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# PART 2—Beneficiation and Extraction of Nonterrestrial Materials

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The group that reviewed options for processing nonterrestrial materials was dominated by industrial materials scientists who tried to identify which processes utilizing space materials could be implemented in the near term.

The most practical process seemed to us to be the extraction of lunar oxygen and the extraction of metals and ceramics from the residues of the reduction process. The growth of space activity will be accompanied by increased demand for liquid oxygen for rocket propellant. In particular, any lunar base activity will require tens of tons of oxygen for each round trip to the Moon. And, of course, the oxygen and the intermediary product water will be needed for life support at the base. The reduced metals and ceramics may be considered byproducts or may develop into primary products. Some of the same processes would be directly applicable to recovery of products from asteroids. We also discussed other processes for directly utilizing asteroid metals.

## Beneficiation and Oxygen Extraction Methods

Reduction of lunar ilmenite with hydrogen imported from Earth was judged to be an oxygen extraction option that could be implemented in the near term. Ilmenite, an iron and titanium oxide, is the most abundant oxide in the samples that have been brought back from the Moon.

Working for Lockheed Corporation at the Johnson Space Center, I reported the successful concentration of ilmenite in an Apollo 11 soil sample, using an electrostatic separator based on a commercial design and operated both in nitrogen and in a vacuum. This was the first research reported on the industrial behavior of actual lunar material. Additional research is needed to determine the characteristics of a system that could operate on the Moon.

A process not requiring beneficiation, because it extracts oxygen from the predominant silicates, was probably the first process considered for extraction of oxygen from lunar materials. The carbothermal process was developed by Sanders Rosenberg and colleagues at Aerojet-General Corporation in the mid-1960s, before we had been to the Moon. They assumed that ordinary rockforming minerals, such as olivine and pyroxene, would be abundant, an assumption that proved to be mostly correct. Rosenberg and his colleagues performed a series of experiments demonstrating actual oxygen extraction from simulated lunar materials. In addition, they designed an oxygen production plant and did a parametric analysis

of mass, power, and cost. David McKay has combined several of their papers from that period and updated the cost analysis to 1989 dollars. This combined paper presents the basic concepts of the carbothermal process, gives the results of some of the laboratory experiments, and includes the design concept for a lunar oxygen plant. The paper is interesting both because of its historical value in presenting a lunar oxygen plant designed before Apollo 11 and also because the basic concept is still viable today as a candidate oxygen plant for a lunar outpost in the early 21st century.

In a review of proposed lunar oxygen production processes, including carbothermal reduction and electrolysis of basalts. Christian W. Knudsen and Michael A. Gibson, of Carbotek, Inc. (Houston), have concluded that hydrogen reduction of ilmenite is the simplest process proposed. Products of the reaction are iron, titanium dioxide, and water; oxygen is then extracted from the product water by electrolysis. Both batch and continuous-flow fluidized-bed processes for the reaction have been described. Although the preliminary results of bench-level tests on the batch process conducted by Richard J. Williams at JSC seemed promising, further engineering work by Knudsen and Gibson indicates to them that only a continuous process is practicable on a large scale.

Knudsen and Gibson also considered hot pressing of the metallic iron and titanium dioxide residues of the reaction into cermet parts and bricks, as outlined by Agosto (1981).

Russell O. Colson and Larry A. Haskin discuss the direct electrolysis of molten lunar material to produce oxygen. In the magma electrolysis process. iron, silicon, or iron-silicon alloys are produced at the cathode and oxygen is produced at the anode. Potential byproducts include ceramics (spinels) and cast-rock products such as bars, beams, and sheets. Colson and Haskin argue that, compared to most other proposed processes, this process requires less energy per unit of oxygen and has the advantage of being relatively simple. Technology challenges include finding container and electrode materials that will withstand the corrosiveness of molten silicates. The work of a number of years has determined some of the fundamental properties of melt conductivity and some of the factors affecting the efficiency of oxygen production.

Solar furnace pyrolysis of lunar basalts in vacuum, as proposed by Elbert A. King and me (1983), is considered another highly promising process for nonterrestrial oxygen production. It does not require reagents imported from Earth. King (1982) demonstrated the process on Earth using terrestrial basalts and samples of the Murchison meteorite heated to approximately 3000°C in a furnace with a solar mirror 2 meters in diameter. Residues of metallic iron and oxides of aluminum, calcium, and titanium indicated the evolution of oxygen and volatile oxides of other elements. A bench-level research program is required to characterize, quantify, separate, and capture the oxygen and other volatiles liberated by the process. Residues of the process include metals (iron), semimetals (silicon), ceramics (Al-Ca-Ti oxides), and feedstocks rich in aluminum oxide for aluminum electrolysis.

Wolfgang H. Steurer, of the Jet Propulsion Laboratory, considered two vapor-phase processes: (1) the volatilization of oxygen by vacuum pyrolysis of oxides and (2) electrostatic separation of metals from high-temperature plasmas of nonterrestrial materials. The high temperatures and reactivities of such processes suggest that the technology will be difficult to develop. However, such processes may prove to be effective.

#### Metallurgy

David F. Bowersox, of Los Alamos National Laboratory, has described a novel anhydrous chloride process, used in the nuclear industry to recover plutonium, which could be adapted to extract iron and titanium from nonterrestrial basalts and ilmenite. All reagents and products of the process are recycled and, because it is waterless, the system is one-tenth the size of an aqueous system with the same metal output. It has the disadvantage of being a metal extraction process that does not directly yield oxygen or water, and it requires chloride salts, which are rare or absent on the Moon. However, as a successful operating anhydrous system that recycles all reagents and products, it merits serious consideration for nonterrestrial application.

Karl R. Johansson has reviewed the literature and found several bioprocesses for the beneficiation of lunar and asteroidal materials by the action of microorganisms. Notably, the extraction of metals by (1) oxidation-reduction reactions, (2) acid leaching, (3) pH alteration, (4) organic complexing, and (5) cellular accumulation of metals due to the action of bacteria on minerals. All these bioprocesses would require stringent radiation and temperature controls in closed aqueous environments having elements in which the Moon is deficient, like carbon, nitrogen, and hydrogen. However, Karl says that the process of microbe-enhanced vat leaching, which is used terrestrially to concentrate copper ores, might be applicable to extracting common lunar metals

like iron and manganese from lunar rocks and soils. In addition, bioaccumulation of metals by microbial cells might be used to concentrate rare (and often toxic) elements like copper, lead, mercury, cadmium, and silver and remove them from biological systems on the Moon. In his paper Karl also mentions metal reduction by anaerobic bacteria, the uptake of silicates by diatoms, and a tantalizing claim in the Russian literature of "silicate bacteria" that concentrate aluminum oxide by freeing silicates from aluminosilicate ores. However, human settlements and early lunar industrial operations would probably have to be well established before controlled bioprocessing systems could be set up in nonterrestrial locations.

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If aqueous processing were to prove practicable, then leaching of useful elements from lunar and asteroidal materials by inorganic acids like hydrofluoric acid, without the introduction of microorganisms, might well be a more direct and productive method of extracting most of the major lunar elements (oxygen, silicon, aluminum, iron, magnesium, titanium) as well as many of the minor ones (sodium, potassium, manganese, chromium, phosphorus). See Criswell (1980).

Metallurgist Constance F. Acton has critically reviewed proposed processes for metal production, using the harsh criterion of

terrestrial commercial viability. Iron in lunar soil may be the most easily obtained metal resource on the Moon through the low energy extraction techniques of magnetics and electrostatics. Its physical extraction presents many challenges, but it is likely to be one of the easier objectives for nearterm lunar resource development (Agosto 1981). Acton points out the need for hard thermodynamic and kinetic data on all the reactions and the necessity for long-term bench-level testing and pilot plant facilitation of the most promising processes. She outlines the extensive effort required to prove a commercial metallurgy process through thermodynamic and kinetic chemical evaluation, bench-level feasibility studies, and pilot plant operations. Her assessments, based on terrestrial experience, of long lead times (20-50 years) and high R&D expenditures (hundreds of millions of dollars) to develop systems are challenges to the designer of lunar materials processing technology. However, the long lead times presume development of processes competitive with those of terrestrial suppliers. That assumption does not apply to the initial provision of products from lunar sources for use in orbit.

John Lewis, of the University of Arizona, presented to our group information on the gaseous carbonyl process. Carbon monoxide is reacted with metal at low temperature to purify it or separate one metal from another. With a relatively low energy input, lunar metallic iron might be purified or trace elements, particularly platinum group metals, could be separated from asteroidal metal. Although considerable terrestrial experience is in hand, adaptation to the space environment remains a challenge for future investigation.

#### **Nonterrestrial Cements**

T. D. Lin, of Construction Technology Laboratories (CTL), the research arm of the Portland Cement Association, proposed to our workshop aroup the manufacture of cement from lunar oxides and in his paper proposes concrete as a building material for a space station and a lunar base. The major constituents of common types of cement occur in lunar highland anorthosites and lunar mare basalts. The high compressive strength and the mass of lunar-derived concrete would make it an effective shield against radiation and micrometeorite impacts and thus a candidate material for orbital and lunar structures. Concrete is fireproof, lends itself to modular construction, and can be reinforced with Moonderived metals and fiberglass to improve its tensional and flexural strength. Lunar cement would also be useful as mortar to assemble

building blocks of other materials, whether imported or nonterrestrial. Common concrete mixtures are about 10 percent water by weight, but drier formulations can be developed and the water can be recovered as the concrete dries. In any case, typical concretes, which consist of 2/3 to 3/4 aggregate materials bonded by cement, retain only 5 percent water when thoroughly dried, which corresponds to only a few tenths of a percent of Earth-derived mass in the form of hydrogen (Cullingford, Keller, and Higgins 1982). Suitable lunar aggregates could readily be obtained by crushing and grading rocks from the lunar surface.

I have proposed a method for concentrating lime (CaO) in lunar materials to Portland cement formula levels, using phosphates that might be lunar derived. A similar process was proposed by Ellis M. Gartner, of CTL, using terrestrial phosphate. Construction Technology Labs received 40 grams of lunar soil from the Johnson Space Center in 1986: from it CTL fabricated a lunar mortar sample, which was tested and proved usable. NASA is interested and the project has attracted favorable attention from the press. The apparent widespread interest in cement as a lunar product and public recognition of it may generate substantial support for its development.

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The consensus of the group working on beneficiation and extraction of nonterrestrial materials was that there is a near-term need for bench-level data on lunar and meteoritic materials processing. such as (1) beneficiation of industrially valuable minerals in lunar soils and disaggregated lunar rocks and meteorites; (2) oxygen and metal extraction processes, like carbothermal reduction of silicates, hydrogen reduction of ilmenite, magma electrolysis, vacuum pyrolysis, and anhydrous chloride reduction, using actual nonterrestrial samples; and (3) formulation of cementitious compounds from lunar oxides and aggregates. The above research should be conducted under conditions approximating, as closely as practicable, the expected operating environment. This work is necessary to make a credible case for nonterrestrial materials utilization to the materials science community.

#### **Recommendations**

 Support for a thorough benchlevel hardware investigation and demonstration of the beneficiation, primarily by electrostatic and magnetic means, of lunar soils and crushed rocks for minerals like ilmenite, anorthite, and pyroxenes, and valuable minor phases like metal, chromite, and phosphate.

- 2. Support for bench-level research and development of the carbothermal process, including additional testing to determine optimum pressure and temperature conditions for feedstocks of various compositions, such as simulated mare basalt and concentrates of ilmenite or pyroxene.
- Support for a thorough investigation of the thermodynamics and kinetics of hydrogen reduction of lunar ilmenite, together with a benchlevel laboratory research project to investigate the workability of hardware designs like those of Richard J. Williams and Christian W. Knudsen and Michael A. Gibson.
- Support for additional studies of magma electrolysis, including laboratory work on the basic process, development of innovative electrodes and containers, and investigation of the effects of feedstock of different compositions. This effort should also include engineering evaluations and plant design concepts.
- Support for a thorough investigation of the thermodynamics and kinetics of solar furnace vacuum pyrolysis of nonterrestrial materials. The effort should

include a bench-level research project to characterize, quantify, separate, and capture oxygen and other volatiles liberated by hightemperature vacuum pyrolysis of lunar and meteoritic materials, as well as thorough characterization and chemical analysis of resulting condensates and residues.

- A literature search and evaluation of the anhydrous chloride process for plutonium reclamation reported by David F. Bowersox and its applicability to production of metals and ceramics from nonterrestrial materials.
- Support for bench-level research on aqueous leaching of nonterrestrial silicates with inorganic acids like hydrofluoric acid, as well as beneficiation by bioprocesses derived from the action of microorganisms on nonterrestrial minerals.
- 8. A study of the application of the carbonyl process to the purification and extraction of lunar and asteroidal ferrous metals.
- Support for the development of cement formulations from lunar materials and cement and concrete fabrication processes adapted to lunar and orbital manufacture and applications.

### References

Agosto, W. N., and E. A. King. 1983. In Situ Solar Furnace Production of Oxygen, Metals and Ceramics From Lunar Materials. In Lunar & Planetary Sci. XIV, Special Session Abstracts: Return to the Moon (March 16) and Future Lunar Program (March 17), pp. 1-2. Houston: Lunar & Planetary Inst.

Agosto, William N. 1981. Beneficiation and Powder Metallurgical Processing of Lunar Soil Metal. In Space Manufacturing 4, Proc. 5th Princeton/AIAA Conf., ed. Jerry Grey and Lawrence A. Hamdan, 365-370. New York: AIAA.

Criswell, David R. 1980. Extraterrestrial Materials Processing and Construction. Final Report on Mod. No. 24 of NASA contract NSR 09-051-001. Houston: Lunar & Planetary Inst.

Cullingford, H. S.; M. D. Keller, and R. W. Higgins. 1982. Compressive Strength and Outgassing Characteristics of Concrete for Large Vacuum-System Construction. J. Vac. Sci. Technol. **20**:1043-1047.

King, Elbert A. 1982. Refractory Residues, Condensates and Chondrules From Solar Furnace Experiments. Proc. 13th Lunar & Planetary Sci. Conf., Part 1. J. Geophys. Res. **87** (suppl.): A429-A434.