Experimental Development and Testing of the Reduction of Ilmenite for a Lunar ISRU Demonstration with ProSPA

Conference or Workshop Item

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. As any frozen water is likely located in hard-to-reach polar regions, other sources of water are being considered. Ilmenite, a common lunar mineral, can be reduced with hydrogen to produce water. This work considers the time and temperature 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:

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

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, successfully trapping and quantifying the produced water [6]. The BDM was used to reduce 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 reaction times.

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 0.3 mmol of hydrogen is added to the system. The furnace is then heated to the desired temperature (850, 900, 950, 1000, 1050 °C) for 0.5 hr and 4 hr 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 and mass spectrometry readings are recorded during each experiment to monitor the reaction and its products.

Results: Preliminary results show with increasing temperature, the rate of reaction increases between 850 and 1050 °C for the first 2 hrs of a reaction. Beyond this point, the reaction rate slows for samples reacted at 1050 °C, likely a consequence of some partial melting of ilmenite that is slowing the reaction. SEM analysis of the reacted grains will be used to investigate this. When the furnace is operating at 1000 °C, oxygen yields of ~0.4 wt.% and ~3.6 wt. % are recorded for reaction times of 0.5 hrs and 4 hrs respectively. However, the reaction is still only ~34 % complete in this 4 hr time-frame. As there is a potential temperature limit of ~1050 °C, longer reaction times and/or variations in hydrogen concentration may be required.

Conclusions and Future work: A static system can successfully produce water/oxygen from 45 mg ilmenite which is reduced by 0.3 mmol hydrogen at temperatures of between 850 and 1050 °C. 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 variations in hydrogen concentrations and if the system can be used to produce water from the reduction of lunar meteorites and Apollo samples.

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