CARBOTHERMAL REDUCTION OF LUNAR MATERIALS FOR OXYGEN PRODUCTION ON THE MOON
REDUCTION OF LUNAR SIMULANTS WITH METHANE

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The utilization of extraterrestrial resources will become a key element in space exploration and colonization of the Moon and Mars in the 21st century. Indeed, the development and operation of *in-situ* manufacturing plants are required to enable the establishment of permanent lunar and Martian bases. Oxygen manufacture for life support and propulsion will be the most important manufacturing process for the first of these plants. The Carbothermal Reduction Process for the manufacture of oxygen from lunar materials has three essential steps: the reduction of ferrous oxide and metallic silicates with methane to form carbon monoxide and hydrogen; the reduction of carbon monoxide with hydrogen to form methane and water; and the electrolysis of water to form oxygen and hydrogen. This closed cyclic process does not depend upon the presence of water or water precursors in the lunar materials. It produces oxygen from silicates regardless of their precise composition and fine structure. In accord with the Statement of Work of Contract NAS 9-19080, Carbothermal Reduction of Lunar Materials for Oxygen Production on the Moon, ORBITEC has placed emphasis on the following issues to gain a better understanding of the Carbothermal Reduction Reaction of lunar regolith and to develop a low-risk, light-weight design for a lunar lander experiment: (1) highly efficient, i.e., greater than 95%, reduction of the lunar simulants with methane; (2) determination of conditions, particularly temperatures, required for initial and complete reduction of the lunar simulants; (3) identification of the products formed, gases and solids; (4) determination of solid product properties; (5) determination of reaction rates and mechanisms; and (6) demonstration of container materials. The most important of these issues were: (1) efficient reduction of the lunar simulants, i.e., JSC-1 and MLS-1A, with methane; (2) identification of the products formed , i.e., carbon monoxide, metals, e.g., iron and silicon, and slags, e.g., complex silicates; and (3) delivery of methane to the surface of the molten simulants without premature pyrolysis of the methane. The results of this empirical research are reported in this paper.

The following activities were conducted: (1) experiment requirements definition; (2) lunar simulant acquisition; (3) small furnace apparatus design; (4) apparatus fabrication, assembly and checkout; (5) experiment execution; (6) determination of reaction efficiency, and (7) reduction product analysis and identification. This paper will focus on (1) experiment execution and methane conversion efficiency and (2) reaction product analysis and identification.

The main components of the test apparatus are the high-temperature furnace (HTF), the quadrapole mass spectrometer (QMS), and the electronic mass-flow control system. The HTF is a vacuum-tight, water-cooled, resistance furnace with graphite heating elements capable of reaching a maximum temperature of 2200 C (3992 F). The temperature was controlled using a state-of-the-art, programmable temperature controller. The temperature control thermocouple (boron/graphite design) is recommended for use up to 1800 C (3272 F). The temperature can be controlled to remain constant at a given level to an estimated average of ± 1 C (1.8 F). The HTF was mounted so it can be rotated from horizontal to vertical in any required orientation. It has a working space of 11.43 cm (4.5 in.) dia by 15.24 cm (6 in.) high (vertical position). It was maintained in the vertical position in all of the experiments and could accommodate two crucibles. The QMS used for product gas analyses was placed next to the HTF. Four electronic mass-flow controllers were used to control and measure flow, three for inlet gases and one for exit gases. All of the gas lines were made of 6.35 mm (0.25 in.) OD stainless steel tubing . Yttria (8 wt%) stabilized (YSZ) crucibles were used in the experiments reported in this paper.

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The chemical process under study was the direct reduction of lunar simulants with methane. In all of the experiments, JSC-1 or MLS-1A, were weighed directly in their YSZ crucibles. The crucible containing the simulant was placed in the HTF on alumina shims on graphite support furniture to eliminate interaction of the crucibles with the graphite. After loading, the gas-cooled methane inlet tube was positioned so that the exit of the tube was immediately above the simulant surface. The HTF was sealed and an argon purge was used at ambient to remove all of the air from the HTF chamber. The power was turned on and the temperature was raised to 1625 C (2957 F) controlled by a predetermined program, i.e, a heating rate of 2.5 C/min.

Argon flow was maintained throughout each experiment from the start of heating to the end of cooling to room temperature. Methane flow was started when the HTF temperature had reached 1500 C (2732 F) and was continued until carbon monoxide evolution, as indicated by the QMS, has declined significantly. The reduction reactionswere allowed to proceed for periods as long as 24 hr. The experimental data included HTF temperature, argon, methane, hydrogen and product gas flow rates, and carbon monoxide QMS peak height. In addition, the power output variables and controller thermocouple readings were recorded. After the completion of each experiment, the crucibles containing the reduction products were visually examined, weighed and photographed immediately. Elemental identifications of the solid products were carried out using a computer aided scanning electron microscope (SEM) with energy dispersive x-rays (EDX) in which SEM photomicrographs and EDX spectra were regularly taken for the various solid phases. Phase identifications were performed using x-ray powder diffraction techniques in which diffraction patterns for various phases were obtained. The results of the experiments are reported in detail in this paper.