IN SITU RESOURCE UTILIZATION (ISRU) TECHNICAL INTERCHANGE MEETING: OVERVIEW OF RESEARCH FOR LUNAR OXYGEN PROCESSING AT CARBOTEK DEVELOPMENT LABORATORIES. J. D. Ortego, Jr., L. L. Sorge, M. Guo-Murray, M. A. Gibson, and C. W. Knudsen, Carbotek, Development Laboratories, 16223 Park Row, Suite 100, Houston Texas 77084, USA.

Abstract: Oxygen production from indigenous lunar material is considered an enabling technology for future solar system exploration. Lunar derived oxygen provides many lunar base program enhancements. A great mass benefit can be derived when Earth return propellant oxidizer is not manifested for transit vehicles traveling to the moon. This results in substantial cost savings to the overall space transportation infrastructure. In addition, lunar produced oxygen can be used to supplement life support systems. Finally, many of the lunar oxygen processes under development produce by-products which are excellent construction materials, rich in iron and titanium, for shielding habitats and lunar surface equipment from cosmic radiation and more lethal solar flares. As a result of the apparent benefits of lunar derived oxygen, NASA has funded research for the development of promising techniques since the mid-1980's in order for the technology to be available for lunar return missions. Carbotek, with funding and technical assistance from NASA Johnson Space Center and the Shimizu Corporation, Space Systems Division, has been developing oxygen producing technology since 1984. This paper describes past and future work by Carbotek on two processes, hydrogen reduction of ilmenite and magma electrolysis.

Carbotek began developing a process for oxygen production from ilmenite-rich lunar deposits in 1984 to support NASA's lunar base operations and large-volume propulsion needs under the technical guidance of Dr. David McKay of JSC. Carbotek proposed the reduction of iron oxide in ilmenite by hydrogen at elevated temperatures in a three-stage fluidized bed reactor. Water vapor formed by the reduction reaction is electrolyzed to produce product oxygen, which is liquefied and stored, and hydrogen, which is recycled to the reactor. Reaction rate, thermodynamic equilibrium, and lunar-gravity fluidization behavior are key factors which have been considered in the development activity. NASA awarded Carbotek two Phase I and one Phase II SBIR contracts, which provided approximately $800,000 for this development work from late 1984 through early 1989. Shimizu Corporation provided approximately $700,000 for the fluidized-bed reactor process development plus the technical support of several of their engineers from 1991 through the present. Two U.S. Patents were granted in 1990, one for the oxygen process and another for a lunar hydrogen production process.

A Plexiglas two-dimensional bed was constructed to measure bubble sizes at lunar gravity on NASA's KC-135 aircraft to develop fluidized bed reactor design criteria. Four lunar simulants were filmed while fluidizing at lunar gravity for digital analysis of the video data. Analysis revealed that a linear relationship clearly exists between the bubble size and vertical bed position. A key observation was the size of the bubbles is inversely proportional to the particle density of the test solids. Solids with low particle density such as glass beads form larger bubbles than the more dense MLS-1 and Florida ilmenite fractions. Also, a theoretical correlation relating bubble rise velocity as a function of bubble diameter and gravity was verified by these tests. A comprehensive data set now exists making it possible to predict the bubble size and rise velocity of numerous lunar starting materials for fluidized bed lunar oxygen processes.

Another objective has been the understanding of standpipe behavior in fluidized beds at lunar gravity and to provide solids friction factors at 1/6 g for predictive design. A total of 10 flights have been conducted aboard NASA's KC-135 variable gravity airplane to aid in the understanding of this behavior. A variety of materials were tested during these flights including glass beads with air , and Florida ilmenite, and JSC-1 and SMZ-1 lunar soil simulants with helium. The flights proved to be successful and the information obtained was used to improve the test hardware and procedures for successive flights. An empirical formula for solids velocity versus friction factor was determined for the glass beads and air. Since the necessary equations contain the gravity term explicitly, this relationship can be combined with existing 1-g data to properly size standpipes for a lunar multi-stage fluidized bed reactor system.

In addition to mechanical design issues, Carbotek has studied the chemistry and kinetics of terrestrial ilmenite and lunar ilmenite. For the first time, water/oxygen production from Apollo program lunar samples

has been experimentally demonstrated. A 10 gram sample of lunar basalt designated 70035/96 was obtained from NASA and used in seven separate reactivity experiments varying temperature and pressure. The experiments gave extremely encouraging results as all ilmenite was reduced in every experiment. Water production was followed continuously during reduction, and these data were analyzed kinetically. Quantitatively, the data were best represented by a shrinking core process in which both chemical reaction and diffusion play significant roles. Reaction rate showed only a 0.15 order dependence on hydrogen concentration, and rate and diffusion constants were such that lunar ilmenites were initially about twice as reactive as terrestrial ilmenites.

With funding provided by NASA Johnson Space Center, Small Business Innovation Research (SBIR) program, and under the technical guidance of Dr. Thomas Sullivan of JSC, Carbotek initiated an aggressive scale-up of the magma electrolysis process invented by Drs. Haskin and Colson. In this process, lunar feedstock is heated to high temperature (1400°C+) to transform the material into a molten state (magma). Using a platinum/rhodium anode, and an iron cathode, the molten feedstock is electrolyzed in a process similar to that of alumina electrolysis. Oxygen trapped in the mineral phase is liberated and forms as gaseous bubbles on the surface of the anode, proceeding to rise through the melt and burst on the surface. The oxygen is carried out of the electrolysis cell by a stream of inert gas where it is separated and collected as a direct product of the process.

Carbotek's scale-up was approximately one thousand, which was a substantial leap in the technology. Key areas of the design included a large high temperature furnace system with material feed and extraction systems; large Pt/Rh anode ring systems capable of variable position; and an electrolysis power and gas analysis system. These challenges alone proved very difficult, but satisfactory design solutions were achieved.

The major obstacle proved to be melt containment. Due to these unforeseen melt containment problems, actual electrolysis runs were not possible. During this work, it was discovered that spinel (in its current form) is not suitable for containing the synthetic oxide melts. Tests performed at Carbotek and JSC suggest that the melt infiltrates the spinel pore space and grain boundaries. Liquid diffuses into the crucible, grain recrystallization and growth occurs and the spinel loses mechanical strength. As a result the spinel becomes more permeable and does not contain the melt. In order to answer the many questions of long duration melt containment, a new research program should be implemented to research materials problems alone. The basis of this work could then lead to further magma electrolysis tests using the equipment and system designed and confirmed by Carbotek.

Carbotek's current work involves funding from NASA, through SAIC, for the design and operation of a 3 in. single-stage batch fluidized bed system. This will be Carbotek's first kg-scale demonstration of the hydrogen reduction fluidized-bed process. In addition, Carbotek has recently been awarded, with Physical Sciences Inc. (PSI) serving as a subcontractor, a NASA SBIR Phase I contract to couple PSI's solar optical waveguide system with Carbotek's fluidized bed reactor.