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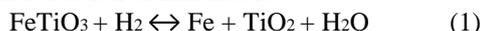
## HYDROGEN REDUCTION OF ILMENITE IN A STATIC SYSTEM FOR A LUNAR ISRU

**DEMONSTRATION.** H.M. Sargeant<sup>1</sup>, F. Abernethy<sup>1</sup>, S. J. Barber<sup>1</sup>, I. P. Wright<sup>1</sup>, S. Sheridan<sup>1</sup>, P. Landsberg<sup>1</sup>, A. D. Morse<sup>1</sup>, M. Anand<sup>1,2</sup> and P. Reiss<sup>3</sup>. <sup>1</sup>School of Physical Sciences, The Open University, Walton Hall, Milton Keynes, <sup>2</sup>The Natural History Museum, London, UK, <sup>3</sup>Technical University of Munich, Germany; E-mail: [hannah.sargeant@open.ac.uk](mailto:hannah.sargeant@open.ac.uk)

**Introduction:** *In situ* Resource Utilisation (ISRU) is a concept that resources can be harvested *in situ*, enabling space exploration missions that would be otherwise economically unfeasible if solely reliant on resources from Earth. Although ISRU paper studies have been undertaken from as early as 1979 [1], more attention has been paid to laboratory and field studies in the last decade as the need for such technologies becomes critical to future exploration missions [2].

Lunar ISRU demonstration and resource prospecting missions are currently in development. ProSPA is a Sample Processing and Analysis instrument being developed at The Open University as part of ESA's Package for Resource Observation and *in situ* Prospecting for Exploration, Commercial exploitation and Transportation (PROSPECT). PROSPECT is envisaged to operate in a high latitude region of the Moon on board the Luna-27 mission in ~2022 [3]. One goal of ProSPA is to perform a proof-of-principle ISRU experiment on the lunar surface. The current work concerns the evaluation of the feasibility to carry out hydrogen reduction of ilmenite as such an experiment.

**ISRU Reaction:** The chemical equation for the hydrogen reduction of ilmenite is:



The reaction has been shown possible at temperatures of 700 - 1000 °C with reasonable yield of H<sub>2</sub>O [4] which is within the temperature constraints of ProSPA. The only reactant needed is hydrogen gas which in the case of ProSPA is supplied from onboard gas tanks, and the products of the reaction are not damaging to the system. It should be noted that for full scale ISRU technologies, hydrogen gas could be sourced from lunar regolith and recycled in the reaction.

The chemical reaction is reversible so the partial pressure of water above the ilmenite must be kept sufficiently low to allow the reaction to proceed to completion. Normally this is achieved by utilising a flow of hydrogen, often by a fluidised bed system, so that as the water is produced it is transported away from the reaction site. As ProSPA is a static (non-flowing) system another technique has been proposed, in which a cold finger is used to draw any produced water away from the reaction site. As water vapour is produced it will diffuse through the system until it reaches the cold finger where it will condense. As a result, the water vapour content near the cold finger will decrease and the remaining water in

the system will continue to diffuse before ultimately condensing at the cold finger. This process will be less efficient than a flowing system in terms of quantity and speed of the removal of water from the reaction site, however 1<sup>st</sup> order modelling suggests that the diffusion of water through the hydrogen reagent gas is fast enough to allow the reaction to proceed [5].

**ProSPA Breadboard Model:** A breadboard model of the ISRU relevant aspects of the ProSPA system has been built at The Open University. A schematic of the breadboard is shown in Figure 1.

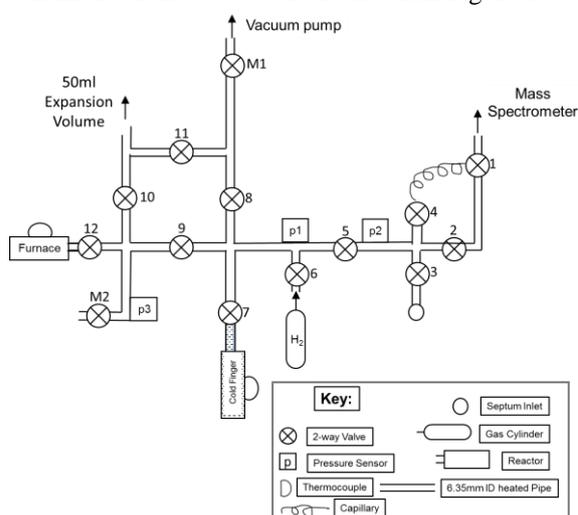


Figure 1 Schematic diagram of the ISRU demonstration breadboard model.

**Water Trapping Study:** The system was initially used to evaluate whether water could be trapped at the cold finger and then re-released. 0.5-5 μl samples of water were injected into the system with the cold finger operating at -180 °C for 1 hour whilst the manifold operated at 115 °C. The cold finger was then heated to 115 °C to release any trapped water into the system and pressure readings were taken in what is defined as the volatile release stage. The results are shown in Figure 2.

The results show that with increasing volumes of water, the pressure reading after heating the cold finger also increases as expected. This suggests that the system is capable of trapping water and making quantitative measurements. However, the pressure of water that can be measured is limited to ~120 mbar. This is likely owing the saturation vapour pressure being reached as some areas of the system are at lower temperatures because of minor imperfections in the heating system (See “Going Forward” below).

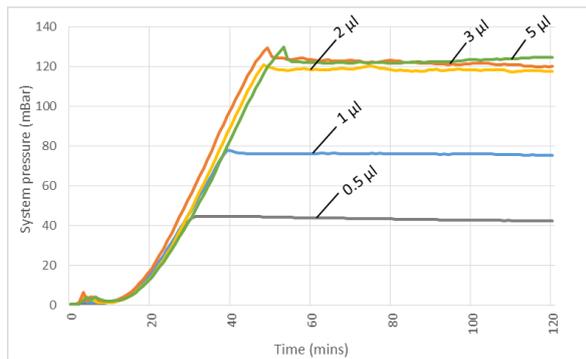


Figure 2 Pressure rise as a result of water released from the cold finger when heated to 115 °C.

**Ilmenite Reduction Study:** Next, the system was used to perform preliminary ilmenite reduction reactions. Since a mass spectrometer was utilised in this study (See Figure 1) this increased the operating volume of the system. A full sample expected to be collected in ProSPA ovens is ~45 mg; this was used to define 100% sample mass. A range of masses of ilmenite was studied from 0 - 100%. No other material was added to the samples. Each sample was reacted with 0.3 mmol of H<sub>2</sub> gas which is sufficient to completely reduce 45 mg of ilmenite. The samples were heated in the furnace to 900 °C in the presence of H<sub>2</sub> for 1 hour whilst the cold finger was operating at -180 °C, and the remaining manifold was heated to 115 °C. Next, any trapped water was released as in the previous study. Both pressure sensor and mass spectrometer data were recorded during the ilmenite reduction reactions.

The pressure sensor data obtained during the volatile release stage from the ilmenite reduction study shows a similar trend to the water trapping study. There is a corresponding increase in pressure with increasing ilmenite mass, albeit with a pressure limit of 18 mbar. This is likely to be a consequence of the introduction of cold spots associated with the mass spectrometer. The equivalent yield of water from each ilmenite sample was calculated assuming that the pressure increase is entirely as a result of water vapour in the system. The yield against sample mass is shown in Figure 3.

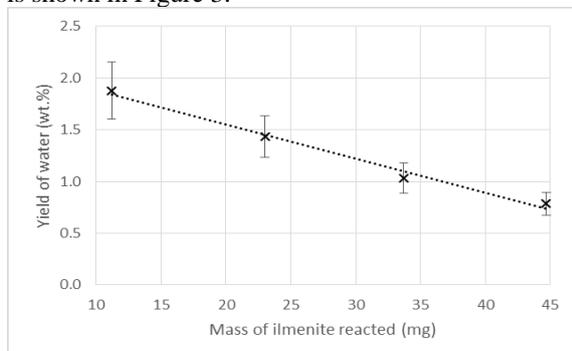


Figure 3 Estimated yield of water with corresponding sample mass as calculated from the pressure rise in the system.

Theoretically, the yield should remain constant at 11.8 wt.% across all sample sizes. The ilmenite reduction study shows that the yield decreases with larger samples with a yield significantly below the predicted value. There are a number of reasons why this could be related to completion of reaction and re-condensation of water in the system. These will be investigated further in future work.

Initial analysis of mass spectrometer data has shown that volatiles additional to water are released during the reaction, likely due to impurities in the ilmenite feedstock. The detection of mass 18 has been a challenge with the current setup due to relatively high levels of background water.

**Going Forward:** The ilmenite grains that were used in this study will be analysed under SEM to determine the extent of the reaction within the grains. A new breadboard model is also in development with the aim of reducing the problems highlighted in these preliminary studies, such as the non-uniform heating of the manifold and high background water signal in the mass spectrometer. The data obtained by the mass spectrometer will be used together with the pressure sensor data to quantify and characterise volatiles released during the reaction.

In terms of lunar application, one potential hindrance to the ilmenite reduction technique is the concentration of ilmenite in lunar regolith. It is thought that ilmenite concentrations in high latitude regions is <1 % [6]. Although the reaction will allow the reduction of other metal oxides, the efficiency is not well known. This will be considered along with other potential ISRU techniques.

**Summary:** ProSPA will perform an ISRU proof-of-principle experiment on the lunar surface. Adapting the well studied technique, hydrogen reduction of ilmenite is the primary reaction being considered to produce water from lunar regolith.

Initial results indicate that water can be trapped and re-released using a static system. Also, ilmenite reduction appears to be possible with the current setup, and the produced water can be detected.

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**References:** [1] Rao D. B. et al. (1979) in Space Resources and Space Settlements, NASA-SP 428, NASA. 257-274. [2] Sanders G. B. and Larson W. E. (2012) *JAE*, 26.1, 5-17. [3] Barber S. J. et al. (2017) *LPS XLVII*, Abstract #2171. [4] Taylor L. A. and Carrier W. D. (1993) in Resources of near-Earth space, University of Arizona Press. 69-108.[5] Sargeant H. M. et al. (2017) *ELS* [6] Taylor L. A. et al. (2010) *JGR*, 115.E2.