REDUCTION OF IRON OXIDES IN SIMULATED LUNAR SOIL USING CARBON AS THE REDUCING AGENT, S.A. Hayes and R.M. Bustin, Lyon College, Batesville, AR 72501 and C.C. Allen, Lockheed Engineering & Sciences Co., Houston, TX 77058

One of the first industrial activities on the lunar base will probably be recovering oxygen from lunar minerals such as oxides. Iron(II) oxide occurring in ilmenite is common in some lunar soils (1). Both hydrogen (2,3) and carbon monoxide (4) have been used as reducing agents for the iron oxides in simulated lunar materials. Lunar ilmenite has also been reduced with hydrogen (5,6). The method described here involves the reduction of iron oxides in lunar simulant using carbon in the form of graphite as the reducing agent. Using MLS-1 simulated lunar soil (7), we have shown that the iron oxides in the soil can be reduced with finely powdered carbon (200 mesh, 99.9999% pure). Although a trace amount of carbon dioxide is produced during the reduction, the majority of the analyzed gas is carbon monoxide. Elemental iron is produced, as evidenced by its magnetic properties and by X-ray diffraction analysis. Samples analyzed by XRD showed no evidence of iron(II) or iron(III) forms but only elemental iron, indicating that the iron oxides had been completely reduced.

The reaction system includes a stainless steel vacuum rack, manometers, a tube furnace, a quartz reaction chamber, and a gas chromatograph equipped with a gas injection valve to analyze the gases evolved during reaction. The MLS-1 and carbon are weighed and mixed thoroughly. The mixture is dried at 120° to release any adsorbed moisture and is transferred to a sample boat which is placed in a large quartz tube. The tube is attached to the sampling line and evacuated. When a pressure of 35 microns is reached, the sample is heated in a tube furnace. At the end of the heating cycle, the evolved gases are cooled to room temperature and injected into the gas chromatograph. The system is calibrated using pure carbon monoxide and pure carbon dioxide. After trying a number of different kinds of sample boats, small quartz boats (pieces of half-inch quartz tubing about two inches in length) have been found to be the most desirable. They do not produce a blank, and they do not react with the sample or with carbon.

In an effort to optimize reaction conditions; studies were done on temperature, relative amounts of MLS-1 and carbon, and heating times. There is no measurable reaction at temperatures below 1100°C, and at 1200°C all the iron oxides appear to be reduced under optimum conditions. (See Figure 1.) Higher temperatures could not be checked because the furnace used in these experiments cannot be run at temperatures greater than 1200°C. To obtain a high efficiency, a large excess of carbon must be used. (See Table 1.) During long heating times (up to four hours), a small amount of gas continues to be released even after all the iron oxides have been decomposed. (See Figure 2.) This same phenomenon was observed during the hydrogen reduction of both simulated lunar soil and actual lunar material (3,5). One explanation could be that the titanium in the ilmenite, $FeTiO_3$, is being slowly reduced and that it goes through several different oxidation states during the course of the reaction, producing different oxides and releasing oxygen (5).

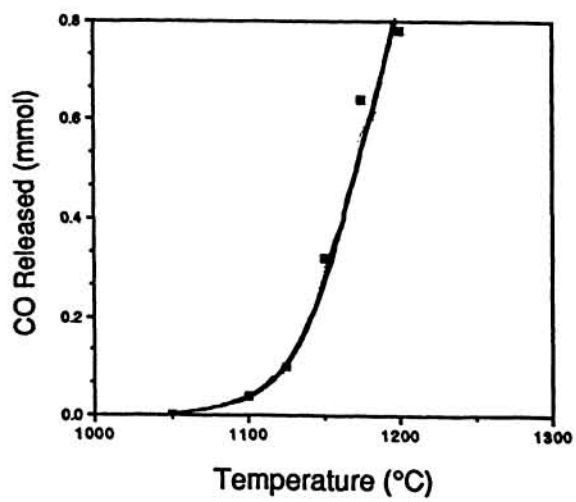

Figure 1. Production of carbon monoxide at different temperatures. Each experiment ran for 30 minutes and used 0.500 g MLS-1 and 0.100 g carbon.

CARBON REDUCTION OF IRON OXIDES IN LUNAR SIMULANT: Hayes S. *et al.*

This research shows that carbon can be used to completely reduce the iron oxides in simulated lunar soil to elemental iron. Research is currently underway to change the procedure so that carbon dioxide (rather than carbon monoxide) will be the product of the reduction. This would allow oxygen to be produced from the carbon dioxide using an electrochemical cell such as that described by Sridhar and Kaloupis (8). Efforts are also underway to construct a system which can accomodate much larger amounts of soil.

Amount of Carbon (grams)	CO Produced (mmols)
.020	.39
.025	.53
.050	.62
.100	.77

Table 1. Production of carbon monoxide from experiments using different amounts of carbon with 0.500 g MLS-1 at 1200°C.

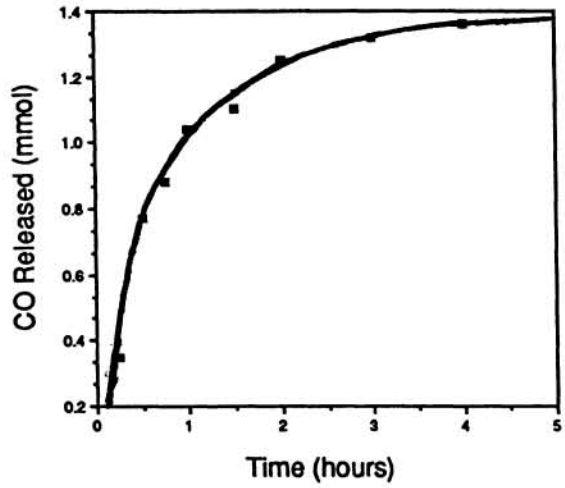

Figure 2. Production of carbon monoxide from experiments of different durations. Each experiment used 0.500 g MLS-1 and 0.100 g carbon and was carried out at 1200°C. Complete reduction of the FeO and Fe_2O_3 in the MLS-1 would produce 1.20 mmol of carbon monoxide.

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