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MULTIDISCIPLINARY RESEARCH LEADING TO  
UTILIZATION OF EXTRATERRESTRIAL RESOURCES

Quarterly Status Report  
October 1, 1967 to January 1, 1968

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

QUARTERLY STATUS REPORT

October 1, 1967 to January 1, 1968

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STATUS REPORT SECOND QUARTER FISCAL YEAR 1968

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

January 1, 1968

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

PROGRESS REPORT

Objective

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 Second Quarter

The core group continued to obtain and analyze information applicable to the program. Following the suggestions of a number of the research task investigators, a greater effort is being made to provide general background information, particularly results from the various lunar probes, to all of the task groups. To help with this effort we are obtaining and distributing multiple copies of some NASA and NASA contractor reports and providing lists of references to articles in the technical journals.

The format of the current status report is changed slightly. A few groups of research tasks have been combined, and some others have been separated, so that the work of each team of researchers is now reported as a single task. It should be kept in mind that each task is a part of a regular program of Bureau research and most members of a research team work only part-time on the NASA task. The total effort involved in the NASA work represents about 15 man-years per year.

In early October, Dr. Jack Green, McDonnell Douglas Advanced Research Laboratories, visited the Twin Cities Center and reported on his investigations of the Galapagos volcanoes. Also, as a member of the team evaluating the results of the chemical analysis of the lunar surface

made by Surveyor V, he discussed the reasons why the team concluded that the material sampled was of basalt-like composition. This result, confirmed by the analysis from the more recent Surveyor VI, is particularly gratifying in view of the emphasis given to volcanic rocks in the Bureau of Mines studies.

Later in October, Atchison and Fogelson visited the USGS Center of Astrogeology, Flagstaff, Ariz., and the NASA Manned Spacecraft Center, Houston, Tex., to discuss the latest interpretations of the data from the Surveyor landings and the Lunar Orbiter missions. Of particular interest to the Bureau's program was the extensive research in geology, geochemistry, and geophysics, related to lunar surface problems, that is being carried out at Houston. Also of interest at the Houston Center were the plans for operation of the Lunar Receiving Laboratory and for establishing an earth resources satellite program.

In November, Atchison visited the Pittsburgh Explosive Research Center and Schultz visited the College Park Metallurgy Research Center to discuss the progress of the research tasks at these centers. Atchison also attended a meeting of the Steering Committee of the Working Group on Extraterrestrial Resources (WGER) at NASA's Washington headquarters. At the same time, Atchison and Schultz conferred with Mr. James Gangler on future plans for the Bureau's program.

Four papers describing the progress of the Bureau's program are planned for presentation at the Sixth Annual Meeting of the WGER at Brooks Air Force Base, Tex., February 19-21, 1968.

#### 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, is being prepared for presentation at the Sixth Annual Meeting of the Working Group on Extraterrestrial Resources at Brooks Air Force Base, Tex., in February.

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

Select and obtain samples of rocks and minerals covering the range of materials likely to be found on the Moon.

### Progress During the Second Quarter

A field trip was made to Big Rock Candy Mountain near Marysvale, Utah, to collect altered latite as a possible substitute for the altered rhyolite previously collected from a site near Bend, Oreg. Because the altered latite is highly fractured and difficult to mine without causing excessive breakage, the altered rhyolite will be retained as a member of the simulated lunar rock suite. While the altered rhyolite is also fractured and not too desirable as a testing material, large blocks can be secured. With very careful selection, adequate test specimens can be prepared. About one-half ton of partly altered latite was collected from the Big Rock Candy Mountain site for possible future tests.

Dr. Paul Lowman of NASA's Goddard Space Flight Center requested samples of the Bureau's rock materials for use at Goddard in a study of simulated Martian soil. Several pounds each of rhyolite, altered rhyolite, semiwelded tuff, granodiorite, dacite, flow basalt, gabbro, serpentinite, and dunite were selected and sent to Goddard.

### Status of Manuscripts

Simulated Lunar Rocks, by D. E. Fogelson, is being prepared for presentation at the Sixth Annual Meeting of the Working Group on Extraterrestrial Resources at Brooks Air Force Base, Texas in February.

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 projects are participating

PROGRESS REPORT

Objective

To incorporate simulated lunar materials into basic fragmentation re-  
 search currently in progress. By this means to determine the composi-  
 tion, elastic, strength, surface, thermal, electrical, magnetic, and  
 explosive shock properties of simulated lunar materials in Earth envi-  
 ronment.

Progress During the Second Quarter

Samples of flow basalt and dacite were prepared for studies of the ef-  
 fect of confining pressure and rate of loading on the strength and elas-  
 tic properties by the Rock Physics laboratory. Previous determinations  
 of the principal symmetry axes were used to obtain property oriented  
 samples.

Preliminary measurements of the magnetic susceptibility of 14 simulated  
 lunar rocks were made by the Explosive Fragmentation group. Two samples  
 of each rock type were measured using a standard magnetic susceptibility  
 bridge. Nonuniformity in the samples limited the accuracy of the results  
 (see table 1) to about 10 percent.

TABLE 1. - Magnetic susceptibility of simulated  
 lunar rocks in earth environment

Rock type	Magnetic susceptibility ( $10^{-6}$ cgs units)
Dunite	400
Gabbro	6,000
Tholeiitic basalt	2,000
Granodiorite	Negligible
Serpentinite	6,000
Obsidian	100
Altered rhyolite	Negligible
Rhyolite	400
Vesicular basalt #1	400
Vesicular basalt #2	500
Dacite	600
Vesicular basalt #3	200
Semiwelded tuff	400
Pumice	Negligible

The Thermal Fragmentation group completed the repeat measurements of electrical dissipation factors being made on several of the simulated lunar rocks in an effort to reduce the spread in the data from the original measurements. Statistical analysis of the results to determine the frequency dependence of the dissipation factors from 20 to 100 megahertz is not yet completed.

Several property measurements were made on the altered rhyolite, following a decision to reinstate this rock in the lunar suite.

#### 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, is being prepared for presentation at the Sixth Annual Meeting of the Working Group on Extraterrestrial Resources at Brooks Air Force Base, Texas in February.

Task title: Chemical reactivity and cold welding of freshly formed surfaces  
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  
William H. Engelmann, Research Chemist  
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 Second Quarter

The thermal conductivity cell or bridge used for sensing changes in gas composition, has been run at five concentration levels of water vapor in an argon carrier gas. The levels were very low, ranging from several torr partial pressure down to the  $10^{-4}$  torr region. The reference gas was argon, dried with magnesium perchlorate, followed by a cold trap maintained at  $-120^{\circ}\text{C}$ . This reference gas was run through the standard half of the bridge or cell which contains two of the four resistance units in a Wheatstone bridge configuration. The emergent gas was then routed through a water vapor saturator, followed by a cold trap maintained at any one of five temperature levels by selected organic compounds at their fusion temperature. These temperature levels established the water vapor levels which then were passed through the comparator half of the cell. The electrical output of this bridge gave values in the small millivolt range at the higher water vapor levels and much less than 1 millivolt at the lowest level. Braided shielding was installed over the wiring harness to the cell, to help to suppress the noise. At the lower temperature levels of the condenser, where the output was under 0.1 millivolt, drifting made reproducible millivolt measurements unattainable. The well-stabilized dc power supply used as input to the Wheatstone bridge network, was supplanted by a 6-volt storage battery. This improved the output characteristics to the extent that we can report the two higher water vapor level responses with some confidence. All five millivolt values as a function of vapor pressure will be graphed and presented in a subsequent report.

Several baseline-determining runs were made with argon containing 0.62 percent water vapor ( $0^{\circ}$  condenser,  $\text{H}_2\text{O}$  partial pressure of 4.6 torr) as the reference gas. A glass-lined ball mill (4-1/2-inch diameter by



5-1/2-inch length), filled with 100 grams of dried 65/100 mesh quartzite, showed adsorption to the saturation point in 80 minutes with a flow rate of 165 cc/min. The mill contained no balls in these runs, the immediate object being to establish the saturation requirements of the surface presented by the starting material.

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 Second Quarter

This past quarter a paper on outgassing of simulated lunar materials in ultrahigh vacuum was presented at the 14th American Vacuum Society Symposium in Kansas City. Work continued on the outgassing studies using 1/4-inch flat samples of material.

It has been found that the friction device would not be adequately supported in the originally designed manner. A new support frame has, therefore, been designed and is presently in the process of being made. This frame will greatly improve the reliability and the capability of the friction device. The external configuration for mounting the drive motor and gear reduction unit of the friction system are being finalized. This should be ready for testing by the time the support frame is received.

Some time has been spent this quarter on equipment maintenance. The Varion ion gauge control, the electron energy control on the mass spectrometer, the Kriesman gauge control, and a rotary force pump for sample pretreatment all went out of operation at the same time. To repair the ion gauge controls a transistor tester had to be purchased or fabricated. A short survey of assembled cost versus parts cost indicated that it should be made locally. The parts were, therefore, obtained and the tester was constructed.

### 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, is being prepared as a Note to the Editor in Review of Scientific Instruments.

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  
Minneapolis, Minnesota  
Date begun: June 1966 To be completed: June 1969  
Personnel: Egons R. Podnieks, Mechanical Research Engineer  
Robert J. Willard, Geologist  
Thomas R. Bur, Geophysicist  
Richard E. Thill, Geophysicist  
Peter G. Chamberlain, Geophysicist  
Kenneth E. Hjelmstad, Geophysicist  
Richard M. Brumley, Electronics Technician

## 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 Second Quarter

Data from the preliminary environmental test program were analyzed this quarter to determine the effects of pressure, temperature, and moisture content on the strength and elastic properties of two simulated lunar rocks, flood basalt and dacite. Property measurements in moderate vacuum ( $10^{-3}$  torr) are providing important background information for the ultrahigh vacuum measurements that will be made soon. For the basalt moderate vacuum, as compared to atmospheric pressure, had little effect on compressive strength or Young's modulus at room temperature, but at 212°F the vacuum environment resulted in an increased value for both properties. For the dacite at both 72° and 212°F the vacuum caused an increase in strength, but no change in modulus.

Both basalt and dacite showed an increasing Young's modulus as temperature decreased from 212° to -250°F, as was earlier reported for compressive strength. Increasing moisture content from ultradry to saturated condition resulted in a small decrease in strength at all temperatures for the basalt. For the more porous dacite increasing moisture content caused a similar decrease in strength at 212° and 72°F, but a substantial increase in strength at -250°F. Further analysis is being carried out to determine the statistical significance of these and other trends shown by the environmental test data.

In conjunction with the environmental tests, acoustic velocities were measured by both pulse and resonance methods on several basalt and dacite

cores. The measurements were made at room temperature under three different moisture conditions, ultradry, room, and saturated. The nondestructive nature of the acoustic measurements permitted using the same cores for all three conditions, eliminating variations due to specimen differences. Comparative results are shown in table 1. The basalt showed only small differences in velocity due to moisture content. The more porous dacite showed substantial differences. Of outstanding interest is the opposite direction of the moisture effect for pulse velocity and resonance velocities.

TABLE 1. - Percentage change in acoustic velocities for room and saturated conditions based on the velocities for the dry condition

Rock type	Pulse velocity		Bar velocity		Torsional velocity	
	Room	Saturated	Room	Saturated	Room	Saturated
Flood basalt	-.1	+1.2	+.1	+.8	-.1	+.6
Devil's Hill dacite	+.3	+10.9	+.4	-3.6	+.4	-7.8

The fracture morphology study of the dacite samples from the environmental test program was continued. A computer program was prepared to help in the statistical evaluation of the effect of the test parameters on the transgranular-intergranular ratio and the areal percentage of minerals at the fracture surface. The computer program carries out the inversion of a 5 by 5 matrix, required in the statistical evaluation.

The application of scanning electron microscopy to the study of rock fracture is being investigated. Dr. Willard visited the demonstration laboratory of the Japan Electron Optics Laboratory Company in Medford, Mass., in December. Several of our rock samples were scanned and various surface features were photographed at magnifications ranging from 100X to 30,000X. The photographs showed the unparalleled capability of the instrument to resolve surface microtopography. Evidence of various deformational features on fractured surfaces of the samples suggest that the scanning electron microscope can provide a means of relating microstructural aspects of rock deformation to gross behavior.

The ultrahigh vacuum chamber is in the process of being adapted for use with the compression testing machine. Facilities were installed to provide easy transportation of the chamber in and out of the testing machine. Both the bellows and the platens were received from their respective manufacturers and have undergone extensive cleaning prior to final installation in the chamber. Rock specimens will be prepared, inspected, and preconditioned during the next quarter, and tests will start either during the third quarter or early in the fourth quarter.

Mr. Egons R. Podnieks has assumed the duties of Project Leader for the Rock Physics laboratory at the Twin Cities Mining Research Center and is carrying out the responsibilities of investigator for this NASA task. Dr. Robert J. Willard has assumed the position of Project Leader for a newly separated Microstructural Analysis laboratory. He will continue his contributions to this task.

Status of Manuscripts

None in progress.

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  
Daryl J. Jersak, Engineering Technician

## PROGRESS REPORT

### Objective

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

### Progress During the Second Quarter

Development of thermal conductivity probes that can be applied to vacuum chamber measurements continued. Planned experiments using the vacuum system associated with the thermal shock furnace were delayed because of the need for developing more experience with the furnace itself and the pressure of other work in the Thermal Fragmentation laboratory.

### 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  
Walter 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 Second Quarter

Thermal expansion measurements on simulated lunar rocks over the temperature range from  $-320^{\circ}$  to  $2,000^{\circ}\text{F}$  were continued. Measurements for ten of the rock types are nearly completed. For most of the rocks the expansion coefficient increases with increasing temperature in a uniform manner. Some of the lower density rocks show anomalous behavior above  $1,000^{\circ}\text{F}$ , with decreased expansion or even contraction taking place. The rocks appear to soften and are weakened at lower temperatures than the usual mine rocks we have studied previously.

### Status of Manuscripts

Flexural Strength of Rock from  $-320^{\circ}$  to  $1,600^{\circ}\text{F}$ , 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 Second Quarter

Laboratory experiments were completed using a diamond drilling system instrumented to measure torque, thrust, and penetration rate. Use of this equipment permitted careful control of drilling parameters as the instrumentation gave continuous readouts on recorders. These tests were an extension of the bench drilling tests previously reported, the purpose being to study the effects of flushing media on drill performance and to investigate the feasibility of utilizing a solid lubricant in lieu of other flushing media in dry diamond drilling.

The tests results, still being analyzed, indicate that a liquid flushing media may have a surface weakening effect on the drilled rock that is absent when air flush is used. Presence of this effect was confirmed by diamond point scratch tests in the laboratory. The use of a solid lubricant in lieu of liquid flushing media was found to minimize the drill torque developed for a given rate of penetration and thereby to conserve energy.

### Status of Manuscripts

Lunar Drilling, by James Paone and R. L. Schmidt, is being prepared for presentation at the Sixth Annual Meeting of the Working Group on Extra-terrestrial Resources at Brooks Air Force Base, Texas, in February.

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

The crushing and classification circuit for producing fine particle materials and simulated lunar rock powder, located at the Auxiliary Laboratory site, was completed during the quarter. A fan and ventilation duct work were installed in the crusher building. In addition, a small cone crusher was purchased and added to the original circuit to reduce the crushed product from the #2-1/2 gyratory crusher to a more suitable size feed for the impact mill. The impact mill feed must be 1/8-inch size to avoid choking the distribution plate inside the mill. The circuit flow as presently installed is shown in figure 1.

Background literature studies of materials handling science and the surface chemistry of fine particles was continued.

The conversion of the "Earth Pressure at Rest" ( $K_0$ ) testing chamber was completed during the quarter. Low stress gages were seated in the loading head and base plate of the chamber to give true applied and transmitted stress data from 0 to 150 psi. Several series of shakedown tests have been run on the chamber using standard Ottawa sand to check out the instrumentation and the Dymec data collection system. The three strain

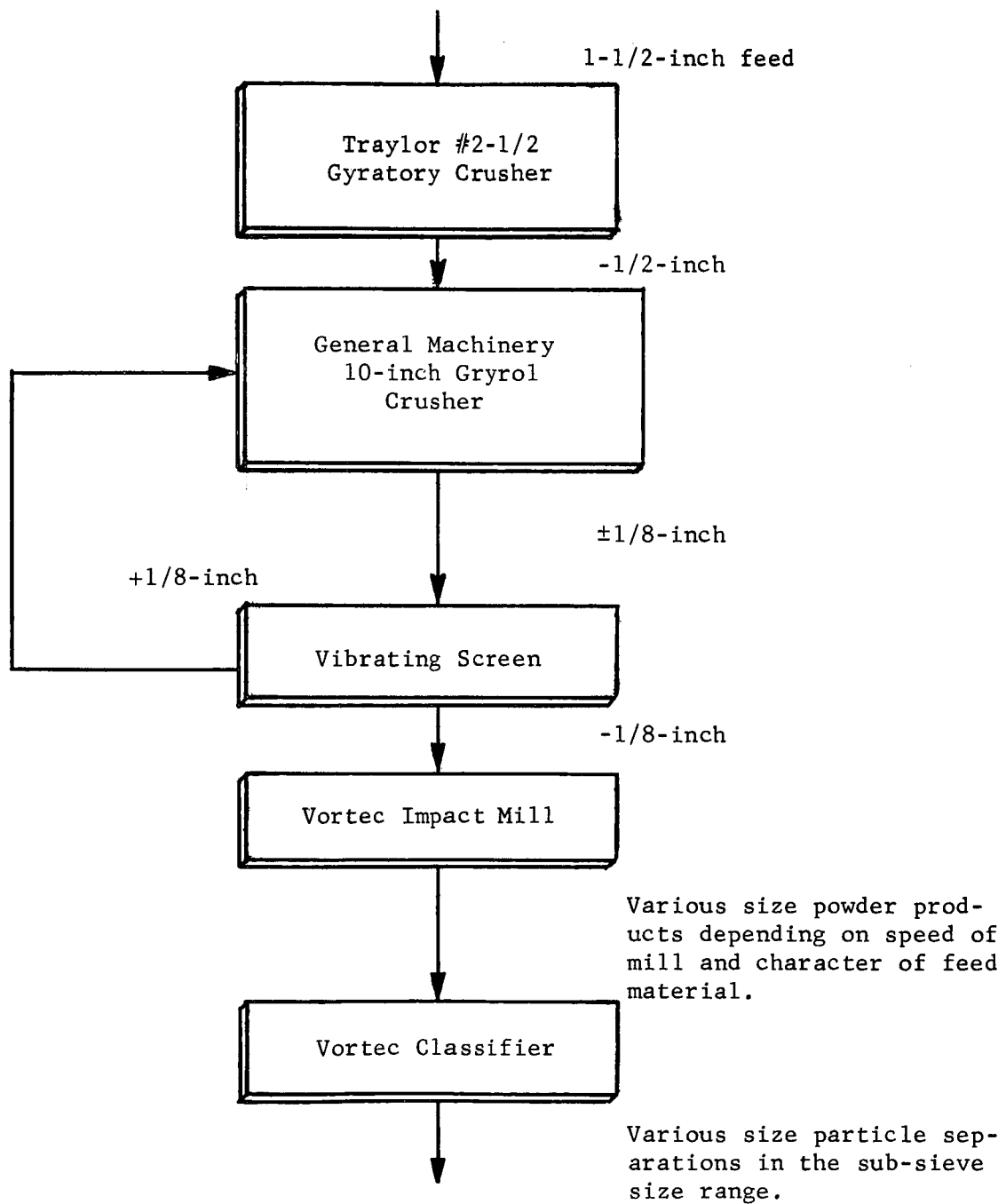


FIGURE 1. - Crushing and Classification Circuit.

gage bridge circuits, which monitor lateral stress, have been modified. During shakedown testing it was observed that the amplifier-milliammeter output was not compatible with the input to the Dymec data collection system. A Sanborn recorder has been substituted for the amplifier-milliammeter system, correcting the problem and also giving a better resolution of the lateral pressures used to null the lateral stress. The linear potentiometer which was to be used for producing a dc current readout of the applied load from the Tinus Olsen testing machine was also found to influence the torsion bar deflection on the testing machine. On the recommendation of the Tinus Olsen distributors a single turn potentiometer will be substituted and mounted with a special antibacklash gear which will give a direct readout from the load dial of the testing machines. This unit has not been received from the company, but was shipped on December 12, 1967. Installation of the turn pot and antibacklash gear will complete the testing equipment.

It was originally planned to start the materials handling test work in the  $K_0$  testing chamber with a silica flour or standard backfill material. However, recent data from the Surveyor space craft indicate a basalt type of lunar surface material of about 50 to 30 microns mean size; therefore, a sample of crushed and milled flow basalt has been prepared and will be substituted in this test work. The simulated lunar material is from the flow basalt formation at Madras, Oregon.

In addition to preparing the  $K_0$  test chamber for testing the friction constants, standard laboratory physical property tests were started to determine specific gravity, grain size distribution, and maximum-minimum density, as well as direct shear tests to determine angle of internal friction at three levels of density. The angle of internal friction developed from the direct shear tests will be correlated with the friction coefficients from  $K_0$  testing.

Samples of the Oregon basalt were also tested for magnetic attraction. The samples were found to be highly magnetic.

#### Status of Manuscripts

None in progress.

Task title: Support for underground lunar shelter  
Investigator: Robert C. Bates, Project Leader  
Location: Spokane Mining Research Laboratory  
Spokane, Washington  
Date begun: April 1966 To be completed: March 1969  
Personnel: Robert C. Bates, Mining Engineer  
Lester J. Crow, Mining Engineer

## PROGRESS REPORT

### Objective

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 Second Quarter

The major part of the equipment needed to conduct laboratory studies was assembled at the main laboratory in Spokane. The equipment was tested to determine its suitability, and the workability of some sulfur aggregate mixtures was investigated. It was found that out in the wind and cold, where these tests were being performed, it was quite difficult to control the temperature of the mix. When the mix was poured into small steel test molds it solidified too rapidly. Further, the odor from the experiment was undesirable in this location.

In view of these problems, a new test area has been established at the Auxiliary Laboratory (former missile site), where experimentation can be continued under more controlled conditions. Complete facilities are being set up to grade, mix and pour the sulfur aggregate. A 200,000-pound compression testing machine has been set up in an adjacent area so that property tests can be made at the Auxiliary Laboratory.

Some of the potential obstacles to the use of sulfur in an extraterrestrial environment have been evaluated. Potential problems imposed by the lunar environment appear solvable by proper design of the placement

system and proper formulation of the mix. However, a detailed evaluation has not been made. A Ph. D. physicist and a Ph. D. physical chemist, part-time faculty employees, have been assigned the task of examining these potential problems and preparing an evaluation report.

Status of Manuscripts

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

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

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 Second Quarter

During the quarter, determination of the sensitivity of explosive charges to initiation by high-velocity spherical projectiles was begun; the velocity for 50 percent initiation has, thus far, been established for 1/2-inch, 25/64-inch, 1/4-inch and 1/8-inch diameter steel balls.

The explosive used was Composition A-5 (RDX with 1 percent stearic acid) pressed as pellets to a density of 1.72 g/cm<sup>3</sup> and 1-5/8-inch diameter by 1/2-inch thick. Two such pellets were stacked to provide a charge length of 1 inch. The anticipated point of impact of the projectile was on the axis of the cylindrical charge; a 4-inch by 4-inch by 1/4-inch thick steel plate was placed 1/4 inch behind the charge to act as a witness. In order to minimize ambiguities in interpretation of a "go" or "no-go," a well-defined 1-5/8-inch hole in the steel plate was considered to result from complete reaction. The Bruceton up-and-down method was employed to determine the velocity capable of producing a 50 percent probability of initiation. The velocity of the steel ball was varied by changing the quantity of propellant in the gun or launcher.

The projectile launcher is a modified 50-calibre anti-tank gun having a smooth 1/2-inch bore to eliminate spinning. A teflon sabot was used to launch all balls having diameters less than 1/2 inch. The sabot was a cylinder, 1/2-inch in diameter and approximately 19/32-inch long, cupped in front to receive the ball and scored in quarter segments to insure breakup by aerodynamic drag. The cup was slightly smaller than the ball to be used and was drilled axially to within 1/8 inch of the rear of the sabot.

The ball velocity was measured 70 cm in front of the explosive charge by timing the interruption of two parallel focused light beams spaced 4 inches apart: As each beam was interrupted by the projectile, a voltage pulse from the light detector (a photovoltaic solid-state diode) was fed to the signal channel of an oscilloscope with a calibrated time base, triggered by the interruption of a third beam ahead of the others.

The data thus far have provided tentative trends between the mass and the velocity that provide a 50 percent probability detonation of the explosive. The data are given in table 1 and figure 1 which also il-

TABLE 1. - Mass-velocity relationships for 50 percent probability of initiation of pressed Composition A-5 charges

Diameter (inches)	Mass (grams)	$v_{50}$ (m/sec)	$m^{0.33} v^2$	$m^{0.24} v^2$
1/2	8.33	918	170	143
25/64	3.95	1030	168	149
1/4	1.04	1215	150	148
1/8	0.13	1447	106	126

lustrate attempts to find a scaling relationship of the form

$$(\text{mass})^A \times (\text{velocity})^B = \text{constant.}$$

For the three largest projectiles, the quantity  $m^{0.33} v^2$  (which has the significance of energy/unit area) is approximately constant, but when the data for the 1/8-inch diameter projectiles are included, the data are better fit by the relationship

$$m^{0.24} v^2 = k.$$

These data are being used to establish a basis for the design of true hypervelocity projectors for which the selection of projectile mass and velocity is not so trivial a matter as with a conventional gun, and which thus cannot be used on a trial-and-error basis. The data obtained at hypervelocities will be extrapolated to predict the probability of initiation by micrometeorites.

Plans are in progress for the readaptation of the 12-foot spherical firing chamber to high vacuum work to continue the work of Ahrens and others in the propagation of blast clouds in a vacuum. The chamber will be sandblasted and coated with a low vapor pressure resin to protect the metal from attack by explosive products. Efforts are being made to procure a pump of suitable ultimate vacuum and pumping rate.



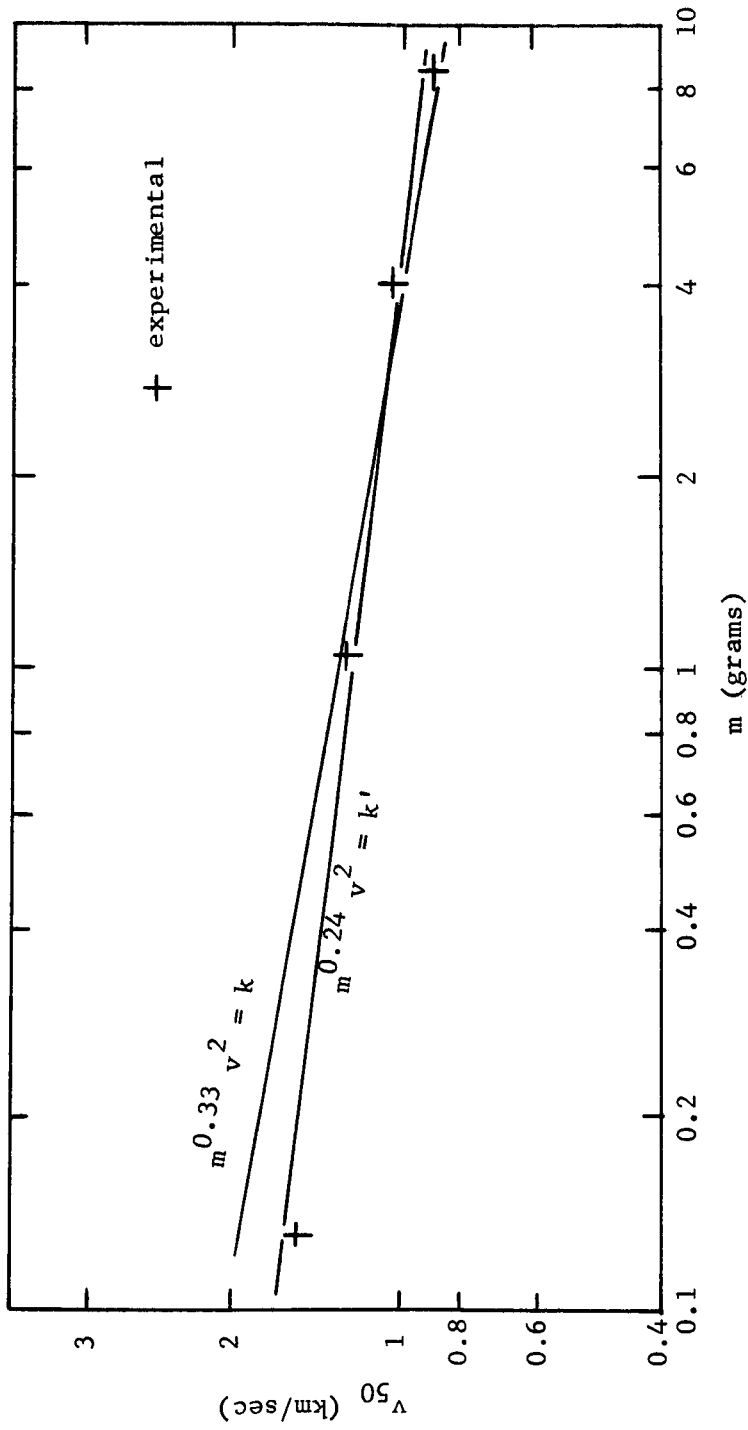


FIGURE 1. - Velocities for 50 Percent Probability of Initiation vs Projectile Mass.

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 Second Quarter

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

### Status of Manuscripts

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

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

## PROGRESS REPORT

### Objective

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 Second Quarter

The goal in the current quarter was to develop a method whereby finely divided graphite and silica powders could be agglomerated into a strong, porous briquet and to study the reduction of the resulting briquets.

Attempts were made to agglomerate silica and graphite powders into a briquet in order to study the effect of the ratio of exposed surface to the total volume of the briquet. The results last quarter indicated that the reaction rate was very dependent on the bed porosity or the reactants particle size. The first briquets were made without a binder by compressing the powdered mixture in a 3/4-inch diameter die at 50 tons per square inch. However, these briquets were very fragile and were so dense that very little reaction occurred in the vacuum furnace at 1,400°C. The reactants were then agglomerated without pressure, using dextrose as a binder. Dextrose, when heated to high temperatures, decomposes to carbon and water. Since water is readily driven off as a gas, the addition of dextrose as a binder and as a volatile filler also adds additional amounts of carbon to the system.

The thermal properties of reagent grade dextrose were first determined. At about 150°C, a foaming syrupy mass was obtained which on further heating became a porous, voluminous charred sinter. The foaming action is due to the escaping water from the dextrose dehydration reaction. This reaction was quite slow even at 500°C. At 600°C constant weight was obtained in less than 4 hours. Theoretically, the weight loss should be 40 percent if all the dextrose is decomposed to carbon. However, about half of the dextrose vaporized out of the reactor so only half of the theoretically available carbon was retained in the charge.

The first batch of briquets were made by dry-mixing 80/120-mesh silica with 33 percent dextrose by weight. Five percent water was added to the powdered mixture and the paste was pressed into cylindrical holes in a teflon mold. The material in the mold was then air-dried to constant weight in 20 hours at 90°C. The briquets were then transferred from the mold to a Pyrex boat and heated in a CO atmosphere at a rate of 25°C per hour up to 300°C and soaked for 16 hours. The temperature was then rapidly increased to 600°C, and held for 6 hours and then cooled. After this treatment, the 33 percent dextrose briquets were found to be deformed, fragile, and very porous. A new batch of briquets were then made by decreasing the percent dextrose to 20 percent and adding graphite to maintain the equimolar SiO<sub>2</sub>/C ratio. These briquets were strong after air-drying at 90°C or after firing at 600°C for 4 hours in a CO atmosphere. The results of these measurements are given in table 1 where the compressive strength is defined as the amount of force needed to crush a briquet, 1/2-inch in both diameter and length, when the force is applied parallel to the axis of the cylinder. When the 20 percent dextrose briquets were placed in the vacuum furnace, they also disintegrated due to explosions caused by carbon monoxide formation.

TABLE 1. - Compressive strengths of dextrose briquets

	Test numbers				
	1	2	3	4	5
Weight of reactants...percent.					
Dextrose.....	33	20.0	20.0	5.1	6.0
Silica.....	67	73.4	73.4	80.7	94.0
Graphite.....	0	6.6	6.6	14.2	0
Calculated molar ratio.SiO <sub>2</sub> /C.	1	1	1	1	8
Sintering temperature....°C...	600	600	1500	1500	1500
Compressive strength.....lbs..	Nil	6	50	14	48
Weight-loss.....percent/hr..	15.2	12.8	12.8	<sup>1</sup> 18.6	7.2

<sup>1</sup> Sample blowout was noticed at end of test.

Attempts were then made to make a stronger briquet by sintering at 1,500°C for 2 hours at 1 atmosphere carbon monoxide pressure. At these experimental conditions, no carbon monoxide would be generated and the temperature would be high enough for the silica particles to fuse together and make a stronger briquet. It can be seen in table 1 that the compressive strength was increased more than eightfold but these briquets still broke up in the vacuum furnace. Other investigators<sup>1</sup> have shown that the physical integrity of refractory oxide briquets decreases as the percent reduction increases. To reduce the reaction rate, a third batch of the briquets were made with only 5.1 percent dextrose and 14.2 percent graphite, and again the briquets broke up in vacuum.

<sup>1</sup> Crowley, M. S. Hydrogen-Silica Reactions in Refractories. Amer. Ceram. Soc. Bull., v. 46, No. 7, 1967, p. 680.

The next approach was to eliminate graphite as this material has a low coefficient of friction. Removing graphite would also increase the number of silica-silica contacts. A 6 percent dextrose - 94 percent silica batch of briquets were made and after firing at 1,500°C in a carbon monoxide atmosphere, strong, grey briquets were obtained. These briquets were able to withstand the vacuum treatment. The foregoing trials indicate that a briquet can be made which will maintain its physical integrity when exposed to vacuum at 1,400°C provided that: (1) very little reductant in the form of dextrose is used, and (2) the briquet is pre-heated in carbon monoxide at 1,500°C.

Since thermally decomposed dextrose undoubtedly forms a different type of reductant at 1,400°C than graphite, a series of vacuum reduction tests were carried out with various proportions of both reductants. The experimental results are shown in table 2.

TABLE 2. - Data for carbothermal reduction of silica with carbonized dextrose or graphite<sup>1</sup>

Test No.	Charge			Discharge			Observed weight-loss, percent
	Analysis, percent		Weight, grams	Analysis, percent		Weight, grams	
	Carbon	Silica		Carbon	Silica		
Carbonized dextrose reductant							
6	1.08	96.7	13.940	<0.01	98.0	12.935	7.2
7	1.70	95.9	12.551	.23	97.5	11.585	7.7
8	2.76	94.5	11.928	.83	96.6	10.728	10.1
9	2.76	94.5	13.341	.80	97.2	11.769	11.8
10	4.66	93.8	10.019	3.06	95.9	8.483	15.3
11	5.72	92.2	12.657	3.82	93.6	10.731	15.2
Graphite reductant							
12	1.4	98	12.295	0.15	96.7	11.509	2.9
13	3.8	96	11.734	.26	97.6	9.177	4.4
14	4.8	95	12.596	1.92	99.3	11.129	2.3
15	7.0	93	12.897	4.43	97.4	11.905	1.5
16	9.1	90	13.142	6.57	95.3	12.134	1.5
17	16.7	83	14.373	14.6	87.3	13.318	1.4
18	16.7	83	14.379	14.8	85.7	13.457	1.3
19	28.6	81	16.765	27.5	72.5	15.964	0.96
20	28.6	81	16.792	27.5	72.5	15.988	.90

<sup>1</sup> These five-hour experiments were performed at 1,400°C and pressures <0.01 torr. The charge consisted of minus 80- plus 120-mesh silica and either minus 100- plus 200-mesh low density graphite or thermally decomposed dextrose.

It can be seen from these results that carbonized dextrose is a better reductant than graphite. This is undoubtedly attributed to the intimate contact of the carbonized dextrose to the silica; since dextrose when heated will liquify and coat the silica particles. This is in agreement with the results of Vertman<sup>2</sup> who reported that various forms

<sup>2</sup> Vertman, A. A., and A. M. Samorin. (Kinetics and Mechanisms of Reduction of Chromium Oxide by Carbon in Vacuum). *Primenenie Vakuuma V Metallurgia*, Moscow, 1958, pp. 132-146.

of carbon have different reducing activities. He showed that soot reduced chromium oxide almost twice as fast as graphite. Saltsbury<sup>3</sup> also stated that charcoal has a higher activity than graphite, probably due to adsorbed hydrogen. The plot of the observed weight loss expressed as the percent oxygen removed per hour from silica against the initial percent solid reductant in the charge is shown in figure 1. When reduction was carried out with carbonized dextrose, curve a was obtained, while curve b represents the reduction data with low density graphite. It is interesting to note that a maximum reaction rate can be achieved with percents of graphite ranging from 3.5 to 4, whereas maximization of the rate with carbonized dextrose, although probably possible, was not achieved by the present set of data. Pellets with dextrose percents higher than about 19 percent (corresponding to 6 percent charred carbon) were difficult to prepare.

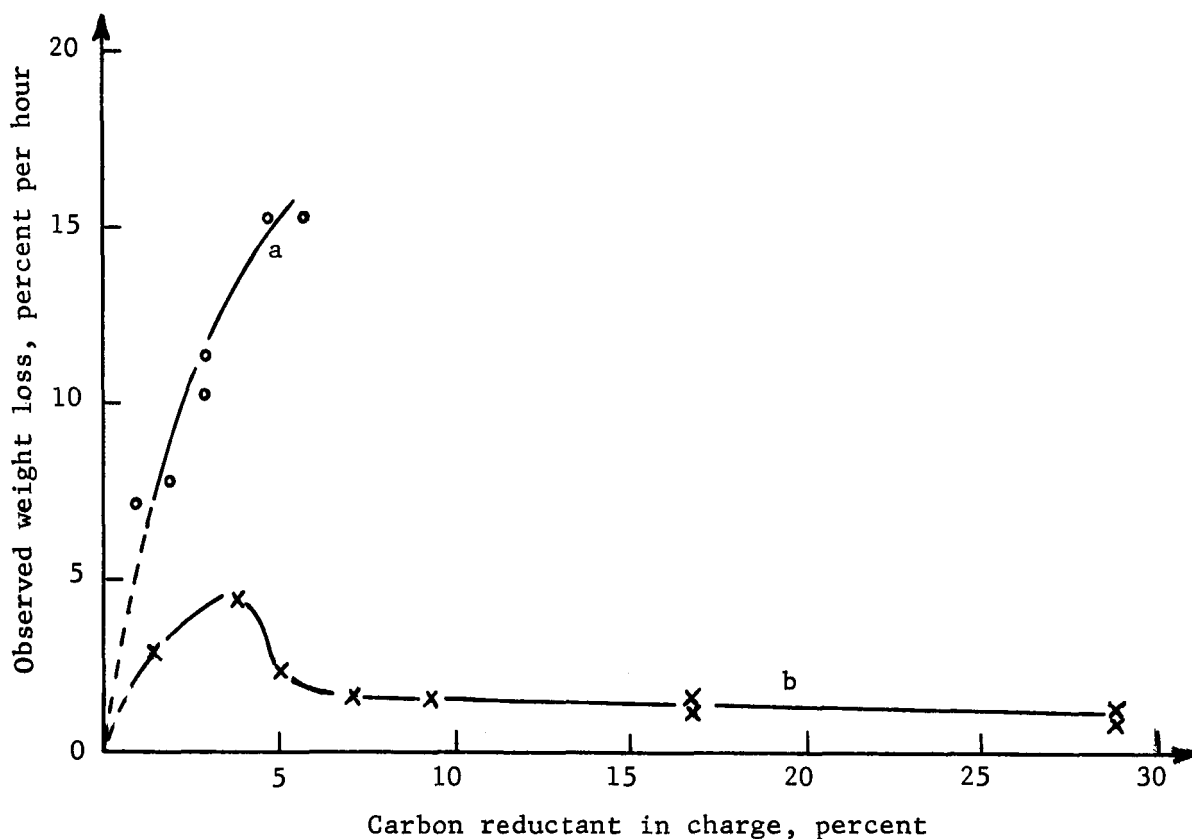


FIGURE 1. - Variation of Observed Weight Loss per Hour with Percent Carbon in the Charge  
 a - Carbonized Dextrose  
 b - Low Density Graphite

<sup>3</sup> Saltsbury, H., and others. Fundamentals of Gas-Surface Interactions. Academic Press, N. Y., 1967, p. 340.

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

Study adsorption and contact electrification in a vacuum and determine their effect on the separability of nonconducting minerals.

### Progress During the Second Quarter

The objectives for the second quarter were to construct a particle recirculating system, and to measure particle flow characteristics when the feed particles are periodically recirculated in an ultrahigh vacuum.

Investigation of the flow characteristics of particles was continued from the first quarter, although a pressure lower than  $10^{-7}$  torr could not be reached.

A systematic investigation of factors which prevent attainment of an ultrahigh vacuum included cleaning of the O-ring grooves of the stainless steel bell jar. The inner groove was found to be poorly machined resulting in the tearing of the O-ring on removal. It was also found that the silver soldering of some items resulted in the penetration of the soldering flux to the vacuum side of the equipment, particularly with the vibration feed-through.

As a result of some defects, the particle recirculating system was redesigned. Repairs were also made on the bearing refrigeration unit permitting bake-out of pump and bell jar.

### 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  
College Park, Maryland  
Date begun: June 1966 To be completed: May 1967  
Personnel: Ray A. Heindl, Supervisory Chemical Research Engineer  
Joseph A. Sutton, Research Chemist  
John D. Corrick, Research Chemist  
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 Second 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

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 Second Quarter

Objectives for the quarter were to continue investigations on the effect of increased cell temperature on the conductivity of silicate-plus-LiF mixtures and to study various physical characteristics of synthesized silicate minerals.

Significantly better electrical conductivity was achieved in various silicate-plus-LiF melts at temperatures of 1,400° to 1,550°C than was attained in previous experiments conducted at 1,200° to 1,300°C. Granodiorite, pumice, serpentinite, and basalt, each containing 10 weight-percent LiF, were 1.5 to 2 times more conductive at 1,400°C than at 1,300°C, while at 1,550°C the same melts were about three times more conductive than at 1,300°C.

Silicate minerals, including wollastonite, anorthite, and diopside were synthesized by fusion of their constituent oxides at temperatures in excess of 1,550°C. Relative electrical conductivities of the minerals were determined, and the effect on the conductivity of a 10 weight-percent LiF addition was studied. With no flux added, anorthite was too viscous at 1,550°C to determine an amperage-voltage ratio; however, under similar conditions, diopside had a 2:1 amp-to-volt ratio, and wollastonite had a 1:1 ratio. Adding LiF to each mineral markedly increased the electrical conductivity, and at 1,500°C both diopside and wollastonite showed an amp-to-volt ratio of about 4:1, while the amp-to-volt ratio in the anorthite-plus-LiF melt was about 2.5:1.

### 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  
Albany, Oregon  
Date begun: April 1966 To be completed: March 1968  
Personnel: Hal J. Kelly, Supervisory Ceramic Research Engineer  
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 Second Quarter

The objectives for this quarter were to obtain samples of the minerals selected for study to prepare purified samples and to continue preliminary DTA and TGA.

Mineral samples of the zeolite, epidote and amphibole groups listed in the first quarterly report have been obtained and concentrates of the pure minerals are being prepared. Samples of the bauxite group, brucite and goethite were not purchased because preliminary tests with minerals on hand showed that they blew out of the crucible during vacuum DTA. Concentrates of analcite, tremolite, and zoisite have been prepared and a differential thermal analysis has been made on analcite. There appears to be a low-temperature endothermic peak at 380°C, but the base line is poor in this temperature range and reactions are hard to detect. There is an exothermic peak at 840°C. Efforts will be made to adjust the controller so that a more uniform heating rate can be obtained at lower temperatures and DTA will be made on more of the new mineral samples.

Epidote was studied most intensively during the second quarter. Weight loss (TGA) in air and vacuum DTA were made. From room temperature to 700°C the weight loss was 0.74 percent. No more weight was lost until 850°C. From 850°C to 1,050°C the sample lost an additional 1.75 percent. The total weight loss was 2.49 percent.

The formula for epidote is  $\text{Ca}_2(\text{Al,Fe})_3(\text{SiO}_4)_3\text{.OH}$ . The ratio of Al to Fe is about 3 to 1. Therefore, the average weight of these ions is about 34 and the molecular weight approximately 475. The loss of one hydroxyl radical would result in a weight loss of 3.79 percent. It appears that 1,050°C is not a high enough temperature to remove all of the hydroxyl radicals when epidote is heated in air. The weight loss up to 700°C is not understood. X-ray analysis of unheated samples and samples heated to 750°C showed only minor differences in X-ray patterns.

DTA made on three samples of epidote showed an endothermic peak at 810°C, a second endothermic peak at 915°C, and the start of a third at about 990°C.

The time in flight mass spectrometer was used to analyze the effluent gases during vacuum decomposition and  $\text{CO}_2$  was detected. This indicated that a carbonate, possible  $\text{CaCO}_3$ , could be present. However, X-ray diffraction and optical examination failed to show the presence of an impurity. The sample was leached in acetic acid, washed, and dried. A DTA run on the leached mineral showed one large endothermic peak at 1,000°C. Future work will be done on the mineral without the carbonate impurity.

The sample of actinolite which was used also has a small amount of carbonate in it. Therefore, it also was leached in acetic acid, washed, and dried. TGA and DTA will be run on the leached material and compared with the results of previous analyses.

One of the major problems with vacuum DTA is that of holding the sample in the crucible during the reaction in which gas or water vapor is given off. To see if a cover on the crucible would help in holding the sample in, disks 1/8-inch thick and slightly smaller in diameter than the inside diameter of the crucible were made. Three .040-inch diameter holes were drilled in the caps to allow the gases to escape. Trial runs were made on calcite and kaolinite. Most of the calcite blew out of the crucible and a satisfactory thermograph was not obtained. Kaolinite, which is less active than calcite, gave a satisfactory thermograph, but a small amount of the sample blew out. There appears to be no advantage in the use of covered crucibles.

During the quarter the X-Y recorder was reconditioned, new thermocouple insulators were installed in the DTA furnace, and a new longer tantalum heat shield was installed.

#### Status of Manuscripts

None in progress.