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VELOX – A DEMONSTRATION FACILITY FOR LUNAR OXYGEN EXTRACTION IN A LABORATORY ENVIRONMENT

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ABSTRACT

The ultimate goal of a permanent human presence on the Moon is discussed intensively within the global lunar community. Obviously, such an effort poses stringent demands not only on the technology but also on logistics, especially considering the important aspects of masses and volume for materials and replenishments of consumables. On-site propellant production (i.e. liquid oxygen) is one of the main needs and would lead to more efficient return-to-Earth or further exploration missions. Additionally, the supply of breathable air and water for the survival of the crew on the lunar surface is also a major aspect. Thus, large effort is put into the development and research of technologies for in-situ resources utilization (ISRU) to drastically reduce the required supply from Earth and to increase the level of autonomy of a lunar outpost. The major resource on the Moon for such a purpose is regolith, which covers the first meters of the lunar surface and contains about 45% of mineralogically bounded Oxygen in terms of mass. By using adequate processing methods of this material, one could be able to extract valuable minerals and volatiles for further utilization.

At DLR Bremen a compact and flexible lab experimenting facility has been developed, built and tested, which shall demonstrate the feasibility of the process by extracting oxygen out of lunar regolith, respectively soil simulants and certain minerals in the laboratory case. For this purpose, important boundary conditions have been investigated such as temperatures during the process, chemical reaction characteristics and material properties for the buildup of the facility, which shall be analyzed within this paper. Since it is one of the most elaborated chemical processes regarding ISRU and has comparably low temperature and energy constraints it has been primarily concentrated on the Hydrogen-reduction process which reduces the iron oxide component of Ilmenite ($FeTiO_3$) within the lunar regolith. Based on the obtained results, a first line-out of a planned superior test set-up and infrastructure with pre- and post-processing units such as feeding and extraction is also presented, as well as an analysis of reaction products with common methods.

This paper will present the first results of DLR efforts regarding these topics. Finally, important aspects of the future development of the processes and technologies are discussed with special consideration of lunar applicability and with respect to environmental conditions as well as mass and energy constraints.

INTRODUCTION

Within the context of European as well as international exploration activities, returning to the Moon – manned or unmanned – is a mid-term goal of the entire space science community. Besides transfer vehicle, lunar landing and surface module developments, in-situ resource utilization (ISRU), in particular Oxygen production, is of much interest for future lunar activities.

About 45 weight-% of the lunar soil is oxygen bounded in minerals like silicates, Calcium oxide or Ilmenite. Ilmenite which is contained in Earth and also Moon basalts consists of Titanium oxide (Rutile) and Iron oxide. It is the major resource for the selected Oxygen extraction process of this project and will be reduced by floating hydrogen and an operational temperature of about 1000° Celsius. At this temperature the iron oxide component releases the Oxygen component which reacts with the gas and produces water vapor. This will be processed by common techniques up to the production of pure Oxygen, either gaseous or liquefied, depending on the final purpose. This process is of special interest, since it is one of the most elaborated chemical processes regarding ISRU and has comparably low temperature and energy constraints.

At the Institute of Space System of the German Aerospace Center (DLR), which is dealing among others with System Analysis and Space Exploration, the project called "Verification Experiments for Lunar Oxygen Production (VELOX)" has been established in order to contribute to the ISRU activities of the international science community.

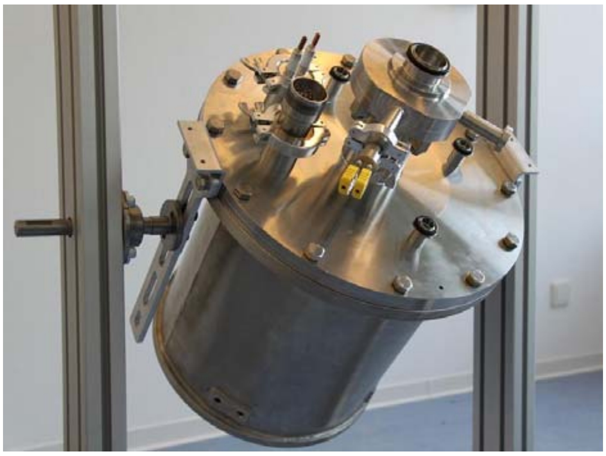

Fig. 1: Reaction chamber with outer casing of the inner loop, feed-throughs and in-/outlets and without feed lines and supporting or measurement devices

In the frame of present study a small demonstration plant which can be seen in Figure 1 has been built which shall allow research on lunar Oxygen production topics here on Earth in a laboratory environment. Furthermore, the activities are driven by the goal to develop a system which is able to be used on the Moon with a minimum set of modifications.

The present paper starts with the project objectives, top-level requirements and a brief description of the reaction process. Furthermore it discusses the main design trades on system level and the derived design, mainly focusing on the reaction chamber since the entire set-up including the entire periphery is not completed yet.

The reactants of the reduction process are described as well as the overall process cycle and its measurements and instrumentation. The first test results of Phase 1 (see Table 1) and the experiment campaigns for later phases are introduced. Finally, the paper summarizes the current activities and explains the next steps

VELOX OBJECTIVES

Since several interests are driving the project and already much effort has been made by other institutions during last years the objective is not only to demonstrate feasibility of Ilmenite reduction.

General feasibility has already been demonstrated and the needs intensively discussed among others by NASA [1], [2] and the Tokyo Institute of Technology together with the Shimizu Corporation [3], [4].

Therefore, the objectives of the DLR test facility and its related experiments can be divided in two different categories.

The primary objectives which are the main project drivers and describe the basic scientific interests need to be fulfilled. They could either be on a short- or long-term basis and are as follows:

I. to extract Oxygen out of lunar (simulated) soil and in a laboratory environment
II. to quantify and compare efficiencies and yield rates for lunar soil simulants and selected mineral compounds by parameter variation
III. to define critical parameter of the Hydrogen reduction process
IV. to perform analyses for efficiency increase opportunities of the Hydrogen reduction process
V. to determine the reaction process and design comparability between terrestrial and lunar applications
VI. to derive clear requirements and forecasts for a future lunar Oxygen production plant
VII. to gain a better understanding of Oxygen extraction techniques out of (synthetic) lunar soil and strengthen DLR competencies on ISRU related topics for space exploration

The secondary objectives which have to be understood as value-adding goals are mainly related to potential future applications:

- to provide a reaction chamber which can be used also for volatile measurements
- to generate a test-bed for comparisons of different Oxygen extraction techniques
- to operate a fully automatic parameter regulation and
- to compare experiments following both the electrical and solar-thermal power supply approaches with co-operation partners

PROJECT PHASES

The project is divided into several phases in order to address the objectives and to fulfill the goals step by step while continuously enhancing the system and its capabilities. There have been six major project phases identified, including the design and set-up phase which are summarized in Table 1:

Phase	Title / Activities
0	Design and Set-up of Core Modules
1	Testing Preparations
2	Regolith Heating Behavior
3	Process Feasibility with VELOX system
4	Efficiency Analyses Campaigns
5	Volatile Measurement campaigns

Table 1: VELOX project phases

In parallel to the Phases 2 - 5, the design of the pre- and post-processing units as well as the definition and integration of the periphery will be completed.

Currently the project is finishing Phase 1 and prepares for Phase 2.

THE REACTION PROCESS

The process which has been selected as most promising for these activities is the "Hydrogen Reduction of Ilmenite". After the Apollo missions have explored the lunar surface and brought back several kilograms of regolith to Earth, the idea of ISRU has been investigated intensively.

During the following years 21 potential processes have been identified as general options to extract Oxygen out of lunar soil [5].

An intensive trade-study considering the maturity of the processes, the experience of the staff, the comparability between Earth and Moon applications as well as technical and safety issues led to the decision to use Hydrogen flowing through the heated soil to produce oxygen. The following equations are representing the basically involved elements and show the two-step chemical reaction process which will be utilized [1]:

$$FeTiO_{3(s)} + H_{2(g)} \rightarrow Fe_{(s)} + TiO_{2(s)} + H_2O_{(g)} \quad (1)$$

$$2H_2O_{(l)} \rightarrow 2H_{2(g)} + O_{2(g)} \quad (2)$$

The first step will be performed by the fully developed VELOX system, whereas the second step represents the electrolysis principle which will be done by additional equipment. Therefore, between these two steps the water vapor has to be cooled down and liquefied in order to use common methods. Different electrolysis processes are outlined in [6].

At DLR some related experiment campaigns with respect to lunar Oxygen production have already been performed using Vacuum Pyrolysis (Vapor Phase Reduction) as a different reaction process [7].

Within this work the extraction of oxygen out of soil simulants was successfully demonstrated, whereas capturing the Oxygen still remained a problem. The Hydrogen reduction process is much more mature and although the yield rates are less than using the Pyrolysis technique, the entire process is feasible.

SYSTEM REQUIREMENTS

The challenge of this and any other similar projects is to combine different environmental and process-related constraints for

- vacuum activities,
- regolith (abrasive dust) handling,
- thermal control (< 1000° Celsius),
- utilization of Hydrogen and
- utilization of inert gas.

Derived from the objectives the following system requirements have been defined:

- The system shall follow a modular design in order to ensure expandability and future modifications.
- The system shall use commercial-of-the-shelf (COTS) components in order to reduce cost and allow simple integration and replacements wherever possible.
- The system shall be easily transportable
- The system shall deal with batch-wise reduced soil samples in order to ensure comparability with precursor studies.
- The system shall provide comparability to potential subsequent lunar facilities in terms of energy demand, Oxygen yield and process cycles.
- The system shall allow the automated regulation of the process parameters such as time, pressure, temperature and volume flow.
- The system shall ensure fail-safe operation with the utilization of hazardous gas (e.g. hydrogen) as well as inert gas.

MAIN SYSTEM DESIGN TRADES

In order to tackle the detailed design and construction phase some overall decisions on system level had to be made. Trades having the highest impact for further design are discussed in the following:

Energy Supply

There were two main options considered to provide the required energy for the reaction process: solar-thermal and electrical energy supply.

Solar-thermal energy supply: Using solar concentration systems like parabolic dishes, heliostats or Fresnel lenses is one way to introduce thermal energy into the feedstock.

Parabolic dishes collect solar rays and generate a focal point in a distance according to their convex bend. Following the simple equation

$$d = \sqrt{\frac{P_{req} * 4}{Is * \eta_{ref} * \pi}} \quad (3)$$

with the assumptions Is = 1350 W/m^2 as solar flux for the Moon and Is = 850 W/m^2 for the Earth in Central Europe [7] and a conservative reflector efficiency factor of $\eta = 0.5$ this leads to an estimation of the reflector diameter d as depicted in Figure 2.

Heliostats like the DLR solar furnace [7] split the bended surface of a parabolic dish into several different tilted mirrors causing the same concentration effect.
Fresnel lenses follow a similar principle but break the light within the vitreous lens body instead of reflecting the solar rays.

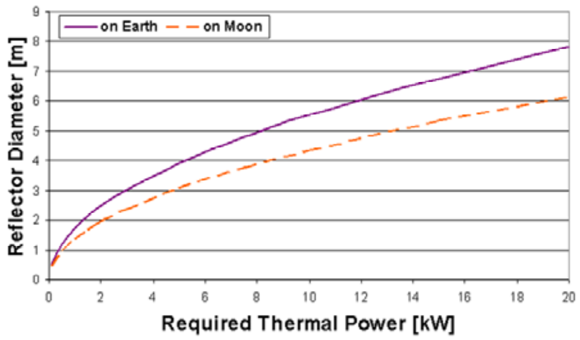

Fig. 2: Comparison of Moon and Earth reflector diameters for given required thermal power demands of the reaction process

These concepts are widely considered for lunar applications because they are acting almost independent without a high rate of additional electrical energy which still has to be provided. A parabolic dish for instance is comparably light and has higher efficiencies compared to a solar array. On the Moon micro-meteoroid impacts would not cause major issues since a dish or mirror with a diameter of several meters provide sufficient area to compensate a certain degradation. During lunar daylight the concentrator system has to track the sun continuously in order to collect the solar rays and to generate a high-energy focal point which most likely requires an additional mirror for guiding the beam into the process chamber.

On Earth constant daylight is not guaranteed. Additionally, the efficiency of the beam is much lower due to the reduced solar flux. However, this effect can be compensated with larger surface areas. At DLR Cologne there is a solar furnace which uses heliostats for solar concentration. This one was used for the mentioned Pyrolysis experiments [7]. During these activities it also has been reported that the extracted volatiles will be released and contaminate the illumination window. This will occur more intense using Pyrolysis than during the Ilmenite reduction due to much higher temperatures, but ascending gaseous molecules generally reduce the solar-thermal supply efficiency and generated additional tensions [7].

Another option for solar-thermal energy supply is using an optical waveguide system. Arrays of several concentrators with additional reflector surfaces feed the focused solar rays into an optical fiber cable which transports the thermal energy into the reaction chamber. Overall system efficiencies of 40-60% seem to be achievable with current existing systems [8].

Electrical energy supply: An alternative to the solar-thermal systems are electrical energy supply system. They require a certain power generation which on the Moon can be realized either by solar arrays, battery systems, fuel cells or radio-isotope generators. No solar tracking is required but additional equipment to transmit the energy from the source to the consumer, i.e. the ISRU plant.

On Earth there is public electricity available which of course reduces the complexity for the presented demonstration facility. In order to introduce the heat into the process chamber, heating elements are required which convert the electrical energy into thermal energy. This will then be transferred primarily via radiation and additionally by convection which will be neglectable on the Moon or in a vacuum here on Earth.

The sample heating occurs from outside to the center of the chamber whereas for solar-thermal systems the heat will be spread from the focal point to the process chamber wall. Figure 3 shows two design concepts for the heating elements which can be used, either as one single heat source for the process chamber or as a single element which could be modified individually:

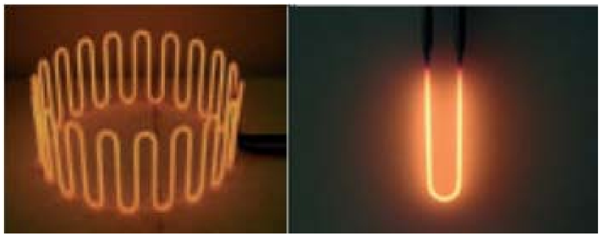

Fig. 3: Electrical heating elements; meander shaped (left) and U-shaped (right) [9]

Future lunar scenarios including habitation and fully Earth-independent human or robotic activities will have large areas of solar arrays available. In fact, since the entire Ilmenite reduction process requires electrical energy supply energy anyway, for e.g. the electrolysis the valves, engines and the instruments, using heating elements reduces the overall system complexity due to the limitation of energy sources to one single technology. Additionally, using energy storage such as secondary battery systems, the facility could be operated with the electrical energy supply system also during night if sufficient insulation is given.

These arguments and the better transportability for Earth applications led to the electrical option as a baseline for VELOX demonstration plant.

<u>Shape of the inner process chamber</u>

Originally, for the inner process chamber a flat bottom surface was foreseen. A fully cylindrical structure would have led to an easy manufacturing process and integration with a meander shaped electrical heating element (left in Figure 3).

This process chamber type, called solid bed reactor, has already been applied within the research of [3], [4]. Since one of the objectives is to investigate potential options for efficiency increase, the design has changed to a reactor shape which also allows a fluidization of the solid reactant. This also has been proposed by [10].

Therefore the bottom surface became conical in order to ensure a smooth transition from the outer wall to the gas inlet.

Fluidization increases the particle-wall and the particle-particle interaction. This reduces the heating time and allows an easier diffusion of the reaction gas.

Since also the heating elements had to be adapted and became a set of eight tilted U-shaped coils leading to a conical set-up it is possible to increase or decrease the distance between the heaters and the process chamber more easily. Figure 4 shows the final arrangement

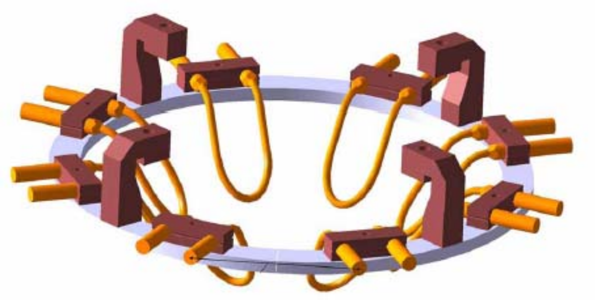

Fig. 4: Tilted coils and conical heater arrangement

For the early experiment campaigns the reduction will follow the fixed-bed-reactor concept but after an additional simulation and testing phase the appropriate gas volume flow for the fluidization of the feedstock will be determined.

Reactor Mounting

The entire Reactor has to be integrated in a test-stand within the laboratory. Additionally, it requires access to almost all sides of the system at any time during the preparation and reaction process. It has been identified that the reactor becomes relatively heavy for Earth test applications to allow pressure differences between the atmosphere, security chamber and inner loop of up to $2*10^3$ hPa which is the design baseline for the intended pressure regulations.

A framework has been designed to hang up the furnace. Instead of a fixed construction the Reactor remains rotatable. Figure 5 shows the arrangement and mounting to the framework. The rotation axis has to be close to the center of mass of the reactor. Therefore two flanges, one at each side, with elongated holes are foreseen to adjust the mounting positions whenever it is necessary.

The advantage of this rotary principle is twofold: on the one hand it allows easy feeding and removal of the feedstock while turning the regolith in-/outlet to the pre-processing (feeding) unit which is mounted above the reactor or to the feedstock collection container which is mounted below the reactor.

Moreover, this mounting principle with an additional stepping motor attached to the rotation axis will help to further increase the efficiency of the reduction itself.

Rotating the reactor smoothly back and forth during the reaction process leads to a feedstock circularization within the inner process chamber. Thereby the particles will have increased particle-wall and particle-particle interaction as also described for the fluidization process. Shorter heating cycles and facilitated diffusion have high potential to increase the oxygen yield and reduce energy and reactant consumption.

One must consider that the rotation has to be limited within a maximum angle of 180 degrees to avoid wire and tube wrapping.

SYSTEM ELEMENTS DESCRIPTION

Since the entire system follows a modular approach there is no need for a complete set-up before starting the first experiments. The description focuses on the main part of the system: the reaction chamber, its design, tasks and applicability.

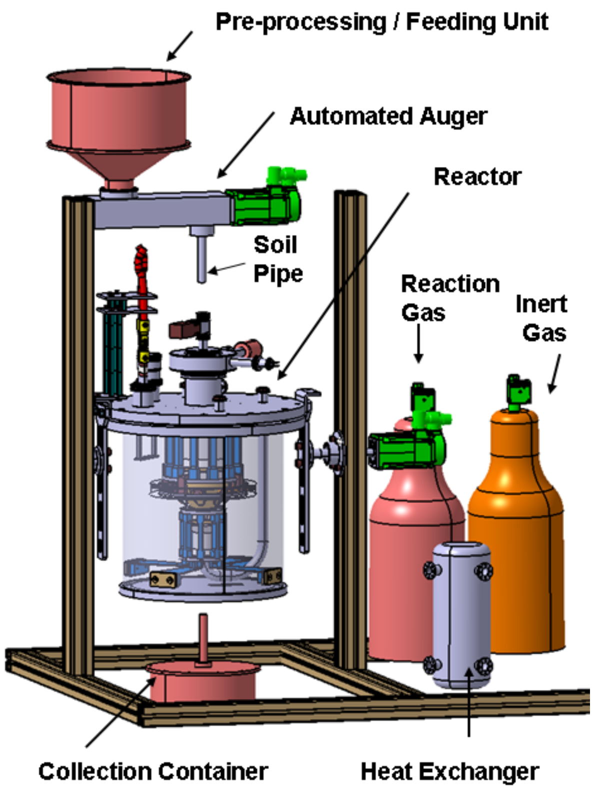

Fig. 5: VELOX system overview

The final set-up of the system, as shown in Figure 5, will consist of the main furnace chamber, a pre-processing and a post-processing unit, gas supply equipment a frame construction and several monitoring, regulation and measurement devices.

Reaction Chamber

The main VELOX system element is the reaction chamber with an inner set-up of parts for the reduction process and an outer casing which acts as a hermetic security chamber.

A three-dimensional design drawing, presented by Figure 6, shall highlight the parts which contribute to the reaction chamber.

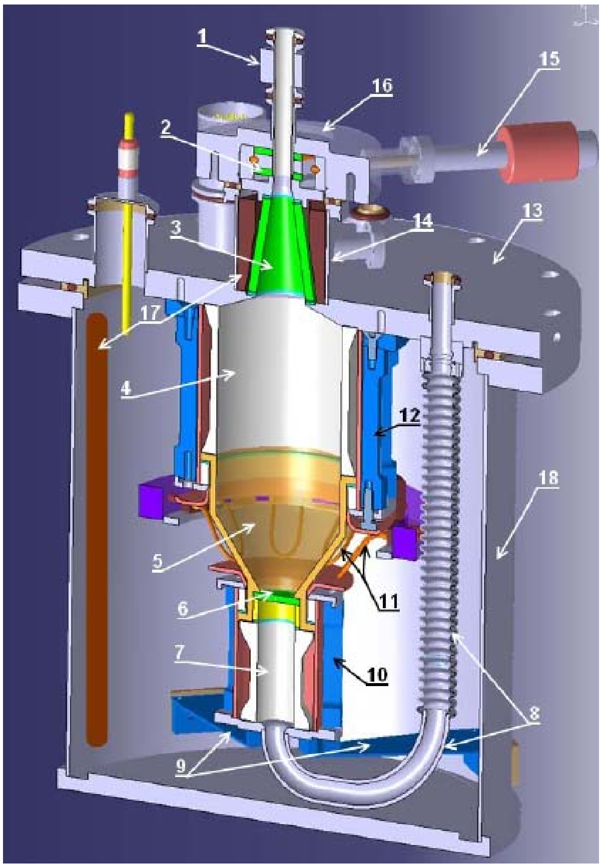

Fig. 6: Cross-sectional view of the reaction chamber with numbered parts

In the following all numbered parts are listed and briefly described with respect to their function:

1. *Gate Valve* – gas tight sealing for regolith inlet/outlet
2. *Slide valve* – prevents for regolith and heat radiation getting in contact with gate valve (1.)
3. *Filter cone* – prevents regolith entering the conduit
4. *Upper ceramic tube* – thermal buffer to reduce heat dissipation from process chamber (5.) to bade plate (13.)
5. *Inner process chamber* – regolith container and heater
6. *Filter plate* – gas permeable ceramic plate
7. *Lower ceramic tube* – thermal buffer to reduce heat dissipation from process chamber (5.) to U- and bellow tube (8.) and heat connectors (9.)
8. *U- and bellows tube* – reaction gas guidance and compensation of thermal elongation
9. *Heat connectors* – transmission of heat flow from the lower tensile rods (10.) to the outer casing (18.)
10. *Lower tensile rods* – support the sealing interfaces of the lower ceramic tube (7.)
11. *Heater elements (coils)* – transmit heat energy to the process chamber (5.) by radiation
12. *Upper tensile rods* – support the sealing interfaces of the upper ceramic tube (4.)
13. *Base plate* – mounting suspension of reaction core module and interface for in-/outlets and feedthroughs
14. *Housing neck* – interface to reaction gas outlet
15. *Linear actuator* – movement of slide valve
16. *Top flange* – housing for slide valve (2.), interface for linear actuator (15.) Regolith in-/outlets and inner thermal probes
17. *Clearance insulation* – radiation protection for outer elements (just indicated in Figure 6)
18. *Outer casing / Security chamber* – hermetic insulation of reaction core module

The heat resistant wall of the cone-shaped furnace chamber, selected for optimal thermal reliability at required operating temperatures and variable internal pressures, is surrounded by eight electrical heating elements which radiate the outer conical surface area of the chamber.

To prevent gas leakage and heat transfer from or to the reactor a supplement metallic outer casing is closing the internal part of the furnace hermetically. All interfaces leading into and coming from the inside of the chamber are required to support gas-tight integration of external located devices.

Since the hydrogen shall pass through the reactor a separate (bend pipe) gas inlet and a gas outlet were integrated to the assembly. This is the main internal loop through the reaction chamber and it is responsible for the Hydrogen reduction process of the filled regolith sample.

During the reduction, the space between the reactor and the outer casing will be filled with inert gas at the same pressure as the reaction chamber. This will be reached by passing through the outer casing by means of the secondary inert gas loop.

The whole construction is closed by a hermetically sealing mechanical locking device for Regolith refilling and removal after each reduction run. One part of the mechanisms ensures protection of the sealing device against regolith grains, whereas the other part is responsible for gas tight sealing of this unit.

The ceramic parts (white) are made of Zirconium oxide which provides a very small thermal conductivity. The inner process chamber (light orange) material is Inconel® Alloy 625 whereas the outer casing and the base plate is made out of X5CrNi18-10 stainless steel.

The filter elements (green) which are self-made gas-permeable ceramics ensure the prevention of regolith entering the gas supply and extraction sections. Most of the other elements are made of stainless steel to be able to deal with the vacuum environment during the air extraction phase and to ensure sufficient tightness.

In Figure 7 the complete inner set-up of the reaction chamber without the security chamber is shown.

Additionally, Figure 8 enables a closer look to the inner set-up components:

Fig. 7: Reaction core module attached to the base plate which is mounted within the reaction chamber frame and connected to a gas tube for the inner loop

Fig. 8: Inner set-up with process chamber and including heat coils, flexible power connections, ceramic tubes as well as the upper arrangement of tensile rods (reverse view)

In order to reduce complexity, the interfaces from the environment to the inner and outer sections of the reaction chamber are placed only on the top side of the module.

They are classified as feed-throughs, which are fixed mounted to monitor and control the process, and inlets/outlets which represent the ways into and out of the system for solid and gaseous reactants. Figure 9 gives an overview of the interfaces.

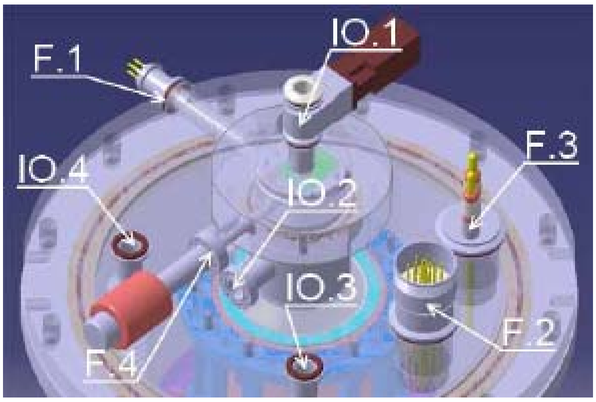

Fig. 9: Design drawing and numbering of in-/outlets and feed-throughs

Feed-throughs (F)

- F.1: Thermal sensors
- F.2: Thermocouples
- F.3: Electrical power supply
- F.4: Linear actuator flange

Inlets and outlets (IO):

- IO.1: Regolith
- IO.2: Reacted gas outlet (inner/main loop)
- IO.3: Inert gas inlet for the outer casing
- IO.4: Reaction gas inlet (inner/main loop)

Pre-processing Unit

The cylindrical structure of the pre-processing unit – which is not fully designed yet - will be made from aluminium alloy. The mineral dosing device for variable mineral feeding is given by a stepping motor, a rotation controllable conveying screw and a small Regolith container for an average Regolith transport capacity of approximately $5dm^3$. The mineral dosing device will be powered by an electric motor.

Post-processing Unit

According to the described architecture above the post-processing unit consists of a heat resistant rectangular container for processed minerals, a feeding and cooling department that transfers gaseous water vapour from the furnace chamber to a heat exchanger, and an embedded glass reservoir to store the condensed water.

The cooling process occurs batch-wise, so that no additional gas circulation is required. This unit is also still in the design phase.

Periphery

Additional equipment surrounding the main elements (pre-, reaction and post-processing unit) are among others:

- Pressurized gas bottles which contain the reaction as well as the inert gas,
- a pressure reduction valve set-up attached at the gas bottle(s) for generating atmospheric pressure ranges,
- vacuum pumps (i.e. rotary vane and turbo molecular pumps depending on the desired vacuum quality) for the evacuation process,

- a collection container for processed minerals,
- Swagelok piping systems and
- measurement devices for e.g. temperature, pressure and chemical analyses.

In Figure 5 a basic system overview shows where most of these components are located.

REACTANTS

Due to the characteristics of the selected process and environmental conditions on Earth solid and gaseous reactants are required.

Feedstock

According to the objectives of this project, several kinds of samples shall be used as feedstock of the reaction process. In order to compare the influence of mineralogical and chemical compositions different simulants like JSC-1A or FJS-1 and minerals like pure Ilmenite ($FeTiO_3$), Iron oxides or basalts will be filled in the process chamber for particular experiment campaigns.

In order to increase the accuracy and comparability of the reaction process considerably the feedstock, in particular the soil simulant has to be adapted to lunar conditions as much as possible. On the one hand there are volatiles implemented in the Moon regolith which do not appear on Earth. Fortunately this inconsistency is not expected to endanger the experiment result since the amount of oxygen is not really influenced. On the other hand soil simulants on Earth contain Iron oxides with higher oxidation states which do not appear on the Moon (i.e. Fe_2O_3), which can be seen in Table 2. Only FeO as part of the Ilmenite is supposed to be reduced with the Hydrogen reduction process. Unfortunately former experiments like [3], [4] indicated that the major reduced mineral is Fe_2O_3.

Mineral Component	Lunar Mare Regolith [wt-%]	JSC-1A Lunar Simulant [wt-%]
SiO_2	47.3	47.6
Al_2O_3	17.8	15.0
CaO	11.4	10.4
MgO	9.6	9
FeO	10.5	7.4
Fe_2O_3	-	3.4
Na_2O	0.7	2.7
TiO_2	1.6	1.6
K_2O	0.6	0.8
Others	0.5	2

Table 2: Mineralogical composition and comparison of a lunar sample [11] and a widely used simulant [12]

Since removing the Hematite (Fe_2O_3) components mechanically is not feasible, the minerals have to be reduced in advance. This would lead to a more representable feedstock. The process which also can realized by the VELOX reaction chamber is the blast furnace process using Carbon monoxide to extract oxygen out of the Iron oxides in several steps depending on the temperature. Two steps and temperatures up to 1000°C, where Carbon monoxide emerges up to 90% out of the initial reactant Carbon dioxide, are required to fulfil the chemical kinetics of following equations [13]:

$$3Fe_2O_3 + CO \rightarrow 2Fe_3O_4 + CO_2 \quad (4)$$

$$Fe_3O_4 + CO \rightarrow 3FeO + CO_2 \quad (5)$$

Further reduction to pure Iron, which is the actual purpose of the blast furnace process, is not desired.

As a first step for the here discussed experiments it is planned to perform this beneficiation process at external institutions who have access to bigger plants and more experience on that topic. Nevertheless, it is aimed to combine this preparation sequences to the Ilmenite reduction process later on within the VELOX system. This also would minimize potential re-oxidation of the feedstock between the blast furnace process and the Ilmenite reduction.

Reaction Gas

The appropriate gas for Ilmenite reduction is Hydrogen (H_2). On the one hand Hydrogen has a very high reactivity, which is its main advantage for the reduction process. On the other hand it is difficult to handle. The utilization of pure hydrogen would lead to a significant increase of safety requirements both for the experiment proceedings and the surrounding infrastructure. Thus, for the first experiments a forming gas with a mixture ratio of 5% Hydrogen and 95% Nitrogen (N_3) will be used. During later phases, after safety verification of the system design and continuous laboratory improvements, the amount of Hydrogen within the reaction gas will be increased in several steps and safety installations will be added subsequently.

Inert gas

Earth's Oxygen-containing atmosphere is a disturbing factor for the Ilmenite reduction process. In order to ensure the absence of additional gaseous oxygen within the reduction process the air has to be removed from the facility. This is done by vacuum pumps. Furthermore, the inner loop will be floated by Nitrogen as part of the forming gas and can be floated with pure gaseous nitrogen prior to the reduction process in order to regulate the pressure level and additional air removal. The outer casing, i.e. the safety cover, will also be evacuated by vacuum pumps and filled with inert gas afterwards for Hydrogen leakage prevention and air introduction into the inner set-up. In general, besides Nitrogen, also Argon or Helium could be used for the creation of the outer oxygen absent environment, whereas Helium is not favorable due to its volatility.

VELOX PROCESS CYCLE CHARACTERISTICS

The goal for the VELOX demonstration facility is to set up a fully autonomous reaction process, which includes feeding of the reactants and removal of the products. One complete sequence requires following steps, as shown in Figure 10 and are listed below:

i. Phase 1: Filling
ii. Phase 2: Reduction preparation
iii. Phase 3: Reduction process
iv. Phase 4: Discharge preparation
v. Phase 5: Discharge

Since the reaction occurs batch-wise the process cycle has to be repeated continuously for long-term operations.

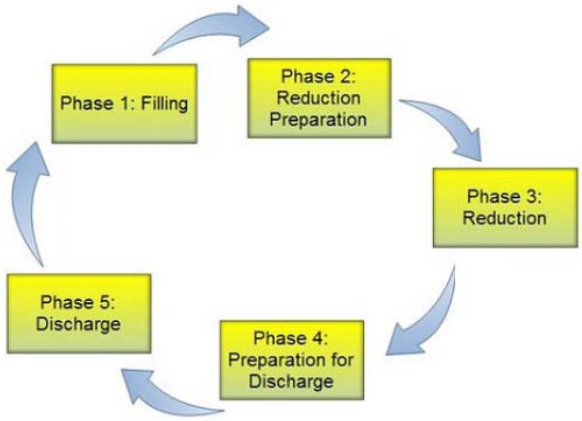

Fig. 10: VELOX process cycle with presenting the main steps

As a first step (i.) the process chamber has to be filled with the feedstock. Therefore the reactor has to be in upright position and the samples will be filled into the inner process chamber by a funnel and filler neck moving downwards from the pre-processing unit through the reactor locking device. This ensures a direct injection of the soil and prevents the sealings from contamination which would be negative for further vacuum generation.
When the chamber is filled properly the pre-processing unit closes and the filler neck will be pulled back.

During the reduction preparation (ii.) pre-heating cycles will remove undesired volatiles out of the soil simulant. For future applications this will also be the blast furnace process phase for feedstock beneficiation. Furthermore the remaining air will be extracted out of the reactor for both, the inner and outer part, using vacuum pumps. Flushing the inner loop as well as the security chamber with inert gas will also be part of the preparation. Detailed sequences will be defined during the first experiments.

The main procedure is the reduction (iii.). The feedstock will be heated up to the selected temperature and the forming gas will enter the inner process chamber. Additionally the entire reactor starts to rotate within a certain angle which is an option for efficiency increase as well as the variation of the gas volume flow in order to realize fluidization of the solid particles. The reduction of the Ilmenite will occur and the water vapor as result of the Hydrogen-Oxygen bond will be pumped out of the gas outlet at the locking device on top of the reactor.

For the preparation of discharge (iv.), the energy supply to the heating elements and the gas supply has to be shut down. The movement of the reaction over its rotation axis also has to be stopped. The remaining gas has to be removed out of the process chamber for further processing, i.e. analyses, cooling and/or removal.

Finally, for discharging the solid reactant (v.), the reactor has to be turned down slowly until it is in upside-down position. The reactor locking device opens and the slag will be removed by the force of gravity supported by shaking of the reactor if necessary. Therefore a similar neck like attached to the pre-processing unit will ensure proper slag depositioning within the collection container. Afterwards the locking device closes again and the reactor moves back to its initial position

In the beginning this entire procedure will be done manually but later an autonomous acting set-up will perform the five described process steps.

MEASUREMENTS AND INSTRUMENTATION

For both, reaction chamber testing and the experiment campaigns, it is mandatory to collect data and analyze the conditions of the reagents, products and the process itself. Consequently, several instruments for real-time measurements as well as equipment for pre- and post-processing are defined.

Housekeeping

Monitoring the process parameters is realized by standard temperature and pressure sensors. In order to achieve knowledge about the thermal distribution, ten flexible thermocouples (TC) are placed at different parts within the outer casing. The TC are attached to the inner set-up, at both sides of a tensile rod, close to the heat connecters, outside of the process chamber and close to the sealings of the ceramic tubes.

The regolith temperature itself will be measured by a pair of redundant Inconel® coated rigid thermocouples which are introduced through the upper filter cone and connected within the feed-through F.1 (see Figure 9). For more complex processes like fluidization there is a need for volume flow measurements. The respective instruments will also be attached close to the gas inlet and outlet.

Since there are two main sections (inner reduction section; outer security chamber) within the system, two different gas types and pressures will occur. The gas pressure of the inner loop is measured outside of the reactor, close to the gas in-/outlet using Swagelok® technology to attach the devices.

Measurement devices of the security chamber pressure level can be attached either analogue to the inner section or directly to one of the two in-/outlets if no gas circulation is foreseen.

Gas and Volatiles Measurements

Since there will be no water production during the early experiment campaigns, which would indicate the success of the reduction process, a mass spectrometer for water vapor analyses has to be attached to the gas outlet (IO.2 within Figure 9). This instrument shall determine the elemental composition of the acquired water vapour and detect the amount of Oxygen which should be extracted out of the soil.

In the future micro electro-mechanical systems (MEMS) technology shall be used in order to reduce mass, which is highly important for lunar applicability, and integration complexity of the measurement devices.

During Phase 5 of the project (volatile measurements of several soil simulants, see Table 1), the gas analysis unit could be replaced or extended by specific volatile detectors.

Mineral Analyses

Basically, three main instruments will be used for mineralogical analyses before and after the reaction, using:

- X-ray fluorescence (XRF),
- X-ray diffraction (XRD) and
- Scanning-electron microscopy (SEM) technologies

The elemental/chemical composition is easily to be defined by XRF measurements, in particular by energy dispersive X-ray spectroscopy (EDS), whereas XRD provides information about the crystalline structure of single mineral phases. Additionally, SEM will visually help to understand the changes occurred during the reaction process and with respect to the phases within the solid reactant.

Determining the oxidation status of the feedstock iron component can be done by Mossbauer spectrometry. Especially on Earth, where different iron phases occur, this is a valuable method for the process verification and interpretation of the Ilmenite reduction results.

For lunar activities these measurements will not be fully applicable since they require bulky instruments and additional electrical energy. The regolith of the location has to be investigated in advance externally (e.g. by rover, humans and their analyses devices). The measurements during the Ilmenite reduction on Earth rather serve as feasibility demonstration than process monitoring. However, using MEMS technology it shall be possible to achieve sufficient data to continuously adjust the process on the Moon as well.

SET-UP TEST RESULTS

During the project Phase 1 which deals with the preparation activities the components of the reaction chamber and the overall system have to be tested with respect to their thermal behaviour and tightness.

The thermal energy supply and distribution is the main driver for the conditions of any material within the system. It has to be analyzed to what extent the thermal energy provided by the heating elements enters the inner process chamber and how the thermal flow evolves during the heating cycles. The first test results are outlined in the following subsection.

Since several gas types are part of the process the tightness of the inner set-up/loop and the outer casing is very important. Therefore vacuum tests also have been performed.

Thermal Testing

Before the heating elements will be fully integrated in the set-up the general heating behaviour has to be analysed. The arrangement of heat elements which is customized for this project are connected by flexible element-to-element bridges and receive the electrical energy by two copper cables mounted at the related feed-through (i.e. F.3 in Figure 9).

One issue is that the resistance of the heating elements is very low and in order to provide about 2 kW of power a very high current of about 200 A would be required. Therefore it is mandatory to have a current-regulated power supply in order to avoid short circuits during the first heating sequences. When temperature increases, the resistance also increases.

This reduces the required current and relaxes the conditions for further heating cycles and temperature increase. During the first test phases it was possible to heat up the system within a few minutes. Figure 11 shows a picture of the glowing heat coils:

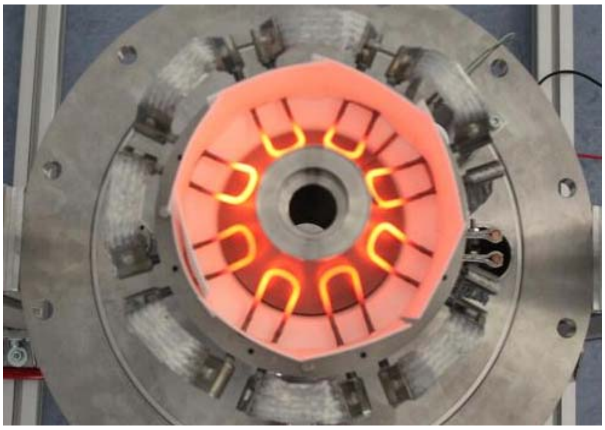

Fig. 11: Heating elements in action (bottom view)

Verifying the heat transfer FEM-Model of the inner set-up, depicted in Figure 12, will be one of the last tasks during the system preparation phase. Therefore a hand-held infrared camera (for tests without the outer casing) as well as the flexible thermocouples will be used.

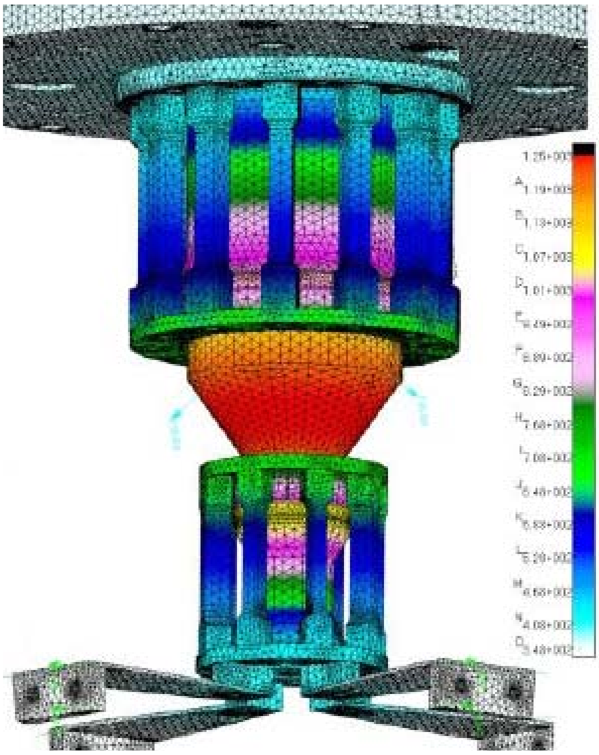

Fig. 12: Simulated thermal distribution using a Finite Element Method (FEM) model

Vacuum Tests

Vacuum generation will be performed prior to the reduction process to reduce additional failure potentials caused by the presence of air. A reasonable vacuum quality and optimum case for the here discussed activities would be fine vacuum for the outer casing and high vacuum for the inner loop. Higher vacuum would lead to a significant increase of design complexity with additional mass and cost impact.

Fig. 13: Test set-up for vacuum generation with a rotary vane pump and two different pressure measurement devices

The preparation tests started with an empty security chamber, followed by the vacuum generation within the inner loop and finally as a combined set-up. For each test one interface serves as a connection to the vacuum pump and at least on as a port for the pressure sensor. Any other interfaces were locked by blank flanges. Figure 13 shows a set-up of the initial tests for the security chamber.

According to the first tests Figure 14 shows the pressure trend which came close to a fine vacuum 2.0 * 10^{-2} hPa after a short period of about 6 minutes within the security chamber. This concludes that the outer casing and its connection to the base plates is ready to handle the low pressure demands for the process preparation.

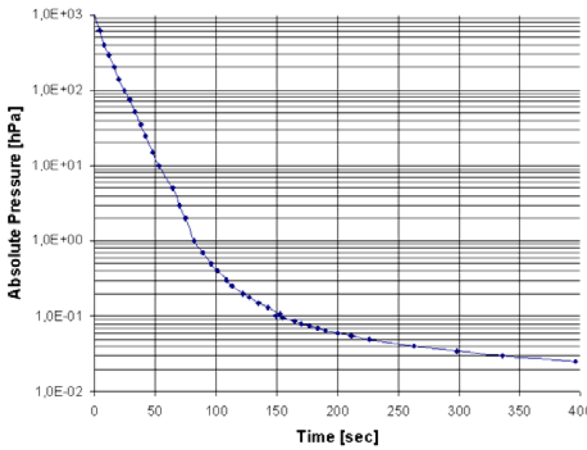

Fig. 14: Trend of pressure reduction caused by air evacuation out of the security chamber

EXPERIMENT CAMPAIGNS

As already indicated during the project phase section within this paper, several experiment campaigns are planned in order to compare the results with respect to certain parameter changes. During the first demonstration campaign (Phase 3) lunar soil simulant will be used in order to compare the work with international precursor activities. For the efficiency analyses campaigns following conditions and parameters will be varied, either individually or in combination:

- Reduction time
- Reduction temperature
- Reduction pressure
- Hydrogen volume flow
- Hydrogen percentage of the forming gas
- Feedstock type
- Amount of feedstock

The parameters time, temperature and pressure will be the first subjects of variation in order to get a feeling of the set-up behaviour and the respective extraction results. The feedstock types like simulants and minerals will differ in chemical compositions as well as in grain sizes.

CONCLUSIONS AND OUTLOOK

Complementary activities of the science community and preliminary test results of VELOX with respect to thermal behavior and vacuum indicate, that the current set-up is prepared to start with the final testing sequences. Primarily, these are (pre-)heating sequences of the feedstock and gas flow analyses, followed by the experiments.

The DLR demonstration facility provides a broad range of applications: the Oxygen production process as well as Iron oxide reduction and volatile measurement experiments for a variety of soil simulants.

The beneficiation process during the blast furnace process for internal activities or with externally prepared feedstock is a great opportunity to come closer to the results with lunar Oxygen production rates.

Fluidization and the rotating reaction chamber both have high potential to increase the efficiency of the current reduction process as already been demonstrated in precursor projects of the science community.

The paper described the main goals and requirements for the VELOX demonstration facility. It discussed the evolution and results of certain system design decisions and briefly described the functionality and tasks of the major components as well as the reactants and supporting devices.

Latest test results and planned experiments gave an insight of the current DLR activities related to lunar Oxygen production.

Constantly considering the process to be applied on the Moon, a lot of effort has been made to adapt the system for such application while still dealing with the different and partly contrary characteristics of the terrestrial environment.

During the next project phase the system will be filled with solid reactants and several thermal couples will measure the behaviors in order to adjust the electrical energy supply units for the reduction process.

In parallel, the post-processing unit will continue its design phase in order to produce water out of the produced gas mixture. This will be the most promising way of demonstration a successful reduction process.

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