REDUCTION OF LUNAR MARE SOIL AND PYROCLASTIC GLASS
C.C. Allen, Lockheed ESC, Houston, TX R.V. Morris and D.S. McKay, NASA/JSC, Houston, TX

Mare soil and orange and black pyroclastic glass were reduced in hydrogen gas at temperatures of 900-1100°C. The experiments support studies of regolith maturation, lunar volcanism, and the production of oxygen on the Moon. The most reactive component in the high-Ti soil was FeO in ilmenite, which was completely reduced to iron metal at all temperatures. Vitreous orange glass crystallized and was partially reduced to iron metal, pyroxene, and minor olivine. Initially devitrified black glass was similarly reduced and converted to iron metal, pyroxene, and minor olivine. The degree of reaction in both glasses increased with temperature.

Lunar Samples. Sample 75061 is a high-Ti mare soil which contains pyroxene, plagioclase, ilmenite, minor olivine, agglutinates, and a trace of glass (1). Sample 74220 is the Apollo 17 "orange soil," an essentially pure pyroclastic glass deposit (1). Orange spheres, which comprise the bulk of this sample, are completely glassy. Black spheres are the same glass partly recrystallized to olivine, ilmenite, and spinel.

Experimental/Analytical. The samples were reduced in a vertical tube furnace, with weight change continually monitored, as in previous tests with lunar simulants (2). All samples were run "as received." Samples (200-300 mg) were placed into the hot furnace and reduced in flowing hydrogen for three hours. The fO2 was kept below the iron-wüstite buffer.

Results - Mare Soil. Lunar mare soil 75061 was reduced in three experiments at 900-1050°C. Sample weights decreased rapidly for the first 20 minutes and then fell more slowly, remaining essentially constant after the first hour. The total weight losses ranged from 2.6-3.0 wt.%, increasing slightly with increasing temperature.

Most of the weight loss was due to reduction of ferrous oxide to iron metal, with concomitant release of oxygen. This sample contained FeO in several phases, including pyroxene, ilmenite, olivine, and agglutinates and pyroclastic glass. The VSM data indicated that 47-54% of the Fe2+ in the starting sample was reduced to alpha iron metal. This was equivalent to the loss of 1.9-2.2% of the starting sample weight as oxygen. The XRD and FeMS spectra (Fig. 1) show that a portion of the remaining Fe2+ was reduced to gamma iron in experiments above the transition temperature of 911°C.

The most reactive phase in this Ti-rich soil was ilmenite. In our experiments the Fe2+ in ilmenite was completely reduced. No ilmenite remained in these samples, to the detection limit of FeMS and XRD. All ilmenite grains examined by SEM showed characteristic phase separation and reduction textures (Fig. 2). Other FeO-bearing phases showed much less evidence of reduction. Olivine grains exhibited small-scale reduction textures in SEM images. Olivine peaks in the FeMS spectra became smaller with increasing temperature. Agglutinitic glass underwent partial reduction, with increasing concentrations of iron metal particles as reduction temperature increased from 900 to 1050°C. Minor reduction of pyroxene was documented in the 1050°C experiment by SEM and FeMS.

These results are in close agreement with data from previous reduction experiments utilizing crushed lunar basalt (3). They also provide an extreme "end member" for studies of reduction as a cause of lunar soil maturation.
Results - Pyroclastic Glass. Pyroclastic glass 74220 was reduced in four experiments at 900-1100°C. Weight loss was rapid for the first 10-20 minutes, and then became more gradual. Total losses ranged from 2.2-5.4 wt.%, increasing steadily with temperature.

The XRD and FeMS spectra of all reduced glass samples were dominated by the peaks of iron metal (Fig. 3). The metal occurred as micrometer-scale blebs scattered throughout each glass particle (Fig. 4). The VSM data indicated that 32-68% of the Fe²⁺ in the starting sample was reduced to alpha iron, with the percentage increasing with temperature. This was equivalent to the loss of 1.7-3.3% of the starting sample weight as oxygen. A significant portion of the remaining Fe²⁺ formed gamma iron.

Orange glass underwent devitrification in all of the experiments. Pyroxene crystals grew steadily with temperature, from less that 1 μm across in the 900°C sample to well over 10 μm at 1100°C. At this temperature the orange glass was totally devitrified to pyroxene and iron metal, plus minor olivine and titanium oxide (Fig. 4). Black glass spheres also underwent striking changes. At higher temperatures ever greater amounts of FeO in the glass and crystals were reduced to metal. Ilmenite was reduced to iron plus titanium oxide and olivine to iron plus pyroxene. In the 1100°C sample, spheres which were initially crystalline were almost indistinguishable from those which were initially vitreous.

Reduction and devitrification experiments on orange and black glass provide constraints on conditions during lunar pyroclastic eruptions. They also support the contention that pyroclastic glass would be an ideal feedstock for lunar oxygen production (4).


Figure 1. FeMS spectrum of mare soil 75061, reduced at 1050°C

Figure 2. Ilmenite from mare soil 75061, reduced at 1050°C

Figure 3. FeMS spectrum of pyroclastic glass 74220, reduced at 1100°C

Figure 4. Pyroclastic glass from 74220, reduced at 1100°C