EXPERIMENTAL DEVELOPMENT AND TESTING OF THE REDUCTION OF ILMENITE FOR A LUNAR ISRU DEMONSTRATION WITH PROSPA. H. M. Sargeant[1], F. Abernethy[1], M. Anand[1,2], S. J. Barber[1], S. Sheridan[1], I. Wright[1] and A. Morse[1]. [1]School of Physical Sciences, The Open University, Walton Hall, Milton Keynes, [2]The Natural History Museum, London, UK. Email: hannah.sargeant@open.ac.uk

Introduction: ProSPA forms part of ESA's Package for Resource Observation and in-situ Prospecting for Exploration, Commercial Exploitation and Transportation (PROSPECT) which is to be used in a high latitude region of the Moon in ~2023 [1]. In addition to determining the lunar volatile inventory, ProSPA will perform a proof-of-principle ISRU water extraction experiment on the lunar surface on samples of ~ 50 mg.

It is of interest to obtain water, and its associated oxygen and hydrogen, on the Moon in order to meet the needs of crewed exploration missions to the lunar surface, and beyond. Frozen water is likely located in hard-to-reach polar regions, so other sources of water are being considered. Ilmenite, a common lunar mineral, can be reduced with hydrogen to produce water. This work considers the temperature and pressure constraints of an ilmenite reduction reaction performed using a static system, suitable for use on ProSPA.

Ilmenite Reduction: Hydrogen can reduce ilmenite to produce water in an equilibrium reaction as follows:

$$FeTiO_3 + H_2 \rightarrow Fe + TiO_2 + H_2O \qquad (1)$$

Ilmenite reduction can be performed at relatively low temperatures of 700-1000°C [2]. This is within the operating constraints of ProSPA. However, the reduction reaction is usually performed in a flowing stream of hydrogen which removes water from the reaction site [3,4]. As a consequence of the static nature of the ProSPA design, a cold finger is used to condense the produced water [5]. A benchtop demonstration model (BDM), used to simulate the ProSPA design, has successfully been used to reduce ilmenite in a static system, trapping and quantifying the produced water [6]. The BDM reduced ilmenite samples (up to 45 mg) at 900°C for 1 hr. Although water was produced from these studies, the reactions did not complete. A new system design was developed with improved thermal control, and is known as the ISRU-BDM. The new system has been used to perform ilmenite reduction tests for a range of temperatures and pressures.

Materials and Methodology: The ISRU-BDM is a sealed vacuum system that operates inside a uniformly heated box at 120°C. A furnace that can reach >1000 °C heats a 4 mm i.d. ceramic sample holder. The cold finger is thermally controlled by heaters and a supply of cooled nitrogen gas.

For each experiment 45 mg (0.3 mmol) of 95% pure ilmenite (average grain size of 170 μm) is baked out to 500°C for 1 hr [6]. Then the desired quantity of hydrogen (120, 210, 345, 420, 580 mbar) is added to the system. The furnace is then heated to the desired temperature (850, 900, 950, 1000, 1050, 1100°C) for 4 hours and any produced water is condensed at the cold finger which operates at -80°C. Finally, the cold finger is heated to 120°C and water released as a vapor. Pressure readings are recorded during each experiment to monitor the reaction and its products.

Results: Preliminary results have shown that with increasing temperature the reaction produces increased quantities of water. When ilmenite is in the solid phase, the maximum yield is 3.40±0.07 wt.% O_2 for a reaction temperature of 1000°C. When heating beyond the melting point of ilmenite, the maximum yield is 4.42±0.08 wt.% O_2 for a reaction temperature of 1100°C. Hydrogen pressures have a twofold effect on the reaction rate. Initially, lower pressures (<120 mbar) are favorable as the produced water easily diffuses through the system to the cold finger. As the reaction proceeds into the grain higher hydrogen pressures (~420 mbar) are required to enable penetration into the grains. SEM and XRD analysis supports the results obtained.

Conclusions and Future work: A static system can successfully produce water from the hydrogen reduction of 45 mg ilmenite. Yields can be calculated from the change in gas pressure, where the maximum yield is obtained at higher temperatures. The required hydrogen pressure should be tailored to the design of the system with low pressures at the start of the reaction followed by higher pressures as the reaction proceeds. Although a static system is not optimized for an ISRU reaction, it is a simple technique that can be used to perform a proof-of-principle reduction reaction of lunar ilmenite in situ.

Future work will consider if the system can be used to produce water from the reduction of lunar meteorites and Apollo samples.

References: [1] Barber S. J. et al. (2018) *LPSC XLIX*, Abstract #2172. [2] Taylor L. and Carrier W. (1993) *Resources in Near Earth Space*, 69-108. [3] Gibson M. A. and Knudsen C. W. (1985) *Lunar Bases and Space Activities*, 543-550. [4] Christiansen, E. et al. (1988) Conceptual Design of a Lunar Oxygen Plant, NAS9-17878. [5] Williams R. J. and Mullins O. (1983) LPSC XIV, Special Session Abstracts 34-35. [6] Sargeant H. M. et al. (2018) *PSS* (in review).

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