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Hydrogen reduction of ilmenite as an ISRU demonstration for ProSPA.

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Introduction: The next step for human exploration in space will begin with a return to the Moon, where In-Situ Resource Utilisation (ISRU) will be crucial in supporting an extended human presence on the lunar surface that is less dependent on the Earth's resources. To determine the feasibility of lunar ISRU we must first understand the resources available. A number of missions are planned for the coming decade including the Luna-27 surface lander, which will provide a better understanding of the ISRU capabilities of the lunar south pole [1].

On board the Luna-27 mission will be ESA's Package for Resource Observation and in-situ Prospecting for Exploration, Commercial exploitation and Transportation (PROSPECT). One of the components of PROSPECT is a Sample Processing and Analysis instrument, known as ProSPA. This miniature laboratory is in development with the aims to perform evolved gas pyrolysis, and stepped pyrolysis or combustion, to release volatiles which can be identified and quantified before undergoing isotopic analysis. Also, an ISRU demonstration is to be carried out by reducing mineral phases in the presence of hydrogen [2]. Together, these processes will enhance our understanding of resources available at the south pole and the feasibility of some extraction processes.

The reduction of ilmenite has long been considered a potentially viable technique to extract water, and consequently oxygen, from the lunar regolith with the purpose of producing fuels and life support gases. One of the ISRU demonstration techniques being considered for ProSPA is the reduction of ilmenite in the presence of hydrogen. The ilmenite reduction process is well understood, however most experiments are performed on a 100 % ilmenite sample with a continuous flow of hydrogen. The ProSPA instrument is limited to a static system and will likely obtain samples with low concentrations of ilmenite. It is the purpose of this study to determine the extent to which the reaction will proceed in the ProSPA system.

Hydrogen Reduction of Ilmenite: Ilmenite, $FeTiO_3$, is a member of the oxide family of minerals, which are the second most abundant minerals on the Moon after silicates [3]. The ilmenite reduction process goes as follows:

$$FeTiO_3 + H_2 \rightarrow Fe + TiO_2 + H_2O$$

The process requires relatively low temperatures (< 900 °C) compared to oxygen extraction from silicates (> 1100 °C), and hydrogen has been shown to be the preferred reducing agent over CH_4, CO, and electrolysis [4].

In order to demonstrate the feasibility of the extraction process in-situ, ProSPA will heat a ~ 30 mm^3 regolith sample in the presence of hydrogen up to 900 °C in one of its ovens. Any water produced will collect at a 'cold finger' before being analysed in the ion trap mass spectrometer. A breadboard model is in development at The Open University to trial the reaction and determine its feasibility for use on ProSPA.

Experimental Set-Up: The breadboard model is comprised of 'off the shelf' parts and the layout is shown in Figure 1. The furnace will be operated at ~ 900 °C and the cold finger will be operated at ~ -190 °C. The connecting pipes will be heated to > 100 °C to inhibit condensation of water between the furnace and the cold finger.

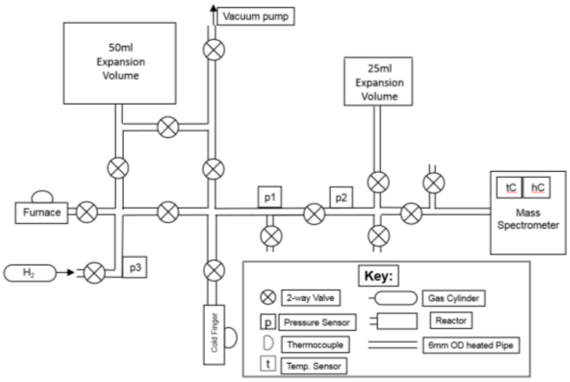

Figure 1 Schematic diagram of the ISRU demonstration model.

Before any experiments are run, the system is to be cleared of contaminants by placing it under vacuum and heating to > 100 °C. A control reading will also be taken from the mass spectrometer to determine the quantity of any remaining contaminants. Similarly, a control reading of a lunar analogue without any ilmenite will be taken, to determine the quantity of any water that is produced from sources other than ilmenite.

Next, simulants doped with ilmenite will be used and the quantities of water produced will be detected. As the concentration of ilmenite is varied, so will the quantity of hydrogen used in the system. The minimum amount of hydrogen will be used to avoid increasing the pressure and limiting the movement of any water produced. This equates to one mole of hydrogen for each mole of ilmenite present.

A number of experiments are envisaged, as well as varying the concentration of ilmenite, such as varying the grain size distribution of ilmenite to determine any effect upon the reaction rate. The number of moles of hydrogen will also be increased to see at which point the movement of water is inhibited.

Reaction rate predictions: If a sample with 1% ilmenite present completely reacts, the quantity of hydrogen required for the reaction also increases as shown in Table 1. Considering these values it is possible to determine the gas flow type within the system. Gas flow can be characterised by its Knudsen number, k_n, which considers the mean free path of particles, λ, and the radius of the pipes, a, as $k_n=\lambda/a$. With a pipe radius of 2 mm, the Knudsen number is in the viscous flow region suggesting the water molecules produced must diffuse through hydrogen before collecting at the cold finger.

Table 1 The effect of ilmenite concentrations on the quantity of hydrogen required and diffusion time of the water molecules produced.

Concentration of ilmenite (%)	Mass of hydrogen required (µg)	Pressure of hydrogen in system (Pa)	Time for diffusion of water molecules (s)
0.5	3.0	731	0.5
1	5.9	1500	1
2.5	14.8	3700	2.5
5	29.6	7300	5
10	59.2	15000	11
50	296.1	73000	51
100	592.1	150000	101

As water molecules are produced they prevent hydrogen from accessing the remaining ilmenite grains and therefore must diffuse through the system before the reaction can continue. The rate of diffusion can be calculated with Fick's law, $J_{AZ}=-D_{AB} * dC_A/dz$, where J_{AZ} is the molar flux of gas A into gas B in the z direction, D_{AB} is the diffusion coefficient, C_A is the concentration of gas A, and z is the distance of diffusion [5, 6]. Applying this equation with the assumption that all of the 1 % concentration ilmenite present has reacted; all the water molecules produced are stored in the furnace volume; and the distance between the furnace and cold finger is 0.5 m, the molar flux of water is $0.23\,\mathrm{mol\ m^{-2}\ s^{-1}}$. The velocity of diffusion is therefore shown to be ~ $0.5\,\mathrm{m\,s^{-1}}$ by dividing the molar flux by the average concentration, resulting in a diffusion time of 1 s. Table 1 shows how the diffusion time increases as the quantity of ilmenite, and therefore quantity of water produced, also increases. There are limits to this analysis due to the assumptions stated above. In reality there will be pressure changes as the water condenses, and the temperature will differ at each end of the system, both of which will significantly affect the diffusion rate.

The other rate controlling steps being considered are the diffusion of hydrogen in the product layer, and the chemical reaction at the interface. It has been shown that for a 100 % ilmenite sample, with a continuous flow of hydrogen (to reduce the effect of water molecules surrounding the sample), the reduction reaction completes in ~ 20 minutes and is controlled by the chemical reaction at the interface [7]. This suggests that with the diffusion rate of water also being considered, the time taken for the reaction to complete will be increased further. It is not yet known how the ilmenite concentration in the soil will affect the reaction rate.

Further considerations:

The grain size distribution of the lunar soil that is to be obtained from the Luna-27 landing site is not known, and it is understood that grain size distribution does affect the reaction rate of the ilmenite reduction process. If the grain size is too large then it would suggest that further beneficiation would be needed by future ISRU technologies in similar locations.

In the lunar mare regions, ilmenite can be present at relatively higher abundances, from 10 – 20 vol% [8] whilst the highlands are relatively depleted with an average concentration of < 1 vol% [9]. The mineralogy of the lunar poles is largely unknown as no direct measurements have been taken to date. However, based on remote sensing data, it is expected that the lunar poles will be mostly comprised of highlands-type material which may strongly limit the applications of this resource extraction technique, necessitating alternative considerations. On the other hand, if this process was applied on the ProSPA instrument it would give a ground truth indication for the ilmenite concentration at the lunar south pole and combined with the results of its other experiments, it will help to determine if the south pole is a viable location for resource extraction.

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