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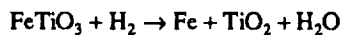
LUNAR RESOURCES—OXYGEN FROM ROCKS AND SOIL. C. C. Allen¹, M. A. Gibson², C. W. Knudsen², H. Kanamori³, R. V. Morris⁴, L. P. Keller⁴, and D. S. McKay⁴, ¹Lockheed Engineering and Sciences Company, Houston TX 77058, USA, ²Carbotek Development Laboratories, Houston TX 77084, USA, ³Shimizu Corporation, Space Project Office, Tokyo, Japan, ⁴NASA Johnson Space Center, Houston TX 77058, USA.

Introduction: Permanent human habitation of the Moon will require the use of local resources because of the extremely high cost of transporting materials from the Earth. One of the first lunar resources to be exploited will be oxygen, for both life support and rocket propulsion [1]. Projected demand for oxygen at a lunar base ranges from tons to hundreds of tons per year.

Among the major-element oxides in lunar materials, the iron-oxygen bond is most easily broken. Some oxygen production proposals concentrate on releasing only oxygen bound to iron. Studies of the TiO₂ system, however, have shown that a portion of the oxygen normally required to make TiO₂ can also be released under highly reducing conditions. Some of the Ti⁴⁺ is reduced to a lower oxidation state, presumably Ti³⁺.

Essentially all the iron and titanium oxide in the lunar regolith is concentrated in three mineral groups: metal oxides (ilmenite and rutile), pyroxene, and olivine. Studies of terrestrial minerals show that the high-temperature reduction of iron oxide in ilmenite is rapid and essentially complete [2]. Pyroxene, olivine, and rutile can also be partially reduced. Compared to ilmenite, the reduction of these minerals is slower and less efficient [3].

The "baseline" process for lunar oxygen production involves reduction of ilmenite [2]. A typical reaction, using hydrogen as a reducing agent, is



The water is decomposed to yield oxygen and the hydrogen is recycled as a reactant.

Most scenarios for oxygen production envision reaction of the lunar soil rather than the rocks. Lunar soil samples show evidence of limited natural reduction in the form of iron metal blebs on and within grains of agglutinate glass [1]. If the impact-driven maturing process has subjected the ilmenite or other minerals in lunar soil to significant reduction, mature soils could be undesirable feedstocks for an oxygen plant.

Experimental: *Lunar and terrestrial basalt.* The first set of hydrogen reduction experiments to use actual lunar material has recently been completed. The sample, 70035, is a coarse-grained vesicular basalt containing 18.46 wt% FeO and 12.97 wt% TiO₂ [4]. The mineralogy includes pyroxene, ilmenite, plagioclase, and minor olivine. The sample was crushed to a grain size of <500 μm.

The crushed basalt was reduced with hydrogen in seven tests at temperatures of 900°–1050°C and pressures of 1–10 atm for 30–60 min [2]. A capacitance probe, measuring the dew point of the gas stream, was used to follow reaction progress.

Experiments have also been conducted using a terrestrial basalt similar to some lunar mare samples. Minnesota Lunar Simulant (MLS-1) contains 13.29 wt% FeO, 2.96 wt% Fe₂O₃, and 6.56 wt% TiO₂ [5]. The major minerals include plagioclase, pyroxene, olivine, ilmenite, and magnetite. The rock was ground and sieved, and experiments were run on the <74- and 500–1168-μm fractions. Experiments were also conducted on <74-μm powders of olivine, pyroxene, synthetic ilmenite, and TiO₂ [3].

The terrestrial rock and mineral samples were reduced with flowing hydrogen at 1100°C in a microbalance furnace, with reaction progress monitored by weight loss. Experiments were run at at-

mospheric pressure for durations of 3–4 hr. Solid samples from both sets of experiments were analyzed by Mossbauer spectroscopy, petrographic microscopy, SEM, TEM, and XRD.

Mature lunar soil. Apollo 17 soil 78221 was examined for evidence of natural reduction in the lunar environment. This sample was chosen based on its high maturity level ($I_p/\text{FeO} = 93.0$). The FeO content is 11.68 wt% and the TiO₂ content is 3.84 wt% [4]. A polished thin section of the 90–150-μm size fraction was analyzed by petrographic microscopy and SEM. **END**

Results: *Lunar and terrestrial basalt.* Reduction of the lunar basalt samples was extremely rapid, with major evolution of water occurring within minutes after the introduction of hydrogen. Mossbauer analysis of the sample reduced at the lowest temperature, 900°C, showed complete reduction of the Fe²⁺ in ilmenite to iron metal.

All the reduced ilmenite examined by SEM displayed phase separation and reduction throughout. Samples reduced at 1000° and 1050°C showed evidence of iron migration to grain surfaces, while the single 900°C sample did not show such evidence.

One olivine crystal in the sample reduced at 1050°C displayed submicrometer iron blebs at the surface and along interior cracks, indicating partial reduction. Other olivine and pyroxene grains in the same sample showed no such evidence. No sign of olivine nor pyroxene reduction was found in samples run at 1000° or 900°C. High-resolution TEM photographs of ilmenite from the 1050°C experiment showed reduction of the TiO₂ to Ti₆O₁₁.

The reduction of MLS-1 basalt by hydrogen at 1100°C was rapid, with over 90% of the weight loss occurring in the first 10 min. The XRD data showed that the reduction of iron oxide in ilmenite and magnetite was essentially complete. The ilmenite displayed 1–20-μm blebs and stringers of iron metal in a titanium oxide matrix. Iron metal was also concentrated on ilmenite grain surfaces. Most ilmenite, even crystals located hundreds of micrometers from grain edges, showed this texture.

Pyroxene and olivine reduced in hydrogen and examined by SEM displayed evidence of very limited reduction. Submicrometer iron blebs were sparsely scattered over surfaces and along internal fractures. Analysis of crystal interiors demonstrated, however, that most of the iron in these minerals was not reduced.

Reduction of TiO₂ was also observed. After three hours at 1100°C the material was completely reduced to Ti₄O₇. The Ti₄O₇ was also a major phase, along with iron metal, in synthetic ilmenite reduced under the same conditions.

Mature lunar soil. Soil sample 78221 was searched for evidence of ilmenite and olivine reduction. The results were negative for the size range examined (90–150 μm). Isolated ilmenite and olivine crystals were texturally and chemically homogeneous to the resolution limit of the SEM. The same was true of crystals incorporated within agglutinates, even though the agglutinate glass contained submicrometer blebs of metallic iron. Thus, even in this very mature soil, oxygen has apparently not been released from ilmenite by natural processes.

Output from a Lunar Oxygen Plant: These first reduction tests with actual lunar material allow estimation of the output from an oxygen plant. The FeO in ilmenite was totally reduced in the 900°C test, and reduction appeared to be complete at higher temperatures also. The oxygen yield from this reduction is 10.5% of the ilmenite mass, or 20.0% of the mass of TiO₂ in this mineral.

The TiO₂ formed by ilmenite reduction, as well as that found as rutile in lunar basalts, can itself be reduced to Ti₆O₁₁ at 1050°C and to Ti₄O₇ at 1100°C. This process yields a maximum of 5.3% of the TiO₂ mass as oxygen.

Small degrees of FeO reduction occur in terrestrial olivine and pyroxene at 1100°C and in lunar olivine at 1050°C. No evidence of

reduction was detected at lower temperatures. We consider these contributions to the oxygen yield to be negligible.

The predicted output from a lunar oxygen plant thus depends on the ilmenite and TiO_2 contents of the soil. In most lunar soils almost all the TiO_2 is incorporated in ilmenite [1]. The maximum oxygen yield therefore will equal 20% of the TiO_2 content if only ilmenite is reduced, and 25% if further conversion to Ti_4O_7 occurs. Lunar soil 78221 contains 3.84 wt% TiO_2 . The maximum predicted oxygen output from a plant using this feedstock is just under 1% of the total input mass. The output from a high-Ti soil such as 75061, with 18.02 wt% FeO and 10.38 wt% TiO_2 [4], is 2.6%.

Concentration or beneficiation of ilmenite would increase the process yield, but not the overall yield. An output of 2.6% means that 38 tons of lunar soil would be required to produce one ton of oxygen. By terrestrial standards this is a small amount of feedstock. A single medium-sized dump truck can hold 40 tons and can be loaded in under 10 min with a front-end loader [6].

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NORTH MASSIF LITHOLOGIES AND CHEMICAL COMPOSITIONS VIEWED FROM 2–4-mm PARTICLES OF SOIL SAMPLE 76503. Kaylynn M. Bishop, Bradley L. Jolliff, Randy L. Korotev, and Larry A. Haskin, Department of Earth and Planetary Sciences and McDonnell Center for the Space Sciences, Washington University, St. Louis MO 63130, USA.

In this work, we identify the lithologic and compositional components of soil 76503 based on INAA of 243 2–4-mm particles and 72 thin sections from these and associated 1–2-mm particles (76502) [1]. We present a statistical distribution of the major compositional types as the first step of a detailed comparative study of the North and South Massifs. The soil sample was collected well away from any boulder and is more representative of typical North Massif material than any single large rock or boulder sample. So far, our examination of the 76503 particles has provided a better definition of precursor igneous lithologies and their petrogenetic relationships [2]. It has enabled us to refine the nature of mixing components for the North Massif <1-mm fines [3]. It has confirmed the differences in lithologies and their proportions between materials of the North and South Massifs; e.g., the North Massif is distinguished by the absence of a 72275-type KREEP component, the abundance of a highly magnesian igneous component, and the absence of certain types of melt compositions found in the South Massif samples.

Results: On the basis of chemical compositions and binocular microscope observations, sample 76503 comprises 30 wt% dark glassy-matrix breccias, regolith breccias, and agglutinates; 29% highland igneous fragments and granulitic breccias; 24% noritic melt breccias; 13% high-Ti mare basalt; 1.5% orange glass regolith breccias and vitrophyre, 0.4% (1 particle) VLT basaltic breccia, and 2% unclassified.

Impact melt lithologies (noritic breccias) are rich in incompatible trace elements (ITE) (Fig. 1) and include very fine-grained crystalline and poikilitic impact-melt breccias, glassy matrix breccias, and regolith breccias and agglutinates that include only impact melt breccia lithologies. The latter may have developed in the regolith higher on the North Massif or prior to the introduction of mare materials into the soil. On the basis of Sc, Cr, Sm, and Eu concentrations, noritic melt lithologies from 76503 and matrices from station 6 and 7 boulders differ significantly from those of stations 2 and 3, except boulder 2, station 2. Among particles from sample 76503, evidence of more than one melt group is lacking (Fig. 2). Most of the melt breccias are tightly clustered compositionally and fall within the field of North Massif melt breccia compositions defined by analyses from the literature (Fig. 2). Those melt breccias having compositions outside this field contain clasts of highland material having low concentrations of ITEs; thus their compositions are displaced toward those of highland igneous lithologies and granulitic breccias.

Highland lithologies that have low ITE concentrations include fragments of shocked and unshocked anorthositic troctolite, anorthositic norite, gabbroic anorthosite, and granulitic breccias of generally anorthositic-norite or anorthositic-gabbro compositions. Coarse single crystals or clumps of several crystals of plagioclase are common in the 2–4-mm range. These are compositionally very similar to plagioclase in 76535 troctolite [4]; however, we believe these, and perhaps 76535 also, are members of a more anorthositic body [2]. We find no igneous particles whose compositions suggest affinity to ferroan-anorthositic suite igneous rocks. Granulitic breccias are generally more pyroxene rich than the samples having igneous textures, and, although they have low ITE concentrations, many are substantially contaminated by meteoritic siderophile elements.

Observations and Implications: Below, we summarize some important features of the distribution of lithologies and compositions of particles in 76503 by comparison to the model distribution of components determined for station 6 <1-mm soil by [3]. Several of these features distinguish this soil from soils of the South Massif. (1) The mass-weighted average composition of the regolith breccias and agglutinates is very similar to the average composition of the station 6 <1-mm fines [3] (Fig. 2). (2) The proportions of components that have been used to model the station 6 soil [3] are similar to the proportions of groups we find in sample 76503 (i.e., the regolith breccias and agglutinates can be well accounted for as a mixture of observed mare basalt and orange glass fragments, noritic melt breccias, and ITE-poor highland lithologies).

The <1-mm fines can be modeled as 51% highlands [36% anorthositic norite and 14% MG component (norite/troctolite mix)], 21% noritic breccia, 21% mare basalt, and 6% orange glass, whereas the proportions of fragments in sample 76503 are 43% highlands, 34.5% noritic breccias, 19% mare basalt, 2% orange glass, and 0.6% VLT basalt (by mass on an agglutinate/breccia-free basis). (3) The proportion of noritic breccias in 76503 exceeds that determined as a mixing component in <1-mm fines by [3]; however, we have included in our particle count noritic breccias whose compositions are skewed toward ITE-poor highland compositions (see Fig 1). Therefore, a portion of the "MG" and "AN" highland components of [3] is taken up in our proportion of noritic breccias. This portion consists of mineral and lithic clasts that, on average, have a composition similar to magnesian granulite or magnesian anorthositic norite [2]. (4) More orange glass was found in the fines model than in the 2–4-mm particles because orange glass particles have a mean size of 40 μm [5] and so concentrate in the finer soil fractions. Particles with orange glass composition in sample 76503 were orange/black glass regolith breccias, not large, individual glass spheres. (5) Magnesian troctolitic