NASA Contract R-09-040-001

# MULTIDISCIPLINARY RESEARCH LEADING TO UTILIZATION OF EXTRATERRESTRIAL RESOURCES

Quarterly Status Report
January 1, 1968 to April 1, 1968

U. S. Bureau of Mines NASA Program of Multidisciplinary Research
Leading to Utilization of Extraterrestrial Resources

QUARTERLY STATUS REPORT January 1, 1968 to April 1, 1968

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STATUS REPORT THIRD QUARTER FISCAL YEAR 1968

U. S. Bureau of Mines NASA Program of Multidisciplinary Research Leading to Utilization of Extraterrestrial Resources

April 1, 1968

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|---|---|---|
| Task title: | Core group activity | |
| Investigator: | Thomas C. Atchison, Senior Research Scientist | |
| Location: | Twin Cities Mining Research Center<br>Minneapolis, Minnesota | |
| Date begun: | April 1965 | To be completed: Continuing |
| Personnel: | Thomas C. Atchison, Supervisory Research Physicist<br>David E. Fogelson, Supervisory Geophysicist<br>Clifford W. Schultz, Metallurgist<br>James Paone, Supervisory Mining Engineer<br>Other Bureau personnel, as assigned | |

## PROGRESS REPORT

### Objective

The objective is to provide the basic scientific and engineering knowledge needed for subsequent development of an extraterrestrial mineral resource extraction, processing and utilization technology for supporting and enhancing the economy of manned lunar and planetary missions.

### Progress During the Third Quarter

The core group continued to obtain, study, and distribute information of importance to the research program. Special effort was placed on analyzing the impact of new information from the lunar probes on the Bureau's research. Atchison participated in a symposium on the Interpretation of Lunar Probe Data at McDonnell Douglas Advanced Research Laboratories, Huntington Beach, Calif., in January.

Chemical composition information from Surveyor V, VI, and VII indicate the Moon is a differentiated body, probably having a hot interior. The lunar surface seems to be composed of a variety of materials with characteristics quite like many earth materials. The trenching devices of Surveyor III and VII demonstrated the existence of lunar surface material similar to very fine particle sandy soil, although somewhat more cohesive than most earth soil of comparable particle size and dryness. Lunar Orbiter pictures reveal a wide variety of topographical features, some undoubtedly caused by meteorite impact, some very characteristic of volcanic activity, and some that indicate other types of tectonic activity. The Surveyor and Orbiter pictures together show evidence of some weathering at scales from the very small to the very large. Theoretical considerations revising upwards the probable lifetime of the lunar atmosphere, together with observations of some topographical features of the lunar surface, are giving rise to a small but growing body of opinion that water in one form or another may be more plentiful than previously believed possible.

In general the lunar probe results confirm the need for information of the type being developed by the Bureau on the properties and behavior of a variety of rocks and minerals that might be excavated, handled, transported, stored, and used in the lunar environment.

A paper was presented in February at the Annual Meeting of the Working Group on Extraterrestrial Resources (WGER) at Brooks Air Force Base, Tex., describing the concept, scope, and present status of the Bureau's research on lunar resource utilization. A brief summary was given of the progress on each of the research tasks making up the program. More detailed reports on several of the tasks were presented in three companion papers.

Tentative plans are being made for a NASA review meeting on the Bureau's program that may be held at Minneapolis the last week in May.

## Status of Manuscripts

Summary of Electrical Property Data on Rock, an informal report by T. C. Atchison, R. L. Marovelli, and R. E. Griffin, was submitted to NASA in July.

Engineering Property Measurements on Returned Lunar Samples, a revised proposal by T. C. Atchison, was submitted to NASA in September 1967.

Proposal for Continuing Bureau Extraterrestrial Resource Utilization Program, by the core group, was submitted to NASA in September 1967.

Bureau of Mines Research on Lunar Resource Utilization, by T. C. Atchison and C. W. Schultz, was presented at the Sixth Annual Meeting of the Working Group on Extraterrestrial Resources at Brooks Air Force Base, Tex. in February and submitted for publication in the proceedings in March.

Task title: Selection and sample collection of simulated lunar materials
Investigator: David E. Fogelson, Project Leader
Location: Twin Cities Mining Research Center
Minneapolis, Minnesota
Date begun: September 1965 To be completed: Continuing

Personnel: David E. Fogelson, Supervisory Geophysicist
Other Bureau personnel, as assigned

## PROGRESS REPORT

### Objective

The objective is to select and obtain samples of rocks and minerals covering the range of materials likely to be found on the Moon.

### Progress During the Third Quarter

A paper on the simulated lunar rocks being used in the Bureau's lunar resource studies was completed and presented at the Sixth Annual Meeting of the Working Group on Extraterrestrial Resources (WGER), Brooks Air Force Base, Tex., in February. The paper outlined the reasons for selecting these rocks and gave the results of some of the property measurements that have been made. It contained complete petrographic descriptions and chemical analyses of each of the rocks, together with photographs of hand specimens and photomicrographs of thin sections.

As a result of the presentation at the WGER meeting and discussions of the Bureau's lunar resource research at other scientific meetings, we have received requests from several additional organizations for samples of simulated lunar rocks for use in their lunar surface research. Samples were furnished during the quarter to the Jet Propulsion Laboratory, Pasadena, Calif.; the USGS Center of Astrogeology, Flagstaff, Ariz.; Martin Marietta, Denver, Colo.; General Dynamics, Fort Worth, Tex.; and Litton Industries, Minneapolis, Minn.

### Status of Manuscripts

Simulated Lunar Rocks, by D. E. Fogelson, was presented at the Sixth Annual Meeting of the Working Group on Extraterrestrial Resources at Brooks Air Force Base, Tex. in February and submitted for publication in the proceedings in March.

Task title: Physical properties of simulated lunar materials
Investigator: Thomas C. Atchison, Senior Research Scientist
Location: Twin Cities Mining Research Center
Minneapolis, Minnesota
Date begun: October 1965 To be completed: Continuing

Personnel: All project are participating

PROGRESS REPORT

Objective

The objective is to incorporate simulated lunar materials into basic fragmentation research currently in progress, and by this means to determine the composition, elastic, strength, surface, thermal, electrical, magnetic, and explosive shock properties of simulated lunar materials in Earth environment.

Progress During the Third Quarter

Average values of thermal expansion coefficient at room temperature were determined for most of the simulated lunar rocks, as part of the study of the effect of temperature on thermal expansion. Average values of dielectric constant and dissipation factor at 100 megahertz were determined for all of the rocks except serpentinite, as part of the study of the electrical properties of the simulated lunar rocks from 20 to 100 megahertz. Results of these three property determinations are shown in table 1.

TABLE 1. - Thermal and electrical properties of simulated lunar rocks in earth environment

| Rock type | Thermal expansion ($10^{-6}/°F$) | Dielectric constant (ratio) | Dissipation factor (ratio) |
|---|---|---|---|
| Dunite | 6.2 | 5.0 | 0.008 |
| Gabbro | 3.9 | 15.4 | .081 |
| Tholeiitic basalt | 4.5 | 11.1 | .099 |
| Granodiorite | 5.6 | 5.5 | .004 |
| Serpentinite | 5.3 | ([1]) | ([1]) |
| Obsidian | 4.1 | 6.3 | .010 |
| Altered rhyolite | ([1]) | 4.8 | .017 |
| Rhyolite | 4.5 | 5.1 | .003 |
| Vesicular basalt #1 | 3.2 | 6.0 | .012 |
| Vesicular basalt #2 | ([1]) | 6.7 | .036 |
| Dacite | 3.1 | 5.0 | .008 |
| Vesicular basalt #3 | ([1]) | 4.2 | .005 |
| Semiwelded tuff | 3.3 | 2.9 | .015 |
| Pumice | 4.5 | 2.3 | .008 |

[1]Not yet measured.

## Status of Manuscripts

Dielectric Constants and Dissipation Factors Between 20 and 100 Megahertz for 14 Simulated Lunar Rocks, by Russell E. Griffin, is being prepared as a journal article.

Impact Pulse Propagation in Rock, by Thomas E. Ricketts and Werner Goldsmith, is being prepared for submission to the Journal of Geophysical Research.

Small-Scale Cratering in Simulated Lunar Materials, an informal report by Dennis V. D'Andrea and Richard L. Fischer, was submitted to the core group in September.

Simulated Lunar Rocks, by D. E. Fogelson, was presented at the Sixth Annual Meeting of the Working Group on Extraterrestrial Resources at Brooks Air Force Base, Texas in February and submitted for publication in the proceedings in March.

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| Task title: | Chemical reactivity and cold welding of freshly formed surfaces |
| Investigator: | Clifford W. Schultz, Project Leader |
| Location: | Twin Cities Mining Research Center<br>Minneapolis, Minnesota |
| Date begun: | January 1966 To be completed: March 1969 |
| Personnel: | Clifford W. Schultz, Metallurgist<br>William H. Engelmann, Research Chemist<br>Ernest Bukofzer, Physical Science Technician |

## PROGRESS REPORT

### Objective

Measure the equilibrium constants for the adsorption of gases on the surfaces of silicate minerals. Relate this quantity to the fractional coverage necessary to inhibit cold welding and to determine the rate at which various other processes inhibit or prohibit cold welding of vacuum-formed surfaces.

### Progress During the Third Quarter

The gas adsorption studies were continued this quarter, using low levels of water vapor in an argon carrier. The small ball mill used in earlier work has been set aside in favor of a larger porcelain mill of about three times the capacity. Leakage rates at the slip connections to the small mill were intolerable, and pressure fluctuations caused by bobbing motions of the gas lines to the rotating mill produced excessive bridge output noise.

The larger procelain mill was charged with 300 grams of 35/48 Wausau quartzite and 4,200 grams of porcelain balls. The water vapor partial pressure of 4.6 torr (ice-point) was established in an argon carrier, and allowed to flow through the charged mill to a steady state bridge output, indicating equilibration with the silicate surface. Rotation of the mill for 100 revolutions @ 80 rpm, produced an adsorption peak and equilibration was reached within 2-1/2 minutes. Under the same conditions, 200 revolutions produced a proportionately larger peak and equilibration required very nearly 5 minutes. At this point the slip joint froze, due to entrance of dust. This is being corrected. We have demonstrated, however, that the system can yield information, at least in the 1-10 torr partial pressure region.

Helium has a thermal conductivity 8.8 times larger than that of argon and is now being calibrated for the system as an alternative carrier gas. The use of argon precludes going down to the dewpoint at the liquid nitrogen temperature, since argon condenses at a 10°C higher temperature level.

### Status of Manuscripts

None in progress.

Task title: Surface properties of rock in lunar environment
Investigator: Clifford W. Schultz, Project Leader
Location: Twin Cities Mining Research Center
Minneapolis, Minnesota
Date begun: January 1966 To be completed: March 1969

Personnel: Clifford W. Schultz, Metallurgist
Wallace W. Roepke, Principal Vacuum Specialist
Kenneth G. Pung, Physical Science Technician

## PROGRESS REPORT

### Objective

Develop data on the fundamental frictional characteristics of mineral surfaces as related to their environments. Correlate the measurements of friction, surface energy, and hardness. Further establish the relationship among these various surface properties and between surface and bulk physical properties.

### Progress During the Third Quarter

This past quarter the friction work has advanced rapidly. The redesigned system has been received from the machine shop and is in the process of being set up for bench testing. The readout system and the mounting for the drive motor and gear reduction unit have both been designed and are ready for calibration and bench testing with the rest of the system. The readout system consists of two full bridges mounted in such a manner that one bridge gives a continuous reading of load while the other bridge gives a continuous reading of displacement due to stick-slip or friction of the probe on the test sample. Both of these parameters will be monitored simultaneously on a two-channel recorder during test runs in the vacuum chamber.

A long-term degassing study of basalt has been conducted during this quarter. A 1-inch cube of material has been continuously under ultrahigh vacuum for a period of 6 weeks. During the first 14 days the system was kept at a bakeout temperature of 130°C without the shroud on. This initial baking did not lower the pressure below $2 \times 10^{-7}$ torr hot. The next 4 days the system was allowed to cool. During this period the pressure dropped to $9 \times 10^{-10}$ torr and would go no lower.

The system was then shrouded and baked for a period of 7 days during which the pressure again rose to $2 \times 10^{-7}$ torr and would not fall as long as it was hot. With the heaters turned off the pressure fell quite rapidly to the low $10^{-10}$ torr range and then started a very slow downward trend until it reached $2 \times 10^{-11}$ torr, the lower limit of the present gages.

Increasing the bakeout capability of the system to the 300° to 400°C range could be expected to reduce the pump-down time but might cause permanent changes in some rocks. Mass spectrometer data taken during the study have not been completely analyzed yet. The most significant result of the study was demonstration that lunar vacuum can be achieved with a basalt sample in the chamber.

## Status of Manuscripts

Mass Spectrometer Studies of Outgassing from Simulated Lunar Materials in UHV, by W. W. Roepke and C. W. Schultz, was presented at the American Vacuum Society Symposium in October and is being prepared for publication in the Journal of the American Vacuum Society.

Vacuum Technology Course, an internal report by W. W. Roepke, is in preparation.

Oil Vapor Sieve Trap for Rotary Mechanical Pumps, by W. W. Roepke and K. G. Pung, was completed for technical journal publication.

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| Task title: | Rock failure processes and strength and elastic properties in lunar environment |
| Investigator: | Egons R. Podnieks, Project Leader |
| Location: | Twin Cities Mining Research Center<br>Minneapolis, Minnesota |
| Date begun: | June 1966     To be completed: June 1969 |
| Personnel: | Egons R. Podnieks, Mechanical Research Engineer<br>Robert J. Willard, Geologist<br>Thomas R. Bur, Geophysicist<br>Richard E. Thill, Geophysicist<br>Peter G. Chamberlain, Geophysicist<br>Kenneth E. Hjelmstad, Geophysicist |

## PROGRESS REPORT

### Objective

Extend current experimental studies of rock failure by such mechanisms as dislocation, twinning, and crack formation to include lunar environment. Extend current measurements of static and dynamic elastic moduli and compressive and tensile strengths of rock to include lunar environment.

### Progress During the Third Quarter

Analysis of the data from the preliminary environmental test program was completed and a paper describing the results was prepared. The paper will be presented at the Tenth Symposium on Rock Mechanics at the University of Texas in May and published in the proceedings of the Symposium.

Preparations for strength and elastic property measurements in lunar vacuum environment were completed. Modifications of the ultrahigh vacuum chamber, the compression testing machine, and the pulse velocity equipment were finished and the combined system was checked out. Tholeiitic basalt, dacite, and semiwelded tuff were selected for the initial experiments, providing a range of physical characteristics. Pulse velocity measurements were made on blocks of these rocks to determine the extent and direction of anisotropy. Cylindrical specimens with their axes parallel to the principal axes of symmetry were prepared and are now being preconditioned. The designed program of measurements will be carried out during the next quarter.

### Status of Manuscripts

Environmental Effects on Rock Properties, by E. R. Podnieks, P. G. Chamberlain, and R. E. Thill, was prepared for presentation at the Tenth Symposium on Rock Mechanics at the University of Texas in May.

Task title: Feasibility of thermal fragmentation studies in vacuum
Investigator: Robert L. Marovelli, Project Leader
Location: Twin Cities Mining Research Center
Minneapolis, Minnesota
Date begun: October 1966 To be completed: September 1968

Personnel: Robert L. Marovelli, Supervisory Mining Engineer
Russell E. Griffin, Electronic Research Engineer
Kuppusamy Thirumalai, Mining Engineer
Sam G. Demou, Physicist

## PROGRESS REPORT

### Objective

Investigate the feasibility of extending current thermal fragmentation studies, including thermophysical property measurements, to lunar vacuum environment.

### Progress During the Third Quarter

Preparations were completed for use of the thermal shock furnace to study the effect of $10^{-5}$ torr vacuum on rock behavior at high temperatures. Experimental work on softening and melting characteristics has begun.

Apparatus for determining emissivity and permeability of porous material at high temperatures was obtained as excess property from NASA Lewis Research Center in Cleveland, Ohio. Complete specifications, instructions, and maintenance manuals for the equipment were also obtained. These will help us in modifying and adapting the system to our use. This equipment will enable us to expand our thermal properties measurement capabilities into a moderately high vacuum environment up to a temperature of 1,200°C.

### Status of Manuscripts

None in progress.

Task title: Thermophysical properties of rock at elevated and reduced temperatures
Investigator: Robert L. Marovelli, Project Leader
Location: Twin Cities Mining Research Center
Minneapolis, Minnesota
Date begun: October 1966 To be completed: September 1968

Personnel: Robert L. Marovelli, Supervisory Mining Engineer
Russell E. Griffin, Electronic Research Engineer
David P. Lindroth, Physicist
Carl F. Wingquist, Physicist
Water G. Krawza, Engineering Technician

## PROGRESS REPORT

### Objective

Extend current studies of the effect of temperature on thermophysical properties of rock at atmospheric pressure to cover the lunar temperature range.

### Progress During the Third Quarter

Thermal expansion measurements on the simulated lunar rocks over the temperature range from -320° to 2,000°F, begun last quarter, were completed. Reduction of the data has not been finished. However, an average coefficient at approximately room temperature was determined for most of the rocks and is presented under the earth environment properties task.

In related Bureau work of direct interest to this task, a flash method of determining thermal diffusivity is being investigated. This method appears very promising in that the values of thermal conductivity and specific heat can be obtained at the same time. The acquisition of conductivity and specific heat are contingent on knowing the exact energy per unit area that goes into the specimen.

The method requires that a specimen, in the form of a small disk, be heated (from 1° to 10°C) by a pulse of radiant energy absorbed at the front surface, that is short in duration as compared to the time required for the heat pulse to reach the rear face. From the known energy input, and the temperature rise at the rear face of the specimen, the three thermal property values are determined.

The apparatus required for this experiment is relatively simple and easily obtained. The only contingency at this time is obtaining a radiant heat source with the following parameters: (1) variable pulse duration, (2) variable energy output, (3) variable beam diameter, and (4) preferably nearly monochromatic radiation. At this time a laser appears to be the optimal radiant heat source. Investigation of this method will continue.

## Status of Manuscripts

Flexural Strength of Rock from -320° to 1,600°F, by R. L. Marovelli and A. Hendrickson, is under preparation as a journal article and a Bureau Report of Investigations.

Task title: Basic problems of drilling in lunar environment
Investigator: James Paone, Project Leader
Location: Twin Cities Mining Research Center
Minneapolis, Minnesota
Date begun: January 1967 To be completed: December 1969

Personnel: James Paone, Supervisory Mining Engineer
Robert L. Schmidt, Mining Engineer
Harold F. Unger, Mining Engineer
Carl F. Anderson, Electronic Engineer
David A. Larson, Engineering Technician

PROGRESS REPORT

## Objective

Investigate various means of removing drill cuttings with and without flushing media in lunar environment. Investigate problems of heat removal and bit lubrication associated with drilling in lunar environment.

## Progress During the Third Quarter

James Paone and R. L. Schmidt monitored tests of an engineering model of the Apollo Lunar Surface Drill (ALSD) performed in a vacuum chamber at the Marshall Space Flight Center, Huntsville, Ala., in late December. High vacuum ($10^{-6}$ to $10^{-7}$ torr) did not appear to have an adverse affect on the operation of the ALSD. Vacuum drill cuttings were returned to the Twin Cities Mining Research Center for size analysis and comparison with cuttings from atmospheric drilling tests.

The atmospheric drilling tests will be conducted at the Twin Cities Mining Research Center when the ALSD engineering model is received from NASA. A drill test stand was constructed in anticipation of this and a series of tests were planned to investigate bit heat generation, penetration rate, and power consumption in simulated lunar rocks.

## Status of Manuscripts

Lunar Drilling, by James Paone and R. L. Schmidt, was presented at the Sixth Annual Meeting of the Working Group on Extraterrestrial Resources at Brooks Air Force Base, Texas, in February, and submitted for publication in the proceedings, in March.

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| Task title: | Effect of lunar environment on behavior of fine particles |
| Investigator: | David E. Nicholson, Project Leader |
| Location: | Spokane Mining Research Laboratory<br>Spokane, Washington |
| Date begun: | April 1966 To be completed: March 1969 |
| Personnel: | David E. Nicholson, Mining Engineer<br>Howard C. Pettibone, Civil Engineer<br>Dennis J. Kelsh, Physical Chemist<br>Fred W. Houghton, Engineering Technician<br>Richard P. Curtin, Engineering Aid |

## PROGRESS REPORT

### Objective

The primary objective is to determine basic physical properties which may influence the handling and transportation of fine particles in a lunar environment as an extension of current studies of fine particle behavior in mine backfill applications. Intergranular static and dynamic coefficient of friction and energy loss will be measured. Flow rates and shear strength at various states of particle packing and at various sizes will be determined and correlated with friction and energy loss properties. This work will initially be performed under conditions of normal earth atmosphere, but will be extended to perform selected tests in ultrahigh vacuum. The work will be correlated with the study of electrostatic properties of granular particles being conducted at College Park and the study of frictional properties of mineral surfaces being conducted at Minneapolis.

### Progress During the Third Quarter

Background literature study of materials handling science, lunar vacuum and temperature environments, and surface chemistry of silicates continued during the quarter.

Samples of flow basalt prepared during the second quarter were used in a complete series of one-dimensional compression tests. Test data are being processed through the 1130 computer at Gonzaga University.

The dry basalt material exhibited a substantial amount of apparent cohesion after compression testing. Samples of pure silica flour, and a ball mill ground basalt prepared by the Twin Cities Mining Research Center, were incorporated into the test program to compare the effect of different particle shapes on the amount of cohesion developed after compression. Visual observation of the samples indicate a lesser or negligible amount of apparent cohesion in the silica and ball mill ground basalt in comparison with the impact milled basalt.

A theoretical analysis of the loose packing of granular material was started, using a simple cubic packing model. This will be used for analysis of the basalt tests.

Direct shear tests were completed on the basalt samples and a series of direct shear tests between the basalt and a hard steel insert were started to develop values of the angle of friction at the sidewall of the one-dimensional compression chamber.

A status report on the fine particle materials handling project was written and sent through channels to NASA. Design of an experiment to perform compression tests under conditions of lunar vacuum and temperature was started during the quarter.

## Status of Manuscripts

Fine Particles Study at the Spokane Mining Research Laboratory, an informal status report by David E. Nicholson and Howard C. Pettibone was submitted to NASA in January.

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| Task title: | Support for underground lunar shelter |
| Investigator: | Robert C. Bates, Project Leader |
| Location: | Spokane Mining Research Laboratory<br>Spokane, Washington |
| Date begun: | April 1966 To be completed: March 1969 |
| Personnel: | Robert C. Bates, Mining Engineer<br>Lester J. Crow, Mining Engineer<br>Colen S. Smith, Mining Engineer<br>Dennis J. Kelsh, Physical Chemist |

## PROGRESS REPORT

### Objective

The objective is to advance ground support technology needed to carry on extraterrestrial activities in support of future space missions, as an extension of terrestrial ground support research. Investigate ground support materials which have good potential for utilization both on the Moon and on Earth. One of the materials selected for this investigation is sulfur. Sulfur materials may provide an economical and effective solution to many terrestrial construction and ground support problems. Sulfur has a high probability of being indigenous to the lunar surface and thus sulfur materials might serve in an equivalent capacity for lunar shelter construction. The immediate objectives are to: (1) compile all available data on the properties and potential uses of sulfur as a support medium; (2) conduct laboratory studies to improve certain properties and to determine the feasibility of certain uses of sulfur materials; (3) formulate design concepts for support systems utilizing these materials; and (4) develop techniques and equipment for fabrication or installation of these support systems in a simulated lunar environment.

### Progress During the Third Quarter

The preparation and testing area at the Auxiliary Laboratory was substantially completed in regards to equipment, adequate ventilation, and storage. Some progress was made in increasing the strength of thermoplastic mixtures of sulfur and a dense flow type of basalt aggregate.

For the specimens tested first the balance of coarse and fine aggregate was held to a given proportion and the amount of sulfur varied. Test specimens having more than or less than the amount of sulfur necessary to fill the voids in the aggregate and produce good workability of the mixture had relatively low uniaxial compressive strengths - ranging around 3,500 psi.

Specimens with a good balance of sulfur still showed a problem of some shrinkage. For the first batch of these cylinders tested in uniaxial compression the top one-third of the cylinder tended to break off, and inspection showed a large flat void near the top surface. The average strength of these cylinders was about 5,000 psi.

By puddling the top surface of the test cylinder until it cooled and thereby reducing the void space, a substantial rise in strength and classic double cone-shaped breaks were produced.

Heat was maintained throughout the whole system in order to keep the temperature of the mix sufficiently above the melting point of sulfur to allow working of the material in the molds a short time before it congealed. Test specimens with equal portions of coarse and fine aggregate, with an average grain size of 3.3 mm and a coefficient of uniformity of 50, and containing about 20 percent by weight of sulfur, produced average strengths of 6,600 psi.

A series of test specimens were made in which the amount of fine material in relation to coarse material was successively increased, with the sulfur being maintained at about 20 percent by weight. Compressive strengths increased with the addition of fine material to an average of 10,300 psi for mixtures of about 10 percent by weight of coarse aggregate and 70 percent by weight of fine aggregate, having an average grain size of 1.7 mm and a coefficient of uniformity of 23.

Evaluation of the potential obstacles to the use of sulfur in an extraterrestrial environment continued. Dennis J. Kelsh submitted an evaluation report on sulfur that points out the temperature and sublimation problems in a lunar environment. Considering the problem of sublimation, we feel that sulfur can be mixed and placed in a closed circuit and that the exposed surface of the sulfur in place can be coated with a non-sublimating material to protect it.

Sulfur is one of the materials being considered for lunar shelter support. Sodium silicate is another material proposed for this purpose. Our original plan was to examine sulfur this year and then examine other potential materials such as sodium silicate later. However, as sodium silicate offers a potential solution to the sublimation problem some attention will be paid to it in the fourth quarter.

## Status of Manuscripts

Support for Underground Lunar Shelter Using Sulfur, an internal background report by Lester J. Crow, was completed in October.

Sulfur as Lunar Shelter Support Material, an internal report by Dennis J. Kelsh, was completed in January.

Preparation and Compressive Testing of Sulfur Aggregate Cylinders, an internal report by Lester J. Crow, was completed in March.

Task title: Use of explosives on the Moon
Investigator: Frank C. Gibson, Project Coordinator, Explosives Physics
Location: Explosives Research Center
Pittsburgh, Pennsylvania
Date begun: July 1966 To be completed: June 1969

Personnel: Frank C. Gibson, Supervisory Research Physicist
Richard W. Watson, Research Physicist
J. Edmund Hay, Research Physicist
Charles R. Summers, Research Physicist
William F. Donaldson, Research Physicist
Elva M. Guastini, Explosives Equipment Operator

## PROGRESS REPORT

### Objective

The objective is to develop a body of knowledge relevant to the use of chemical high explosives under lunar environment. Immediate goals are to determine the hazards associated with the storage, handling, and use of explosives in an environment characterized by high vacuum, extreme temperature cycling, and a flux of small hypervelocity particles, and to establish techniques for minimizing these hazards.

### Progress During the Third Quarter

During this quarter, determination of the sensitivity of explosives to initiation of detonation by simulated micrometeoroid impact has been continued. The object of the current research is to develop a scaling relationship of the form $M^{\alpha} V^{\beta} = K$,
where M is the projectile mass,
V is the projectile velocity,
$\alpha$, $\beta$, and K are constants,
and to extend this relationship as far as possible in the direction of smaller projectile masses and higher velocities in order to enable extrapolation with some confidence to projectiles having meteoric velocities.

Experimentally the projectile velocity for 50 percent initiation probability has been established for 1/2-, 25/64-, 1/4-, and 1/8-inch diameter projectiles; the results have been fitted to the empirical relationship between projectile mass and threshold initiation velocity.

The acceptor explosive used was Composition A-5 (RDX with one percent stearic acid), pressed as pellets to a density of 1.72 g/cm$^3$, 1-5/8-inch diameter by 1/2-inch thick. Two pellets were stacked and oriented so that the trajectory of the projectile was normal to the face of the charge and the point of impact was at the center. A steel plate 4-inch by 4-inch by 1/4-inch thick was placed 1/4 inch behind the acceptor as a detonation indicator. For projectiles down to 1/8-inch in diameter, a 50-caliber gun and sabots were used; however, it was found that with the 1/16-inch projectile and a full load of the fastest-burning propellant, a maximum velocity of only 1.9 mm/μsec could be obtained; this was insufficient to cause initiation.

An exploding foil type launcher, in which the projectile is driven by the plasma produced by a capacitor discharge, was tried but velocities of about 1,500 m/sec were found to be maximum when the voltage on the capacitor bank was limited to 20 kv.

Attention is now being given to a solid explosive launcher. Balls of sizes 1/16-inch and 1/32-inch in diameter are being cast into the base of cylinders of Pentolite. At velocities up to 3.0 mm/μsec the projectiles seem to remain intact. Some results are shown in table 1.

TABLE 1. - Pentolite launcher using 1/16-inch balls

| Diameter (inches) | Charge dimension Length (inches) | Velocity (mm/μsec) |
|---|---|---|
| 0.50 | 3 | 0.92 |
| 0.75 | 4 | 2.66 |
| 1.00 | 3 | 2.63 |

The data for the gun-launched projectiles are given in table 2 and figure 1. Good agreement has been obtained with the values $\alpha = 0.277$, $\beta = 2$, $K = 1.515$ for the four projectile sizes employed thus far. However, attempts to extrapolate this relationship are subject to the following limitations: (1) the smallest diameter projectile is still of the order of the critical diameter of the explosive used; (2) the duration of the shock is probably long compared to the reaction time of the explosive; and (3) the projectile velocity is still subsonic with respect to the explosive. For true micrometeorites, all of these conditions will change so that reducing the mass by a factor of 64 may require an increase in velocity of more than a factor of 1.78.

TABLE 2. - Mass-velocity relationship for 50 percent probability of initiation of pressed Composition A-5 charges

| Diameter (inches) | Mass (grams) | $V_{50}$ (mm/μsec) | $M^{0.277}V^2$ |
|---|---|---|---|
| 1/2 | 8.33 | 0.92 | 1.51 |
| 25/64 | 3.95 | 1.03 | 1.55 |
| 1/4 | 1.04 | 1.22 | 1.49 |
| 1/8 | 0.13 | 1.63 | 1.51 |

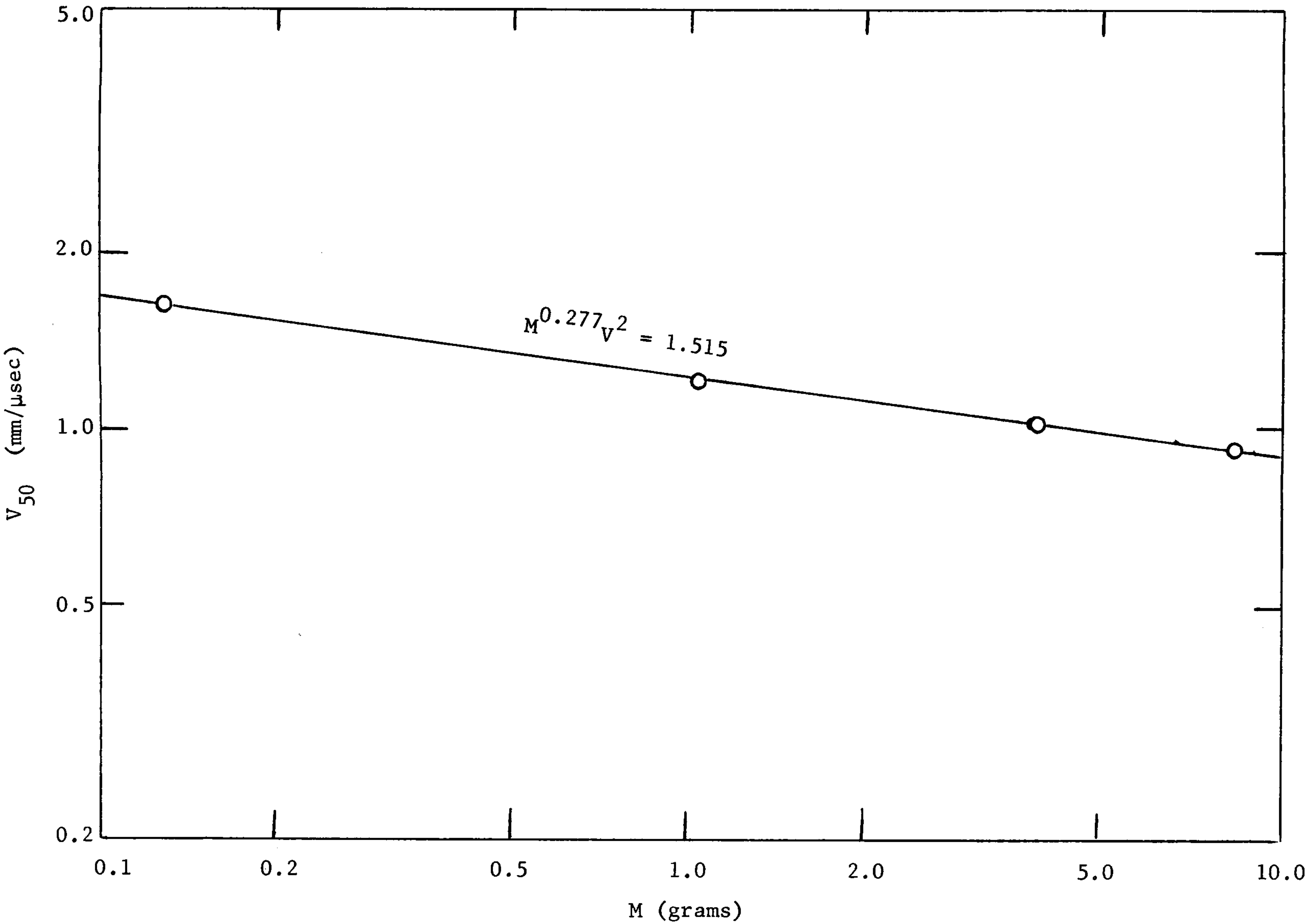


FIGURE 1. - Velocities for 50 Percent Probability of Initiation Versus Projectile Mass.

## Proposed Facility for Investigation of Expansion of Detonation Products in High Vacuum

Considerable interest, both theoretical and practical, is attached to the study of the expansion of the products of a detonation in a high vacuum. For practical purposes a high vacuum will be defined as one in which the mean free path of a molecule is greater than the significant dimensions of the apparatus. The mean free path ($\bar{\lambda}$) is a function of the absolute temperature (T), the absolute pressure (P), and collision cross section ($\sigma$) of the molecules, being given by $\bar{\lambda} = (\sigma\ N)^{-1} =$

$$(\sigma\ N_0\ n)^{-1} = \left(\sigma\ N_0\ \frac{P}{RT}\right)^{-1} = \left(\frac{RT}{\sigma\ N_0}\right)\frac{1}{P},$$

where
- N is the number of molecules per unit volume,
- $N_0$ is Avogadro's number,
- n is the number of moles/unit volume,
- R is the universal gas constant.

Assuming a value for $\sigma = 2 \times 10^{-15}$ cm$^2$, and using $R = 6.23 \times 10^4$ torr-cm$^3$/mole/deg, T = 300°K, this yields $\bar{\lambda} = \frac{0.0156}{P}$ cm, where P is expressed in torr. Thus, if measurements are to be conducted over distances of the order of a hundred cm or so, the required vacuum will be of the order of $10^{-4}$ torr.

Small-scale experiments (spanning a few tens of centimeters) concerning the flow of detonation products in a vacuum have been reported by several investigators. However, there appear to be discrepancies and it is not yet certain whether the results will obey the expected scaling relationships between charge mass and distance from the charge; thus, the need for larger-scale experiments is indicated.

Two facilities for performing these experiments exist at this Center: one of these, a sphere with a diameter of 12 feet, would require expensive modification in order to adapt it to a pumping system of adequate capacity, and will not be further discussed here; the other is a cylindrical vessel with a diameter of 7 feet and a length of 37 feet (capacity$\approx$ 1,400 cu ft and a working pressure of 125 psi). This vessel has a number of advantages for the purpose intended, including a 24-inch flanged access and photography port, a 20-inch flange for connections of a large diffusion pump, a number of smaller flanges for instrumentation ports, and a large dimension for investigating effects at long standoffs. The maximum useful dimension of the chamber is still a matter of speculation, since, if the charge is detonated near one end of the chamber, a shock due to impingement of the products on the near end and the side walls may overtake the products expanding toward the far end. If the charge is positioned at the center it is, however, very unlikely that this would occur before the leading edge of the products cloud traversed several meters.

The required pumping capacity for this vessel cannot be calculated in advance due to lack of knowledge concerning the effective leak rate and the outgassing after a firing; however, in the opinion of several persons with experience in this area, it would be prudent to plan for a 20-inch diffusion pump, and a roughing pump/fore-pump of approximately 100 cfm capacity. If the 12-foot sphere is used, the pumping capacity requirements would be similar, as the volume is comparable (900 cu ft).

## Status of Manuscripts

None in progress.

Task title: Volcanism and ore genesis as related to lunar mining
Investigator: Rolland L. Blake, Project Coordinator
Location: Twin Cities Metallurgy Research Center
Minneapolis, Minnesota
Date begun: June 1966 To be completed: May 1967

Personnel: Rolland L. Blake, Geologist
Others as assigned

## PROGRESS REPORT

### Objective

Study the genesis of ore deposits and the occurrence of minerals associated with volcanic activity here on Earth. Study the effects of the lunar environment and other environments on mineralization and ore genesis. Bring together the pertinent information found in the literature on these subjects and define those specific areas where additional work is needed.

### Progress During the Third Quarter

Work on this task ended in fiscal year 1967, except for the completion of a summary report and recommendations for further studies which are still in progress.

### Status of Manuscripts

Ore Deposits in Volcanic Rocks with Lunar Extrapolation, by R. L. Blake, was presented at the Sixth Annual Meeting of the Working Group on Extraterrestrial Resources at Brooks Air Force Base, Tex. in February and is being prepared for publication in the proceedings.

| | | |
|---|---|---|
| Task title: | Reduction of silicates with carbon | |
| Investigator: | Sanaa E. Khalafalla, Project Coordinator | |
| Location: | Twin Cities Metallurgy Research Center<br>Minneapolis, Minnesota | |
| Date begun: | June 1966 | To be completed: May 1969 |
| Personnel: | Sanaa E. Khalafalla, Supervisory Research Chemist<br>Larry A. Haas, Research Chemist<br>Howard W. Kilau, Chemist<br>Thomas H. McCormick, Physical Science Aid | |

## PROGRESS REPORT

### Objective

The objective is to determine the optimum reaction rate criteria for extraction of oxygen from simulated lunar materials in a vacuum. The major emphasis of this research is to determine the kinetics and mechanism of the carbothermal reduction of siliceous materials in a high temperature vacuum furnace.

### Progress During the Third Quarter

The objectives for the third quarter as set in the work plan anticipated some initial rate and kinetic data measurements with a new thermogravimetric vacuum furnace. Delivery of that furnace was delayed for nearly three months and studies were accordingly postponed. Instead, the time during this quarter was utilized to complete the reactant stoichiometric ratio studies using 0.18 mm diameter high-density graphite and to compare these results with the data previously obtained with 0.10 mm diameter low-density graphite. Also, a literature survey on probable lunar silicates was made and preliminary studies were conducted using selected silicate minerals and graphite in the available vacuum furnace.

Previous results indicated that the degree of interaction of graphite and silica is strongly dependent on the reactant particle size. Below 0.08 mm diameter, the gas permeability of the charge appeared to be rate controlling. Experiments to investigate the effect of reactant ratio were performed with low-density graphite. This form of graphite was very fragile and difficult to obtain in a mono-size fraction (5 percent of the particles were less than 0.05 mm diameter). To ascertain that previous reactant ratio experiments were performed above the critical particle diameter, a new series of experiments were conducted with high-density, briquetted graphite with average particle diameter of 0.18 mm. In experiments with low-density graphite, the number of moles of one of the reactants was fixed while the other was varied, as shown in tests 1 through 12 in table 1.

TABLE 1. - Effect of reactants ratio on the carbothermal reduction of silica

| Test No. | Silica in charge, percent | Moles of reactants | | $SiO_2/C$ molar ratio | Total weight, grams | Weight loss in 5 hrs, percent |
|---|---|---|---|---|---|---|
| | | Silica | Carbon | | | |
| | | Low-density graphite, (0.10 mm) | | | | |
| 1 | 38.5 | 0.025 | 0.200 | 0.125 | 3.9 | 1.8 |
| 2 | 55.6 | .050 | .200 | .25 | 5.4 | 2.4 |
| 3 | 71.4 | .200 | .400 | .50 | 16.8 | 4.8 |
| 4 | 71.4 | .200 | .400 | .50 | 16.8 | 4.8 |
| 5 | 83.3 | .200 | .200 | 1.00 | 14.4 | 6.4 |
| 6 | 83.3 | .200 | .200 | 1.00 | 14.4 | 7.2 |
| 7 | 83.3 | .200 | .200 | 1.00 | 14.4 | 7.2 |
| 8 | 90.9 | .200 | .100 | 2.00 | 13.2 | 7.7 |
| 9 | 90.9 | .400 | .200 | 2.00 | 26.4 | 7.1 |
| 10 | 93.0 | .200 | .075 | 3.00 | 12.9 | 7.7 |
| 11 | 95.2 | .200 | .050 | 4.00 | 12.6 | 11.6 |
| 12 | 97.6 | .200 | .025 | 8.00 | 12.3 | 7.2 |
| | | High-density graphite, (0.18 mm) | | | | |
| 13 | 20.0 | .048 | .096 | .050 | 14.40 | 1.5 |
| 14 | 40.0 | .096 | .720 | .133 | 14.40 | 8.1 |
| 15 | 60.0 | .144 | .480 | .300 | 14.40 | 12.1 |
| 16 | 60.0 | .144 | .480 | .300 | 14.40 | 12.5 |
| 17 | 83.3 | .200 | .200 | 1.000 | 14.40 | 22.0 |
| 18 | 83.3 | .200 | .200 | 1.000 | 14.40 | 22.0 |
| 19 | 90.0 | .216 | .120 | 1.800 | 14.40 | 22.9 |
| 20 | 95.0 | .228 | .060 | 3.800 | 14.40 | 17.4 |
| 21 | 97.0 | .233 | .036 | 6.470 | 14.40 | 11.2 |

In tests 13 through 21 with high-density graphite, the total charge weight was fixed and the number of moles of each reactant were varied to obtain a wider spectrum of molar ratios. Figure 1 depicts the dependence of the 5-hour weight loss on the percent of the reactants. It is clear that maximization of the reaction occurs at about 90 percent silica. Curve _a_ of figure 1 exhibits a sharp drop in reactivity when the amount of low-density graphite increases from 5 to 7 percent. This can perhaps be explained by the onset of gas permeability in controlling the reactivity of the mixture.

When the data in figure 1 were replotted on a molar ratio basis, figure 2 was obtained. A plateau was observed in curve _a_ and _b_ at a molar ratio of about 2. This is consistent with the stoichiometry of the following reaction: $SiO_2 + 2C \rightarrow Si + 2CO$. The sharp increase in reactivity with low-density graphite between a molar ratio of 3.5 and 4.2 as depicted in curve _a_ corresponds to the same sharp decrease observed with the same reductant in figure 1.

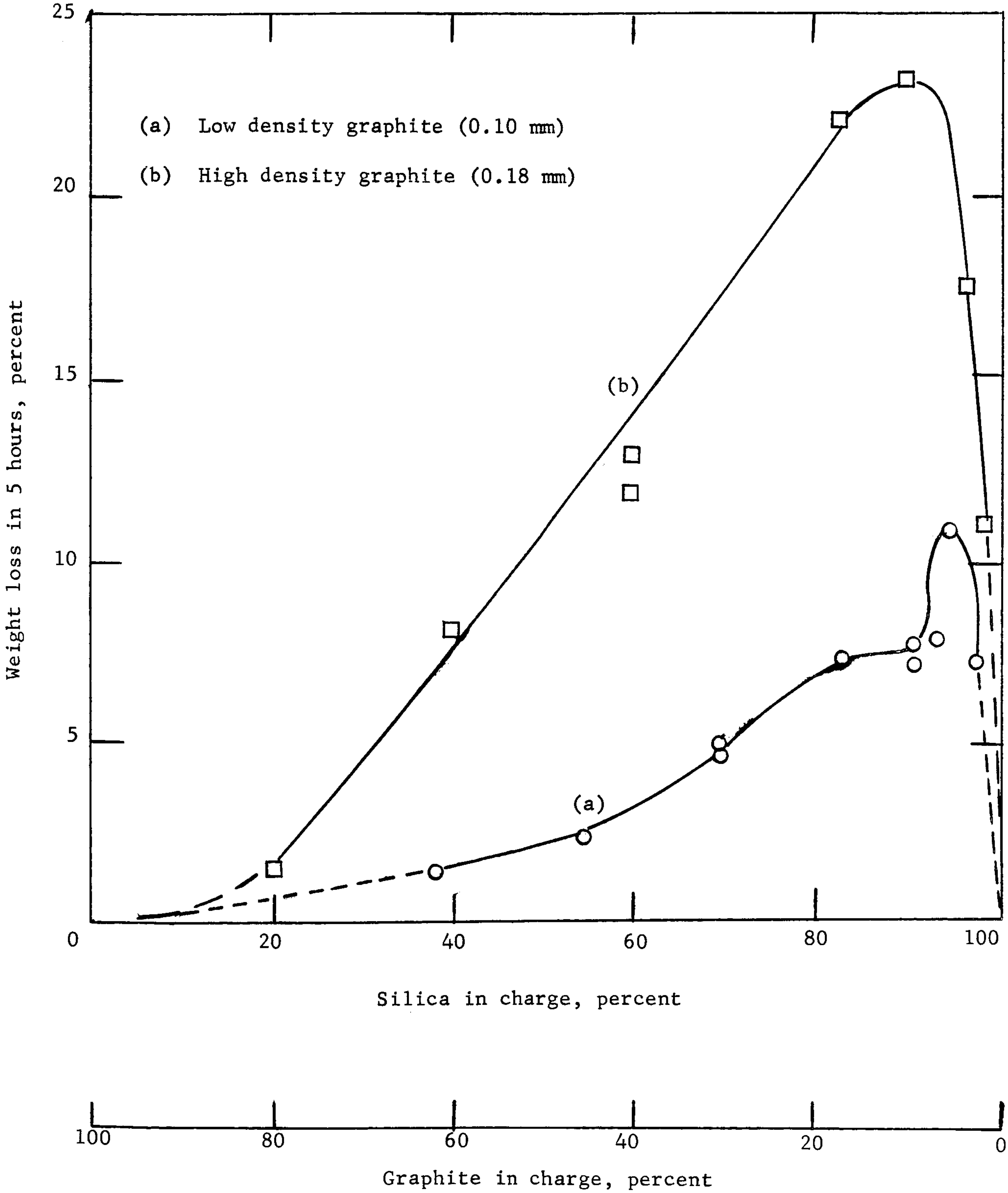


FIGURE 1. - The Weight Loss Dependence on the Amount of Silica (0.18 mm) and Graphite in the Charge.

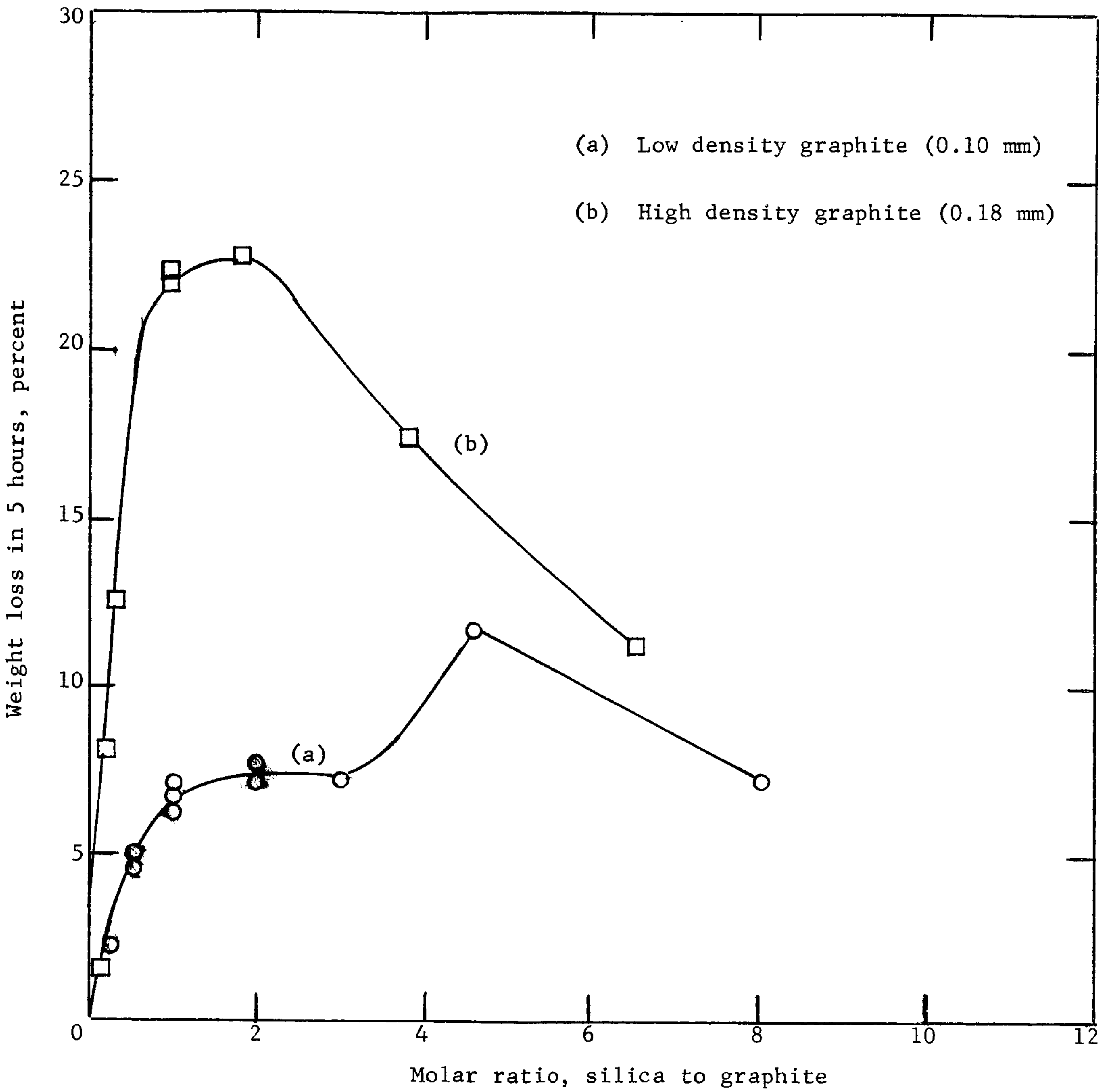


FIGURE 2. - Effect of Reactants Molar Ratio on the Carbothermal Reduction of Silica Under Vacuum.

During this quarter investigations were also initiated on silicate minerals since the objective of this study is to determine the feasibility of generating an oxygen-containing gas by interaction of carbon with simulated lunar rocks. Green[1] postulated that lunar rocks are composed of the igneous rock-forming minerals such as quartz, feldspar, pyroxene, nepheline, biotite, and hornblende. About fifty igneous rocks can be classified by combinations of the above minerals in different ratios.

Before delving into these complex silicate minerals, considerable work was performed on the simplest mineral, quartz. The optimum experimental conditions have been determined for quartz. Structurally, both silica and silicates have the same basic Si-O tetrahedron and only the linkage patterns in forming the framework are different. The most logical method of grouping over a thousand silicates would be by structure. The properties of a solid depend not only on the number and kinds of atoms but also on their arrangement. Table 2 is arranged such that the degree of linking increases as one moves down the chart. Nesosilicates have isolated $SiO_4$ tetrahedra and are linked together only by cations. Tectosilicates, on the other hand, have a framework of $SiO_4$ tetrahedra and are linked together on all four oxygen corners. In addition, as one progresses toward the framework silicates, there is an increasing amount of substitution of $Al^{3+}$ for $Si^{4+}$ due to their similarity in ionic radius. Besides aluminum entering the structure to form aluminum silicates, other cations ($A_m$) attach on the outside of silica tetrahedra. Any cation can be substituted as long as the total positive charge is sufficient to produce electron neutrality. The feldspars, which constitute two-thirds of the igneous rocks,[2] usually have three or more cations.

Individual minerals were selected from these mineral groups and procured from Southwest Scientific Company. The materials were comminuted, sized, and purified by visual and magnetic means. The purified silicate, -60 to +70 mesh size (0.21 mm mean diameter) was blended with the same size high-density graphite: The mixed reactants were placed in a 1.9-inch diameter molybdenum crucible and heated for 5 hours at 1,400°C in a vacuum of <0.01 torr. The weight loss obtained for the different silicates are shown in table 3. There appears to be little correlation of silicate structure and weight loss. The largest weight loss was obtained with pyroxene which is a simple chain silicate with two oxygen-shared and two unshared. The amount of reaction obtained with the layer silicates, which have three shared oxygens and one unshared, was about the same as obtained with the framework silicate (quartz) which have all the oxygens shared. Another variable that must be considered is the melting point of the silicate. One would expect liquid silicates to react differently with graphite than solid silicates because the crystal structure breaks down on melting and the rate-controlling mechanism is usually different in liquid-solid reactions.

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[1] Green, J. The Geology of the Lunar Base. Space Sciences Laboratory, North American Aviation, Inc. SID 61-358, 1962, 127 pp.

[2] Wells, A. F. Structural Inorganic Chemistry. Third Edition, Oxford Press, 1962, p. 808.

TABLE 2. - Classification of silicates

| Silicate class | Structural arrangement | General formula | Mineral group | Typical mineral | Igneous rock group containing this silicate |
|---|---|---|---|---|---|
| Nesosilicates or Orthosilicates | Separate $SiO_4$ tetrahedra | $A_m(SiO_4)$ | Olivine | Forsterite | Dunite<br>Gabbro<br>Basalt |
| Sorosilicates or Pyrosilicates | Two tetrahedra sharing 1 oxygen | $A_m(Si_2O_7)$ | Melilite | Gehlenite | Uncompahgrite |
| Cyclosilicates or Metasilicates | Closed rings of tetrahedra, each sharing 2 oxygens | $A_m(Si_6O_{18})$ | Wollastonite | | |
| Inosilicates | Continuous single chains sharing 2 oxygens | $A_m(SiO_3)$ | Pyroxene | Augite | Gabbro<br>Basalt<br>Andesite |
| | Continuous double chains sharing 2 or 3 oxygens | $A_m(Si_4O_{11})(OH)_2$ | Amphibole | Hornblende | Peridotite<br>Granite<br>Monzonite |
| Phyllosilicates | Continuous sheets of tetrahedra, each sharing 3 oxygens | $A_m(Si_2O_5)(OH)_4$ | Clay | Kaolinite | |
| | | $A_m(Si_4O_{10})(OH)_2$ | Talc | Talc | Soap stone[1] |
| | | $A_m(AlSi_3O_{10})(OH)_2$ | Mica | Muscovite | Granite |
| Tectosilicates | Continuous framework of tetrahedra, each sharing all 4 oxygens | $SiO_2$ | Quartz | | |
| | | $A_m(AlSiO_4)$ | Feldspathoids | Leucite | Syenite |
| | | $A_m(AlSi_3O_8)$ | Feldspar | Orthoclase<br>Labradorite<br>Oligoclase<br>Anorthite | Granite<br>Dacite<br>Diorite |

[1] Formed by hydrothermal alteration of an igneous rock.

TABLE 3. - Experimental results for the carbothermic reduction of silicates

| Test No. | Structure | Mineral | Silicate analysis, % | | | Weight loss, pct | Physical state at 1,400° C |
|---|---|---|---|---|---|---|---|
| | | | $SiO_2$ | $Al_2O_3$ | LOI[1] | | |
| 22 | Chain | Pyroxene | 57.9 | 1.6 | 0.3 | 75.7 | Liquid |
| 23 | Layer | Kaolinite | 53.8 | 38.8 | -0.3 | 30.4 | Solid |
| 24 | Framework | Fisher quartz | 99.0 | <0.05 | 0.6 | 29.0 | Solid |
| 25 | Do. | Wausau quartz | 96.0 | 1.4 | 0.3 | 41.2 | Solid |
| 26 | Do. | Feldspar(1792)[2] | 64.9 | 19.5 | 0.1 | 3.2 | Liquid |
| 27 | - | Mullite | 22.2 | 76.2 | <0.1 | 23.8 | Solid |
| 28 | - | Alumina(120)[2] | 0.95 | 96.2 | 0.1 | 14.2 | Solid |
| 29 | - | Alumina(1139)[2] | <0.1 | 98.3 | 0.7 | 12.2 | Solid |

[1] Loss on ignition at 1,400° C in 5 hours.
[2] Mineral manufacturer's identifying number.

The best correlation in the reduction data was found with the percentage of $SiO_2$ in the silica-alumina and aluminum silicates, provided the mineral maintained its solid state of aggregation at 1,400° C. Figure 3 shows the variation in the five-hour weight loss with the silica percent in the aluminum silicate mineral. It is evident that carbothermal reducibility increases almost linearly with the percentage of silica in this case. Striplin[3] reported that the siliceous bauxite ores reduce more easily than pure bauxite. From these results it appears that the O-Si bond is easier to break than the O-Al bond, in the reduction of solid aluminum silicates. This is consistent with thermodynamic data on the standard heats of formation of the two oxides, thus while $\Delta H_f^o$ of alumina is 245 kcal per mole, the value for silica is only 195 kcal per mole. The data so far obtained suggest that the total carbothermal reducibility of complex silicates seems to obey the additivity rule among the reducibility of the constituent oxides, provided no change in state of aggregation is encountered. Further work is needed to substantiate this preliminary finding.

[3] Striplin, M. M., Jr. and W. M. Kelly. Aluminum-Silicon Alloys by Electrothermal Reduction of Slag with Coke. Chem. Eng. Progress, v. 43, No. 10, 1947, p. 571.

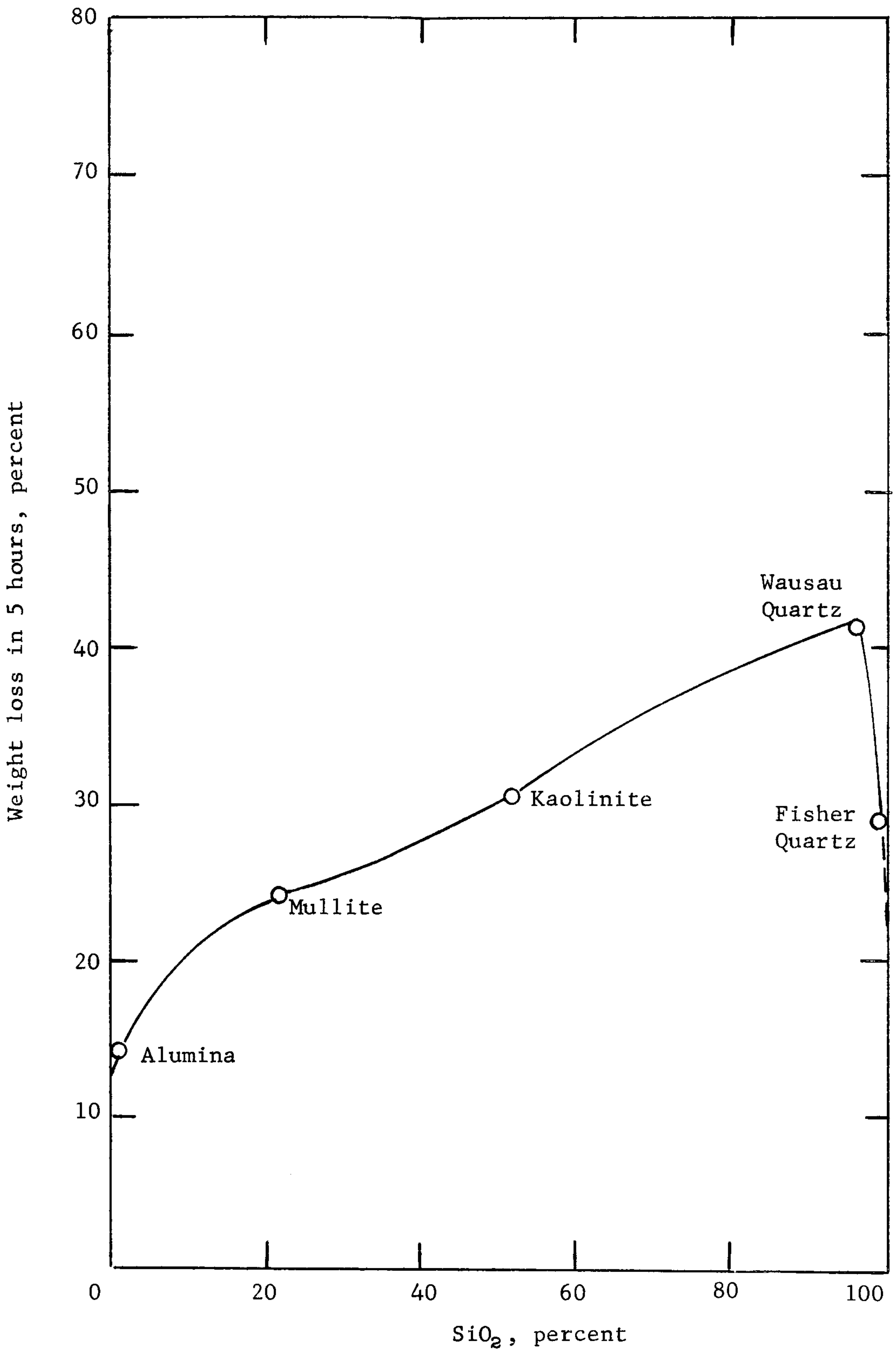


FIGURE 3. - Effect of the $SiO_2$ in the Silicate on the Weight Loss.

## Status of Manuscripts

The Effect of Physical Parameters on the Reaction of Graphite with Silica in Vacuum, by L. A. Haas and S. E. Khalafalla, is under preparation as a Bureau Report of Investigations.

Task title: Magnetic and electrostatic properties of minerals in a vacuum
Investigator: Foster Fraas, Project Leader
Location: College Park Metallurgy Research Center
College Park, Maryland
Date begun: June 1966 To be completed: May 1969

Personnel: Ray A. Heindl, Supervisory Chemical Research Engineer
Foster Fraas, Metallurgist

## PROGRESS REPORT

### Objective

The objective is to study adsorption and contact electrification in a vacuum and determine their effect on the separability of nonconducting minerals.

### Progress During the Third Quarter

The objectives for the third quarter were to determine whether the electrification changes, if they occur, are the result of the removal of adsorption layers or changes in the lattice imperfections at the surface.

Work on the project during the third quarter emphasized the attainment of an ultrahigh vacuum, since this is a prerequisite to any further progress. With removal of the vibration feed-through, the vibratory feeder, and the feed hopper, pumping for 27 hours resulted in a vacuum of $2.4 \times 10^{-7}$ torr. The equipment inside the bell jar constitutes a dirty system, difficult to evacuate since it is constructed of unpolished stainless steel, and in many cases with lap and bolted joints. However, this obstacle can be eliminated by a long bake-out period. Starting at atmospheric pressure and pumping for a relatively short 2-hour bake-out period plus a 4-hour cooling time, the vacuum was $9.2 \times 10^{-8}$ torr. During bake-out the temperature of the bell jar ranged from 150° to 200°C. The turbo-molecular pump was also baked with a heater supplied by the pump manufacturer.

The delays of design and construction, a characteristic of projects requiring items not available on the market, has plagued this project. A new vibration feed-through which was constructed with heliarc welding by a contractor, was found to be severely damaged during shipment. This item has been returned to the contractor for new construction. The damage also required the purchase of a new bellows. Measurements of contact electrification and flow characteristics will resume as soon as the vibration feed-through is delivered.

### Status of Manuscripts

None in progress.

| | |
|---|---|
| Task title: | Biological production of sulfuric acid |
| Investigator: | Joseph A. Sutton, Project Leader |
| Location: | College Park Metallurgy Research Center<br>College Park, Maryland |
| Date begun: | June 1966 To be completed: May 1967 |
| Personnel: | Ray A. Heindl, Supervisory Chemical Research Engineer<br>Joseph A. Sutton, Research Chemist<br>John D. Corrick, Research Chemist<br>Jerry M. Carosella, Microbiologist |

## PROGRESS REPORT

### Objective

Establish the limiting environmental conditions for the survival of bacteria of the genus *Thiobacillus*. Determine the rate of sulfuric acid production within these limits. Conduct a literature survey and visit such laboratories as may be necessary to establish the state-of-the-art in the use of bacteria in any stage of a life support system in an extraterrestrial environment.

### Progress During the Third Quarter

Work on this task ended in fiscal year 1967 and results were reported in the annual status report. Preparation of a more detailed summary report and recommendations for further bacteriological studies is under consideration.

Task title: Electrowinning of oxygen from silicate rocks
Investigator: Donald G. Kesterke, Project Leader
Location: Reno Metallurgy Research Center
Reno, Nevada
Date begun: June 1966 To be completed: May 1969

Personnel: Thomas A. Henrie, Supervisory Metallurgist
Donald G. Kesterke, Metallurgist
Freddy B. Holloway, Physical Science Technician
John D. Lafontan, Physical Science Aid

## PROGRESS REPORT

### Objective

The objective is to determine the feasibility of obtaining elemental oxygen from silicate minerals by electrolytic methods, for use by the Earth inhabitants of the Moon. Emphasis will be directed toward the determination of essential physical and electrochemical properties of silicate and silicate-base melts containing various amounts of halide salts. Complementary investigations will be made to find suitable nonreactive crucible and anode materials for use in silicate melts, or in melts containing halides.

### Progress During the Third Quarter

The objectives for the quarter were to initiate a study of the properties of fluoride-base mixtures, containing relatively small proportions of silicate rocks, and to determine the compatibility of platinum-group metals or alloys with these mixtures.

Experiments were conducted to determine the physical characteristics of fluoride-base, silicate-bearing mixtures at 1,000° to 1,250°C. A melt containing 70 weight-percent $BaF_2$, 5 weight-percent LiF, and 25 weight-percent basalt-plus-sinter showed good electrical conductivity, low viscosity, and negligible fuming at these temperatures, and was used for anode-melt compatibility evaluations. Iridium, platinum, and a platinum-rhodium alloy were tested at 1,250°C. In a 30-minute static test, the iridium was not attacked; however, the other two metals dissolved. Further testing was conducted in which direct current was applied to iridium specimens connected as the anode in an electrolytic cell. At 1,250°C, with a current of 12 amperes and an emf of about 4 volts, the rate of iridium dissolution was about 0.1 g/amp hr. Highly encouraging results were achieved in experiments conducted at lower cell temperatures. At 1,100°C, with a current of 12 amperes and an emf of about 5 volts, no measurable corrosion of the anode occurred, while at 24 amperes and 10 volts, the corrosion rate was only 21 mg/amp hr. Studies of this type will be extended to other silicate-bearing fluoride mixtures.

### Status of Manuscripts

None in progress.

| | |
|---|---|
| Task title: | Stability of hydrous silicates and oxides in lunar environment |
| Investigator: | Hal J. Kelly, Project Coordinator |
| Location: | Albany Metallurgy Research Center<br>Albany, Oregon |
| Date begun: | April 1966 To be completed: March 1968 |
| Personnel: | Hal J. Kelly, Supervisory Ceramic Research Engineer<br>Raymond L. Carpenter, Research Physicist |

PROGRESS REPORT

## Objective

The long-range objective is the determination of the energy requirements for dissociating silicate and oxide minerals to recover oxygen and/or water. The immediate objective is to investigate the stability under high vacuum and elevated temperature of some silicate and oxide minerals employing differential thermal analysis (DTA) and thermogravimetric analysis (TGA).

## Progress During the Third Quarter

The objectives for the quarter were to continue the DTA and TGA studies of the minerals which have been obtained and purified.

The samples of actinolite and epidote, which were found to contain small amounts of carbonate, were purified by acid leaching and washing. TGA studies have been started on these samples.

A TGA run was made on the leached sample of actinolite. There was no significant weight loss below 1,000°C for the leached material, but unleached material had a weight loss of 3.54 percent between room temperature and 800°C. The low-temperature weight loss is due to the decomposition of the carbonate impurity. There is still a small amount of the carbonate in the sample that could not be removed by leaching. This caused a small weight loss of 0.38 percent below 1,000°C.

TGA runs were made on the minerals of the epidote group, epidote and zoisite. Both samples behaved similarly. The major weight loss, which is due to the loss of lattice water, took place between 940° and 1,060°C. The total weight loss for the two minerals also was similar; epidote lost 2.04 percent and zoisite lost 1.97 percent. The weight loss curve for zoisite agrees with the DTA data of MacKenzi.[1] His curve shows an endothermic deflection beginning at 938°C and reaching a peak at 998°C. The

[1] MacKenzi. The Differential Thermal Investigation of Clays. Mineralogical Soc., London, England, 1957, 377 pp.

DTA he published on epidote shows that the endothermic reaction takes place in steps beginning at 912°C; two small arrests occur at 938° and 987°C and reach a peak at 1,018°C. The TGA data did not show these two arrests, but the weight loss started at about the temperature of the first arrest on the DTA curve.

Vacuum DTA has been made on tremolite. Two sizes of starting material were used. No peaks appeared on the thermograph for the fine-size fraction, minus 200 mesh. Two runs were made on the 100/200 mesh material, and these showed two endothermic peaks at 855° and 1,707°C.

New-type tantalum crucibles with 1/4-inch deep thermocouple well instead of the 1/2-inch deep well have been made. These crucibles hold more sample and, if the peak area is proportional to the sample mass, should produce larger peaks on the thermograph. When the 100/200 mesh tremolite was run in the new crucibles, there was no indication of thermal activity on the thermograph.

To check the effect of the design of the crucible on the size of the reaction peak, five runs were made on kaolinite which had been prefired to 750°C. This material had one exothermic reaction at 980°C.

The results of these tests show that on this type crucible the area under the curve is proportional to sample size in the range of sample weight from 0.30 gms to 0.45 gms. A similar series of tests will be made with the new-type crucibles to see if there is a range of sample weights in which the area under the curve is proportional to the sample weight.

## Status of Manuscripts

None in progress.