

ULTRA-HIGH VACUUM THERMAL AND CARBOTHERMAL REDUCTION OF LUNAR REGOLITH SIMULANTS FOR OXYGEN AND CONSTRUCTION MATERIALS.

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Introduction: The establishment of a permanent presence on the Moon is fraught with challenges and perils to human survival at the most basic level. The lunar environment is harsh and devoid of the most rudimentary elements that are required for habitation, including readily accessible O₂. In the absence of an atmosphere and a strong magnetic field, inhabitants are also subjected to deadly radiation, including galactic cosmic rays and dangerous particles produced in solar particle events, that threatens long-term survival by increasing the probability of developing cancers or other major health problems [1]. Hence, lunar resources must be exploited to address these challenges for sustained habitation on the surface of the Moon. Such a potential resource is lunar regolith that consists primarily of metal or metalloid oxides in the form of complex minerals, resulting in regolith that is ~45% O by weight [2]. Oxygen is beneficial for rocket propellants and life-support systems, whereas metals or metalloids are useful as construction materials for scientific and industrial infrastructures, and human habitations that are protected from deadly radiation exposure.

This work experimentally explores two thermochemical processes to reduce regolith into oxygen and metals/metalloids in ultra-high vacuum conditions: direct thermal and carbothermal. These processes require high temperatures, which can be efficiently supplied by concentrating solar technologies. The Moon is ideally situated to exploit solar irradiation as it has no atmosphere preventing attenuations [3], has a declination angle of only 1.5° [4], compared to Earth's 23°, resulting in little seasonal variation in solar irradiation, and has a long lunar day (~14 Earth days [3]) with some regions that are permanently irradiated. The primary limitation is the dilute irradiation (average direct-normal solar irradiation on the Moon is ~1361 W/m² [5]). However, this is overcome by concentrating solar irradiation to provide the necessary radiative heat fluxes to drive the thermochemical processes.

Three types of simulants were 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 [6]; LMS-1 is a simulant representing the average or generic compositions of mare soils located at the darker cratered regions of the Moon [7]; and LHS-1 is a

simulant representing the average or generic compositions of highland soils located at the lighter regions of the Moon including the South Pole [8].

Methodology: Experiments were run in an ultra-high vacuum chamber that was sealed and evacuated via a turbomolecular pump connected in series with a mechanical vacuum pump to ~10⁻⁹ torr to mimic the low-pressure conditions on the lunar surface. Powdered samples of ~5 mg were placed on a thin Mo foil which was resistively heated to the desired power (P) and held isothermally before cooling down to ambient. Heating/cooling rates of 0.01 V/ μ s and 0.01 A/ μ s were used for voltage and current, respectively. Sample temperature was indirectly measured by an infrared pyrometer. The chamber was equipped with a quadrupole mass spectrometer (QMS) to measure produced O₂(g) or CO(g). Two experiments were conducted for each simulant without sample change. A copper foil was mounted ~3mm above the sample to capture vapor evolution during each experiment. The Mo sample holder and both Cu foils for each set of experiments were further investigated via solid-state surface material characterization to identify leftover sample and deposited vapors, respectively. Energy-dispersive X-ray spectroscopy (EDS) was used to identify elemental presence and X-ray photoelectron spectroscopy (XPS) was used to identify oxidation states and possible chemical compositions of detected elements.

For direct thermal reduction experiments, thermodynamic analyses at ~10⁻⁹ torr were used to determine operating temperatures (T) for targeted metal vapor production. Table 1 lists the experimental P and measured T as well as targeted T and metal production from thermodynamics. SiO(g) was targeted instead of Si(g) because Si(g) production (requires $T < 2000$ °C) was not theoretically possible within the temperature limits of experiment setup. Isotherm durations were shortened at higher T s to avoid deterioration of Mo foil.

Table 1. Operating P and measured T by pyrometer for targeted T and metal vapor production, and corresponding experimental isotherm duration.

Simulant	P , W	T , °C		Target metal	Isotherm duration, min
		Target	Measured		

LMS-1	613.2	1170	1182-1195	Mg(g), SiO(g)	60
	730.0	1380	1234-1245	Al(g), SiO(g)	30
LHS-1	835.9	1300	1304-1313	Ca(g), SiO(g)	30
	985.5	1500	1484-1498	Al(g), SiO(g)	5

For carbothermal reduction experiments, simulants were mixed with activated carbon at a C-to-simulant ratio of 0.5, representing the excess C needed to reduce all contained oxides/minerals to metals or metalloids. $T > 1200$ °C and $T > 1400$ °C were chosen for the first and second experiment, respectively, for each simulant because previous work [6] found that $T > 1200$ °C is needed for vapor production in carbothermal reduction of simulants with stoichiometric C amounts.

Results:

Direct thermal reduction. QMS data showed no significant $O_2(g)$ production, likely due to favorable recombination of compounds with the released O_2 in the gap between the sample holder and Cu foil at lower temperatures. Hence, separation must be considered in reactor design for direct thermal heating, either via electrothermal methods in which a high-temperature O_2 membrane is used to separate out the oxygen, rapid quenching, or by adding reductant (*e.g.*: C, H_2) to the reduction process to avoid recombination.

EDS detected significant amounts of Na, Fe, K, and P on Cu foils for LMS-1 experiments with larger evolved amounts from the higher P experiment. Si and Mg were detected on the Cu foil for the higher P experiment, indicating that these compounds required $T > 1200$ °C for evolution. XPS inferred that the Na detected by EDS is likely metal. Other compounds were not detected due to the shallow depth of analysis for XPS compared to EDS. EDS and XPS analyses of the leftover sample from LMS-1 experiments indicated the presence of Mg metal, MgO, FeO, TiO_2 , aluminosilicate and CaO.

Na, Fe, and K were also significantly detected by EDS on Cu foils of LHS-1 experiments with smaller evolved amounts from the higher P experiment. P was only detected on the Cu foil of the lower P experiment, likely due to its low quantity in the simulant causing evolution completion. Significant Si and some Mg were also detected on both Cu foils with larger evolved amounts as temperature increases, indicating the need for elevated temperatures for complete evolution of these compounds. Sample segregation was evidenced on the leftover sample between unevolved Si and Al as shown in Figure 1. The O scan, evidence of oxides, indicated incomplete reduction.

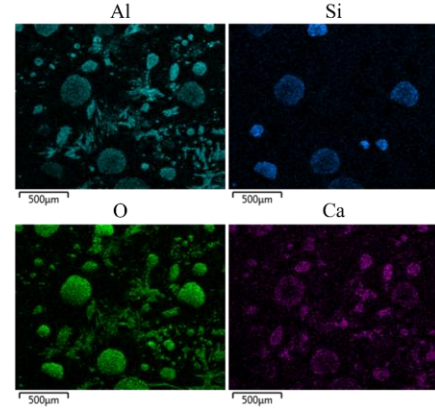


Figure 1. Elemental scan of Al, Si, O, and Ca by EDS for direct thermal reduction of LHS-1.

Carbothermal reduction. $CO(g)$ was significantly detected by the QMS, indicative of carbothermal reduction of LHS-1. EDS detected significant amounts of Fe, Na, and K in the $T > 1200$ °C experiment only, indicating the completion of evolution of these compounds. Si, Mg, and Ca significantly evolved during both experiments with larger amounts of Ca in the $T > 1400$ °C experiment. Material characterization on the leftover sample yielded similar results shown in Figure 1, in which Al, Ca, and Si segregation was evidenced.

Conclusions: Separation mechanisms to avoid compound recombination with oxygen are needed for direct thermal heating of regolith. For carbothermal reduction of regolith, $CO(g)$, the reaction byproduct, could be further processed for oxygen yield. Production of Na, Fe, K and P compounds were possible at lower temperatures whereas Si, Mg, and Ca required elevated temperatures. Al remained in the leftover sample and sample segregation was evidenced by material characterization, indicating the possibility of Al separation and production upon the evolution of other compounds. This work deepened the understanding of direct and carbothermal reduction of lunar regolith as a function of temperature in ultra-high vacuum conditions for oxygen and metal/metalloid production.

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