

EXPERIMENTAL STUDY ON CARBOTHERMAL REDUCTION OF LUNAR REGOLITH SIMULANTS FOR METAL/METALLOID PRODUCTION.

S. Kaur¹, B. B. Barbre², W. J. Ready³, T. M. Orlando⁴, and P. G. Loutzenhiser⁵, ¹Georgia W. Woodruff School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, Georgia, USA, 30332-0405 (ssingh648@gatech.edu), ²Georgia W. Woodruff School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, Georgia, USA, 30332-0405 (bbarbre6@gatech.edu), ³Georgia Tech Research Institute, Georgia Institute of Technology, Atlanta, Georgia, USA, 30332-0826 (jud.ready@gtri.gatech.edu), ⁴School of Physics and School of Chemistry and Biochemistry, Georgia Institute of Technology, Atlanta, Georgia, USA, 30332-0405 (thomas.orlando@chemistry.gatech.edu), ⁵Georgia W. Woodruff School of Mechanical Engineering, Georgia Institute of Technology, Atlanta, Georgia, USA, 30332-0405 (peter.loutzenhiser@me.gatech.edu).

Introduction: The harvesting of lunar resources via *in-situ* resource utilization (ISRU) is vital for lunar habitation and subsequent space exploration efforts. The extraction of valuable metals and metalloids contained in lunar regolith are of particular interest, including Si, Fe, Ti, K, and Ca, which are integral for the development of perovskites for optical glass and other industrial and scientific infrastructures [1]. These metals and metalloids are bound with oxygen as oxides, which are contained in natural basaltic glass, plagioclase, olivine, and other minerals [2], necessitating chemical reductions. Solar-driven carbothermal reduction potentially exploits CH₄ found on the Moon via cracking with C deposited on the surface and H₂ used for other applications [3]. Activated carbon was used in this work as the reducing agent to eliminate the secondary process of H₂ and extend the analysis validity to different sources of carbon to prepare for other fixed carbonaceous sources, *e.g.*, human feces [4], becoming available on Moon in the future due to human presence.

Compositions of lunar regolith simulants. Three types of simulants are studied in this work: (1) JSC-1A, (2) LMS-1, and (3) LHS-1, where JSC-1A is a mare simulant simulating the Apollo sample 14163 [5]; LMS-1 is a simulant representing the average or generic compositions of mare soils which are located at the darker cratered regions of the Moon [6]; and LHS-1 is a simulant representing the average or generic compositions of highland soils which are located at the lighter regions of the Moon including the South Pole [7].

Methodology: All simulant types were mixed with activated carbon at C to JSC-1A, C to LMS-1, and C to LHS-1 of 0.332, 0.319, and 0.344 respectively, representing the exact C needed to reduce all oxides/minerals in the simulants to metals or metalloids. These mixed samples were used in all experiments.

Tube furnace experiments. Thermal heating of sample in an inert environment (100% Ar) was conducted in a tube furnace to simulate the low O₂ environment on lunar surface and the setup is shown in Figure 1. Ultra-high purity Ar flow passed through an O₂ trap and was controlled by a flow controller (FC).

The mixed sample was thinly spread on a Mo foil which was then placed on a Zr₂O₃ crucible to minimize contact between sample and the Al₂O₃ tube. At the exhaust end of the tube, four layers of W meshes were mounted to capture volatiles leaving the sample during experiments for subsequent material characterization. A mass spectrometry (MS) and a gas chromatography (GC) were hooked to the exhaust line to measure gas evolution and the exhaust gas was eventually purged into the fume hood.

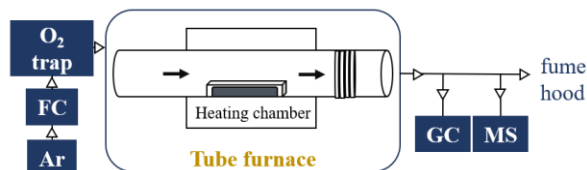


Figure 1. Schematics outlining the setup of tube furnace experiments.

Ultra-high vacuum experiments. Thermal heating of sample in an ultra-high vacuum chamber was conducted to simulate the low-pressure lunar conditions. The experimental setup is shown in Figure 2, where the system was continuously pumped via a turbomolecular pump (TP) and a mechanical vacuum pump (MVP) to maintain vacuum of $\sim 10^{-12}$ bar. The mixed sample was mounted on a Ta strip as a dense layer of ~ 10 mm² area via H₂O suspension (dried in air at 300 K) and heated to temperatures up to 2000 °C via different temperature profiles. Cu foils were mounted ~ 3 mm above the sample to collect volatiles for subsequent material characterization to identify volatile production as a function of temperature. The setup was also equipped with a quadrupole mass spectrometry (QMS) to measure gas evolution, specifically CO, a by-product of carbothermal reduction.

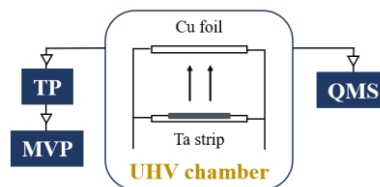


Figure 2. Schematics outlining the setup of ultra-high vacuum experiments.

Solid-state surface material characterization. Energy-dispersive X-ray spectroscopy (EDS) was used to identify elemental distribution of samples before and after the tube furnace experiments and elemental presence and distribution of volatiles collected by W meshes and Cu foils for tube furnace and ultra-high vacuum experiments, respectively. X-ray photoelectron spectroscopy (XPS) and X-ray diffractometry (XRD) were used to identify chemical compositions, and transmission electron microscopy (TEM) was used to identify particle morphology of samples after tube furnace experiments.

Results:

Tube furnace experiments. The temperature profile and normalized ionic current of mass 28 (CO) measured by the MS are shown in Figure 3 for JSC-1A + C. CO evolution increased with increasing temperature and then decreased at temperatures ≥ 1500 °C, indicating that majority of reactions occurred below this temperature. Mass balance indicated ~37% of sample mass was evolved from the sample as CO and volatiles.

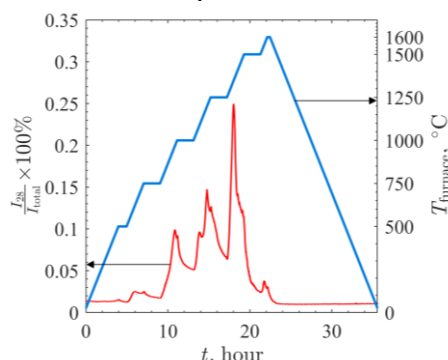


Figure 3. Normalized ionic current of mass 28 (CO) as a function of experiment time and furnace temperature.

Ultra-high vacuum experiments. In carbothermal reduction of JSC-1A, CO was detected by the QMS for all experiments, indicative of reactions. The reaction rates increased with increasing temperatures up to 1500 °C, in agreement with thermodynamic predictions. However, the reaction rates decreased for the 1750 °C experiment likely due to previous volatile evolution at lower temperatures. An increase in reaction rates between the 1750 °C and the 2000 °C experiment was observed likely due to other reactions becoming favorable, e.g., production of Si(g).

Solid-state surface material characterization. EDS results of post-experimental sample for the carbothermal reduction of LMS-1 in the tube furnace are shown in Figure 4. Elemental scans indicated the formation of Mg- constituents and showed reactions with Al- and Ca- constituents.

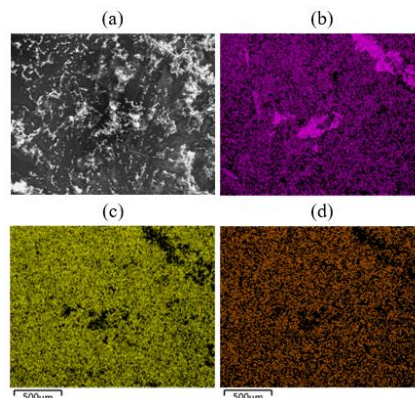


Figure 4. EDS-captured (a) sample image and elemental scan of (b) Mg, (c) Al, and (d) Ca of sample after tube furnace experiment using LMS-1 + C.

In the ultra-high vacuum experiment for JSC-1A + C, Fe-, Al-, Mg-, and Si- deposits were identified on the Cu foils for experiments at or above 1250 °C up to 2000 °C. The Fe-, Al-, and Mg- were released between 1250 °C and 1500 °C. The amounts of Si- varied as a function of temperature indicating variations of constituents evolving from the sample. The Si- released at lower temperatures was likely in the form of SiO(g) whereas the Si- released at higher temperatures was likely in the form of Si(g).

Conclusions: The carbothermal reduction of JSC-1A, LMS-1, and LHS-1 via activated carbon was demonstrated in inert and ultra-high vacuum conditions that mimicked the lunar surface conditions. Identification of post-experimental samples and volatile production as a function of temperature and simulant types gives insights into the metals or metalloids produced by carbothermally reducing lunar regolith. Quantification of volatile production in inert conditions describes the feasibility of metal or metalloid production. This work lays a foundation and pathway to further investigate, design, and optimize reactors for carbothermal reduction of lunar regolith.

Acknowledgements: This work was carried out as part of Lunar in-situ Atomic Resource Utilization Experiment (LISA RUE) which was directly supported by the Georgia Tech Research Institute (GTRI). Special thanks to Brant M. Jones for configuring the ultra-high vacuum experimental setup.

References:

- [1] Crawford I. A. et al. (2015) *Prog. Phys. Geogr.*, 39, 137-167. [2] Papike J. J. et al. (1982) *Rev. Geophys.*, 20, 761-826. [3] Balasubramaniam R. et al. (2010) *Int. J. Miner. Process*, 96, 54-61. [4] Bittencourt F. L. F. et al. (2019) *SN Appl. Sci.*, 1, 1077. [5] Cannon K. (Cited 2022) <https://shorturl.at/ABV19>. [6] Tech S. R. (Cited 2024) <https://shorturl.at/hjq06>. [7] Tech S. R. (Cited 2024) <https://shorturl.at/pGOTX>.