Experimental setup for the demonstration of thermal processing of lunar regolith for the extraction of solar wind implanted particles via a solar powered cavity receiver

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One of the enabling technologies in long-term perspective for human space exploration is in-situ resource utilization (ISRU). When dealing with the prospect of future manned missions to Moon and Mars the use of ISRU seems useful and intended. The activities presented in this paper focus on lunar ISRU. This basically incorporates both the exploitation of lunar oxygen from indigenious rock and the extraction of solar wind implanted particles (SWIP) from regolith dust. Currently the the Institute of Astronautics (LRT) is examining possibilities for the extraction of SWIPs, which may provide several gaseous components (such as H_2 and N_2) valuable to a human presence on the Moon. This experiment, LUISE (LUnar ISru Experiment), will comprise a solar powered thermal process chamber for regolith heating. The proposed thermal extraction chamber uses an insulated double wall cavity receiver for heating, with regolith being filled into the gap between the two walls. The goal is to assess the thermal behavior of regolith within the process chamber. An experimental setup with two cylinders stacked into each other has been developed and built. Heating is accomplished by a molybdenum heater located within the inner cylinder. The overall setup is exposed to high vacuum during the complete heating run. With this simplified geometry we will heat several hundred grams of JSC-1A, a widely used lunar regolith simulant, to a temperature of 1000°C. The data gained during the testing is used to determine the influence of regolith layer thickness, thermal connection between the inner and outer wall of the cavity on the heating duration until steady state, power demand and overall heating homogeneity within the regolith.

Introduction

Lunar ISRU is a field of research that has the goal of investigating future solutions, which allow mankind a sustainable presence on the Moon. Ideas for in-situ resource utilization range from the use of bulk regolith as a radiation shield to the production of mission critical consumables like oxygen, water or propellants. In pursuing these efforts the lunar regolith, which is the finely grained layer of dust-like material that covers the lunar surface, acts as the common source. This also demarcates a central difference between lunar and martian ISRU due to the lack of a lunar atmosphere, whereas Martian efforts strongly rely on the utilization of atmospheric components (like CO_2). When it comes to lunar consumables production, there are two principal directions namely production of lunar oxygen and extraction of solar wind implanted particles (SWIP) from the regolith. The field of lunar oxygen production currently benefits from a very active research being conducted over the last 5 year [1, 2]. Our research focuses on the much smaller and less well developed field of SWIP extraction. This field promises the recovery of a wider range of elements from the lunar regolith such as hydrogen, carbon, nitrogen or even water, which has arisen to become a promising resource in the last years [3]. It is supposed that extraction of those resources could be achieved by thermal desorption [4].

Due to the extremely thin lunar atmosphere the surface covered in regolith is directly exposed to the solar wind. As a result a constant stream of low keV/amu ions from the sun impinges on the lunar surface. This energy is sufficiently high that solar wind ions penetrate the finely grained regolith surfaces to a depth of several 1000 Å [5, 6], where they come to a rest in the mineral lattice. Heating the particles causes the SWIP to move towards the particle surface and subsequently to be released there.

The motivation of the lunar resource experiment (LUISE) project is the demonstration of in-situ extraction of SWIP from lunar regolith. In order to address the possibilities for utilizing SWIP and water in future lunar exploration, a spacecraft design study has been conducted at the Institute of Astronautics at TU München, which resulted in a concept for a solar thermally powered experiment for demonstration of SWIP extraction.

LUISE as a demonstrator for a thermal oven concept

The vision behind LUISE was the investigation of extracting SWIP from a sample of several hundred grams of regolith by applying solar energy directly to an oven, without utilizing any assisting fluids or reactants. The process foresees to heat the regolith sample to a temperature between 900°C-1000°C and wait for the SWIP to be removed from the regolith grains. Subsequently, the released gas will be analyzed in order to assess which gaseous species evolved and in which quantity.

The scope of the ongoing research at the LRT focuses on the evaluation of the thermal performance of the solar-concentrator/oven system. The major objective of this solar-concentrator/oven system is to achieve a low thermal gradient within the regolith while heating the sample to the desired temperature band. Thermal analyses on several oven concepts for solar thermal irradiation lead to a cavity receiver design. Among the initially conceived concepts were solutions which utilized different column-occupied pots, a heat exchanger fluid loop heated by a parabolic trough and a double-walled cavity receiver. Simulations performed on both, the column-occupied pot and the fluid loop concepts, showed insufficient thermal performance with regard to the requirements. The fluid-loop heat-exchanger concept suffered from a massive loss of enthalpy while the fluid (Helium) was being transported into the oven-heat exchanger, even with strong insulation applied to the piping. The column-occupied pot showed strong thermal gradients within the regolith filling as well as high re-emission of the irradiated solar flux. A gold-coated dome-like cap offered improvements by reducing re-emission, but lead to great resemblance to a cavity receiver concept. Due to the increasing similarity of this concept to a cavity receiver, it was decided to lead further investigation on to a detailed analysis of that concept.

Figure 1 depicts the final concept derived from this study. It comprises a double-walled cavity receiver in a spherical shape (Figure 1- on the right). The regolith is resting in the gap between both cavity walls during processing. In order to achieve and maintain the required temperature, the cavity receiver is clad in a ceramic insulation material with low thermal conductivity. The inner sphere will be irradiated by solar power coming from the solar concentrator (see Fig 1, left). There are three independent openings in the cavity receiver. The first one is used for irradiation and the second one is used as an orifice for the regolith intake. The third opening serves as vent to lead the evolved gas to the analysis unit. The gas analysis method to be used is under investigation with GC/MS being one potential conservative option.

Performance and Concept evaluation

Parametric analyses for irradiation flux and insulation were calculated with ESATAN-TMS. The simulations were performed on a sphere cavity with 45 mm inner diameter. Both cavity walls thicknesses were set to 5 mm and regolith to 20 mm. In this configuration the regolith acts as insulation to the cavity receiver. The cavity was positioned into a temperature controlled hot box at 120°C. The cavity's 25 mm diameter opening is facing towards the sun for the irradiation flux. Ambient space was set to 3 K. Parameters for irradiation were varied between 100 W and 800 W and insulation thicknesses ranging from 5 mm to 50 mm. Insulations thermal conductivity was constant over temperature at 0,3 W/m^2*K. Gas evolution within the heating process has not been included as it would have an impact on the thermal conductivity of the regolith. The analyses were done with both a high conductivity regolith model [7] and a low conductivity model ([8]). Low conductivity in the regolith and minimum thermal conduction between the cavity walls yields high temperatures as well as very high gradients in the regolith layer. For a low conductivity regolith material model and no conductance between inner and outer wall, the cavity receiver insulation only has a small effect on maximum regolith temperature. The regolith exhibits high temperatures with high gradients. Use of a high thermal conductivity model shows an analogous behavior, with gradients being slightly lower. This reflects the problematic insulator-like behavior of regolith in vacuum. In order to compensate for this insulation character of the regolith a deliberate conductive connection between the cavity walls was added to the model. The analyses were repeated for a low conductivity regolith model with a conductive interface between inner and outer wall and the results are shown in Fig. 2. The conductive interface creates a short circuit to the regolith layer. Gradients within the regolith are reduced at the cost of heat flux bypassing the regolith. This shifts the insulating effect from the regolith and onto the actual insulation layer.

Results obtained from ESATAN-TMS simulation on a simple geometric model of a sphere-shaped cavity lead to three conclusions for such a system, which were considered in the following design phase for the LUISE laboratory experiment (see Fig. 3):

- Temperature and gradients within the regolith layer strongly depend on the applied layer thickness and the conductivity law applied to regolith
- Gradients will decrease with lower power irradiation and increased thermal insulation
- Gradients can be reduced by connecting the inner and outer wall of the cavity receiver to each other, and thus generating a thermal short circuit, at the cost of extra insulation.

The desired temperature band (900°C - 1000°C) appears rather small and sensitive to deviations in both irradiation flux and insulation properties. Additional factors of uncertainty are the regolith behavior over temperature and pressure, as we are trying to evolve gas from the material. Again, those have been neglected for initial worst case simulations, but we expect them to have a non-negligible positive influence on the system. From these results we anticipate that solar heating of regolith is a feasible concept, but robustness and system stability are challenging. For this reason we have decided to test the concept with an experimental setup, which allows us to verify our models and to determine the performance and sensitivity of the cavity receiver.

Impact of regolith conductivity on thermal performance

Additionally it became obvious, that the thermal properties of regolith will act as a major design driver for the thermal design of such an oven chamber. As measured in Apollo samples, the lunar regolith behaves as a super-insulator, with thermal conductivities as low as 15 mW/m^2*K at approx 25°C [9]. Some evaluation of the thermal conductivity over temperature has been done on lunar samples in vacuum and 1 bar atmosphere with varying temperature, limited to temperatures ranging from -170°C to 130°C [10]. In our experimental setup, temperatures far beyond these known values will be reached. In addition, there will be a constant change in interstitial pressure within the regolith layer due to the expected gas evolution. Therefore it is necessary to evaluate the thermal conductivity as a function of both temperature (20°C to 1000°C) and pressure (10e-4 mbar to 1 bar).

It is expected, that the thermal conductivity of lunar regolith over temperature and pressure will follow a set of equations proposed by [8]. A numerical simulation approach using this approach is currently investigated at the Lehrstuhl für Thermodynamik (LTD/TUM).

Experimental Setup

We have designed an experiment for verification of our simulations and for measurement of JSC-1A thermal conductivity in vacuum. It allows for the variation of the power input to the cavity (representing the "irradiated solar flux"), cavity insulation, conductance between cavity walls and regolith layer thickness (see Table 1). Together with the regolith thermal conductivity these features have a primary influence on the stated three conclusions for cavity receiver behavior. The components and a cut-through are shown in Figure 3. The environmental simulation is depicted in Figure 4 as a block diagram. The method to be used in the calculation of the thermal conductivity from this experiment as well as the results will be reported separately. Further aspects envisaged to be learned from this experiment are the required power demand for heating, as well as the transient behavior during heating and cooling. Moreover, this experimental setup provides several challenging sets of issues that are connected to ISRU technologies. Relevant issues are the handling of regolith, its adhesion to surfaces, clogging and poor flowability. Moreover there is a great uncertainty on the behavior of the material at elevated temperatures close to its melting point at approx. 1100°C. Maintaining such high temperatures in a high vacuum does also impose several technological challenges on material selection, thermo-mechanical stress and material creep.

The focus has been put on the following design objectives, which will help to assess the thermal behavior as a first step. Gas evolution and analysis are being neglected in the first step of this experiment. We are well aware, that the used JSC-1A does release water and CO_2 during heating [11]. The impact of interstitial gas pressure is currently being investigated at LTD. Our experiments will be performed on “vacuum pre-burned” JSC-1A, which does show a reduced gas evolution in vacuum (see Fig 5), due to the irreversibility of this process [11].

Table 1: Geometrical variation options for testing

Regolith layer thickness	Conductive I/F	Insulation thickness
5 mm	None (insulated)	40 mm

15 mm	Spacer ring on top	80 mm
	Spacer ring top/bottom	

Experiment design

The experiment will be tested in a relevant environment. This is achieved by testing inside a vacuum chamber (approx. 10e-5mbar) equipped with a water cooled shroud controlled at 10-25°C. The vacuum chamber itself is equipped with lead-throughs for water, electrical power and Type K thermocouples. Pressure inside the vacuum chamber is tracked by an Oerlikon pennivac PTR 90. Cooling water flow into the cold shroud is measured. Figure 4 shows the complete installation of the environmental simulation.

The spherical cavity receiver is represented by cylinders. There are two outer cylinders and one inner cylinder for testing. The inner cylinder is stacked into one of the outer cylinders, which allows a 5 and 15 mm regolith layer. The outer cylinders are equipped with pressure venting holes; the inner cylinder is closed against the regolith but open against the surrounding vacuum. The option for various conductive interfaces between cylinder walls is achieved by adding spacer rings made of insulation material or titanium. Boron nitrate must be applied to each metal/metal contact surface to prevent diffusion welding. The choice for titanium has been made against ceramics (e.g. alumina) due to cost reasons. Titanium's getter behavior at high temperatures will not allow for a later amendment of gas analysis to this experiment. Insulation is established by Rath KVR 502/242 high purity Al_2O_3 ceramics. It will be stacked as intersecting ring segments which allow either 40 mm or 80 mm insulation to the cylinders. Heating of the cavity is done by a custom 50VDC / 20A HTM-Reetz molybdenum electro-resisting heater, inserted into the center of the inner cylinder. The option of irradiating the power into the cylinder cavity has been investigated, but had to be discarded, due to the desire for thermal conductivity measurement. This measurement demands for a maximum closure of the insulation to prevent heat losses, which would not have been possible by irradiating into an open cavity. Temperature measurement is achieved with thermocouples (type K, Omega UK) sunk into grooves oriented parallel to the isothermal lines. The thermocouples are distributed along the circumference and in different heights in order to assess the temperature distribution. Signals for temperatures, power output to heater and cooling water flow rates are processed using a NI cRIO connected to Labview.

Preliminary Results

Currently the titanium elements of the experiment are in production, as well as the molybdenum heater with the ceramic insulation. Full testing is to be expected to yield results by the mid of 2011. The environmental simulation (vacuum chamber, process cooling and the cold-shroud) have been finished and successfully tested for the designed functionality, as well as data acquisition and sensor application. Initial testing of the behavior of JSC-1A under the expected environmental conditions during oven operation (high-vacuum and high temperatures) has been performed in both a vacuum tube oven and inside of the vacuum chamber. Subjecting JSC-1A to a high-vacuum and temperatures above 600°C causes an irreversible material change. Fig. 5 shows the pressure evolution for a given heater pre-setting with a maximum temperature of 700°C. The diagram depicts the original material behavior as a continuous line. There are three distinct pressure peaks. The first can be explained by the evaporation of residual water in the low temperature range. Both peaks at elevated temperatures can also be reproduced by a linear heating ramp without dwelling phases. As reported by [11] [12] the gas release experienced in the elevated peaks can be attributed to the release of CO_2 and stronger bound water. The gas pressure peak in the 700°C dwell phase coincides with the color change of JSC-1A under vacuum treatment. This appears to be the temperature range, which causes the CO_2 release by mineral decomposition. Repeating the heating cycle on a vacuum pre-heated sample, without prior exposure to air will yield a pressure release as depicted in Fig. 5 by the dashed line. This confirms the irreversibility of the vacuum heat treatment. Only very little pressure changes can be observed, with the exception of a short pressure increase during the 400°C dwell phase. Storage of vacuum pre-fired samples in a high purity argon atmosphere will allow the stimulant to resorb small amounts of gaseous material. The dramatic pressure increase in the 700°C dwell phase is only found in the untreated sample. Further increase of temperature above 700°C will additionally cause sintering of the material, which causes a thin brittle crust covering the loose material. Exposing the material to temperatures above 1100°C will yield a brittle brick-like material.

Testing of Ti Gr5 (two separate bars, pre-loaded by titanium screws) under test conditions in a Carbolite tube-vacuum oven (1000°C, 5 h curing, 10e-4 mbar) has shown the problem of diffusion welding. For this reason boron nitrate will have to be applied to each metallic contact within the oven cylinders.

Initial testing done with stacked alumina crucibles with 500 g of JSC-1A filled into the gap and a self made electro-resistive Kanthal heater with 230 V AC power has been conducted in the vacuum chamber. Figure 6 (left and middle) shows the crucibles equipped with the Kanthal heater in the cold shroud before and after heating. During heating a spontaneous pressure increase caused a massive ejection (approx. 80g) of JSC-1A to both the inside and outside of the unsealed crucibles. This ejected JSC-1A was found covering almost every surface within the vacuum chamber. Material thrown into the inner crucible caused a local overheating and melting of JSC-1A on the heater. This eventually lead to arc discharging and ultimately to a loss of the heater. (see Fig. 6, right). This experience will be used to improve the testing with the experiment hardware.

Summary

The idea behind LUISE is based on the desire for demonstrating the extraction of SWIP in-situ by means of solar energy. A concept for a thermal extraction oven has been developed and its behavior has been simulated to derive a set of critical parameters. Some of these parameters can be governed directly, e.g. insulation or internal conductance. Others are depending on widely unknown values, such as regolith thermal properties. The overall concept appears feasible but with some challenges concerning robustness and stability of the thermal design. We have derived an experimental setup with environmental simulation for model verification of our cavity receiver concept as well as for the measurement of regolith thermal conductivity. Initial testing of our environmental simulation has been successful. Preliminary experiments on heating JSC-1A show a challenging material behavior, such as dust covering, mass ejection or even damaging of critical elements. Full experiments with the described experimental hardware will have to be prepared against such failures. Results of the described experiment will be published after finishing the test campaign.

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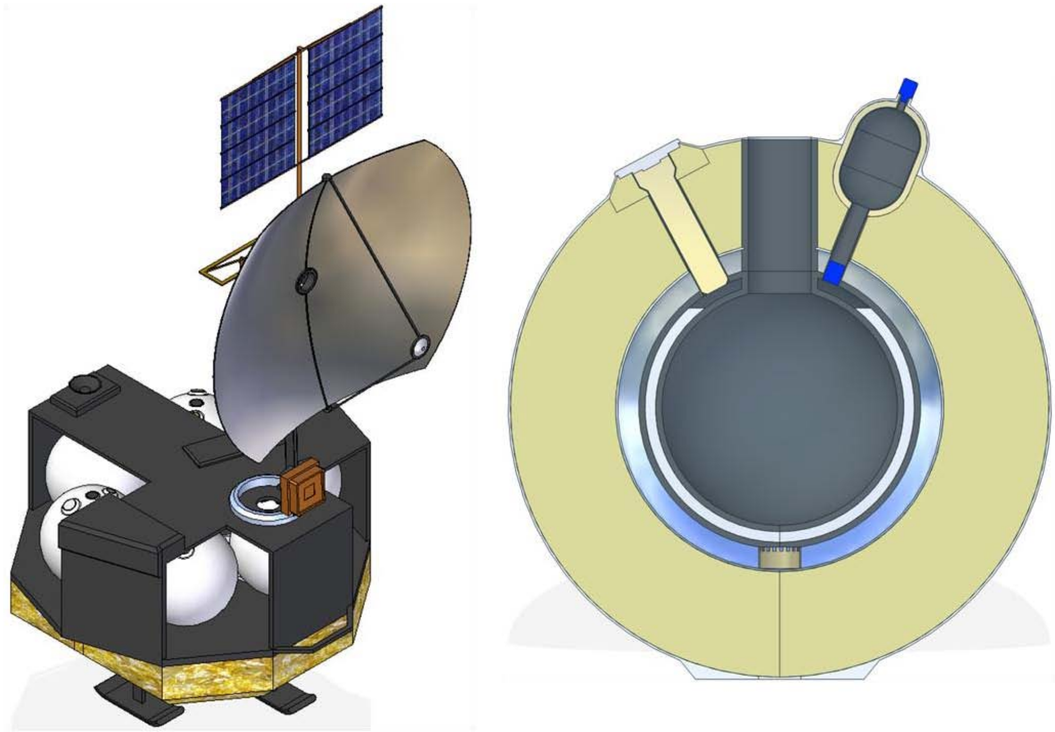

Fig. 1: left: Concept for the solar-thermal in-situ extraction of SWIP from lunar regolith; right: Cut through cavity receiver concept study, depicting double walled structure, with regolith filling, insulation and released gas venting.

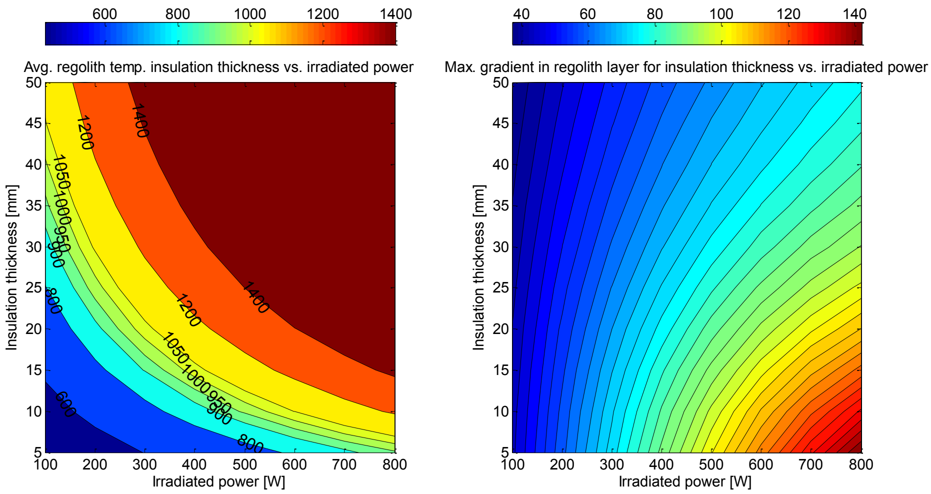

Figure 2: Temperatures and thermal gradient for low regolith thermal conductivity model, good conductive connection between cavity walls

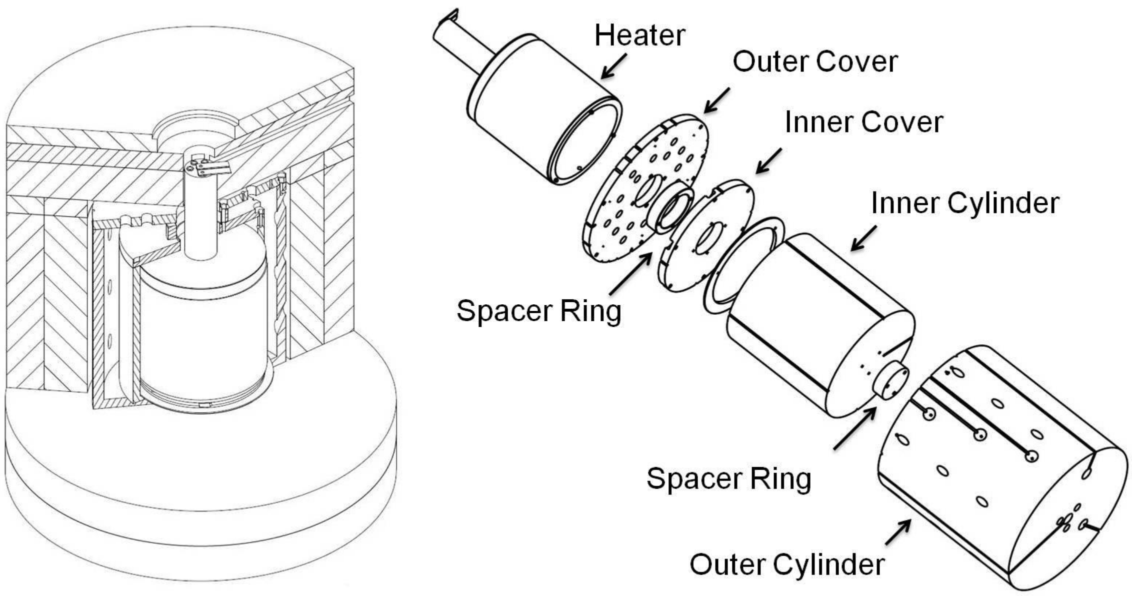

Figure 3: left: Cut through experiment, depicting molybdenum heater, inner and outer titanium cylinder and insulation; right: Explosion of stacked cylinders showing heater, covers, spacer rings and cylinders (insulation not displayed)

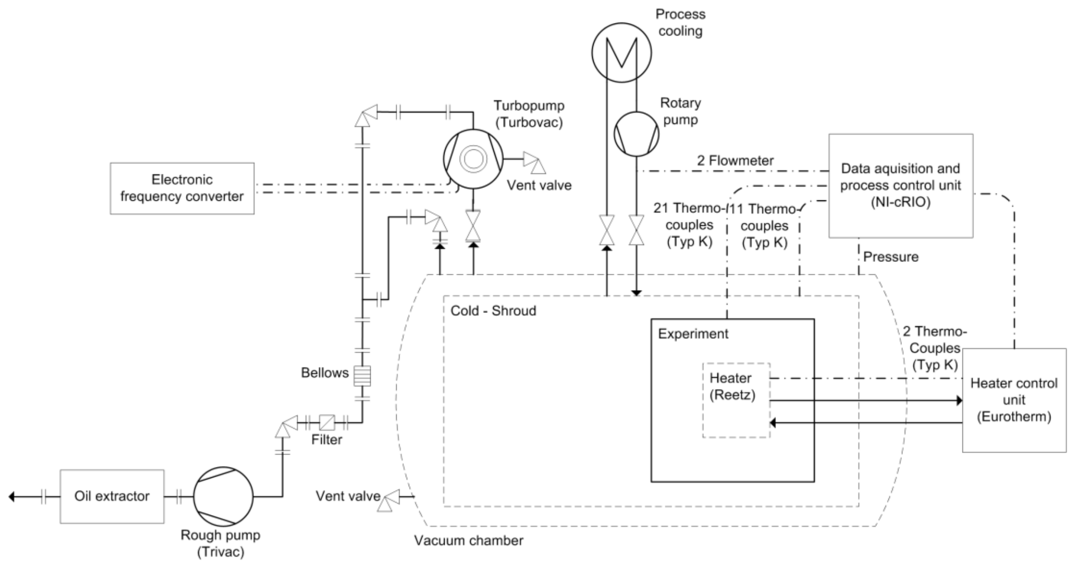

Figure 4: Environment simulation for the experiment with data aquisition

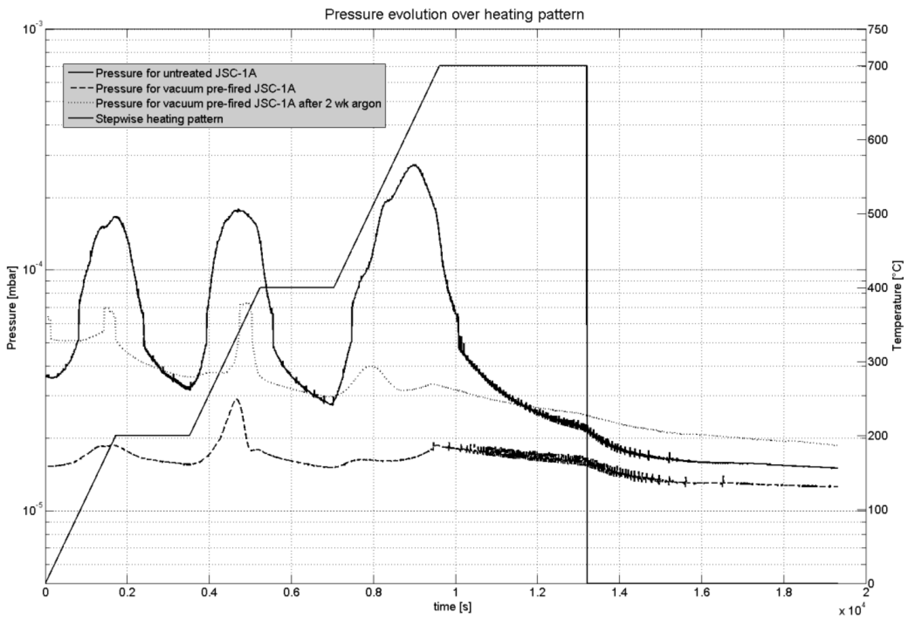

Figure 5: Pressure evolution for stepwise heating of untreated "fresh" JSC-1A, vacuum pre-heated JSC-1A and vacuum pre-heated JSC-1A after 2 weeks pure argon exposure

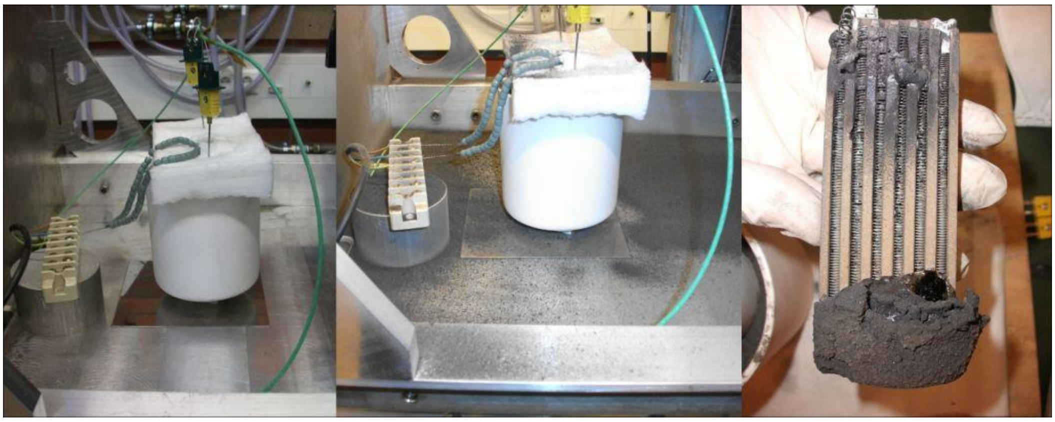

Figure 6: Preliminary testing with 2 stacked alumina crucibles in vacuum chamber (left: before, middle: after heating to 400°C in vacuum) showing the destroyed heater with molten JSC-1A at the bottom.

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