contribute to these benefits are discussed below:

<u>Anode corrosion</u>

In other molten salt electrolysis processes, the anode is exposed to a pure oxygen and molten salt environment at high temperature, which is very challenging from a materials compatibility point of view. The solid oxide membrane between the salt and anode removes this problem.

<u>Oxygen separation</u>

Since the anode is “behind” the solid oxide membrane the oxygen is separated from the molten salt. The solid oxide membrane therefore prevents the pure oxygen gas from reaction with the reactor vessel or the reduced regolith, i.e., no oxygen is lost to corrosion with the reactor vessel and the quality of the reduced regolith is improved. Contamination of the produced oxygen with inert gas from the reactor or molten salt is eliminated due to the near-infinite selectivity of the solid oxide membrane for oxygen.

<u>Energy efficiency</u>

As another key benefit, parasitic currents, e.g., due to electronic conduction, are eliminated since the electronic conduction path from the cathode to the anode is cut by the solid oxide membrane. Consequently, higher voltages and current densities are possible and the energy efficiency of the reaction increases. To the best of our knowledge, no other anode technology can achieve these results because all other anodes produce gases in the electrolyte that may react with the metals produced, the electrodes, or the reaction chamber.

<u>System Benefits</u>

Benefits on the level of a ROXY system arise from the much simpler architecture and lower complexity of a ROXY-based reactor compared to a classical molten salt electrolysis reactor:

- The need for a gas system to manage/recirculate the inert gas, including gas storage, compressors, instrumentation and piping, is eliminated.
- A dedicated system to separate the inert gas from the produced oxygen, and a salt particle filter are not needed as the solid oxide membrane already performs this function
- The leak rate requirements for the reactor cover are drastically reduced, since the process does not need a process gas, i.e., the function of the cover seal is limited to the containment of the salt vapor. Also, the lower pressure differential between reactor and environment will have benefits with respect to container material selection and wall thickness, and therefore system mass.
- Due to its low volatility, the evaporation of the specific ROXY salt electrolyte mixture will be very limited. The evaporated salt electrolyte will be contained by the reactor. Salt loss due to evaporation which could be a major concern is therefore not an issue.
- Since the presence of molecular oxygen in the salt bath is essentially eliminated, corrosion of reactor elements due to unwanted oxidation reactions will be drastically reduced.
- A dedicated system to analyze the composition of the produced oxygen, such as a mass spectrometer is not needed, since the solid oxide membrane is highly selective, and high-purity oxygen is directly produced. Note that YSZ-based oxygen ion pumps are considered for medical applications.
- Due to the elimination of electronic conduction in the salt bath, the efficiency of the reaction is increased and the power required by the reactor is reduced.
- Due to the compact reactor design, the mass, accommodation and thermal control requirements are reduced.
- Few active elements such as sensors and actuators are needed to monitor and control the reactor, leading to lower requirements for the control electronics.

2.1.3 Process Diagnostics

For a comprehensive understanding of the electrochemical extraction of oxygen and metals from regolith and for the future development of larger systems, such as a pilot plant, it is crucial to be able to diagnose the process and its main elements. In particular, since the composition of the regolith at the landing site is not known a priori, there is some uncertainty regarding the interaction of the lunar regolith with the salt electrolyte. During the reduction runs, the electrolyte may change its properties, such as composition, electrical conductivity, viscosity, etc. These changes may influence mass and charge transport processes and affect the performance of the electrolytic cell and therefore must be understood to evaluate regolith reduction runs on the Moon. These and other effects can be determined by electrochemical modeling of the cell performance assisted by various potential-current measurements including frequency dependent electrochemical impedance spectroscopy (or EIS) measurements.

The principle of EIS is shown in Figure 10: the cell voltage is modulated by a small amplitude and varying frequencies, in a typical range between 1 Hz and 100kHz. The current response of the system is measured, and the complex impedance of the system is evaluated as a function of frequency, from which the physical and electrochemical phenomena that occur in the electrochemical cell can be characterized.

Electrochemical impedance spectroscopy is therefore the method of choice for process diagnostics. It is an established method for characterization of electrochemical systems. Extensive experience with EIS for process diagnostics has been gained by the team over many years with the SOM process. The ROXY process enhanced by electrochemical impedance spectroscopy is therefore ideally suited for the extraction of oxygen from lunar regolith and for scientific investigations of the reduction process.

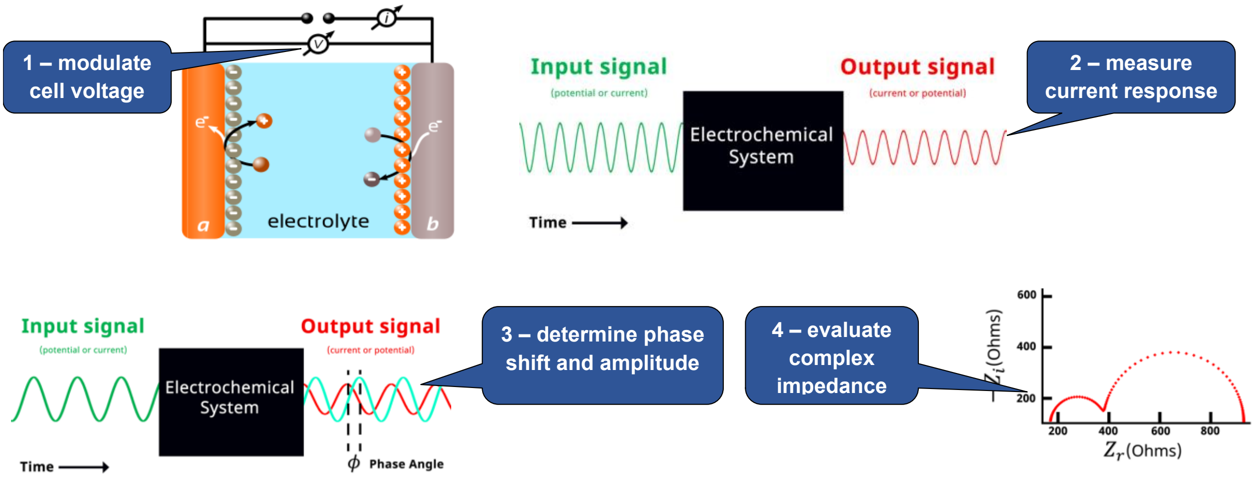


Figure 10: Principle of electrochemical impedance spectroscopy

2.2 Mini-ROXY: The Next Step Towards Resource Efficiency

Mini-ROXY is a process that incorporates all beneficial features of the ROXY process, specifically porous metal cathodes, an optimized fluoride salt electrolyte, and YSZ solid oxide membrane anodes, in a design implementation optimized for compactness and very low resource requirements. This is achieved by using the YSZ membrane not only as an anode, but also as the crucible for the molten salt electrolyte. As a result, the reactor vessel normally required in molten salt electrolysis is eliminated and the size and mass of the system is significantly reduced. A Mini-ROXY system therefore offers the possibility to achieve lunar demonstration objectives with a very compact set-up. Furthermore, the concept is very flexible, i.e., different system sizes are possible. Such a Mini-ROXY system can be accommodated on a wide range of rovers and landers. Finally, a Mini-ROXY system can be modularly expanded with additional elements, such as diagnostic systems to characterize the regolith composition.

The principle of the Mini-ROXY setup is shown in Figure 11, which provides a schematic representation of ROXY and Mini-ROXY setups: The standard ROXY setup includes a cathode cup and a YSZ tube, which are placed in a reactor vessel that also contains the molten salt. In Mini-ROXY, the cathode cup is inserted into the YSZ tube, which also serves as a crucible for the molten salt. The reactor vessel is therefore eliminated in the Mini-ROXY setup, resulting in a significant reduction in the size of the cell. The figures are intended to illustrate the principle and are not to scale.

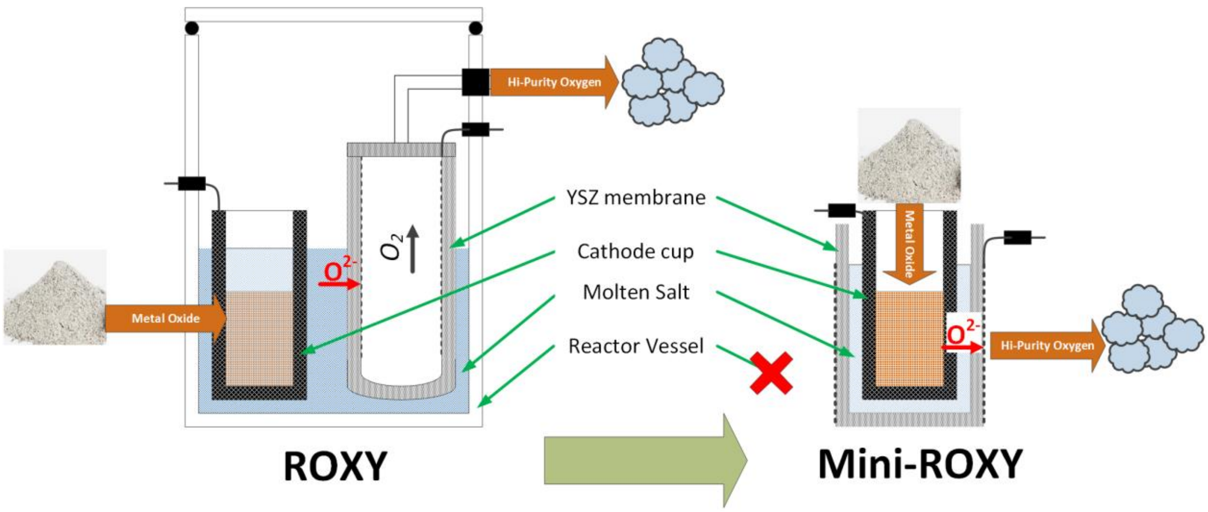


Figure 11: Schematic representation of ROXY and Mini-ROXY setups

Mini-ROXY System Concept

The system concept of a facility based on the Mini-ROXY process is shown in Figure 12. One or more cartridges are integrated in a reactor body. The number and size of the cartridges can be tailored to meet requirements on oxygen production rate. The thermal insulation around the cartridges can be adapted to the available resource budgets for the system, in particular mass and power. System power consumption can be reduced by adding more thermal insulation; however, this comes with a mass penalty. The flexibility that is achieved with this concept allows tailoring the Mini-ROXY principle to different mission scenarios from lunar demonstration to larger-scale pilot facilities.

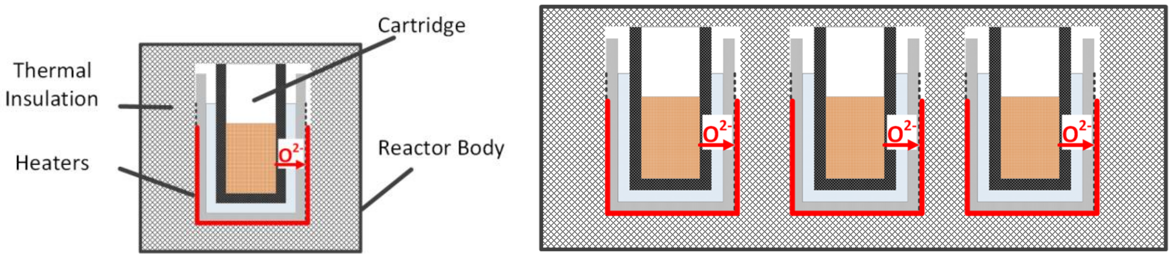


Figure 12: System concept of a facility based on the Mini-ROXY process. The simplest configuration includes one cartridge integrated in a reactor body that provides heaters, thermal insulation, etc. (left). This can be expanded by integrating several cartridges into one common reactor body (right).

Lunar Demonstration

Benefits for a lunar demonstration arise from the fact that a Mini-ROXY lunar demonstrator will be small and can therefore be flexible in terms of accommodation. This will allow the demonstration and characterization of oxygen production on the Moon in a cost-effective manner, and due to its low complexity, it will allow a rapid development cycle. Mini-ROXY is therefore very attractive for small-scale targeted science investigations in the context of a lunar demonstration because it has a unique set of features:

- Full implementation of the ROXY process on a small scale.
- Demonstration of oxygen and metal production from regolith
- Moderate process temperature (<850°C)
- State-of-the art process diagnostics by electrochemical impedance spectroscopy
- Simple and low-cost solution
- Small cartridge as core element
- Lunar model is small and compact and can therefore be accommodated on a lander or rover
- Low resource requirements of lunar model
- Designed as the basis for a rapid development cycle of a low-cost lunar model to demonstrate oxygen production on the Moon
- Modularly expandable with other equipment, e.g., for regolith and oxygen processing and diagnostics
- Possibility to operate a Mini-ROXY demonstrator as part of a larger facility or as part of an ensemble of facilities that may be provided by external partners. This includes, for example, rovers, systems for regolith extraction and processing, and further processing of the extracted oxygen, see also chapter 3 for more details.

From the above, it is clear that Mini-ROXY provides a unique opportunity to quickly and effectively conduct a lunar demonstration of oxygen production from lunar regolith.

Scalability

It may appear counter-intuitive to miniaturize a process when the long-term objective is a scale-up to industrial production levels. However, the relevant figures of merit are the ratios of reactor mass/size/power to the amount or rate of material produced. This is where the large advantage of the Mini-ROXY concept is: by eliminating or miniaturizing elements that are not really contributing to the performance of the process, these figures of merit are improved.

A scale-up of the process can be achieved by using a large number of cartridges that are only moderately increased in size, according to the principle shown in Figure 12. With this approach, the cartridges of a Mini-ROXY lunar demonstrator can be considered the nucleus of a future scaled-up facility, and therefore the Mini-

ROXY demonstration size facility is relevant for scale-up. It has been shown that the energy efficiency of scaled up ROXY facilities is higher than that of a demonstrator. This is addressed in more detail in chapter 0.

In summary, Mini-ROXY provides unique benefits both for a lunar demonstration and for future upscaling, as shown in Figure 13.

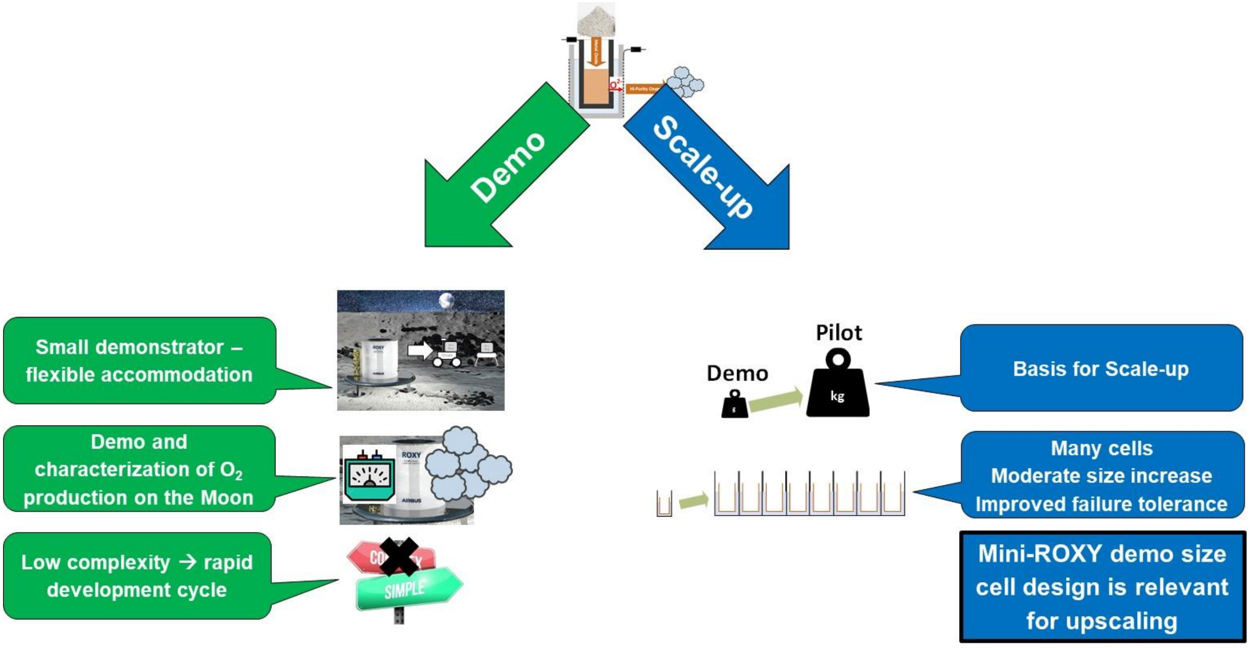


Figure 13: Mini-ROXY Benefits for Lunar Demonstration and Upscaling

3 Mini-ROXY Lunar Demonstration Mission

A lunar demonstration is one early step towards the development of larger scale capabilities on the Moon, as shown in Figure 14. A lunar demonstration should therefore be:

- relevant for later upscaling
- flexible in terms of accommodation, i.e., small and compact
- limited to the demonstration needs (focus on feasibility and knowledge gain for later upscaling)
- quickly implementable
- affordable

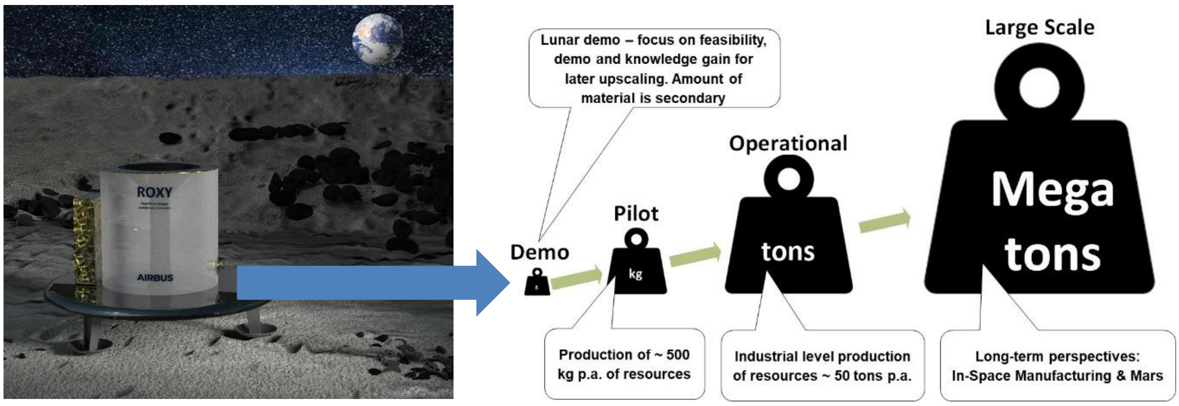


Figure 14: Lunar demonstration in a larger context. Left: Artist´s concept of a lunar Mini-ROXY demonstrator, right: Lunar production scales.

With this in mind, a Mini-ROXY lunar demonstration mission has been conceived that takes advantage of the features of Mini-ROXY introduced in the previous chapter. The mission concept is based on a minimum viable product approach that is discussed in section 3.1. The mission objectives that have been derived from this approach are presented in section 3.1.1 which also includes a comparison with the ISRU demonstration mission objectives defined in the NASA LIFT-1 RFI. The implementation approach for a basic version of the demonstrator is then presented in section 3.1.2.

The resulting architecture of the lunar demonstrator is compared to the building blocks of the Notional NASA ISRU Oxygen Extraction Reference Demonstration Concept defined in the NASA LIFT-1 RFI in section 3.2.

A key issue for the justification of any ISRU demonstration on the Moon is the question which of the steps of the end-to-end process chain are dependent on the lunar environment, in particular the reduced gravity and the presence of true lunar regolith. This is covered in section 3.3, followed by a complementary analysis in section 3.4 of the process steps that at NOT dependent on gravity and regolith and can therefore be covered by terrestrial qualifications.

Section 3.5 provides high-level technical data of the Mini-ROXY lunar demonstrator and a compliance matrix with the generic CLPS lander payload interfaces as defined in the PRISM-3 call.

Section 3.6 provides an in-depth discussion of scientific mission objectives for each major building block or process step of the Mini-ROXY lunar demonstrator, starting with an assessment of how much each of the building blocks is dependent on gravity and/or the properties of true lunar regolith.

3.1 Small is Beautiful: Mini-ROXY Minimum Viable Demonstrator

The need for a lunar demonstration arises because not all factors that have an impact on the process steps can be adequately simulated on Earth. This applies in particular to any process steps that are dependent on gravity, on the properties of true lunar regolith, or both.

A key objective and justification of a lunar demonstration mission is therefore to demonstrate the extraction of oxygen from regolith with the ROXY process under lunar conditions as a first step towards larger scale lunar ISRU facilities. The main goal of such a demonstration is to characterize the oxygen production process using state-of-the-art process diagnostics. To achieve this goal, it is not necessary to produce a large amount of oxygen. Minimizing the amount of oxygen to be produced to the demonstration objectives therefore opens up the possibility of designing a cost-optimized lunar demonstration mission with a simple, compact and low-mass demonstration facility – such as the Mini-ROXY system. This leads to the definition of a minimum viable product. A key mission objective is therefore to optimize the cost/benefit ratio of the mission.

On the other hand, even a minimum viable product needs to demonstrate that the ROXY process will work under lunar conditions, in particular at 1/6 g and with true lunar regolith as feedstock. And finally, the demonstration should provide the knowledge to develop larger-scale ROXY facilities.

The basic version of the Mini-ROXY lunar demonstrator has been conceived as a minimum viable product with these considerations in mind. It is the result of a radical simplification exercise, with features that have been reduced to the bare minimum. The resulting concept provides a low-complexity starting point for defining the scope of the lunar demonstration. Features may be added to this basic version depending on mission, science, programmatic, and technical objectives and constraints.

3.1.1 Mission Objectives

The mission objectives that have been derived from the minimum viable product approach are presented in section 3.1.1.1 below. For reference, the mission objectives defined in the NASA LIFT-1 RFI are provided in section 3.1.1.2. A comparison and compatibility assessment of both sets of mission objectives is provided in section 3.1.1.3.

3.1.1.1 Mini-ROXY Lunar Demonstration Mission Objectives

Primary Mission Objectives

- Demonstrate the extraction of oxygen from regolith with the ROXY process as a first step towards larger scale lunar ISRU facilities
- Develop a payload design that meets the process viability criteria, in particular low resource requirements, compactness, simplicity
- Develop a payload design that will optimize the benefit-cost ratio of the demonstration mission, i.e., allow for a cost-effective mission with a payload that can be developed rapidly, while meeting the scientific requirements of the demonstration mission
- Develop a payload design and operational concept that will provide information needed for future scale-up of the process by including state-of-the-art process diagnostics
- Develop a modular payload complement architecture that will support collaboration with third parties that could provide elements of the mission or payload complement
- Actively pursue international collaboration, both in terms of hardware provision and scientific collaboration
- Develop a payload design that allows measurement of the amount of produced oxygen based on the oxygen ion current through the YSZ membrane

Secondary Mission Objectives

- Perform beneficiation of the raw regolith, e.g., in terms of grain size distribution, chemical or mineralogical composition to support systematic studies of the reduction process as a function of feedstock properties
- Perform independent in-situ measurements of oxygen flow rate and purity
- Develop a payload design that will support sample return of processed regolith and extracted oxygen to Earth (Note that this applies in particular to man-tended missions)
- Develop a payload design and operations concept that contributes to the objectives of the MEFAM concept, i.e., the Metals Factory on the Moon (briefly introduced in chapter 7), by addressing some of the steps in the end-to-end metal product value chain, to the extent possible with regards to other mission constraints.
- Perform a ground-based characterization of processes to extract metals and alloys from regolith.

Note on metal extraction: Extraction of reduced regolith from the cartridge on the Moon is not included in the mission objectives since it will increase the complexity of the payload. Instead, this feature will be subject to a ground demonstration campaign. On the other hand, the concept of the lunar demonstrator includes the capability to perform multiple regolith reduction runs, see section 3.1.2 for details.

3.1.1.2 NASA Mission Objectives – from LIFT-1 RFI

The following mission objectives are stated in the NASA LIFT-1 RFI:

<u>Primary Technology Objectives and Demonstration Concept</u>

- The end-to-end process of extracting, producing, and storing oxygen from lunar regolith involves a series of operations that will occur without surface human interaction. Figure 18 (from NASA LIFT-1 RFI, reproduced in section 3.2 below), NASA ISRU Oxygen Extraction Reference Demonstration Concept (provided as reference only), depicts the functional steps needed to both perform these operations as well as to understand the performance of the oxygen extraction process and system chosen. The green boxes depict what NASA considers as the minimum functional elements that should be included in the oxygen extraction demonstration.

- ISRU demonstration operations only need to be performed within a single lunar day to minimize costs. The number of processing operations and cycles performed should be tied to addressing: (a) performance with actual regolith in lunar environment and (b) repeatability and processing durations. A regolith processing cycle is defined as: (i) feeding unprocessed regolith in the processing reactor, (ii) performing regolith processing to extract oxygen to a predefined extraction performance amount, and (iii) discharging processed regolith to allow for new unprocessed regolith to be introduced into the reactor. A minimum of three (3) regolith processing cycles should be performed, with additional cycles highly encouraged.

- All demonstration hardware should fit on a single lunar lander with pre-integration and checkout of the oxygen extraction from regolith module.

- The ISRU demonstration should include sufficient instrumentation to determine performance with actual lunar regolith versus Earth performance data using lunar simulants.

- For minimum risk and cost, the lunar regolith used for initial processing could be obtained at the lander location.

Ancillary Objectives

Optional objectives are additional infrastructure objectives that may or may not be directly relevant to this demonstration or could be successfully demonstrated through other means. Ancillary objectives are functions that will need to be implemented for a fully operational system and would enhance information on how well this demonstration performed and provide additional insight into how to improve in future demonstrations.

Utilize regolith geotechnical, mineral, and elemental instrumentation to:

(1) allow for comparison with lunar simulants,

(2) allow for better understanding of the minerals reduced during oxygen extraction operations, and

(3) provide science information on the distribution of regolith minerals around the landing site.

Measure the types and amounts of gases released before, during, and after each regolith processing cycle.

3.1.1.3 Comparison between Mini-ROXY Mission Objectives and NASA Mission Objectives

NASA mission objectives as stated in the LIFT-1 RFI mission objectives are comprehensive, addressing all steps of the end-to-end process of extracting, producing, and storing oxygen from lunar regolith. On the other hand, Mini-ROXY mission objectives are limited to the demonstration of the oxygen extraction, and it is assumed that functions that are not linked to the “core” process of regolith reduction will be performed by equipment external to the Mini-ROXY reactor.

On the process level, the objectives are similar, with the exception of the repeatability which is part of the NASA mission objectives while Mini-ROXY mission objectives do not include in-situ extraction of the processed regolith. These functions are planned to be tested on ground. However, a capability to perform the extraction could be added to the basic version of the Mini-ROXY demonstrator, and would lead to impacts in terms of complexity, development schedule and cost.

3.1.2 Implementation Approach

The following section describes the implementation approach of the basic version of the Mini-ROXY lunar demonstrator by identifying the main building blocks of the demonstrator, followed by a section that provides a mapping of these building blocks to the functional blocks for an ISRU demonstration as identified in the NASA LIFT RFI.

Mission Elements

This project will provide a Mini-ROXY lunar demonstrator that will receive and process the regolith, produce the oxygen, characterize the process and the product oxygen – as indicated by the green box (core part/this project) in Figure 15.

Other mission elements such as the lander, regolith acquisition and transfer to the Mini-ROXY reactor as well as the characterization of the regolith feedstock are assumed to be provided by external partners – as indicated by the orange box (partner-provided) in Figure 15. This may include regolith beneficiation.

The Mini-ROXY demonstrator provides the diagnostics to evaluate the oxygen production rate. Oxygen purity will be very high and is driven by the physical properties of the YSZ membrane, see below for details. Oxygen purity sensors are therefore not included in the demonstrator. Such sensors and other downstream oxygen management equipment could be added to the scope of the mission. Such functions could be provided either by the Mini-ROXY demonstrator or by external equipment - as indicated by the blue box in Figure 15.

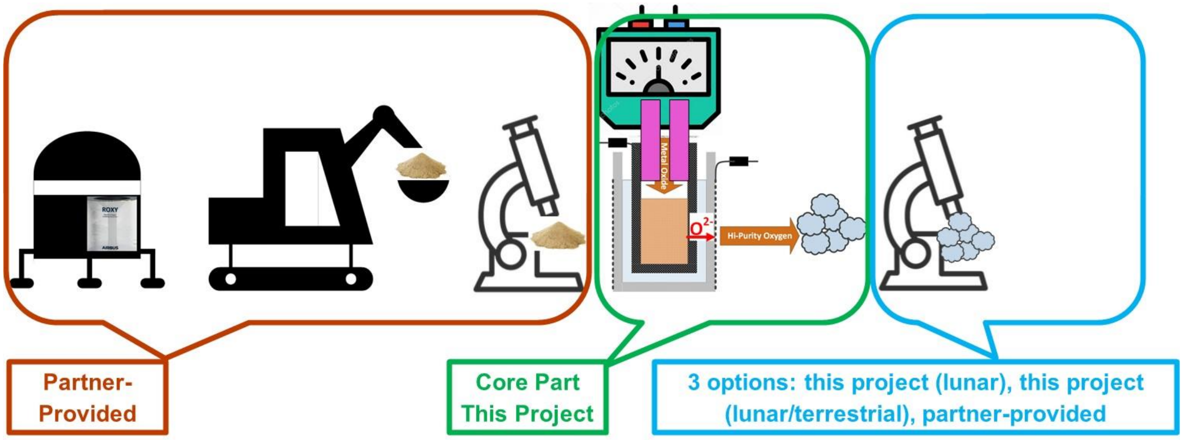


Figure 15: Mini-ROXY lunar demonstration mission elements

Dimensioning of the reaction volume

The focus of the demonstration is on feasibility and process analysis. Rather than producing a certain amount of oxygen, the process is therefore dimensioned to produce only an amount of oxygen that is required for a meaningful demonstration. A few hours of electrolysis will be required to gather data. Running the electrolysis for 10 hours at an electrolysis current of 0.5 amps will produce about 1 standard liter of oxygen, which in turn corresponds to about 3 grams of regolith. Cartridges are therefore designed to accept about 3 grams of regolith, plus margin.

Duration of the process demonstration on the Moon

Many landers are not designed to survive the lunar night. Mini-ROXY lunar demonstrator surface operations are therefore limited to less than one lunar day, which is well compatible with the process design introduced above.

Redundancies

The original Mini-ROXY concept includes one cartridge. It is considered that the mission risk is increased if only one cartridge is available. Therefore, the Mini-ROXY lunar demonstrator will include 3 cartridges for redundancy and risk reduction, as shown in Figure 12 in section 2.2 above. Note that multiplying the cartridges is also the principle that will be use to scale up the process later on.

Process and Product Diagnostics

The main diagnostics objectives are to:

1. understand the properties of the regolith feedstock,
2. understand in detail the thermophysical and electrochemical processes that contribute to the overall electrochemical reduction process, and
3. the properties of the produced oxygen.

These objectives are addressed as follows:

1. Relevant regolith feedstock properties include chemical and mineralogical composition as well as the particle size distribution. It is assumed that the characterization of these properties is performed by external equipment.
2. Detailed characterization of the reduction process: Electrochemical impedance spectroscopy (EIS) is a versatile and in-depth method for process diagnostics of electrochemical cells, is well established and is

therefore adopted as the prime characterization method for Mini-ROXY. The principle of EIS is illustrated in section 2.1.3.

3. Oxygen characterization: A simple way of determining the oxygen production rate, which is already available in the system, is to measure the current through the YSZ membrane. Oxygen ions are the only charge carriers that can move through the YSZ, so the current is a measure of the amount of oxygen that passes through the membrane per unit of time. This principle leads to the simplest possible configuration without the necessity to collect the oxygen in a volume or container. In this implementation, the oxygen is released directly into the lunar vacuum. A gas system for oxygen capture and analysis is therefore not necessary. Alternatively, it is possible to measure the amount/rate of oxygen produced with a gas flow sensor. This requires the oxygen to be collected, i.e., this option would require a gas system, which in turn increases the mass of the system. Furthermore, such a gas system could be extended by an oxygen sensor to determine the purity of the oxygen produced on the moon. However, it is known that YSZ has an almost infinite selectivity with respect to oxygen, see section 2.1.1.1 for details.

<u>Cartridge configuration and exchange, materials extraction and sample return</u>

The cartridge is the element in which the reduction takes place. It is a tubular structure that contains the YSZ crucible, the cathode cup and the salt, and is accommodated in an overall housing, as introduced in section 2.2. The regolith is inserted into the top of the tube via a funnel. Before the salt is melted, the tube is sealed to reduce evaporation of the salt. Once the process has started, oxygen ions are generated by regolith reduction and conducted through the YSZ membrane. At the anode, molecular oxygen is generated and released to the lunar environment or to optional systems for gas analysis and/or storage.

The cartridges of the ground-based laboratory model can be removed manually after reduction. The generated material can then be extracted. After this process, the cartridge can be reprocessed, prepared for the next reduction run and reinserted into the reactor. This manual process means that the cartridges of the ground-based laboratory model can be reused.

The cartridges of the basic version of the lunar demonstration model, on the other hand, are intended for single use in order to reduce a batch of a few grams of regolith. An extraction of the processed regolith on the moon and/or exchange of processed cartridges are not absolutely necessary for achieving the demonstration objectives. They are therefore not included in the basic version of the lunar demonstrator. Adding such features will increase the complexity of the demonstrator. The impact will depend on the feature, i.e., material extraction from the cartridges and/or exchange of processed cartridges, and the type of mission, i.e., robotic or man-tended.

While for a robotic mission, the addition of such features would significantly increase the design complexity, they could be more easily performed in case of a man-tended mission, wherein extraction and replacement of processed cartridges are performed by an astronaut. Extracted cartridges could then be returned to Earth for post-flight analysis.

The configuration of the cartridges and options for cartridge exchange in the ground test campaign, for a robotic mission (basic version) and for a man-tended mission (basic version) are shown in Figure 16. The operational workflow for a man-tended mission with cartridge exchange and sample return is shown in Figure 17.

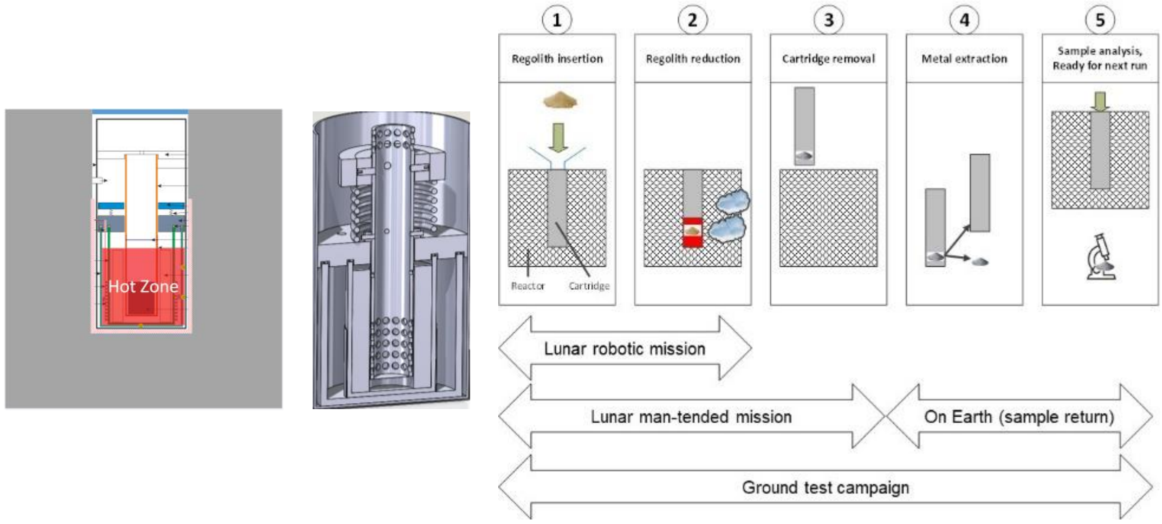


Figure 16: Left: cartridge configuration with hot reaction zone at bottom, heaters and thermal insulation, center: cartridge design, right: options for cartridge exchange in the basic version of the demonstrator for: a lunar robotic mission, a lunar man-tended mission with sample return, the ground test campaign.

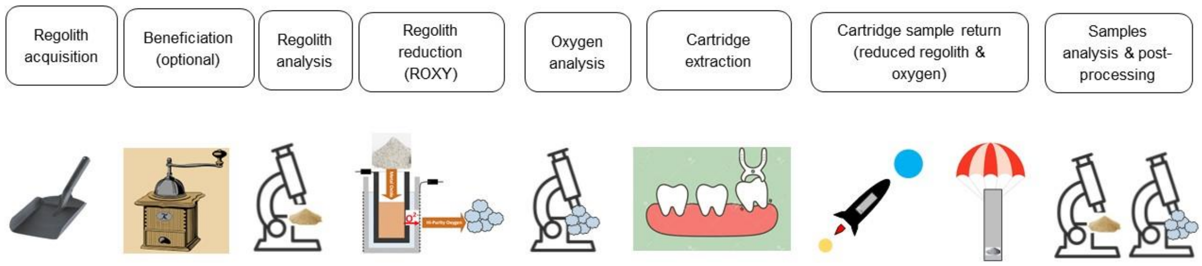


Figure 17: Operational workflow for a man-tended mission with cartridge exchange and sample return

<u>Resource constraints from moon lander, in particular mass, power/energy, and volume</u>

A Mini-ROXY demonstrator could be accommodated on a large number of moon landers or on a man-tended mission. The capabilities of these carriers in terms of payload mass, volume and power are generally significantly higher that what will be needed for a Mini-ROXY lunar demonstrator. It is therefore intended to design the Mini-ROXY lunar demonstrator for low resource needs such that it can be accommodated on a number of carriers as a secondary payload. The such achieved flexibility will increase mission options for the Mini-ROXY lunar demonstrator.

It is considered that the resources, in particular mass and power, are not independent: a low power consumption can be achieved by adding more thermal insulation around the cartridges, but this will obviously have a mass penalty. This means that the design can be optimized in different directions. In order to define a baseline design of the demonstrator, it is therefore necessary to assume a reference lander with defined resource constraints. For the present project phase, the generic CLPS lander as defined in the PRISM-3 call is used as reference. See also section 3.5 for more information on technical data of the demonstrator and compliance with lander capabilities.

3.2 Mini-ROXY Lunar Demonstrator Building Blocks

This section provides an assessment of how the functions provided by the Mini-ROXY lunar demonstrator relate to the building blocks that are considered necessary for a lunar oxygen extraction demonstration. The reference that is used for this assessment is the NASA ISRU Oxygen Extraction Reference Demonstration Concept, published as part of the LIFT-1 RFI, shown in Figure 18.

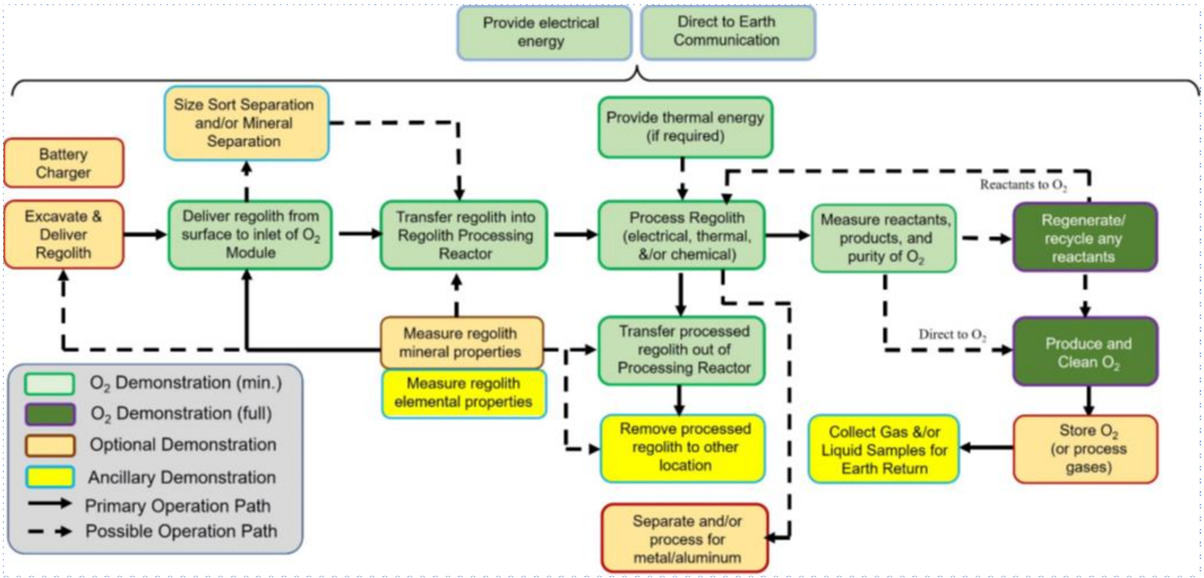


Figure 18: Notional NASA ISRU Oxygen Extraction Reference Demonstration Concept. From: REQUEST FOR INFORMATION: 80HQTR24L002_LIFT1 - Lunar Infrastructure Foundational Technologies-1 Demonstration.

A mapping of the high-level functions of the Mini-ROXY lunar demonstrator to these building blocks is shown in Figure 19, and discussed in detail below.

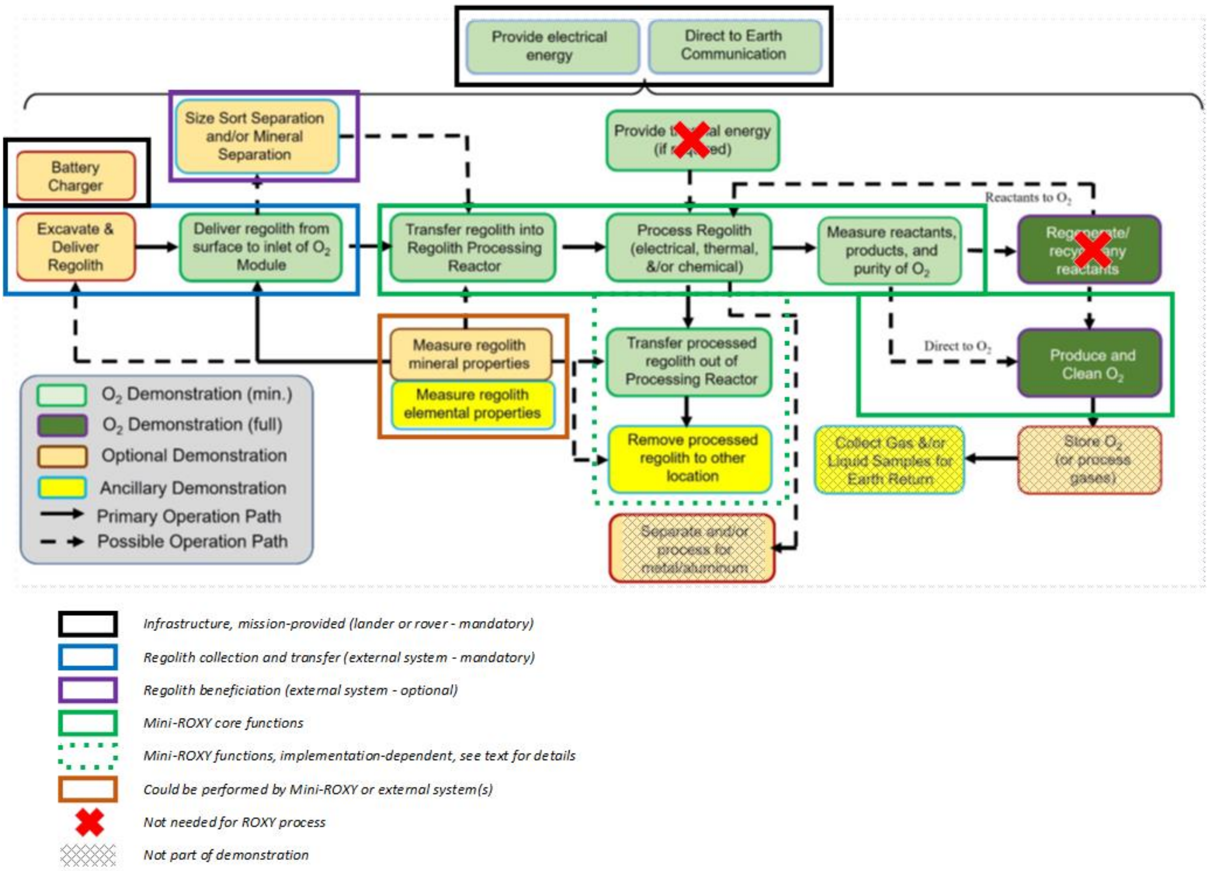


Figure 19: Mapping of Mini-ROXY demonstrator high-level functions to NASA LIFT-1 building blocks

1. Infrastructure functions (black boxes)

These functions are mandatory for the mission. These functions are assumed to be provided by the mission, i.e., the lander or rover, and are not part of the Mini-ROXY demonstrator.

2. Regolith collection and transfer to Mini-ROXY demonstrator (blue boxes)
These functions are mandatory for the mission. These functions are assumed to be provided by other equipment or mission elements, and are not part of the Mini-ROXY demonstrator.

3. Regolith beneficiation functions (purple box)
These functions are optional for the mission. Beneficiation of the regolith would add value to the mission, for example the regolith reduction process could be investigated as a function of regolith composition and/or grain size distribution. These functions are assumed to be provided by other equipment or mission elements, and are not part of the Mini-ROXY demonstrator.

4. Measure regolith mineralogical/chemical composition (orange box)
These functions are mandatory for the mission. The knowledge of which material is processed by Mini-ROXY is considered essential to evaluate the performance of the process. These functions could be performed by the regolith collection and transfer equipment, or a potential regolith beneficiation equipment. It is assumed that the measurements could be an integral part of such equipment. Alternatively, the measurements could be performed by Mini-ROXY.

5. Transfer and process regolith, produce and characterize oxygen (solid green boxes)
These are the core functions of the Mini-ROXY lunar demonstrator. The regolith is transferred from the receiving interface of the system to the cartridges. Regolith in the cartridges is reduced to metal (alloys), and the resulting oxygen is separated, purified, and characterized in terms of production rate by the YSZ membrane. In the basic version of the Mini-ROXY lunar demonstrator, the produced oxygen is released to the lunar environment. The basic version could be extended to provide the oxygen to downstream external systems for oxygen storage etc.

6. Transfer processed regolith out of reactor, remove to other location (dotted green boxes)
The transfer of processed regolith out of the reactor is not part of the basic version of the demonstrator, but could be added in an advanced version. The extraction of processed cartridges by an astronaut and return of processed cartridges to Earth could be an interesting option for a man-tended mission. See discussion in section 3.1.2.

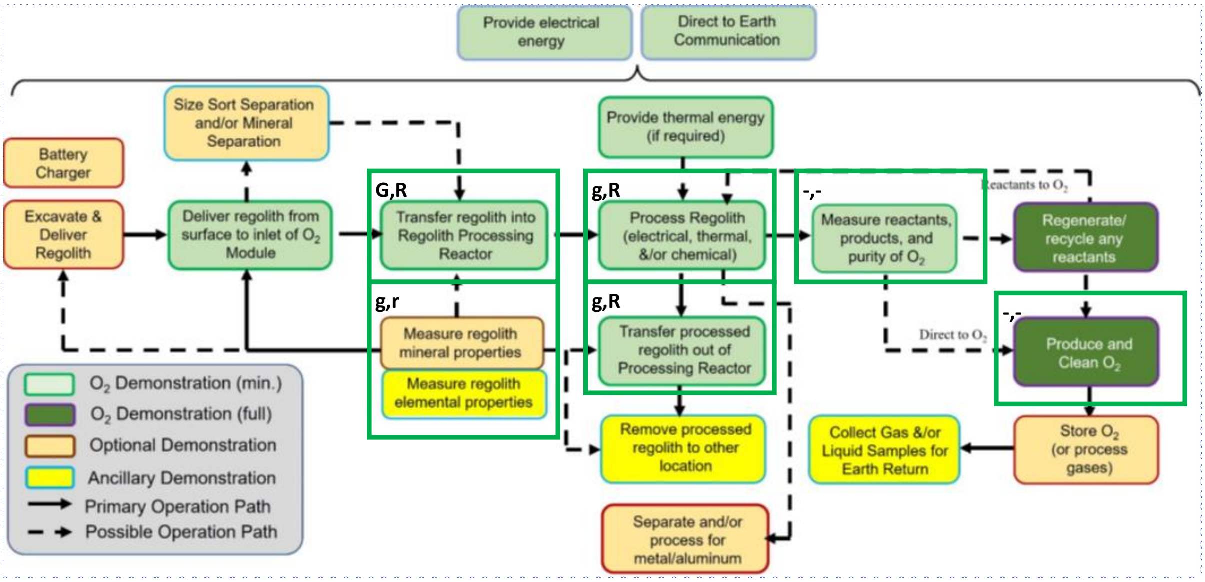
7. Functions that are not needed for a Mini-ROXY demonstration (strikethrough boxes)
Thermal energy: the cartridges will be heated by resistance heaters with power from the lander, so an independent source of thermal energy such as solar concentrators is not needed. Regeneration of reactants: this is not applicable since the separation of the oxygen is an intrinsic function of the YSZ membrane, and the salt electrolyte will not be consumed.

8. Functions that are not part of the Mini-ROXY demonstration (cross-hatched boxes)
Oxygen storage is not needed for the demonstration, but could be added as needed. Post-processing/refinement of the processed regolith is not needed for the demonstration, but could be performed on Earth in case of a man-tended mission with sample return capability. See also chapter 0, which provides a discussion of the end-to-end metal value product chain.

3.3 Dependencies of Process Steps on Reduced Gravity and Properties of True Regolith

A key issue for the justification of any ISRU demonstration on the Moon is the question which of the processes are dependent on the lunar environment, in particular on the reduced gravity and the presence of true lunar regolith. This is assessed for the core Mini-ROXY functions, i.e., the green boxes in Figure 19 above.

In general, the dependencies on gravity and regolith properties are categorized as strong or weak. The result of the assessment is provided below and is summarized in Figure 20.



G: strong dependency on gravity expected
g: weak dependency on gravity expected
R: strong dependency on properties of true regolith expected
r: weak dependency on properties of true regolith expected
-: no dependency on gravity or regolith expected

*Figure 20: Mini-ROXY core process steps and categorization them in terms of strong (**G**) or weak (**g**) dependency on gravity, and strong (**R**) or weak (**r**) dependency on regolith properties*

a. Measure regolith mineral properties &/or elemental properties
Gravity dependency: weak
Regolith dependency: weak
The measurements can be done by non-contact methods, such as Raman spectroscopy. The results of the measurement will of course be dependent on the properties of the regolith, but the performance of the measurement is not expected to significantly depend on gravity or regolith properties.

b. Transfer regolith into Regolith Processing Reactor
Gravity dependency: strong
Regolith dependency: strong
The transfer involves moving the regolith from a funnel-type interface at the top of the Mini-ROXY demonstrator into the cartridges, whereby it must be ensured that a defined small amount of regolith will arrive at the reaction volume of each cartridge. It is known that regolith handling is dependent on gravity and the properties of the regolith such as electrostatic charging, agglutination etc.

c. Process regolith (electrochemical reduction – ROXY process)
Gravity dependency: weak
Regolith dependency: strong
The ROXY process will electrochemically reduce the regolith and transfer the oxygen through the YSZ membrane. As a result, the major gravity dependency of molten salt electrolysis processes that is due to the bubbling of molecular oxygen in the salt electrolyte is eliminated. Therefore, only a weak gravity dependency of the process is expected. On the other hand, the process is known to depend strongly on the material that is subject to reduction, for example in terms of sintering, evaporation of metals with high vapor pressure, interaction with the salt, etc., so there is a strong dependency on the properties of true lunar regolith. This is one of the major reasons why a lunar demonstration of the ROXY process is highly relevant for the development of future larger-scale systems.

d. Measure reactants, products, and purity of oxygen
Gravity dependency: none
Regolith dependency: none
A high oxygen purity is obtained due to the intrinsic selectivity of the YSZ membrane for oxygen ion conduction, which neither depends on gravity nor on regolith properties. This is why the demonstration of oxygen purity is not performed on the moon, but is rather qualified by ground-based testing. The measurement of oxygen production rate takes advantage of another intrinsic property of the YSZ membrane, which neither depends on gravity nor on regolith properties.

e. Produce and clean oxygen
Gravity dependency: none
Regolith dependency: none
See above for oxygen purity and regolith reduction.

f. Transfer processed regolith out of processing reactor
Gravity dependency: weak
Regolith dependency: strong
The transfer of processed regolith out of the reactor involves three steps: 1 - removal of the regolith container (cathode cup) from the cartridge, 2 - removal of the processed regolith from the cathode cup, and 3 - separation of the processed regolith from the remaining salt in the cathode cup. In particular the second and third steps strongly depend on the properties of the (processed) regolith, and should therefore be investigated in detail to gain knowledge for future larger-scale systems. It is therefore considered that it could be advantageous to perform these steps on Earth, however, this implies a sample-return capability of the mission. The gravity dependency of steps 1 and 2 is expected to be weak, while the gravity dependency of step 3 will depend on the details of the process. Currently, the preferred process for salt-metal separation is hot centrifuging, for which it could be argued that gravity dependency is weak.

3.4 Keep it Simple: Lunar Demonstration vs. Terrestrial Qualification

Following the same reasoning adopted in the preceding section, process steps that are not dependent on gravity and regolith do not need to be demonstrated on the moon and can therefore be covered by terrestrial qualifications. For a ROXY system, this includes in particular the measurement of the oxygen production rate and the oxygen purity, as discussed above (process step *d* and *e*).
Ground-based qualification testing will therefore show the high purity of the produced oxygen and the reliability of the measurement of the produced amount of oxygen based on the YSZ ion current.

The extraction of processed regolith from the cartridge after processing is another case: it is expected to be weakly dependent on gravity but including this function would significantly increase the complexity of the lunar demonstrator. It could therefore be considered to cover this function either by ground-based qualification testing or by post-flight analysis of processed cartridges. The latter would require a man-tended mission with sample return capability. Alternatively, the function could be added to the lunar demonstration.

3.5 Mini-ROXY Lunar Demonstrator Technical Data

In the frame of the current project, a first design concept of a basic version of the Mini-ROXY lunar demonstrator is under development. This design will be further detailed and can be used as a starting point to develop alternate configurations with e.g., additional functions such as metal extraction, cartridge extraction etc.

Functions included in the basic version are described in section 3.2. High-level design features are as follows:
- 3 cartridges
- 1 liter of oxygen per cartridge
- < 1 lunar day ground ops
- Advanced process diagnostics via EIS
- Mass ~ 30kg

The design concept is illustrated in Figure 21.

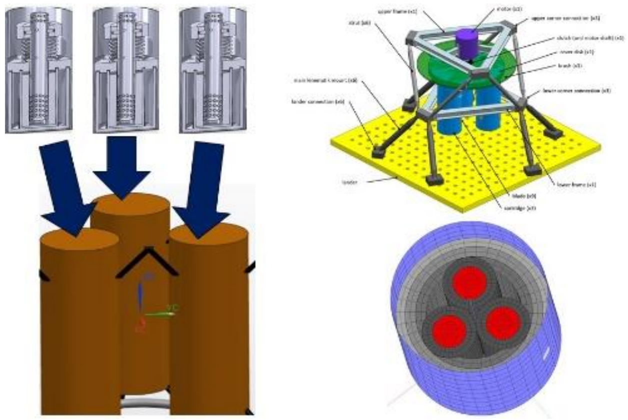


Figure 21: Mini-ROXY lunar demonstrator, early design concept. Left: 3 cartridges, top right: structural concept, bottom right: thermal model

A Mini-ROXY lunar demonstrator based on this design concept will be compatible with the Payload Interface Requirements defined in the PRISM-3 call for a generic CLPS lander, as shown below:

PRISM-3 Requirement	Mini-ROXY Lunar Demonstrator Compliance
Landing site requirements	Yes, site agnostic
Proposed suites shall not exceed 50 kg, including any required accommodation hardware.	Yes, about 30 kg including all margins
Communications:	
Wired RS-422 for both Lander and Rover	Yes, wired on lander is baseline
Wireless: 2.4 GHz IEE 801.11n for both Lander and Rover	Yes, is possible
Data Rate Downlink: average rate of ~300 kbps during operations (minimum downlink rate of 18 out of every 24 hours) for both Lander and Rover	Yes, date rate will be much lower
Power:	
28Vdc Unregulated for both Lander and Rover	Yes
Minimize number of power channels for both Lander and Rover (e.g., <4 preferred, unless more are justified to address compelling science objectives)	Yes, 1 or 2 channels
150 Watts Maximum Steady State Power for Stationary Lander	Yes
Less than 200 Watts Startup Transient Power for Stationary Lander	Yes
20 Watts of Keep-Alive Power for All Payloads, inclusive of electric and thermal management	Not applicable, lunar night ops not foreseen
25 Watts Maximum Steady State Power for Rover while Driving	Not applicable, accommodation on lander assumed
50 Watts Maximum Steady State Power for Rover while Stationary	Not applicable, accommodation on lander assumed
Thermal for both Lander and Rover:	
Mounting – Adiabatic (Acceptable – must specify radiation FOV)	Yes, possible
Mounting – Conductive (Acceptable – must specify dissipation)	Yes, possible
Structural Guidance for both Lander and Rover:	

Random and Sine Vibe: General Environmental Verification Standard (GEVS) levels	Yes
Shock and Acoustics (GEVS)	Yes

3.6 Mini-ROXY Lunar Demo Mission Science Objectives

Even though a lot of information has been gained on lunar resources such as regolith through previous and ongoing ground-based experiments and missions, there are still knowledge gaps of how best to extract and process lunar regolith into usable metal products and resources such as oxygen. In particular, there are significant uncertainties on the properties of lunar regolith. Some of these properties can be determined by established diagnostics methods e.g., to determine the chemical and mineralogical composition of the material. However, the key questions for developing methods for oxygen and metals extraction from regolith are: how will the regolith interact with the extraction method under lunar conditions, and how can the extraction method be optimized for future large-scale lunar facilities? A detailed understanding of the regolith reduction process when applied to true lunar regolith under lunar environmental conditions such as reduced gravity is therefore indispensable for future developments in this field.

This leads to the following key scientific objectives for a lunar demonstration of the ROXY process:

A. Understanding the properties of the feedstock by analyzing the feedstock and determining its rheological, physical, mineralogical, and chemical properties.
B. Understanding the regolith reduction process, its performance and limitations, under lunar environmental conditions, in particular related to reduced gravity, vacuum, charging atmosphere and true regolith. This will allow comparison with terrestrial tests and model predictions, and analysis of likely impacts on alternative sites, feedstocks and production scales.
C. Understanding the characteristics of the product, particularly the amount and purity of oxygen produced.

These key science objectives have been mapped to the building blocks introduced in section 3.2 of a Mini-ROXY lunar demonstrator to formulate five specific objectives. Objectives are therefore to characterize and understand the physical and chemical processes are associated with the following building blocks:

1. Transfer of regolith from inlet of Mini-ROXY demonstrator to cartridge
2. Mineralogical and/or elemental characterization of the regolith
3. Design of an optimized salt electrolyte for regolith reduction
4. Diagnostics for an in-depth characterization of the reduction process
5. Transfer of processed regolith out of cartridge

Each specific science objective is detailed in the following sub-sections.

3.6.1 Transfer of Regolith from Inlet of Mini-ROXY Demonstrator to Cartridge

Gravity dependency: strong
Regolith dependency: strong

Manipulation of granular materials on the lunar surface has been highlighted as one of the Grand Challenges in soft matter physics in a 2021 NASA report. Before that, multiple Apollo engineers and astronauts, for example John Young, anticipated that "dust is the number one concern in returning to the Moon". On-ground, granular materials are common across industries; but the transfer of ordinary processes to lunar regolith poses the issue of rheology, or flow-behavior, of lunar regolith in its native environment. The complexity of lunar regolith rheology is threefold: the granular material itself exhibits distinct physical properties, characterized by sharp and abrasive particles with a significant amount of fines; the reduced gravity alters particle interactions, making cohesive forces predominant; and the singular lunar environment, encompassing low gravity, high vacuum, and electrostatic charges from solar winds, has a complex influence on regolith behavior. When developing a transfer mechanism for regolith, all these effects have to be taken into account. Three aspects in particular:

First, the nature of regolith itself: lunar regolith exhibits distinctive physical and flow properties. The regolith consists of highly cohesive particles, including fines that extend down to nanometric sizes. Small particles display elevated cohesion, notably attributed to van der Waals forces becoming predominant. These particles are highly abrasive, as they haven't undergone rounding or polishing through erosion, resulting in angular shapes. The physical entanglement of nonconvex particles significantly contributes to an effective cohesion effect. Combining the abrasive nature and the tendency to adhere to surfaces, lunar regolith can pose serious hazards to processing hardware. While the abrasiveness might degrade mechanical lifetime, the cohesiveness might lead to regolith sticking in unwanted places during transport.

Second, besides the role of gravity in controlling the flow of granular materials remains a crucial, yet poorly understood question. Experiments and simulations on Earth have shown that granular rheology is highly dependent on gravitational acceleration, with the emergence in the last decade of a pressure-dependent empirical rheology. If granular flows are strongly influenced by pressure, their rheology is gravity-dependent, as it is influenced by the pressure due to particles' own weight. Rheological experiments conducted under microgravity, albeit sparse, exhibit a higher peak strength of the granular bed, a higher friction angle, and generally unstable behavior with exacerbated periodic instabilities. This can be understood as a lower ratio of weight to surface energy of a particle under lower gravity, making cohesive forces predominant and allowing particles to easily fly and stick to each other or nearby hardware. The lack of a strong directional secondary force field (that due to gravity) also hinders flow. Moreover, clogging (or jamming) occurs at different packing fractions under varying gravitational accelerations and for different granular materials (depending on particle size distribution, surface friction, particle shape, etc.). The risk of clogging in funnel-like hardware must be foreseen, and geometry adapted accordingly. In case a clogging event occurs, suitable strategies need to be in place to resolve the issue and restart the flow (i.e., mechanical agitation).

The third challenge is the Lunar atmosphere: first, the strong electrostatic charging present on the Moon due to solar wind and triboelectric charging, and second, the lack of an atmosphere to dissipate surface charging once it is developed (high vacuum of ~3×10^{-15} atm or 0.3 nPa). Electrostatic forces can trap particles in-flight, leading to inelastic collisions and adhesion of particles to each other or nearby surfaces. Very small particles interacting with solar winds can form dirty plasma: charged particles suspended and interacting through Coulomb forces. Electrostatics also play a role in the formation of aggregates. When forming aggregates, the individual elements of the particulate medium become these aggregates instead of single particles, modifying the size, shape, and mechanical properties of each element, which also modifies how they interact with each other and with hardware. Note that charging (notably the exact nature of charge carriers) and its influence on granular flows, remains in debate even on-ground. Finally, under vacuum, there is no humidity in the granular material, which also completely modifies its behavior: it changes particles' charging properties and eliminates the presence of interstitial fluid and cohesive capillary forces among particles. To counter aggregate formation from electrostatic charges material the material inlet needs to be designed to allow the regolith to electrically discharge.

Finally, the interactions between these effects are considerable, representing a fourth challenge in predicting regolith flow-behavior from Earth. On-ground, existing granular models rely on thousands of years of empirical knowledge, which we lack for the Moon. Gravity – specifically Earth's gravity – is included in current rheological models, often implicitly. This gravity dependence also necessitates taking care when testing the transport mechanism on Earth. While regolith simulant might be good at emulating the chemical or spectral characteristics of lunar regolith, it will not properly reproduce the flow behavior in lunar gravity.

In the framework of the DLR-funded GRIS (Granular Rheology In Space) project, a set of semi-empiric models accounting for the effects described above is currently developed. This includes the influence of cohesion and electric charges, leading to particle aggregation and cluster formation. The GRIS project guides the development of instruments and procedures for planetary missions and ISRU applications. The hardware developed for GRIS also provides a low-gravity lunar testing environment where individual designs for funnels, inlets, etc., can be validated to work under lunar conditions. To test these models and verify their accurate predictions for large-scale technologies like ROXY, there is a need for further experimental results *on the lunar surface*, ensuring the safety lunar installations and most importantly, of the crew.

In conclusion we suggest using the modeling developed in GRIS to identify critical parts of the regolith transport mechanism and test these parts in an appropriate lunar-like environment especially under lunar gravity.

3.6.2 Regolith Beneficiation - Optional

Gravity dependency: strong
Regolith dependency: strong

The objective of beneficiation is to obtain a regolith feedstock that is optimized for the downstream processes. Beneficiation may therefore include a number of process steps to alter the regolith composition and produce the desired feedstock, such as a particle size separator followed by magnetic and electrostatic separators. The particle size separator filters out larger rock particles leaving behind uniformly sieved regolith. The magnetic separator segregates the ferromagnetic agglutinates and metallic dust particles from the bulk regolith that enables the electrostatic separator's operation. The final product of the beneficiation process is an enriched feedstock with the desired particle size as well as mineral composition. The feedstock composition can be adjusted to match the defined requirements by manipulating the process parameters thereby keeping the system adaptable. A laboratory demonstrator of such a system was developed at DLR Bremen to process lunar regolith and produce feedstock enriched with the desired target mineral. The experiments demonstrate up to three-fold increase in the grade of target mineral with room for improvement upon optimization. The current system has a processing capacity of about 20 $g*min^{-1}$. With a scaled version of the system, a wide range of processing capacities are possible along with a similar range of possible feedstock composition and particle size characteristics.

The lunar environment is extremely hostile compared to the Earth's environment and therefore plays a major role for any in-situ material processing equipment. The primary challenge on the lunar surface for beneficiation is the reduced gravity which is about 1/6th of the Earth's gravity. The dry separation techniques used for beneficiation are driven by the gravitational force. This will probably lead to increased process time as the particles take longer to fall through the different stages of beneficiation. Also, the vacuum environment on the lunar surface can affect beneficiation processes such as electrostatic separation which is affected by the atmospheric conductivity, presence or lack of air molecules, environmental humidity and time taken to pass through the electrostatic field. All of these factors need to be studied with in-situ demonstrators as the theoretical analysis alone is not conclusive of real-world behavior of the system. Another challenge that will arise while working with the lunar regolith is dealing with the natural charge that it carries. The Apollo missions reported high levels of electrostatic charges in the regolith particles that made them stick to the space suits as well as the equipment. This needs to be considered for regolith processing systems such as beneficiation where this charge might cause an increase in the residual regolith due to excessive surface adhesion. In addition, it may also affect the electrostatic performance of the system depending on the charge polarities. Therefore, a demonstration mission to the lunar surface to test and validate the beneficiation technologies in conjunction with the metals and oxygen production reactors are highly important to enable an efficient and sustainable ISRU infrastructure.

3.6.3 Mineralogical and/or Elemental Characterization of the Regolith

Gravity dependency: weak
Regolith dependency: weak

Raman spectroscopy is used to create a structural fingerprint that can be used to identify molecules. Raman spectroscopy has been used for decades to analyze lunar samples because it is a suitable tool for detecting minerals commonly found on the Moon, including olivine, pyroxene, feldspar and silicate glasses. Full spectral scans of a sample can be completed in typically one hour, allowing for use during short lunar lander missions or multiple measurements of multiple samples of material. All major minerals on the Moon can be detected, and in many cases their composition can be quantified. Raman spectroscopy is therefore suitable for the chemical and mineralogical characterization of the regolith source material for the ROXY process, including the detection of condensed volatiles. In combination with fiber optic probes, a Raman sensor enables non-invasive chemical analysis in situ in a compact setup compatible with the constraints of a lunar demonstration mission, in particular with small budgets for mass, energy and volume as well as high temperatures. Thus, a system based on it could be integrated into a Mini-ROXY facility.

Note: an exhaustive treatment of the subject will be prepared by Robert Hyers from Worcester Polytechnic Institute, Worcester, USA, in a response to the Mini-ROXY RFI, until 6 January 2024. This section will then be updated accordingly.

3.6.4 Design of an Optimized Salt Electrolyte for Regolith Reduction

Gravity dependency: weak
Regolith dependency: strong

Background: Molten salts are composed of ionic species having structures that are not very well understood. These melts, although mostly un-optimized, are employed in many important industrial applications: (a). SOM-Based Electrolytic Processes for Metal and Oxygen Production; (b). Energy Conversion and Storage Systems; (c) Materials/Containment Disposal; and (d). Advanced Next Generation Molten Salt Reactors for Nuclear Power. These melt systems are multi-component and complex and are often used at high temperatures. Acquiring accurate property data are time consuming, expensive, and sometimes experimentally very difficult. Often empirical strategies are employed to identify salts for a particular application. For instance, the salts for SOM-based electrolysis are selected such that its properties like ionic and low electronic conductivity, melting point, viscosity, and vapor pressure are comparable to other molten salts used in metal electrolysis such as the AlF_3-NaF cryolite salt used for Aluminum electrolysis. To meet these requirements, the empirical strategy has been to consider systems containing multiple Group I and Group II fluorides and select compositions that have melting points between 650-1100 °C. CaF_2-MgF_2 eutectic composition has often been used as a baseline composition for SOM applications due to low volatility and good compatibility with the yttria-stabilized zirconia membrane (SOM). It has a melting point of 975 °C which can be further lowered by adding LiF. Its properties and stability in contact with the oxygen-ion-conducting membrane are tailored by making compositional changes for a particular SOM application. It is found experimentally that as the optical basicity in the flux approaches that of the SOM, the degradation of the SOM is mitigated and is greatly reduced when the flux acidity with respect to the SOM is neutralized. These approaches are not optimum nor ideal but lead to workable solutions. Since structure and properties are intricately related, structural information can be used instead to make property predictions and help in designing the optimum salt for a given application. With the advent of advanced characterization techniques, specifically high-energy X-ray scattering and absorption, it is now possible to combine these experiments with various types of structural simulation techniques such as *ab initio* and machine-learning based molecular dynamic simulations to obtain detailed structural information and relate them to their thermophysical and thermochemical properties. Such techniques will be employed to obtain optimum salt for SOM-based regolith reduction by the Mini-ROXY process.

ROXY Relevance: Such a "ROXY salt" needs to have melting point between 650-750 C so one can employ commonly available stabilized zirconia membranes for regolith reduction at temperatures less than 850 C, viscosity less than 0.1 Pa.s for effective mass transfer and salt penetration into the cathode cup during reduction and removal of salt from the cup after reduction to separate and extract the reduced regolith, electrical conductivity greater than 2 S/cm with ionic transference number greater than 0.9 to obtain low cell resistance and high reduction efficiency, low volatility on the order of 10^{-9} - 10^{-16} atmospheres to maintain low salt loss, and no corrosive interaction with the membrane material. Since the gravity dependence of these properties are weak, they are also relevant for low-g applications and can be used for scaleup process modelling in the lunar environment.

The Optimal Salt for ROXY: The aim of the structural studies will be to characterize local ordering and speciation in the molten salt. Determining the speciation and complexation in molten salts is paramount to understanding the chemistry of these salt systems. This can be achieved by High Energy X-Ray Diffraction (HEXRD) which employs 100 keV X-rays to achieve high Q-range measurements while penetrating through relatively large quantities of melts and the containment crucible. HEXRD can characterize the fluid structure of molten salts from approximately 2 to 20 Å. As a result, HEXRD is capable of providing information over the length scales of interest for liquid structures. This research will characterize the X-ray structure factor and yield the corresponding Pair Distribution Function (PDF) of different complex ions in the ionic melt. The existence of these complexes has significant implications on modeling melt thermodynamics and their thermophysical properties. Detection of the complex ions will be achieved by observing the variations in the PDF that occur in molten salts having different compositions. HEXRD of the bulk salt solution will provide sufficient signal intensity for yielding the PDF of complex ions in these systems. Although no single experimental technique can fully solve a liquid-state structure, PDF analysis is one of the most powerful tools for gaining insights into both the local and long-range atom-atom interactions. The technique is also directly comparable to theory and simulation. In addition, one can potentially use resonant x-ray scattering which allows us to change the scattering power near an absorption edge to isolate the scattering around a given atomic species. Neutron scattering could be potentially interesting as well because of its great penetrating power. Our ongoing collaboration with Argonne National Laboratory (ANL) and Brookhaven National laboratory will give us access to their x-ray synchrotron facilities to make such measurements.

Thermophysical properties (viscosity, surface tension, density, diffusivities, vapor pressures and electrical conductivity) of these salt systems will be measured as a function of temperature employing glovebox compatible high-throughput devices that are designed and fabricated at Boston University. Computational tools such as density functional theory (DFT) calculations, molecular dynamic (MD) simulation, *ab-initio* calculations, etc. can be brought to bear to understand the structural characterization results and develop predictive capability of relating the structure to its properties. For instance, we can compute pair distribution function, and spectroscopic and transport properties of these systems using MD with model potentials and large-scale machine-learning techniques.

The gathered physical property information will be correlated to the structural information such as speciation, network structure and size, bonding, and coordination chemistry. For instance, the viscosity is related to the salt network size and structure, heat of melting and vaporization to bonding and interactions, electrical conductivity to transport properties of dominant species, density to volume change due to mixing and interaction of species and hence non-ideality, volumetric expansion as a function of temperature to various thermodynamic properties, and vapor pressure to speciation and their bonding interactions. The proposed study will yield structural information that will enable us to determine structure-property relationships and develop a science-based methodology for optimum selection of ionic melts and process conditions for SOM-based electrolysis of regolith.

3.6.5 Diagnostics for an In-Depth Characterization of the Reduction Process

Gravity dependency: weak
Regolith dependency: strong

A thorough understanding of the reduction process when applied to real lunar regolith is essential for future advances in the field and is therefore of great importance.
For a comprehensive understanding of the electrochemical extraction of oxygen and metals from regolith and for the future development of larger systems, such as a pilot plant, it is crucial to be able to diagnose the process and its main elements. In particular, since the composition of the regolith at the landing site is not known a priori, there is some uncertainty regarding the interaction of the lunar regolith with the salt electrolyte. During the reduction runs, the electrolyte may thus change its properties, such as composition, electrical conductivity, viscosity, etc. These changes may influence mass and charge transport processes and affect the performance of the electrolytic cell. They must therefore be understood to evaluate regolith reduction runs on the Moon.
Effects that are relevant in this context include:

- The total ohmic resistance of the cell including the resistances of the yttria-stabilized zirconia (YSZ) membrane, the salt electrolyte, the electrodes, the external lead wires, and the contact resistances associated with all the interfaces.
- The activation polarization, i.e., the overpotential required to overcome the activation energy barrier for the charge transfer reactions occurring at the electrode interfaces.
- The anodic concentration overpotential, which is due to the oxygen concentration gradient across the diffusion layer at the anode interface.
- A cathodic concentration overpotential due to diffusion-limited transport of oxygen occurring at the regolith (oxide)-salt interface.
- Reduction efficiency loss because of electronic conductivity in the salt due to soluble and transition metal ions.
- The impact of changing salt composition over time on all of the above effects.

These effects can be determined by electrochemical modeling of the cell performance assisted by various potential-current measurements including frequency dependent (electrochemical impedance spectroscopy or EIS) measurements. The principle of EIS is illustrated in Figure 10. The state of the art includes modeling of the equivalent circuit of the cell and monitoring changes in the model circuit elements, which considers all known mechanisms associated with current flow, including:

- The dissociation of the desired oxide.
- The dissociation of impurity oxides.
- All polarization losses occurring in the cell including ohmic (resistive) and non-ohmic (mass and charge transfer).

In particular, the electrochemical measurements and approach include:

- Potentiodynamic scans between the electrodes @ 1-5mV/s which allows determination of the dissociation potentials of the oxides in the regolith through discontinuities in the measured current.
- Potentiostatic holds between electrodes at the desired dissociation potential of the oxide to measure the electrolytic current, analyze oxygen evolution, process stability, and underlying changes in the cell system.
- Potentiostatic holds in the salt between two electrodes at potentials less than the dissociation potential of any oxide to monitor the electronic current (electronic transference number in the salt) as a function of electrolysis time.
- EIS to determine the ohmic resistance of the system and also analyze charge and mass transfer polarization processes occurring at the electrodes.
- Use of the EIS data to analyze the Distribution of Relaxation Times. Each process element in the equivalent circuit model, including those occurring at the electrode interfaces, will have a frequency-dependent time constant. Determining this will help model and monitor the changes in the system.

The potentiodynamic and potentiostatic measurements are DC measurements. EIS requires AC measurements that measure the current response as a function of frequency in the range from 1 Hz to 100 kHz with small (10-20 mV) potential amplitude (see Figure 10 in section 2.1.3). These measurements can provide a fundamental understanding of the process, including:

- Dissociation potentials of the oxides in the regolith.
- Dissociation of the impurity oxides (undesired oxides) dissolved in the salt electrolyte.
- Effect of composition of the regolith.
- Effect of compositional changes in the salt over time.
- Rate of oxygen production and the corresponding metal reduction.
- Reduction rates of the individual oxides that make up the regolith.
- Identification of process parameters to achieve a sequential or selective reduction of the individual oxides that make up regolith in order to obtain a tailored metal alloy product.
- Electronic conductivity of the salt electrolyte caused by either the intrinsic electronic conductivity or dissolved metal ions in the salt electrolyte.
- Mass transfer limitations or polarization, for example associated with a diffusion-limited oxygen transfer from the regolith into the salt.
- Charge transfer processes occurring at the electrodes.
- Equivalent circuit model of the various resistive contributions in the electrolysis cell.
- Optimized process model for scale-up.

Using these experimental characterization techniques on earth and in the lunar environment, the overall process, its limitations and also the impact of gravity can be better understood. The undesirable characteristics can be identified and improved or mitigated. For example, the presence of impurity oxides in the salt electrolyte can reduce the purity of the metal product. Electronic conductivity of the salt electrolyte can reduce the Faradaic current efficiency for metals production and contribute to YSZ membrane degradation, depending on the applied potential. Regular small current reversals can mitigate this problem. The explicit expression of each resistive (ohmic and non-ohmic) element or polarization losses of the electrolytic cell provides direct information on how to lower the losses and contributions to resistance. Electrochemical cell diagnostics will thus provide a general guideline for the design of optimal regolith reduction electrolytic cells for future larger scale applications such as pilot plants and operating plants.

3.6.6 Transfer of Processed Regolith out of Cartridge

Gravity dependency: weak
Regolith dependency: strong

It is expected that retrieval of the reduced material from a Mini-ROXY reactor will require three steps:
1. Removal of the cup containing the processed regolith from the Mini-ROXY reactor,
2. Separation of the processed regolith from the remaining salt electrolyte, and
3. Removal of the processed regolith from the container after separation.

Further details on each step are provided here below:
1. Cup removal

After electrolysis, the processed regolith is contained in the cathode cup. To remove the cathode cup from the reactor, an advanced version of the basic cartridge will allow for vertical movement of the cathode cup without disturbing the other components of the reactor and particularly the electrolysis cell.
The cathode cup is made of porous metal to ensure molten salt electrolyte exchange and diffusion of oxygen ions during reduction. However, due to the porosity, some loss of the fine fraction (< 10 µm) of the processed regolith through the pores of the cup during movement is a concern. The amount of loss depends on the speed and type of movement. The loss of processed regolith obviously decreases the material yield for a reduction run. However, this lost material will also enter the molten salt electrolyte and can accumulate over time. This may change the functional properties of the salt such as conductivity or optical basicity, which are important to maintain the proper operation of the reactor. Contamination with processed regolith may therefore result in the need to replace the salt after a certain number of cycles. This is clearly a concern for a scaled up and repeatable process. Therefore, the loss of fine material and its accumulation in the salt must be minimized.
The envisaged solution consists of two elements, one upstream of the Mini-ROXY reactor, and one that is part of the Mini-ROXY reactor:
The first element is the separation of the fine fraction of the regolith after excavation and before introduction into the reactor, i.e., by size sorting that could be performed as part of a beneficiation step. It is expected that the amount of the fine fraction with particle sizes below a certain threshold value could by reduced significantly by size sorting. The threshold value will depend on the properties of the cathode cup, in particular on the pore size, pore structure (e. g. tortuosity) and total porosity. Threshold values in the range from 10 µm to 50 µm appear reasonable for this step.
The second step is then achieved by the cathode cup which will need to contain essentially all of the size-sorted regolith. For this to be achieved, the pore sizes of the cathode cup must be smaller than the minimum particle size of the size-sorted regolith. Metallic structures with designed porosity can be produced by a variety of processes, such as metal foam replication, placeholder techniques, electron beam melting or laser drilling, the last of which can be used to produce thousands of small holes per minute with high precision and accuracy. Pore sizes on sub-micrometer length scales are achievable which would be appropriate for this application.
If losses of regolith (both processed and unprocessed) per cycle can be reduced to a level many orders of magnitude below the critical contamination threshold, many cycles of the reduction process can be run without the need to replace the salt electrolyte. Investigations of the effect and acceptable levels of contamination on the salt electrolyte are ongoing in the present project.
From the above discussion, it is concluded that this step will depend on gravity to some extent, and will strongly depend on the properties of regolith.

2. Salt-metal separation

Due to capillary action in the spaces between the particles of the processed regolith, some salt electrolyte will remain intermixed with the processed regolith in the cathode cup. Residual salt may compromise the properties of the metallic material and make it more difficult to use in downstream manufacturing steps such as additive manufacturing. The remaining salt would also be removed permanently from the reactor, necessitating the eventual replenishment of salt. It is therefore imperative to achieve a very high degree of separation of the salt and processed regolith. Once again, care must also be taken not to contaminate the salt with processed regolith, since it will be returned to the reactor.
Several possible technologies for separation have been investigated in the ROXY development campaign:
Separation by chemical means was shown to be difficult due to the negligible solubility of fluoride salts in most organic or aqueous solvents. Solvents such as acids and bases would also attack the processed regolith, changing the chemical composition or re-oxidizing it.

Mechanical separation by crushing and sorting are made difficult by the similar density of the salt and at least some of the metallic phases produced by reduction of regolith. The particle size distributions of salt and reduced material after crushing also show significant overlap, making it difficult to separate the phases by sieving or other size-dependent processes.

The most promising approach so far was centrifugation at a temperature above the melting point of the salt electrolyte (> 700 °C). This requires a porous, temperature- and corrosion-resistant filter that lets molten salt pass freely and holds back the processed regolith. Since these requirements are identical to the requirements for the porous cathode cup, the cathode cup itself (alternatively, a container produced by the same manufacturing processes) can be used for centrifugation.

For ground-based testing, a centrifuge operable at more than 800 °C and up to 1000 RPM was designed and built in the ROXY development campaign. First tests showed significant, but not yet sufficient separation of metallic particles and salt. The test activities are continuing in the current Mini-ROXY project, with the goal of identifying centrifugation parameters (such as time, rotational speed, and temperature) for optimum salt removal. Regarding the design of the porosity of the container, the same remarks as for step 1 apply. If sufficient separation is achievable by centrifugation, the design, construction, and testing of a centrifuge suitable for lunar use will be incorporated into the further stages of the Mini-ROXY development program.

From the above discussion, it is concluded that this step will depend on gravity to some extent. The driving force for the salt-metal separation will be the centrifugal force which will be much larger than Earth or Moon gravity. Dependency on regolith properties is expected to be strong.

3. Metal extraction

Finally, the processed regolith, now separated from residual salt, must be removed from the cathode cup. Due to the tubular shape of the cathode cup in the Mini-ROXY design, this can be achieved by pushing the material out of the cathode cup, e. g. with a rod-like device. Again, it is expected that this step will depend on gravity to some extent, and will strongly depend on the properties of regolith.

4 Development Status

Mini-ROXY is based on the same electrochemical process as the established ROXY process but uses a different technical implementation (see section 2.2). Therefore, technical risks are not associated with the underlying process, but to the technical details of the implementation. These issues have been addressed through modeling and bench testing. Modeling provides design criteria on issues such as process temperature, YSZ crucible dimensions, and cathode cup dimensions required to achieve the desired performance and oxygen production capacity. Bench testing was used to confirm the performance of the proposed design. The electrochemical performance, which determines the achievable oxygen production rate, was confirmed to be in line with expectations.

In addition, various technical issues have been investigated in a bench test campaign, such as the differential thermal expansion of the materials, electrical performance of the current collector, containment of the produced oxygen, containment of the regolith, and salt capacity of the YSZ crucibles. The feasibility of the concept was confirmed, and it was also shown that the design of the cartridges will achieve the intended performance in terms of oxygen production rate (1 liter in 10 hours) with a generous margin of 100%.

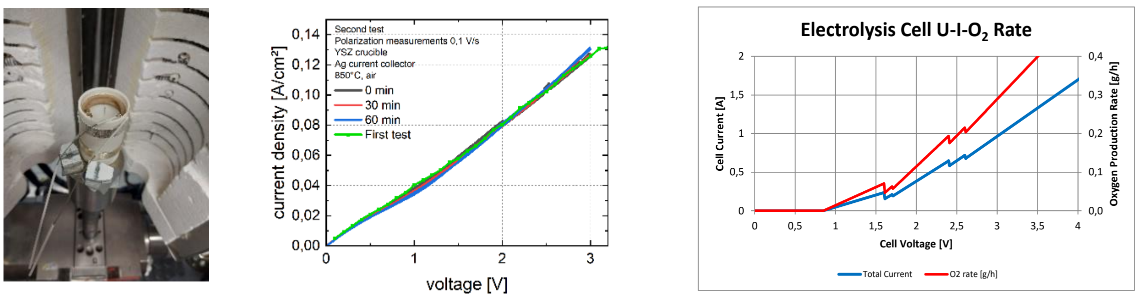


Figure 22: Left: Anode resistance measurement setup, center: current density, right: results of electrochemical process modeling.

5 Ongoing and Future Work

In the frame of the recently started project funded by the German Federal Ministry for Economics and Climate Action, a Mini-ROXY ground model will be built which will be used to perform an end-to-end ground test campaign of the process. In parallel, a Mini-ROXY lunar demonstration mission is being prepared by detailing the design of the Mini-ROXY lunar demonstrator, preparing a design, development and verification plan for the lunar demonstrator and assessing lunar mission opportunities. The main work items of this project are shown in Figure 23.

Current hardware activities include a second breadboard campaign to address relevant issues in setups that are close to the final design, plus tests to prepare the ground test campaign, such as measurements of the salt evaporation rate, pre-test to optimize the separation of the processed regolith (simulant) from the salt, etc.

System engineering activities for lunar mission preparation are organized in a “design campaign” that will be completed in March 2024. The intended result of this campaign is a design baseline of the Mini-ROXY lunar demonstrator that responds to the mission and payload requirements, and a development plan and schedule to prepare the flight model of the Mini-ROXY lunar demonstrator.

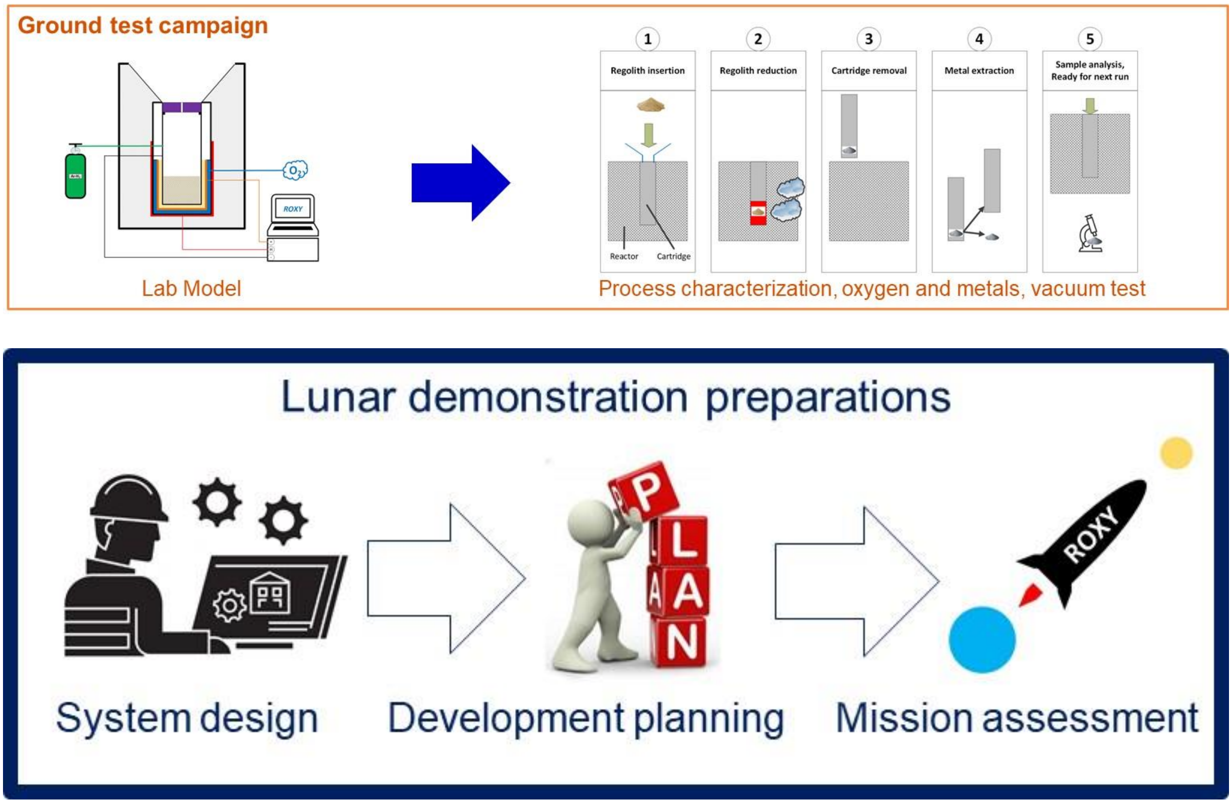


Figure 23: Main work items of current Mini-ROXY project. Top: Lab model and ground test campaign, bottom: lunar model design, development planning and mission assessment.

6 Scalability

A ROXY Pilot Plant would represent a key milestone in the overall exploration strategy to enable a sustainable human presence on the Moon. The follow-on of the near-term Mini-ROXY Demonstrator Mission will be the establishment of a Pilot Plant scaled up to demonstrate the production of about 1/100th of the oxygen needed in the fully operational scenario. Such a pilot plant could be sized to produce about 500 kg of oxygen from regolith per year. In this chapter, the scalability of the proposed design to this scenario is assessed for all major aspects of the operation from excavation to production. A pilot plant will need to demonstrate a range of capabilities, such as excavation from multiple sites, recycling of reactants, sustaining longer term operations, accumulating and storing large amount of regolith as well as the process products (oxygen and metal powders), self-sufficient operation, waste disposal, surviving and/or operating during lunar night periods, safe operation from ground and/or from the lunar surface, etc.

Sourcing

The mass of raw regolith required for production scales proportionally with the target mass of oxygen/metal. As such, robotic means to prospect and source the material need to be designed accordingly. A production rate of 500 kg of oxygen per year, and assuming that collection is only possible during lunar daytime, requires the robotic agents to collect roughly 20 kg of regolith every 24 h. This operation is feasible with a combined scouting and excavation robot, with material taken from the landing site's vicinity. To produce larger amounts of oxygen, such as 50 t per year, the robots would need to acquire around 2 t of regolith every 24 h. In this context, robotic teams consisting of specialized scouting and excavation robots are beneficial.

One of the boundaries for scaling the ROXY plant is the maximum acquisition rate of the excavation robots during the lunar day. To increase this rate, the level of on-board autonomy for each robotic agent is of high importance. Introducing autonomously performed tasks, such as negotiation of obstacles, avoiding high-slip terrain, performing prospecting and excavation, and returning to a dedicated offloading point at the production plant, increase the robotic agents' operational efficiency.

Metal and Oxygen Production Rate

The most important limiting factors for a scale-up of the electrochemical reduction process are the anode current, the ohmic resistance of the salt electrolyte, and the reaction rate at the cathode. For a subscale pilot plant which will produce 500 kg of oxygen per year, the oxygen production rate and thus the reaction rate at the cathode will have to be increased by more than 2 orders of magnitude compared to the lunar demonstrator.
The anode current is limited by the maximum current density, and the overall active area. The ion current density of standard YSZ membranes is reported to be limited to about 1 A/cm², which is reduced significantly to arrive at a design value that takes into account derating and robustness. If the established technology is used, the total current must be increased by increasing the active area, which can be achieved by increasing the number of anodes, their diameter, and/or active height. If combined, a reactor design can be implemented that is less than one order of magnitude larger in both diameter and height than the reactor of the lunar demonstrator model. Using a large number of cells operating in parallel will also be beneficial in terms of failure tolerance. The principle is illustrated in Figure 24. High-performance anodes based on advanced membrane and/or current collector materials could be used to downsize the system even more since higher current densities can be achieved with these technologies.

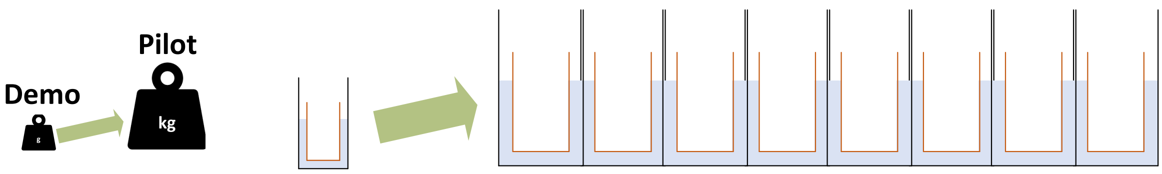


Figure 24: Mini-ROXY scaleup principle: reaction cells are moderately increased in size; the main part of the scaling factor is achieved by running a large number of cells in parallel.

Electrochemical reaction parameters and feedstock properties

The ohmic resistance of the salt is due to the ionic conductivity for oxygen ions, which generally follows an Arrhenius law. The temperature dependence of the conductivity will set a lower limit for the operating

temperature. Therefore, the salt electrolyte needs to have a high oxygen ion conductivity at the operating temperature. At the same time, a low electronic transference number is needed.
The reaction rate at the cathode is limited by the speed of the reaction itself, plus transportation effects related to the progression of the reaction front, and the diffusion of the oxygen ions. These effects depend a lot on the grain size distribution, since the reduction reaction is a surface phenomenon. Optimizing the sample preparation to enable high reaction rates is therefore key to upscaling. In addition, a robust technology to repeatedly remove the processed regolith from the cathodes and the reactor will be needed (see section 3.6.6).

Energy Efficiency
Another key parameter is the energy efficiency of the process, i.e., the energy required to produce 1 kg of oxygen. This figure of merit includes the electrolysis power, which can be easily calculated from the cell voltage, the oxygen production rate (equivalent to the oxygen ion current), plus an allowance for ohmic losses at the supply lines of the cell. It also includes the power needed to heat the reactor to its operating temperature. If, for simplicity, only the power needed to maintain the reactor at its operating temperature is considered, then this power is about one order of magnitude larger than the electrolysis power for a well-designed lunar demonstrator. This situation changes if the system is scaled up using the features as outlined above. Since the size of the reactor will only increase moderately but the electrolysis current will increase by more than two orders of magnitude, the ohmic losses due to the electrolysis current alone may provide enough heat. Details will depend on the thermal design of the reactor.

For large systems such as a pilot plant, the electrolysis power is larger than the power needed to heat the system. As a result, such a large system will be self-heated, i.e., the heat to maintain the reactor at the process temperature will be provided by the thermal dissipation in the salt. Effectively, the salt will be used as a resistance heater due to its ohmic resistance against the oxygen ion current that is passing through. The exact figures depend on the design and process details and are not important for this conclusion. What it means is that the energy efficiency of a large-scale system is much larger than the one of a small lunar demonstrator system, and essentially determined by the thermodynamic parameters of the reaction.
A ROXY Pilot Plant would even produce excess heat, since the heat dissipated by the electrolysis current is larger than the thermal energy needed to maintain the operating temperature. This excess heat could be used for other applications, such as thermal energy storage for the lunar night. With this, the "net" power consumed by the electrolysis is further reduced, the energy efficiency of the ROXY Pilot Plant is further increased, and the power that is needed to operate the system is used very efficiently. Combining a ROXY Pilot Plant with a lunar heat storage facility as shown in Figure 25 would be very attractive and would provide a novel solution for lunar energy storage.

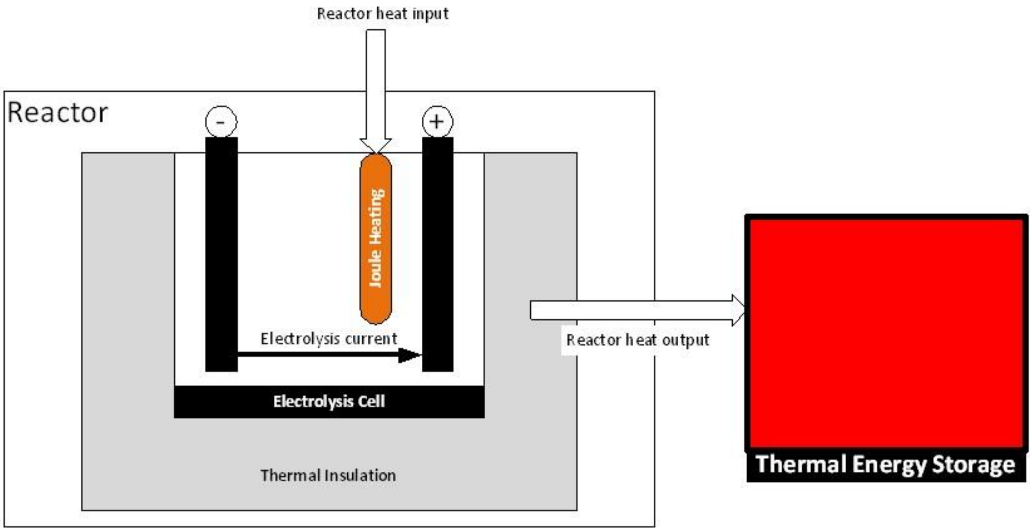


Figure 25: Maximizing energy efficiency by combining a Mini-ROXY reactor with a lunar energy storage system

7 Outlook: The End-to-End Metal Product Value Chain in Space

In the global context of resource utilization, ROXY is a key element in the value chain that ranges from the raw material to finished products, and includes the following major steps:

- Mining
- Beneficiation
- Reduction
- Manufacturing

These issues are addressed in the following sections.

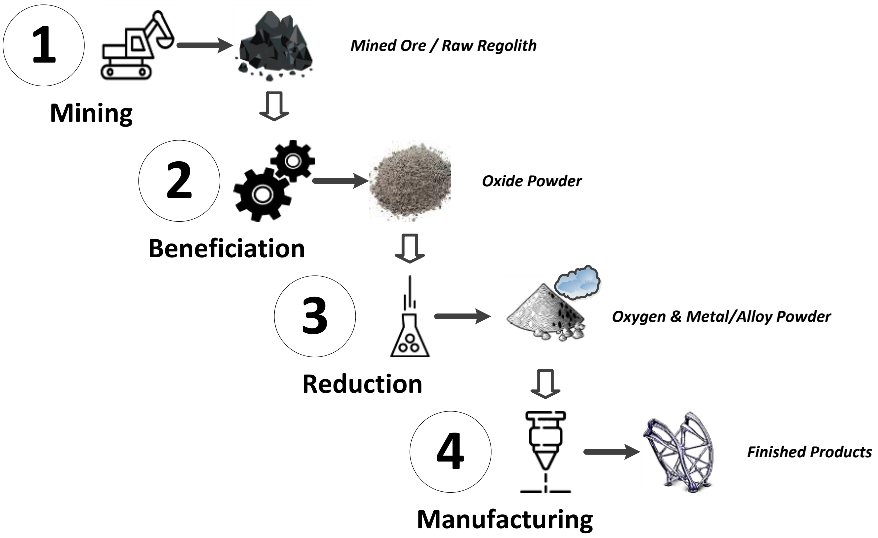


Figure 26: The ROXY Value Chain Part 1: From Regolith to Products

7.1 The Need for a Metal Factory on the Moon

The production of metal-based products on the Moon will require appropriate technologies for the main process steps along the end-to-end metal product value chain, including excavation and mobility, beneficiation, electrochemical reduction, metals extraction and refinement, and manufacturing, including adequate energy supply. This buildup will likely be a staged process starting from initially very limited capabilities both in terms of production capacity and output product quality. A comprehensive scenario for this capability buildup which includes the main process steps and is based on projections of technical capabilities for applications on the Moon is therefore required.

Electrochemical reduction processes like ROXY are preferred processes to extract metals from regolith. The obtained alloys can be used as structural material for colonies, as feedstock for metallic 3D printers, and more. Electrolyzing the regolith powder into oxygen and metals provides two very important materials from an essentially unlimited source.

Plans to colonize the Moon depend heavily on automated construction and manufacturing. Development in 3D printing and additive manufacturing has grown significantly in the last decade, and new additive manufacturing techniques designed to print metal parts in low and zero gravity environments are being developed. The two commonly adopted metal 3D printing techniques are selective laser sintering/melting and electron beam melting. Both techniques are very promising for applications on the Moon.

For additive manufacturing in deep space, i.e., under microgravity conditions, wire-fed metal 3D printers offer a much better alternative for manufacturing parts and structures. The raw material for the wires could be prepared on the Moon from processed regolith by powder-based 3D printing technologies.

Another very interesting application of metal powders is the use as an energy carrier, with applications ranging from temporary energy storage for mobile and stationary applications to rocket propellant. A metal powder energy carrier is a mass-efficient closed loop system and therefore very attractive for lunar applications, for example for applications that require power during the lunar night. The combustion of the metal powder will require the oxygen produced during the reduction of the regolith. After combustion of the metal powder, the resulting metal oxide is reduced again to metal powder using the available electrochemical reduction processes. A closed loop system will then only need to cover the losses of powder and oxygen during the various process steps.

7.2 Capability Buildup and Lunar Constraints

The first hardware sent to the Moon will be high-tech equipment built on Earth. However, the high launch costs will impose strict mass limits, so it will likely have limited manufacturing capability. Therefore, the early lunar infrastructure ISRU facilities will produce crude “mongrel alloys” of iron, aluminum, titanium, silicon and trace metals, and potentially a slag of unreduced oxides. The properties of these mongrel alloys have not been measured yet, but they are expected to demonstrate some ductility and improved tensile strength compared to just melted or sintered regolith.

Hardware constructed from those alloys will need to be massive to add strength and make up for their poor mechanical properties. This will be partially offset by the reduced forces in low lunar gravity. Subsequent generations of metal refineries will add processes and material streams to improve the properties of the materials. In this context it will be very interesting to develop technologies which will allow production of materials with engineered properties from raw regolith, in order to meet a variety of product requirements for products "Made in Space" from solar cells to construction materials. This will ideally require capabilities to extract the main metallic constituents from the regolith, such as Si, Fe, Al, Ti, Mg. If this cannot be achieved completely, capabilities to extract alloys with properties more favorable for the intended applications than the crude mongrel alloy will be needed. During the early phases of lunar production capabilities, the desired materials properties will need to be reconciled with the limited capabilities of the emerging lunar infrastructure, in particular in terms of the cost of transporting the facilities from Earth.

The end-to-end value chain for production of metal products from raw regolith on the Moon comprises the major steps mining - beneficiation - reduction - manufacturing, as shown in Figure 26 above, along with applications of metal powder fuel for both stationary applications and as rocket fuel as shown in Figure 27 below.

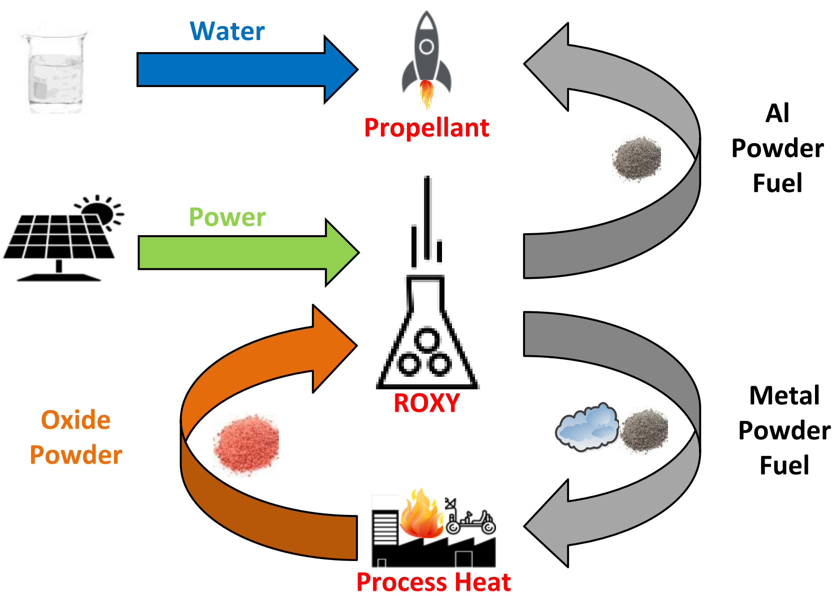


Figure 27: The ROXY Value Chain Part 2: From Metal Oxide Powder to Fuel, A Fully Carbon-Free Circular Economy Approach for Extraterrestrial and Terrestrial Applications

It is expected that no single element or technology in the value chain can provide the capability to extract the main metallic constituents from the regolith completely. The combination of electrochemical reduction technologies with mining, beneficiation, refinement and additive manufacturing is therefore addressed in an integrated approach. An attractive, i.e., economically viable lunar manufacturing technology, must meet the following criteria:

- Environment: Compatibility with lunar environment, i.e., vacuum, 1/6 g, high/low temperatures, radiation, lunar dust
- Input: Compatibility with raw regolith, and/or beneficiated material from upstream process steps.
- Performance: Capability to produce as many useful materials as possible, i.e., a variety of materials/products with engineered properties or in a state that may be easily refined for use in downstream processes
- Process design: Minimized need for high- temperature process steps, choice of simplest possible processes
- Output: High production efficiency, i.e., output to input mass ratio
- Autonomy: Capability to operate with high degree of autonomy, with limited ground supervision
- Mass and Energy: Potential for compact and low-mass design implementation, and minimized energy requirements. Re-use or storage of heat/energy for other processes.
- Consumables: Minimization of consumables, such as reactants or replacement parts to minimize the input from Earth
- Scalability: Scalability to production plant level

These criteria apply to each process step and to the entire value chain.

Another major contributor to the cost of operating equipment on the Moon is the power required to do so, as the power supply equipment will also need to be transported to the Moon, at least in the initial phases of the lunar capability buildup. In later phases, energy may be produced and stored using material produced locally.

A quantitative viability evaluation of a lunar process can be done with a limited set of metrics that address both the performance and the effort of a given process step and the entire system Table 1. For this evaluation, effort/cost is measured in terms of transportation mass and power requirements.

System indicators include an aggregation of the process level figures and further consider any system elements that are not directly attributable to an individual process. Product indicators on the system level address the sum of the end products produced by the system, i.e., the end-to-end process chain. Consumables include both materials consumed by the process, such as gases or fluids, and the replacement of life-limited items during sustained operations.

Item	Unit	Definition
System Production Rate	Mass Out/Time	Rate of product(s) production
System Feedstock Mass Efficiency	Mass Out/Mass In	Ratio of product(s) to feedstock mass
System Mass Efficiency	System Product(s) Mass Production Rate / System Mass	Ratio of product(s) mass to system mass
System Consumable Mass Efficiency	Product Mass / Needed Consumables Mass	Ratio of product(s) mass to consumables mass
System Energy Efficiency	Product Mass / Energy	Ratio of product mass per energy

Table 1: Key Performance/Viability Metrics for Lunar Production Equipment

The initial investment needed to establish a production capability on the Moon is mainly determined by the system mass, as discussed above. The time until break-even is therefore determined by the ratio of the system mass to the rate of production. The economic viability of sustained operations is determined by the ratio of the mass of produced products to the mass of consumables needed to sustain the operation.

Note that the achievement of the break-even point is expected only when a pilot plant and/or an operational plant is implemented, while the potential precursor (i.e., demonstrator) would be aimed at providing the knowledge needed to develop scaled-up systems.

7.3 Raw Material and Production Scales

The raw material that will be used as input material to the process chain is the fine-grained regolith that covers most of the lunar surface. The production scales that are considered in this assessment are aligned with the Global Exploration Roadmap and Space Agency plans, as follows:

Phase 1 - Demonstration:
Total output: 1 g – 1 kg of useable materials/metal products per lunar day

Phase 2 - Pilot Plant:
Total output: 500 kg of useable materials/metal products per year

Phase 3 - Operational Lunar Plant
Total output: >50 tons of useable materials/metal products per year

7.4 Main process steps and technologies

The main process steps of the end-to-end metal produce value chain and candidate technologies are shown in Figure 28 below, and include the following:

1. Excavation and transportation of the raw regolith (A), and delivery to a downstream system (B)
2. Beneficiation of the raw regolith to improve its properties as needed by the downstream processes, and delivery to the regolith reduction system (C)
3. Electrochemical reduction of the regolith with a ROXY or Mini-ROXY process, extraction of oxygen and metals/alloys (D)
4. Extraction and Refinement of the raw alloys to prepare metals with improved composition and physical properties (E). One direct application of this step could be metal powder that is used as an energy carrier.
5. Manufacturing of metal-based products on the Moon, to produce lunar products and/or feedstock for in-space manufacturing, such as wires for a wire-fed metal printer that can operate in microgravity
6. Manufacturing of metal-based products in deep space.

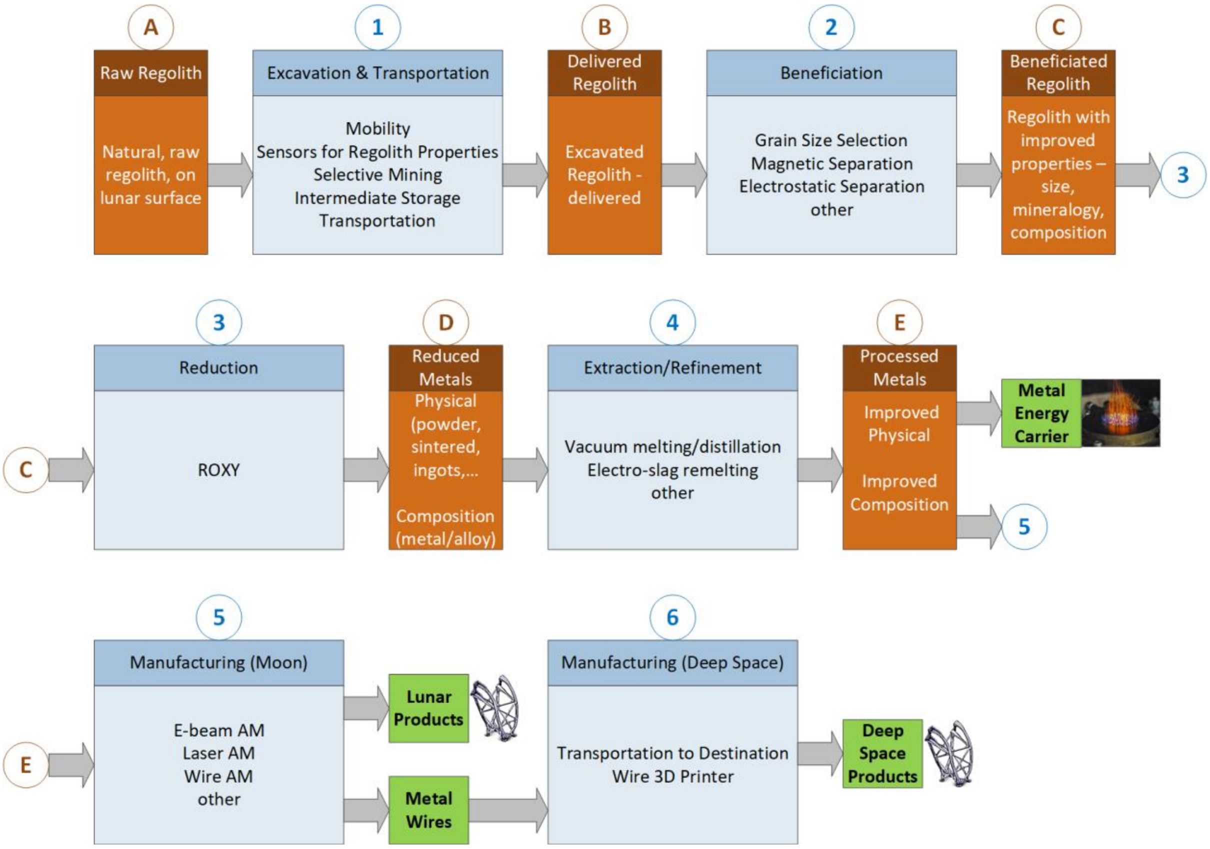


Figure 28: Process Steps and Technologies (blue), Materials (orange) and Products (green)

The overarching objective of ISRU is the production of useable products from local resources, which is the final result of the value chain shown above in Figure 26.The oxide to metal and oxygen conversion which is performed by ROXY is the most important step in this value chain which produces high-purity oxygen directly, plus metal powder. Producing useable products from the metal powder will require at least one more step, shown as #4 "extraction/refinement”.

Additive manufacturing will be ideally suited as a manufacturing technology in this context since the metal powder produced by ROXY can be directly used as an input material. It will therefore be beneficial for the overall objective of ISRU to develop in-space manufacturing (ISM) technologies in conjunction with ROXY.

Relevant technologies and process steps are discussed here below:

<u>Robotic Operations & Autonomy</u>
A high level of autonomy of the robotic operations related to ROXY, such as prospecting (e.g., safe traversal of unstructured terrain, inspection of surface composition), sourcing (e.g., automated excavation and transport of material), and deposition of the processed material (e.g., robotic landscaping and construction) is a key enabling technology for future ISRU missions. Onboard autonomy drastically increases the machines’ utilization rate and allows for task precision that is impossible to reach through human operation today. Those technologies are perfectly in-line with current terrestrial megatrends, such as those found in construction robotics, with immense carryover potential amongst the industries.

<u>Regolith Mining</u>
Regolith acquisition technologies will be required to survey and access the lunar surface, interact with the lunar regolith, acquire and transfer it to the processing plant, while being remotely commanded/ tele-operated.

In this context, selective extraction of regolith will be very interesting to provide raw material with certain enrichments, such as iron, to the processing plant. This will necessitate the development of sensor-technical solutions for the process-oriented material characterization. Data obtained will enable the extraction to be controlled in order to provide the raw material required for the subsequent process steps in the best possible way. Such information will be of interest for controlling the input material to the subsequent process and optimize its efficiency.
Characterization of the lunar regolith before and after acquisition may include techniques like Raman spectroscopy, mass spectrometry, alpha-particle x-ray spectrometry, etc. In addition, a first beneficiation step may be included to filter our large particles which are not suitable for the reduction process.

Beneficiation
Mineral processing of the raw regolith will be used with the objective to improve its properties. Various processing technologies are available and applicable to the particle size range of raw regolith, for screening/classification (separation of particles by size), separation and concentration. Size separation can be accomplished by e.g., sensor-based sorting or vibrating screens. Dry concentration can be performed by e.g., magnetic, density, and electrostatic methods. Further pre-processing steps could include the evaporation of volatiles by heating of the regolith before insertion into the reactor.

Metals Separation and Refining
Following the oxide reduction which is covered in detail in the preceding sections, the separation of metals from each other or the removal of impurities will be required to produce materials with desirable properties. Therefore, the obtained alloy could be processed by diverse refining methods, such as melt filtration, electro slag re-melting in an electron beam furnace, fractional crystallization in zone refining furnaces etc. As a product, metal slabs and ingots can be produced for further applications. These final alloy products can also alternatively be ground to powder with better properties than the one obtained by electrolysis and potentially be more suitable for 3D printing.

Additive Manufacturing by Electron beam Melting (PBF-EB)
PBF-EB is a powder-bed-based technology, which creates high density parts by selectively melting the powder in a layer-by-layer way. The process atmosphere is vacuum, which would make this technology a preferential choice under lunar conditions. This main feature has been beneficial for other reasons in the terrestrial use already:

- highly reactive metals (e.g., Ti, Al) and alloys can be processed,
- outgassing of impurities can take place,
- residual pores do not contain gases, therefore post-processing by hot isostatic pressing (HIP) can eliminate porosity and leads to higher quality and
- a high degree of thermal insulation is provided which makes it possible to keep the elevated process temperature at the desired levels.

The other main features, which makes PBF-EB unique among AM processes is the use of an electron beam with comparatively high beam power, which is used for pre-heating and melting of each powder layer. Pre-heating helps to reduce thermal stresses, because the build chamber can be held at elevated temperatures during the build process. Furthermore, it leads to a comparatively low amount of needed support structures, because mechanical stability is ensured by the pre-heated powder. Due to both facts, materials which are prone to cracking (e.g., tool steels, wear-resistant materials, Ni-base alloys) can be processed. The high energy density enables the densification of otherwise challenging materials, like refractory metals or copper and its alloys.

Laser-based AM from Metal Powder
Since processed regolith from the ROXY process seems to be ideally suited for powder-based additive manufacturing (AM), suitable AM processes and related product outcomes need to be investigated in detail. As laser-based AM (Laser powder bed fusion) today represent one of the industrially most evolved technology, the usability of AM of ROXY powders has to be investigated. This includes the characterization of ROXY powders with respect to composition, particle size and shape distribution, and flowability. These properties then need to be compared to similar powders from alternative production routes. LPBF processed powders are used to investigate their general processability, and process outcomes in terms of material integrity and quality, specifically with respect to composition, microstructure, and strength.

AM from metal wires - ISS 3D printer technology for In-Space Manufacturing under Microgravity
The additive manufacturing from metal wire is compatible with microgravity and thus also useable in deep space. From the metal/alloy output of the ROXY process, a couple of additional steps are required to feed a wire-based metal 3D printer from in-situ resources:

- Evaluate the performance and compatibility with an AM fusion process of the mixed metal aggregate (Si+Fe+Al+Ti+Mn+Ca…)
- Optional: Develop a process to separate all metal constituents and reform the desired alloy, or at least remove undesired elements polluting the fusion process.
- Extrude the metal in a wire-based form usable by the 3D printer, e.g., from cylindrical metallic ingots prepared in a previous step.

Castings from Regolith
Castings on the Moon can be made from recycled or moon-extracted metals with little effort and maximum use of local resources. Regolith can be used as a casting mold for metals and alloys to produce spare parts, tools and larger structures. The metals or alloys from the ROXY process would be a good candidate for such investigations.

Very Large Space Structures
One example of a very large space structure that has been discussed for a long time is a sun shader to deflect a percentage of solar sunlight into space, using mirrors orbiting around the Earth as an alternative way to fight the climate crisis. It would require material that is sourced on the Moon and manufactured in-situ, including structures and thin films. Thin films can be produced from both regolith and reduced regolith using the aerosol deposition method. The technologies discussed above would enable such applications.

8 Benefits for Planet Earth

Low resource consumption and essentially zero emissions make the ROXY process also very interesting for terrestrial use, e.g., for the production of metals. Historically, metal production has been very resource-intensive and has led to a significant contribution to the total global carbon dioxide emissions. For example, steelmaking accounts for up to 5 % of the total global CO_2 emissions. Additionally, many metals are produced with processes that emit large amounts of perfluorocarbons (PFCs), which have a tremendous greenhouse gas potential, about 6500–9200 times of CO_2. Specifically, in the metal product value chain from mined ores - ore concentrates - oxides - metals - alloys - finished products, the most energy-intensive step is usually the oxide to metal conversion. This step typically requires carbothermic, metallothermic, or halide reduction of the oxides.
In addition to the environmental impact, the financial advantage of carbothermic steelmaking is expected to diminish soon with the fast exhaustion of fossil resources. Meanwhile, electricity production is expected to transfer from combustion of fossil fuels to conversion of the more sustainable renewables, particularly solar energy. This inevitable transition of energy supply will force the metallurgical industry to take urgent actions to either accept existing or develop new and efficient electrolytic processes. SOM-based processes similar to ROXY have been applied industrially for rare earth metal production. Adoption of these processes resulted in reduced energy consumption, very low labor, low to zero emissions, and dramatically higher conversion yield.
Furthermore, metals are excellent sustainable energy carriers with very high energy densities. When metal powders burn, the energetic heat is released, while no other combustion product other than solid metal oxide is formed. The technology has been demonstrated on pilot plant level (100kW), further scale-up is ongoing. The solid combustion product can easily be captured for recycling by, for example, ROXY. When the metal oxide particles are reduced back to the metallic particles using renewable energy, the metal can act as a dense, carbon-free fuel. The metals and metal oxides are non-toxic and can be safely stored and transported.

A deeper understanding of granular flow phenomena as discussed in section 3.6.1 will also help improve life on Earth, as it could potentially be used to reduce the energy requirements of granular material transportation in a wide range of ubiquitous industrial processes. Successful execution can also be used to help manufacturing industries improve their energy efficiency here on Earth.

9 Heritage and Team

9.1 Heritage

The ROXY process is closely related to the SOM process developed by Uday Pal from Boston University over the last 20 years with funds from the NSF, DOE and others.

In the metal product value chain from mined ores to finished products, the most energy-intensive step is usually the oxide-to-metal conversion. Metal producers, such as primary aluminum, ferroalloy, iron and steel, lead, magnesium, and zinc, employ carbothermic, metallothermic, halide reduction, and electrometallurgical methods, to produce the metals and in the process generate significant amounts of pollutants including greenhouse gases (GHG). In the US alone, these industries produce more than 100 million metric tons of CO_2 Because of its impact on the environment there is an urgent need to decarbonize the metal producing industries. Electrolysis offers the opportunity to substitute clean electrical energy for fossil fuel energy sources. But commercial electrolytic oxide reduction processes require carbon anodes to electrolyze oxides dissolved in halides and that emits CO_2 and perfluorocarbons with very high global warming potential. As of this writing, no oxygen-producing inert anode technology has yet been scaled beyond a single pilot electrolysis cell.
Through sponsored research from the National Science Foundation and the Department of Energy, Pal pioneered a cost-effective, energy-efficient and low-emissions approach that uses an oxygen-ion-conducting solid oxide membrane (SOM) as a selective electrolyte between the molten salt and the anode for the generic production of metals and alloys. The SOM-based technology produces metals (Me) and/or their alloys at the cathode and pure oxygen gas at the anode from their respective oxides (MeOx) through direct electrolysis of the oxides.
Pal's work (NSF Award 1210442) on YSZ membrane stability during SOM electrolysis using eutectic molten salt composition in the CaF_2-MgF_2 system demonstrated that membrane stability can be greatly enhanced by matching the component activity and optical basicity in the salt with that of the membrane. Pal also showed (NSF Award 0457381) that the oxide solubility in the salt can be controlled through tailored additions of more stable alkali oxides and monitoring the heat of mixing; higher heat of mixing promotes oxide solubility and vice-versa.
Pal has partnered with Sun Edison to investigate SOM process scaleup for solar grade Si production (NSF Award 1601583). Fundamental studies on structure-property relationship of molten salts for the SOM process (NSF Award 1937829) has helped extend Pal's collaboration with Airbus Defense and Space (Friedrichshafen, Germany), TU Bergakademie (Freiberg, Germany), Fraunhofer Institute for Manufacturing Technology and Advanced Materials (Dresden, Germany), and the German Space Agency.

The ROXY process was invented by Airbus and has many similarities with the SOM process. For example, it uses SOM-type anodes to extract oxygen and metals from regolith. The process elements and technical implementation have been optimized for regolith reduction. The team from Airbus, Uday Pal, and Fraunhofer IFAM has developed ROXY to its current state over the last couple of years.

The end-to-end ROXY process, i.e., oxygen extraction from regolith simulants, has been tested in various implementations of the process. Different anode types, cathode materials and geometries were tested at temperatures between 850 °C and 1050 °C. The process was found to work as predicted by the model calculations. Oxygen was extracted from the regolith, and it was shown that the amount of oxygen corresponded to the measured oxygen ion or electrolysis current. From this and from an analysis of the regolith reduction characteristics, it was concluded that regolith can be completely reduced by ROXY with a current efficiency close to 100 %. It was further shown that the energy efficiency of the process is very high and essentially determined by the thermodynamic and kinetic parameters of the process. The advanced concept Mini-ROXY has been invented in response to the need for an affordable lunar demonstration of the ROXY process. Mini-ROXY is the next step towards resource efficiency, is quickly implementable and provides a basis for scaled-up ROXY facilities.
In September 2023, a project focused on a ground-based test campaign of the Mini-ROXY technology and a lunar mission concept has started (see chapter 5). The project also includes the TU Bergakademie Freiberg as a partner and is funded by the German Federal Ministry for Economic Affairs and Climate Action.

As part of his work on DLR project (ERICA) Dr. Kollmer has developed a low gravity test platform for use in the ZARM Bremen droptower. The platform consists of a vacuum chamber mounted on a linear stage inside a droptower capsule. Once the droptower capsule is in zero gravity the linear stage can accelerate, creating – with high precision – the desired gravity level. Since it uses a linear motor instead of a centrifuge no centrifugal force gradient or Coriolis forces can disturb the experiments. The vacuum chamber is suitable for housing regolith and has power and data feedthroughs, a mechanism to release sample material from the top of the chamber and a high-speed camera to monitor its inside. Currently the low gravity test platform is used in the DLR GRIS project (in collaboration with Dr. D'Angelo) to study funnel flow. From these funnel flow experiments frictional properties of regolith in low gravity are determined. Dr. Kollmer also currently uses the platform in the DLR ELISA project where the mechanical properties of charged regolith aggregates and packings are studied.

9.2 Team

Airbus is Europe's largest and most innovative defence and space company. We create innovative, effective space and defence solutions and services for our customers, driving our industry forward. Our portfolio is truly inspirational: we created and operate Europe´s Columbus laboratory and help our partners go to the Moon and Mars. Around the world, our satellites keep people connected and protected; our exploration to the far reaches of our solar system helps us understand the origins of life. Our strength lies not only in our renowned product excellence but in Airbus' mastery of complex systems. This is the key to unlocking many of the world's challenges – from connecting the world and its aircraft to enabling real-time defence of the future. We are always ready to challenge the status quo: whether in self-financing the next generation of very-high-resolution imaging satellites or developing manufacturing concepts to produce hundreds of satellites a year. Through all our endeavours, we strive for the highest performance with our sights set on becoming the world leader in space solutions.
For the last decades the department TSI (Microgravity Payloads) of Airbus DS GmbH in Friedrichshafen has been a world leader in the development and operation of scientific payloads for microgravity experiments.
Through its participation as Prime Contractor and system engineer in numerous payload development projects for the Space Shuttle and ISS, Airbus DS has developed vast experience in all relevant science fields. This includes materials science, fluid science, fundamental physics, life science, and life support systems of closed habitats in space and in submarines, including its relevance and expendability to energy systems and thermal systems, including regenerative fuel cell systems. Airbus DS has developed the external Columbus platform Bartolomeo and offers it to users through a commercial partnership with ESA.
Airbus DS development of materials science facilities for the ISS including the Materials Science Lab MSL, and the Electromagnetic Levitator EML, which allows in-situ investigations of levitated liquid metal droplets at temperatures of up to 2000°C under ultraclean process conditions, and has led to the invention of the ROXY process.
Achim Seidel has over 33 years of professional experience in the development, operation and management of payloads and instruments for space missions. He holds a Masters´ Degree in Space Sciences from Florida Tech and a Ph.D. in Physics – Materials Science from Karlsruhe University.
Achim has worked as system engineer and project manager for the electromagnetic levitator payloads Tempus and EML, the Materials Science Laboratory and has managed a variety of ISS payload development projects such as ACES and Cryosystem, covering the entire life cycle of the payload from conception to mission operations. Based on his background in Solid State Physics, he has acted as the interface to the science community for all aspects regarding experiment objectives, payload requirements, design and development, and accommodation of the experiments into the payload.
Achim has invented the ROXY process and leads the Airbus-internal R&D project to further develop the related technology for applications on Moon, Mars, and Earth.

Prof. Uday Pal is a professor at the Division of Materials Science and Engineering and Department of Mechanical Engineering of **Boston University**.
The overarching theme of Professor Pal's research is to utilize materials-based solutions to the critical environmental and energy crises confronting us. Electrochemical Devices convert and utilize chemical and electrical energies at high efficiencies and are thus eminently suited for many applications resulting in reduced greenhouse gas emissions. Life and cost of these devices and systems are principal barriers to their commercialization.
On-going research in Professor Pal's laboratory include: solid oxide fuel cells, solid-oxide-membrane based electrolytic cells for converting waste to hydrogen, hydrogen storage materials, solid-oxide-membrane based inert anodes for green syntheses of energy-intensive metals, and devices based on mixed-ion-electron-

conducting oxide membranes for generating and separating pure hydrogen from hydrocarbons enabling CO_2 sequestration.
Prof. Uday Pal is the inventor of solid oxide membrane (SOM) electrolysis, an inexpensive, energy-efficient, environmentally friendly, one-step method that he has developed over the past 20 years to separate pure metals from their oxides. SOM electrolysis continuously feeds metal oxide into a molten salt bath, where electricity splits it into metal and oxygen gas.
Conventional metals production technologies employ a lot of carbon-based energy sources to reduce oxides, and generate significant amounts of pollutants, carbon dioxide and other greenhouse gases. SOM electrolysis promises to substantially decrease energy consumption and eliminate carbon and other environmentally harmful emissions associated with reducing oxides to metals, all for less cost. So far Pal has developed and applied his patented SOM electrolysis technology to produce magnesium, titanium, silicon, aluminum and other energy-intensive metals from their oxides.

The Fraunhofer-Gesellschaft based in Germany is the world's leading applied research organization. Prioritizing key future-relevant technologies and commercializing its findings in business and industry, it plays a major role in the innovation process. The Fraunhofer-Gesellschaft's interdisciplinary research teams turn original ideas into innovations together with contracting industry and public sector partners, coordinate and complete essential key research policy projects and strengthen the German and European economy with ethical value creation. International collaborative partnerships with outstanding research partners and businesses all over the world provide for direct dialogue with the most prominent scientific communities and most dominant economic regions.
The Fraunhofer-Gesellschaft currently operates 76 institutes and research units throughout Germany. Over 30,000 employees, predominantly scientists and engineers, work with an annual research budget of €2.9 billion. Fraunhofer generates €2.5 billion of this from contract research. Industry contracts and publicly funded research projects account for around two thirds of this sum. The federal and state governments contribute around another third as base funding, enabling institutes to develop solutions now to problems that will become crucial to industry and society in the near future.
Fraunhofer IFAM, Branch Lab Dresden, one of the leading institutes in powder metallurgy, conducts fundamental and applied research for solution-oriented material and technology development. It focuses on innovative sintered and composite materials, functional materials as well as cellular metallic materials for energy technology, mobility and medical technology. Hydrogen technology plays a key role in the field of energy technology.
For more than 20 years, the Branch Lab Dresden has been engaged in research on technologies to produce porous metallic materials. The department represents the world's largest competence center in this field. Numerous production processes with a wide variety of structure types have been established on a laboratory and pilot plant scale, which enable the production of cellular metallic materials with a wide range of pore size (5 µm to 10 mm) and porosity (50-97%).
During the development of the ROXY technology, Fraunhofer IFAM has closely collaborated with Airbus Defence and Space and the extensive ROXY investigator network, focusing on technological issues such as material compatibility, cathode development, electrolysis cell set-up and testing as well as safety issues. Testing facilities for a variety of implementations of the ROXY processes are operated in the institute's laboratories. The Fraunhofer IFAM contribution to the Mini-ROXY design is based on the significant heritage and experience from those activities. The institute is the coordinator and lead of the currently ongoing Mini-ROXY project. At Fraunhofer IFAM Dresden, **Georg Pöhle** and **Christian Redlich**, both holding Ph.D.´s in Materials Science, are dedicated to the design, development and testing of materials and components for ROXY. They are supported by experienced technical staff for the operation of furnace technology and safety facilities as well as in-house experts on physical, chemical, and mechanical analysis and characterization.

The **German Aerospace Center (DLR)** is the research center for aerospace as well as the national space agency of the Federal Republic of Germany. Around 10.000 employees are doing research in a broad spectrum of topics concerning aviation, space, energy, traffic, security and digital services. Their missions extend from fundamental research until the development of innovative applications and products for tomorrow.
The DLR Institute of Space Systems designs and analyzes future spacecraft and space missions (launchers, orbital and exploration systems, and satellites), and assesses them with regard to their technical performance and cost. It applies state-of-the-art methods of multi-disciplinary engineering in system design and analysis – for example, a computerized system for concurrent design.

The DLR Institute of Space Systems has a wide range of facilities, personnel and experience associated to the manufacturing, integration and testing of whole spacecrafts, payloads and subsystems. These include general facilities like a certified clean room for hardware assembly and integration, thermal and mechanical test laboratories, electronical and mechanical workshops, as well as specialized facilities such as:

- Concurrent Engineering Facility (CEF) dedicated to system and mission feasibility studies for phase 0, pre-phase A and phase A.
- Planetary Infrastructures Laboratory dedicated to performing experiments in the field of ISRU, including regolith beneficiation.

Key personnel:

- **Dr. Paul Zabel** holds a PhD in aerospace engineering with a focus on ISRU and Life Support Systems. He has been involved in different positions in various research and development projects over the past 10 years, among which the EU-funded ISRU project LUWEX is the latest. Since 2021, he is leading a research team dedicated to ISRU technology development at DLR.
- **Kunal Kulkarni** is a PhD candidate at DLR focusing on the development and optimization of beneficiation technologies to process planetary regolith. He also has experience in producing lunar regolith simulants.
- **Luca Kiewiet** is a PhD candidate at DLR focusing on the development and validation of technologies for water extraction from icy- regolith.
- There are also several highly-experienced systems engineers present in the institute which work on demand for different projects.

Dr. Jonathan Kollmer at **<u>Universität Duisburg-Essen</u>** has over a decade of professional experience in microgravity experiments related to granular materials. Trained as a physicist (Universität Bayreuth) he did his Ph.D. at Friedrich-Alexander-University Erlangen Nürnberg during which he developed vibration dampening devices for microgravity applications based on granular materials. He worked as a PostDoc at North Carolina State University where he was Co-PI of the NASA funded EMPANADA (Ejecta-Minimizing Protocols for Applications Needing Anchoring or Digging on Asteroids). Since 2018 he is a researcher and teacher (academic council) at Universität Duisburg-Essen where he is involved with several German Space Agency (DLR) funded projects on the behavior of granular materials in low gravity. These projects include the ARISE (and follow up CHAP) payload on ISS as studying electrostatically charged grains in the context of planet formation. He is PI of the ERICA (Experiments on Rebounding Impacts and Charging on Asteroids) and ELISA (Experiments on Low-gravity Impacts and Settling of Agglomerates) projects studying the properties of cohesive regolith packings in low gravity in the ZARM Bremen Droptower. He further is PI of EDAM (Experiments on the Deposition of Agglomerates on the Moon) studying granular flow on a commercial rocket flight & PI of GRIS (Granular Rheology In Space) researching the flow behavior of regolith in reduced gravity of parabolic flights and the ZARM GTB-Pro. As part of GRIS he is collaborating with a sister project at FAU Erlangen-Nürnberg and an additional collaboration with NASA Glenn Research Center is currently being initiated. Jonathan is also involved in the ESA topical team VIP-Gran and a consultant for ESA in the soft matter facility definition team for post-ISS planning.

Dr. Olfa D'Angelo's work at **<u>Erlangen-Nürnberg University</u>** focuses on the rheology of complex fluids, including granular materials, and the influence of gravity on their flow-behavior. Following her Ph.D. at RWTH Aachen and the German Aerospace Center (DLR) on "Powder-Based Additive Manufacturing for Space Applications", which resulted in two patented technologies, she continues her research in the field of materials physics under low-gravity conditions. She has successfully led several teams in gravity-related experimental campaigns, including drop tower, parabolic flights, and centrifuge, and puts this experience at the service of ESA as a consultant in the soft matter facility definition team for post-ISS planning. As co-leader of the GRIS project, her current interests include the development of new technologies, notably towards ISRU and space exploration, and understanding the behavior of soft matter under low- and microgravity.

10 Intellectual Property

The following patents are relevant for the ROXY and Mini-ROXY technologies:

Title	Owner	Patent Number	Date	Status
Electrolysis apparatus for the electrolytic production of oxygen from oxide-containing starting material	Airbus Defence and Space	US-Patent 11479869	12.10.2020	Active
Elektrolysevorrichtung zur elektrolytischen Produktion von Sauerstoff aus oxidhaltigem Ausgangsmaterial	Airbus Defence and Space	EP 3812483	24.10.2019	Active
Method and system for extracting metal and oxygen from powdered metal oxides	Airbus Defence and Space, Fraunhofer-Gesellschaft, Prof. Uday Pal	US-Patent 17509394	25.10.2021	Pending
System and method for extracting oxygen from powdered metal oxides	Airbus Defence and Space, Fraunhofer-Gesellschaft, Prof. Uday Pal	US-Patent 17509431	25.10.2021	Pending
Oxygen-Producing Inert Anodes for SOM Process	Boston University	US-Patent 8658007	25.02.2014	Active
Method and apparatus for producing solar grade silicon using a SOM electrolysis process,	Boston University	US-Patent 10266951	23.04.2019	Active
Reactor Device for Converting Powdered Metal Oxides and Conversion System Comprising Same	Airbus Defence and Space, Fraunhofer-Gesellschaft, Prof. Uday Pal	US-Patent 18386075	01.11.2023	Pending

Table 2: Relevant patents for the ROXY and Mini-ROXY technologies

11 Conclusions

The ROXY process that has been invented by Airbus and has many similarities with the SOM process invented by Boston University meets the ISRU viability criteria, and is therefore considered to be the core of a future large-scale lunar oxygen and metal product value chain. A first major step will be the demonstration that ROXY processes will work in the lunar environment, under reduced gravity and with true lunar regolith as feedstock. Meeting this objective does not require that a large amount of oxygen is produced. This opens up the possibility to design a cost-optimized lunar demonstration mission with a simple, compact and low-mass demonstration facility, provided that a miniaturized process can be designed. The Mini-ROXY process has been invented, conceived, and validated by Airbus in collaboration with Boston University and Fraunhofer IFAM to meet those objectives.

Current gaps in our knowledge on the properties of lunar regolith and the gravity dependency of the processes associated with the Mini-ROXY process are addressed by a multi-disciplinary international science team. This team has unique experience in the areas of the ROXY process, the regolith and gravity dependency of the associated process steps, and in developing space equipment for materials science applications. The team is ideally positioned to take the next steps in the development of lunar ISRU technologies, starting with a lunar demonstration of the Mini-ROXY process.

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FAU Erlangen-Nürnberg heritage work is funded by the German Space Agency DLR.

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13 Selected Publications

1. Seidel, A., Häming, M., Monchieri, E., Adrian, A., Joumel, P., Quadbeck, P., Fereres Rapoport, S., Pal, U., Spierings, A., "An electrochemical viable lunar ISRU process for oxygen and metal production and related benefits for terrestrial applications", 72nd International Astronautical Congress (IAC), Dubai, United Arab Emirates, 25-29 October 2021, IAC-21-A3, 2C3
2. A. Seidel, M. Altenburg, E. Monchieri, F. Strigl , P. Quadbeck , C. Redlich, U. Pal, “ROXY - An economically viable process to produce oxygen and metals from regolith”, 51st International Conference on Environmental Systems 10-14 July 2022, St. Paul, Minnesota, ICES-2022-140
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