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DEPARTMENT OF THE INTERIOR

BUREAU OF MINES

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MULTIDISCIPLINARY RESEARCH LEADING TO UTILIZATION OF EXTRATERRESTRIAL RESOURCES

Quarterly Status Report
October 1, 1968 to January 1, 1969

TWIN CITIES MINING RESEARCH CENTER

Walter E. Lewis, Research Director

NASA Contract R-09-040-001

MULTIDISCIPLINARY RESEARCH LEADING TO UTILIZATION OF EXTRATERRESTRIAL RESOURCES

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

QUARTERLY STATUS REPORT October 1, 1968 to January 1, 1969

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STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

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

January 1, 1969

Task title:	Background analysis and coordination
Investigator:	Thomas C. Atchison, Program Manager
Location:	Twin Cities Mining Research Center Minneapolis, Minnesota
Date begun:	April 1965 To be completed: Continuing
Personnel:	Thomas C. Atchison, Supervisory Research Physicist Other Bureau personnel, as assigned

PROGRESS REPORT

Objective

The objective of the program is to help provide basic scientific and engineering knowledge needed to use extraterrestrial mineral resources in support of future space missions. Under this task, background and supporting studies and coordinating and liaison activities for the program are carried out.

Progress During the Second Quarter

Evaluation of current information on the lunar surface continued. Detailed results of the Surveyor VII landing in the lunar highlands are not yet available. However, preliminary results indicate no great differences in surface characteristics from those found in the maria landings. Good progress in the Apollo program is causing added effort in the many scientific studies related to the returned lunar samples expected from the first manned landing. We have provided samples of our simulated lunar rocks to several investigators concerned with these studies.

D. E. Fogelson and B. L. Vickers completed a trip through Oregon and northern California to replenish our supply of the 14 simulated lunar rocks being used in the Bureau's program. The supply now on hand should be adequate for the next 18 months.

In connection with the Surveyor simulation studies on our 14 simulated lunar rocks in progress at the Jet Propulsion Laboratory, several property studies have been carried out here on fine particle samples of the rocks. Dr. Rolland L. Blake, of the Metallurgy Research Center Petrographic laboratory, has made detailed studies of the magnetic minerals in the rocks; Joseph L. Condon, of our Explosive Fragmentation laboratory, has measured magnetic susceptibility; Russell E. Griffin, of our Thermal Fragmentation laboratory, has measured dielectric constant; and Ernest Bukofzer, of our Chemically Assisted Fragmentation laboratory, has measured specific surface. Some of the results are shown in table 1.

Status of Manuscripts

Proposal for Continuing Bureau Extraterrestrial Resource Utilization Program, by T. C. Atchison, was submitted to NASA in July.

TABLE 1. - Properties of powdered simulated lunar materials

Rock type	Solid rock bulk density (g/cc)	Powder bulk density (g/cc)	Intrinsic density (g/cc)	Magnetic susceptibility (10^{-6} cgs units)	Dielectric constant (ratio)	Specific surface area (m^2/g)
Dunite	3.19	1.69	3.17	60	-	4.8
Gabbro	3.11	1.70	3.22	3500	3.31	.6
Tholeiitic basalt	2.84	1.45	2.95	1400	2.87	.8
Granodiorite	2.58	1.45	2.69	30	-	.6
Serpentinite	2.56	1.43	2.73	3500	-	3.8
Obsidian	2.39	1.16	2.40	50	-	.5
Altered rhyolite	2.36	1.19	2.63	30	-	3.3
Rhyolite	2.35	1.04	2.60	220	-	1.4
Vesicular basalt #1	2.25	1.37	2.86	340	2.85	.8
Vesicular basalt #2	2.22	1.52	3.02	350	-	1.3
Dacite	1.98	1.31	2.47	460	-	.5
Vesicular basalt #3	1.52	1.31	2.84	260	-	.7
Semiwelded tuff	1.15	1.02	2.46	270	-	1.2
Pumice	.76	1.20	2.40	10	-	1.4

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title:	Chemical reactivity of freshly formed surfaces
Investigator:	Clifford W. Schultz, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis, Minnesota
Date begun:	April 1966 To be completed: June 1969
Personnel:	Clifford W. Schultz, Metallurgist William H. Engelmann, Research Chemist

PROGRESS REPORT

Objective

The objective of this task is to define the equilibrium state of mineral surfaces with respect to adsorbed gases. Further, it is intended that existing adsorption isotherms be clarified in the low pressure end of the scale.

Progress During the Second Quarter

This project has been recessed during the first and second quarters. Work planned will be completed in the third and fourth quarters.

Status of Manuscripts

None scheduled until the fourth quarter.

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title:	Surface properties of rock in lunar environment
Investigator:	Wallace W. Roepke, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis, Minnesota
Date begun:	April 1966 To be completed: July 1969
Personnel:	William H. Engelmann, Research Chemist Wallace W. Roepke, Principal Vacuum Specialist Kenneth G. Pung, Physical Science Technician

PROGRESS REPORT

Objective

The goal of this task is to measure the surface properties of rocks and rock-forming minerals in a simulated lunar environment. Because friction is an important feature of many mining operations, the immediate objective is to measure the coefficients of friction of several mineral and mineral-metal (tungsten carbide) pairs in ultrahigh vacuum.

Progress During the Second Quarter

Initial runs have been made with the friction apparatus in the ultrahigh vacuum system this quarter. Initial erratic performance was traced to slack in the loading mechanism which allowed a change in load as the sample was moved past the probe. After corrective measures had reduced the slack to a tolerable level, it was found that the drive motor was operating too far down on its power-torque curve, allowing the motor itself to run in a start and stop manner. The driving motor for rotating the samples was connected to the atmospheric side of the ultrahigh vacuum rotary feed-through by a flexible shaft. The drive mechanism was redesigned using two large and two small pulleys in a configuration such that the motor operates further up the slope of the torque curve while allowing the desired output speeds for the samples of 0.48 to 4.8 degrees per minute radial velocity. The apparatus is now performing satisfactorily.

Two glove boxes for environmental control have been received from Ft. Detrick, Md. surplus. One was transferred to the Rock Physics laboratory. The other has been stripped down, thoroughly cleaned, and is in the process of being modified with valving, a flow meter, and pressure and temperature gauging to provide both a controlled storage and work area.

For using our CO_2 laser with the ultrahigh vacuum chamber, germanium disks, antireflection coated on both sides, have been ordered as infrared laser windows, together with special Pyrex window blanks for mounting of the germanium windows.

One acquisition needed for employing laser surface cleaning techniques in the friction studies is a suitable radiometer. The type needed will use a copper or mercury doped germanium detector having a broad-band reception between 2 and 18 micrometers with a peak in the 10 to 11 micrometer region. Response must be 2 millisecond or better with a sensitivity of $\pm 0.5°C$. This means the device must use a chopped reference for sensitivity with a modulated input to get rid of background noise and Cassegrainian optics for focusing. Infrared work at 10.6 micrometers has gained interest only in the last 2 or 3 years with the advent of the CO_2 lasers. The newness of this type of equipment, plus the response and sensitivity that we require, results in a high cost, beyond our current budget. Because the radiometer is critical to both the friction studies and the thermal property studies, we hope to lease the required equipment.

Preparation of the new laser laboratory adjacent to the ultrahigh vacuum laboratory is nearly complete. This new area will allow a more integrated use of the laser and the vacuum system for both surface and thermal property studies. This area will be ready for use early in the next quarter.

A considerable amount of time was spent providing consulting services for others this quarter. Discussions have been held with personnel at the Spokane Mining Research Laboratory concerning the problem of adapting their experimental procedures to ultrahigh vacuum tests. To preclude severe design and instrumentation difficulties, it was agreed that a simplified test design would be worked out.

Discussions have also been held with personnel at the Pittsburgh Explosives Research Center concerning the applicability of two large steel chambers to explosives research in high vacuum. A visual inspection of these facilities and a discussion of the desired techniques indicated that either of these chambers will be adequate for the high vacuum they require in their research.

The cooperative effort with the Mechanical Fragmentation laboratory on the ultrahigh vacuum drilling apparatus is continuing, as is the cooperative effort with the Fabric Analysis laboratory on the scanning electron microscope facility. Other cooperative effort has included work for the Metallurgy Research Center here. Some vacuum evaporated coatings were prepared for a new microscopy technique being perfected by Dr. R. L. Blake. The Quad 250 mass spectrometer head being used on the vacuum furnace for the carbon reduction studies was contaminated to the point where it needed a complete overhaul. The extremely delicate unit needed to be checked for electronic as well as physical alignment. The unit was completely disassembled, acid etched, assembled, and realigned with a new filament being installed.

Status of Manuscripts

Inexpensive Oil Vapor Trap for Use with Rotary Vacuum Pumps, a journal article by W. W. Roepke and K. G. Pung, was published in the International Journal of Vacuum in August.

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

Anion Suppressor Grid for Mass Spectrometer, an invention report by W. W. Roepke and K. G. Pung, was submitted to the Washington office.

Vacuum Technology Course, an internal report by W. W. Roepke, has been temporarily placed in suspension.

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

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 Richard E. Thill, Geophysicist Peter G. Chamberlain, Geophysicist Rollie C. Rosenquist, Engineering Technician

PROGRESS REPORT

Objective

The objective of this project is to study the effect of ultrahigh vacuum on rock deformation and failure processes at the macrostructural and the microstructural level and to measure rock strength and elastic properties in ultrahigh vacuum environment.

Progress During the Second Quarter

The alphatron vacuum gage was installed in the ultrahigh vacuum system and several pumpdowns were made with the gage performing satisfactorily. All associated equipment to be used with the vacuum system for the rock property tests was serviced and calibrated. A spring and guide rod assembly was designed to eliminate atmospheric pressure loading of the specimen through the bellows prior to actual testing.

A newly acquired glove box was modified for bake-out and storage of rock specimens before they are tested in the vacuum chamber.

The rock property test program in vacuum originally scheduled to begin during this quarter will be underway early in the third quarter.

Specifications were prepared for purchase of a scanning electron microscope to extend Center capability in surface microtopography and fracture analysis. Invitations for bids have been sent to prospective suppliers. Contract award is scheduled for early next quarter and delivery and installation is expected by the end of the quarter.

Status of Manuscripts

Environmental Effects on Rock Properties, by E. R. Podnieks, P. G. Chamberlain, and R. E. Thill, has been submitted for publication in the Proceedings of the Tenth Symposium on Rock Mechanics.

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title:	Feasibility of thermal fragmentation studies in vacuum
Investigator:	Kuppusamy Thirumalai, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis, Minnesota
Date begun:	October 1966 — To be completed: June 1969
Personnel:	Joseph M. Pugliese, Geophysicist Kuppusamy Thirumalai, Mining Engineer Russell E. Griffin, Electronic Research Engineer Sam G. Demou, Physicist

PROGRESS REPORT

Objective

The objective of this task is to investigate the feasibility of extending thermal fragmentation studies to lunar vacuum environment.

Progress During the Second Quarter

Preliminary results of laser irradiation on obsidian showed differences in fragmentation effects at different levels of incident heat energy similar to those reported last quarter for other simulated lunar rocks. Due to the nearly homogeneous glassy matrix, obsidian was selected as a desirable addition for tests on the effect of vacuum in the thermal studies.

Eight 1/2-inch diameter obsidian cores, 5 inches long, were drilled with a known orientation out of the same block and experiments were conducted to establish the influence of 10^{-5} torr vacuum on the limits of thermal softening. Similar experimental conditions were maintained for nitrogen atmosphere and vacuum conditions. The test results show that the progress of thermal softening is essentially the same in nitrogen atmosphere and in vacuum, with a softening point of 870°C. Among the rocks tested thus far, obsidian showed the least variation between vacuum and atmospheric conditions. These results confirm the previous findings that a vacuum atmosphere should not have an adverse effect on the process of thermal fragmentation.

An estimate of the effect of vacuum on linear thermal expansion appears to be necessary for a complete analysis of thermal stress behavior in vacuum environments. A noncontact system of expansion measurements was designed using angular determinations of the sample dimension from outside the vacuum chamber. Efforts are being made to obtain a precision electrotheodolite on loan from the Defense Logistics Supply Center for exploratory tests with angular measurements.

Attempts to carry out differential thermal analysis up to 1,100°C in granodiorite, obsidian, flow basalt, and dacite to ascertain the reaction at different levels of temperature up to the softening points were unsuccessful because of erratic behavior of the equipment. These experiments will continue as soon as the equipment is operating properly.

Further studies in thermal fragmentation of lunar rocks by laser irradiation were delayed until the installation of the laser and optical equipment in the new laser laboratory is completed.

Status of Manuscripts

None scheduled until the fourth quarter.

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title:	Thermophysical properties of rock over lunar temperature range
Investigator:	David P. Lindroth, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis, Minnesota
Date begun:	October 1966 To be completed: June 1969
Personnel:	Joseph M. Pugliese, Geophysicist Russell E. Griffin, Electronic Research Engineer David P. Lindroth, Physicist Carl F. Wingquist, Physicist Walter G. Krawza, Engineering Technician

PROGRESS REPORT

Objective

The objective of this work is to extend current studies of the effect of temperature on thermophysical properties of rocks at atmospheric pressure to cover the lunar temperature range.

Progress During the Second Quarter

The disks of Al_2O_3 were prepared for testing by means of the flash method to determine the thermal properties of diffusivity, conductivity, and heat capacity. Initially, microminiature grid thermocouples were emplaced on one face of the specimen. These thermocouples proved to be inadequate with respect to response time, and the diffusivity values obtained showed an unacceptably large scattering. As a result the thermocouples will not be used.

Through the courtesy of Barnes Engineering Co., a low cost infrared radiometer was tried. The response time of the instrument was 50 milliseconds with an accuracy of ±2.5°C. Experiments with this radiometer showed that we will need a greater speed of response, preferably down to 1 or 2 milliseconds. Also an improved accuracy and a greater temperature range will be required. An important finding from these experiments is that we are experiencing much higher temperatures at the sample surface, for a given amount of input energy, than we had previously suspected. The effort on the flash method will be reduced until we can acquire the necessary infrared radiometer with adequate response time and temperature range.

At the request of Jane de Wys of the Jet Propulsion Laboratory, Pasadena, Calif., dielectric constant measurements at a frequency of 30 megahertz were made on fine particle samples of gabbro, flow basalt, and vesicular basalt #1. These values are of interest in connection with simulated footpad magnet experiments comparing results with Surveyor V, VI, and VII lunar landing results. Results of the measurements are shown in table 1, page 3.

Electrical and mechanical installation of the emissivity measuring apparatus obtained from NASA surplus was completed and some essential vacuum system instrumentation was ordered. Trials of various components of the apparatus have revealed problems that need correcting to bring the equipment up to full operating status. Some preliminary testing of simulated lunar material should be possible before the end of the third quarter.

Status of Manuscripts

Dielectric Constants and Dissipation Factors for Fourteen Rock Types Between 20 and 100 Megahertz, by R. E. Griffin, is under preparation as a journal article.

Thermal Expansion Measurements of Simulated Lunar Rocks, by R. E. Griffin and D. P. Lindroth, is under preparation as a journal article.

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title:	Basic problems of drilling in lunar environment
Investigator:	Robert L. Schmidt, Project Leader
Location:	Twin Cities Mining Research Center Minneapolis, Minnesota
Date begun:	January 1967 To be completed: December 1969
Personnel:	William E. Bruce, Supervisory Mining Engineer Robert L. Schmidt, Mining Engineer Carl F. Anderson, Electronic Engineer Robert R. Fumanti, Engineering Technician

PROGRESS REPORT

Objective

The objective is to study the basic problems of particle adhesion, heat removal, and bit lubrication associated with drilling in a lunar environment.

Progress During the Second Quarter

The feasibility study of an ultrahigh vacuum drilling test to be performed in one of the Center's vacuum chambers was completed and the decision was made to proceed with the construction of a test apparatus. The purpose of the experiment will be to determine if particle adhesion in a vacuum exists to such an extent that mechanical cuttings removal systems now contemplated for lunar drilling would be ineffective. The drilling experiment will be first performed in atmosphere and then in ultrahigh vacuum. High speed movies and torque measurements will be taken in both environments.

During the second quarter a layout drawing of the drill apparatus was completed and purchase orders were placed for the required components.

Status of Manuscripts

None scheduled until the fourth quarter.

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

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 John J. Mahoney, Laboratory Electrician Elva M. Guastini, Explosives Equipment Operator

PROGRESS REPORT

Objective

The task objective is the acquisition of basic knowledge leading to the solution of potential problems involved in the use of chemical high explosives in the lunar environment, specifically: (1) the stability of explosives and explosive devices in the lunar environment; (2) the problem of exposure of explosives and explosive devices to (micro)meteoroid impact; and (3) the propagation characteristics of the blast wave (products cloud) in the lunar atmosphere.

Progress During the Second Quarter

Experimental studies of condensed phase detonations at reduced pressures were continued. The preliminary work with 1.0-inch diameter spheres of Composition A-5 was temporarily abandoned because of the difficulties encountered in synchronizing the Kerr Cell instrumentation with the initiation of the spherical charges. These difficulties arose from stray electromagnetic radiation from the exploding-wire back-lighting source and the high voltage used to initiate the charge. To circumvent some of these difficulties, additional experiments were conducted using the high-speed framing camera capable of 1.25 million frames a second with a minimum exposure time of 0.14 μsec. A special electric blasting cap is used for the initiation and synchronization of the explosives event to the camera. The blasting cap or detonator is too large to be used with the 1.0-inch diameter spherical charges and as a consequence 1.0-inch diameter x 2.0-inch long cylinders of Composition A-5 were used in the framing camera studies. The charge has a 1/4-inch diameter by 1-3/4-inch long axial cavity to contain the detonator which was 3 inches long. The functioning of the detonator is such that initiation takes place at the midpoint of the charge allowing 1.0 inch for the buildup of steady-state detonation. This is probably adequate for the explosive composition used. The charges were positioned near the center of either 12- or 22-liter spherical flasks which were evacuated with a 260 liter/sec turbo-molecular pump.

To date 14 firings have been completed over the pressure range from 1.0 torr to 1.8×10^{-7} torr. Photographic observations were made of the charge profile and in some cases a 45-degree mirror was employed to simultaneously view the free end of the charge and the impact of the detonation products on the walls of the flasks in the direction of detonation. In most cases the experiment was back-lighted with an exploding wire located behind a paper diffusion screen. Black and white as well as color film was employed; all firings were made at a camera mirror speed of 4,000 rps corresponding to a framing rate of 10^6 pictures/sec with an interframe time of 1.0 μsec and a minimum exposure time of 0.16 μsec.

The entire series of photographs have not been analyzed in any great detail but the following tentative conclusions can be made. The velocity of the fastest detectable ejecta is approximately 22 km/sec and is constant over the pressure range from 10^{-3} to 10^{-7} torr. This material is invisible against the background lighting and its presence is indicated by a bright impact glow on the wall of the flask off the end of the charge. An opaque products cloud, suggesting the presence of free carbon, is also observed. This cloud has a velocity of approximately 8 km/sec in the direction of detonation which is also sensibly constant over the pressure range of 10^{-3} to 10^{-7} torr. At pressures lower than 10^{-3} torr there is no visual evidence of a shock wave; however, at 1×10^{-3} torr a faint blue shock wave that envelops the opaque products cloud is observed. The shock wave has a velocity of approximately 22 km/sec. The opaque products cloud, in this case, still maintains a velocity of about 8 km/sec in the direction of detonation. At 1 torr the shock velocity has decayed to about 13.5 km/sec while the opaque products cloud apparently maintains a velocity of about 8 km/sec. Firings are currently being carried out at ambient pressure to establish the lower velocity limit. Another observation that has some pertinence to the program is that the self-luminosity of the detonating charge is very rapidly quenched at low pressures. The exact time for this to occur is not known but it is less than the 1 μsec interframe time used in the experiment.

A 38,000-liter tank, 7 feet in diameter and 37 feet long is being examined for suitability to the lunar problem. The vessel, designed for a working pressure of 125 psig, had been used as a sump for slurried coal in a project involving coal transportation; consequently, the interior was contaminated severely with respect to its application to high vacuum research. A thorough clean-up by brushing and sweeping the inner surface followed by a fairly long pumping time with a 17 cu ft/min pump produced a vacuum of the order of 200 microns. This value was acceptable for the intended tests since it fell in the range of the thermocouple vacuum gage.

The testing procedure was to coat all gasketed joints, nozzle attachments, and seam welds with a sealant and then, when a vacuum in the thermocouple gage range had been achieved, to methodically dissolve the resin with acetone. If a leak was present a rise in pressure would first be indicated on the nearest of several thermocouple gages placed along the length of the vessel. The predominant leaks were apparently in the gasketed closures that included eight openings 6 inches or more in diameter, the largest having a diameter of 24 inches. No leaks were found when the welds were flushed with acetone. In order to determine that the sensitivity of the detection system was adequate, finite amounts of acetone were injected into the vessel in the end opposite the pump; response of the gages was considered acceptable in that a rise in pressure of 20 microns was observed in 2 seconds near the injection point for 1 cc of acetone and, at the opposite end (37 feet), a rise of 20 microns occurred in 20 seconds when 2 cc of acetone were injected into the vessel. A pumpdown curve is being obtained by admitting dry air and monitoring the rate of decrease in pressure for comparison with the theoretical pumping performance of the system.

Status of Manuscripts

None in progress.

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

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: June 1969
Personnel:	David E. Nicholson, Mining Engineer Maynard O. Serbousek, Structural Engineer David F. Stafford, Physicist Robert W. Carnes, Engineering Technician Richard P. Curtin, Engineering Technician

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 particle 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 include 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

Electrical work on the crushing and milling circuit was completed during the quarter. A high-speed photocell tachometer was installed on the Vortec impact mill. This will improve our control of the simulated lunar rock powders produced by the mill.

A new stock of samples of the simulated lunar rocks was collected in October at the Oregon and California rock sites. About two drums of rock samples were obtained from each site.

Literature search and study on Duffy Mindlin theory of granular media, Mohr-Coulomb theory, and applications to bin flow problems were continued. Dr. Sidiq M. Dar, Professor of Mechanical Engineering at Gonzaga University, is assisting in the analysis of a loose packing model problem in one-dimensional compression from Duffy Mindlin theory.

Planned work on the measurement of handling properties in moderate vacuum was delayed. Tests and data analysis will be continued during the next quarter.

A preliminary design for a torsional type shear testing device to be used in the ultrahigh vacuum experiments at the Twin Cities Mining Research Center was completed. Final design and construction of the device will be worked out in cooperation with Twin Cities personnel during the next quarter.

Status of Manuscripts

Progress Report on the Lunar Materials Handling Project, an informal report by David E. Nicholson, is under preparation.

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

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

Personnel: Robert C. Bates, Mining Engineer
Lester J. Crow, Mining Engineer
Edward W. Parsons, Mining Engineer
Colen S. Smith, Mining Engineer

PROGRESS REPORT

Objective

The objective is to advance ground support technology in areas having both terrestrial and extraterrestrial application. Research will be conducted on ground support materials which have potential for utilization both on the Earth and the Moon.

Progress During the Second Quarter

Work on the use of thermoplastic mixtures of sulfur and fine basalt particles as a grout has been substantially completed. A series of laboratory experiments were carried out using a good gradation of fine (1/2-inch to 10 percent-200 mesh) basalt particles with molten sulfur. The molten sulfur contained the expansion additive developed in the first quarter. The net result was a mixture that expanded sufficiently to compensate for most of the normal shrinkage of the sulfur. The average unconfined compressive strength of the material was about 6,000 psi as compared to 9,400 psi with a good combination of fine basalt particles and sulfur.

Some experiments have been conducted using sodium silicate with additives as a bonding agent and by itself as a sealant on the surface of sulfur-concrete cylinders. The low curing temperatures used so far with sodium silicate (less than 27°C) have not yielded a good sealant, but it is too early in the experimental sequence to develop trends.

Analysis of other materials for use as supports is continuing. Part of this work is based on information summaries such as found in Evaporation Effects on Materials in Space by L. D. Jaffe and J. B. Rittenhouse, J.P.L. Technical Report No. 32-161. Sublimation, evaporation, or decomposition rates of many materials are too high for use in permanent supports that are exposed to high vacuum conditions. Results of this analysis will be summarized in the third and fourth quarters.

Work on formulation of design concepts and devising technqiues and equipment for fabrication or installation of support systems using sulfur and other materials is continuing.

Status of Manuscripts

Strengths of Sulfur-Basalt Concrete, by Lester J. Crow and Robert C. Bates, is being prepared as a Bureau of Mines Report of Investigations.

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title:	Electrowinning of oxygen from silicate rocks	
Investigator:	Donald G. Kesterke, Acting Project Coordinator	
Location:	Reno Metallurgy Research Center Reno, Nevada	
Date begun:	June 1966	To be completed: May 1969
Personnel:	Donald G. Kesterke, Metallurgist Freddy B. Holloway, Physical Science Technician	

PROGRESS REPORT

Objective

The objective is to determine the feasibility of electrowinning elemental oxygen from silicate rocks, as one phase of multidisciplinary efforts to develop basic knowledge for using lunar mineral resources in support of space missions.

Progress During the Second Quarter

The objectives this quarter were to continue electrowinning experiments to prepare oxygen from silicates, emphasizing a study of the effect of different silicate-fluoride compositions on cell performance, and to conclude investigations of various refractory materials for use as electrodes.

Research continued to electrowin oxygen from a mixture having a composition of 69.4 weight-percent BaF_2, 5.6 weight-percent LiF, and 25.0 weight-percent basalt plus sinter. Electrolysis was conducted at 1,150° to 1,200°C, with 23 to 65 amperes applied to the cell at an emf of 14 to 20 volts. Analysis of the gases in the vicinity of the cell showed 3.5 to 4 percent oxygen.

In subsequent experiments, the silicate content of the electrolyte was increased, and electrolysis was conducted in a bath composed of 60.1 weight-percent BaF_2, 4.9 weight-percent LiF, and 35.0 weight-percent basalt plus sinter. Cell temperature ranged from 1,150° to 1,225°C. Electrolysis current was 25 to 33 amperes, and the emf ranged from 14 to 17 volts. The oxygen content over the cell in the initial experiment was about 2.5 percent. However, in two succeeding electrolyses in which the bath was reused, the maximum oxygen content over the cell dropped to 1.7 percent, then to 0.3 percent. During these experiments, fuming from the cell at 1,200°C was quite heavy. In an attempt to alleviate this, the bath composition was changed to permit electrolyzing at lower temperatures. The electrolyte composition in weight-percent was 48.5 BaF_2, 16.5 LiF, and

35.0 basalt plus sinter. Electrolysis was conducted at 1,050° to 1,100°C with 35 to 55 amperes of current applied to the cell at 17 to 19 volts. Fuming was significantly reduced but still remained at a noticeable level. Oxygen content above the cell increased to more than 3 percent during the initial experiment. However, as with the preceding experiments, cell performance deteriorated on reuse of the electrolyte, and in three succeeding electrolyses, the maximum oxygen concentration over the cell was 2.0, 1.8, and 1.3 percent, respectively.

Efforts continued to find suitable refractory materials for use as electrodes. Zirconium diboride and silicon carbide were evaluated as anode materials in experiments conducted at 1,100° to 1,200°C. The ZrB_2 corroded off at the bath level after about 30 minutes of electrolysis, while the SiC lasted for 1 hour. With both materials, oxygen production was negligible. In a subsequent experiment, ZrB_2 was used as the cathode material, but again, severe corrosion occurred after a short electrolysis. From these and previous results, the most suitable electrode combination used to date consists of an iridium anode and a silicon carbide cathode.

Status of Manuscripts

None in progress.

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

Task title:	Reduction of silicates with carbon
Investigator:	Larry A. Haas, Project Leader
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

The objective is to determine the optimum reaction rate criteria for extracting oxygen from possible lunar materials in a simulated lunar environment. 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 objectives for this quarter were to determine the degree of carbothermal reduction of fayalite and its mixtures with silica and to investigate the effect of small amounts of metal oxides on the kinetics for silica reduction.

The carbothermal reduction of the silica-iron oxide system has been studied at various temperatures and silica contents. Iron silicate (fayalite) was made by blending minus 200-mesh powders of wustite and silica at a molar ratio of 2 and fusing this mixture in an inert atmosphere at 1,190°C for 1 hour. The fused sample was then ground to minus 60- plus 70-mesh. Microscopic and X-ray diffraction analysis indicated the principal mineral was fayalite. Twelve grams of fayalite were mixed with 2.4 grams of graphite and heated in a vacuum for 5 hours. The effect of temperature on the degree of reduction is shown in table 1. Only a small amount of reduction occurred at 1,100°C as is evident from the 0.9 percent weight loss and the small amount of carbon monoxide and metallic iron formed. However, at 1,130°C, the weight loss was 22.4 percent and 64 millimoles of carbon monoxide was formed. Because SiO_2 does not reduce appreciably at this temperature, the oxygen must have been extracted from the iron oxide.

The reduction of fayalite was also studied in the presence of various amounts of silica as it is usually associated in nature with large amounts of silica. The data in table 1 show that the amount of reaction as measured by the weight loss increases with decreasing amounts

TABLE 1. - Reduction data of silicon and iron oxides in vacuum

Mineral or oxide	Temp, °C	Oxide charge analysis, percent			Five-hour weight loss, percent	Carbon monoxide formed, millimoles	Residue analysis		
		SiO_2	Fe^{+2}	Fe^{+3}			Fe°, millimoles	$\frac{Fe^{+2}}{Fe^{+3}}$, pct	$\frac{Fe^{\circ}}{\Sigma Fe}$, pct
Fayalite	1100	21.6	52.0	5.6	0.9	0	0	-	0.4
Do.	1130	21.6	52.0	5.6	22.4	64	7.7	-	3.6
Do.	1200	21.6	52.0	5.6	≈50(1)	(2)	(2)	-	(2)
Fayalite and silica	1300	79.2	15.2	.5	11.3	45	30	-	88
	1300	61.2	25.1	4.8	22.2	37	29	-	78
	1300	22.1	52.0	5.6	(1)	(2)	(2)	-	(2)
Wustite	1100	-	66.1	10.3	54.0	163	77	-	75
Magnetite	1100	-	23.6	48.0	30.5	152	77	-	50
Hematite	1100	-	.05	70	33.2	161	107	-	71
Hematite[3]	800	-	.05	70	.03	-	-	0.001	-
Do.	1000	-	.05	70	.045	-	-	.07	-
Do.	1100	-	.05	70	2.1	-	-	.53	-

[1]Reaction too fast and sample blow-out occurred.
[2]Not available.
[3]No graphite added.

of SiO_2 or decreasing melting point of the mineral. Iron oxides reduce much easier than silicon dioxide even if they are present as double oxides. About 75 percent of the iron can be extracted from a fayalite-silica-graphite mixture at 1,300°C in a vacuum. Iron oxides without silica were reduced much more readily than iron silicates.

Hematite was also heated in vacuum in the absence of graphite. The last three rows in table 1 show the effect of temperature on the hematite conversion to magnetite under vacuum. It is evident that by vacuum thermal dissociation alone, hematite can be completely converted to magnetite at 1,100°C in 5 hours.

The study of the effect of small amounts of alkali and alkaline earth metal oxides on the carbothermal reduction of silica was studied during this quarter. Silica, minus 140- plus 240-mesh, was mixed with about 5 percent of various oxide additives. The mixture was moistened with distilled H_2O and then heated slowly in a programmed muffle furnace for 20 hours at the rate of 100°C per hour up to 1,400°C. This was done to minimize the sublimation of the alkali oxides and to incorporate the additive in the silica matrix. Even then some of the volatile oxides did sublime, as indicated by the data in table 2, where a considerable sodium and potassium loss from the intended 5 percent level was recorded in the charge analysis. The manganese additive was only 4 percent of the original mixture before preparation. The solid mass from the muffle furnace was ground to minus 70- plus 100-mesh and 12 grams of this size fraction were blended with 2.4 grams of similarly sized graphite. The mixture was then heated rapidly to 1,400°C in the recently acquired vacuum furnace and its weight was followed with time using a quartz spring balance. In view of the suspected sublimation of the alkali oxide constituent, even after the careful digestion in the muffle furnace, blank tests were run with the graphite reactant missing, and indeed, significant weight losses and relatively smaller blanks were obtained with calcium and magnesium oxide under these high vacuum conditions. In each case the blank test was subtracted, point by point from the carbothermal kinetic curve obtained in presence of graphite, and the curves in figure 1 were obtained. It is immediately discernible that calcium and magnesium, when present at concentration levels around 5 percent, promote the reduction rate of silica. On the other hand, small amounts of sodium and potassium retard the reduction rate of silica. It is interesting to note that 5 percent lime more than tripled the carbothermal reduction rate of silica, whereas 3 percent magnesia almost doubled this rate. Whether the ionic size of the additive exerts an effect on the reaction acceleration or not, will be tested by planned experiments using strontium and barium oxides as additives.

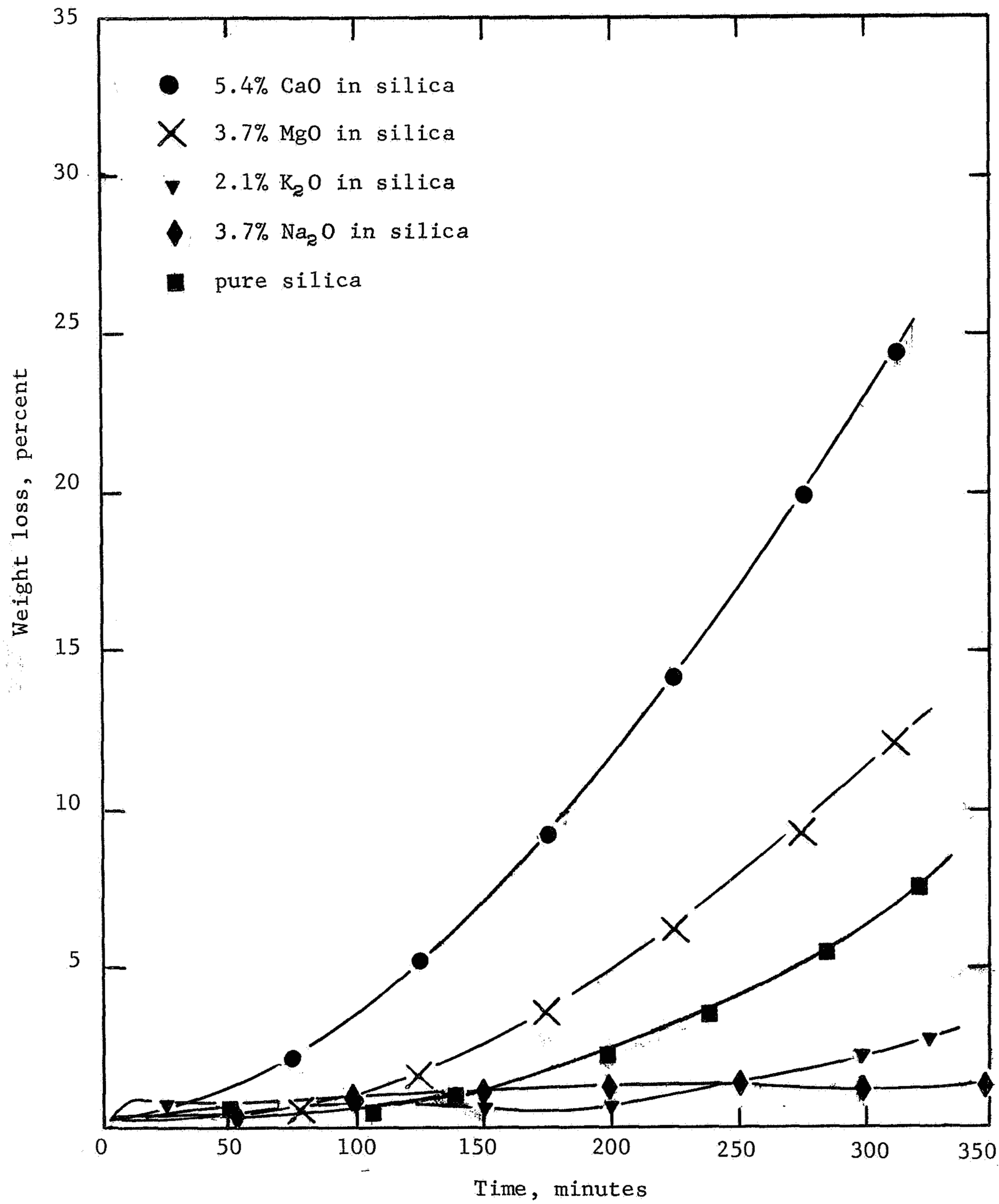

FIGURE 1. - Effect of Alkali and Alkaline Earth Oxides on the Carbothermal Reduction of Silica in Vacuum and at 1,400°C.

TABLE 2. - Experimental carbothermal reduction data of silica with various additives

Type of charge	Charge analysis, percent			Constituent loss, percent		
	Additive	Silica	Graphite	Additive	Silica	Graphite
Graphite	-	-	100	-	-	0.20
Silica	-	100	-	-	0.20	-
$CaO + SiO_2$	5.43	94.68	-	0.00	1.34	-
$CaO + SiO_2 + C$	4.98	78.16	16.67	.00	19.47	44.67
$MgO + SiO_2$	3.65	95.08	-	.00	.31	-
$MgO + SiO_2 + C$	3.04	79.23	16.67	.00	9.94	25.49
$K_2O + SiO_2$	2.05	95.51	-	95.73	.96	-
$K_2O + SiO_2 + C$	1.71	79.59	16.67	90.58	1.21	20.07
$Na_2O + SiO_2$	3.65	93.20	-	75.52	.79	-
$Na_2O + SiO_2 + C$	3.04	77.66	16.67	32.45	.41	7.16

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, was published as Bureau of Mines Report of Investigations 7207.

Carbothermal Reduction of Solid and Liquid Siliceous Minerals in Vacuum, by S. E. Khalafalla and L. A. Haas, is being prepared for presentation at the Annual Meeting of AIME in Washington, D.C. in February.

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

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: May 1969
Personnel:	Hal J. Kelly, Supervisory Ceramic Research Engineer Ray L. Carpenter, Research Physicist

PROGRESS REPORT

Objective

The objective of this project is to investigate by differential thermal analysis (DTA) and thermogravimetric analysis (TGA) the stability of some silicate and oxide minerals in air and under high vacuum at elevated temperatures. The determination of the energies required to dissociate oxide and silicate minerals and recover oxygen and water is the long-range objective.

Progress During the Second Quarter

Further improvements were made in the operation of the DTA equipment. New thermocouples and insulators were installed and the alumina used as the neutral material has been replaced with a type that produces a more stable base line. With these improvements the platinum calorimeter performs satisfactorily in air or vacuum.

A calibration run with calcium carbonate showed that the equipment gives a response of 38.6 calories per square inch when a dual channel recorder with a chart speed of 15 inches per hour is used. DTA runs made with a recorder range of 200 microvolts, instead of the 500-microvolt range usually used, showed that satisfactory thermographs could be recorded at this amplification. The base line noise was about 4 microvolts, representing a substantial improvement over past performance. This range can be used for reactions of small thermal energies.

DTA runs were made on epidote and zoisite in air and in vacuum. The heat of decomposition for these minerals was calculated for the runs in air and found to be 26.7 and 75.5 calories per gram, respectively. This result for epidote represents a correction for the higher value reported last quarter. The data from the runs in vacuum showed the peak temperature was higher in vacuum than in air for epidote but lower for zoisite. Repeat runs will be made on these minerals to see if this effect can be verified.

DTA runs were made on analcite, heulandite, stilbite, and apophyllite. All minerals produced thermographs with peaks in the low temperature range, below 500°C. The thermographs agreed with those found in the literature.

Status of Manuscripts

None scheduled until the third quarter.

STATUS REPORT SECOND QUARTER FISCAL YEAR 1969

Bureau of Mines NASA Program of Multidisciplinary Research

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: Ronald A. Munson, Research Chemist
Foster Fraas, Metallurgist
Anderson Walls, Laboratory Technician

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 Second Quarter

The previously reported tests on microcline, olivine, and hornblende in a size range of minus 35- plus 65-mesh were repeated in the size range of minus 65- plus 150-mesh. Results are summarized in table 1, together with the results reported last quarter on the smaller size quartz. Some single particle layer adhesion on the vibrating feeder near the feed hopper was noted for microcline and quartz, and at the discharge end of the vibrating feeder for olivine. This adhesion corresponded to the behavior usually obtained in low humidity atmospheres.

Changes in contact electrification in the atmosphere can be attributed to the changes in relative humidity, a higher contact electrification being obtained as a result of the lower surface conductivity in the lower humidity atmosphere. The increase in electrification which occurs in an ultrahigh vacuum would be beneficial to the electrostatic method of mineral separation.

Contact electrification changes between the first pass in vacuum and the second pass with its intervening 100°C bake-out may be noted. For both the minus 35- plus 65-, and the minus 65- plus 150-mesh particle sizes, there is little change in the positive electrification of olivine. The remaining minerals, except for microcline in the finer size, have a decrease in negative electrification value. At the low temperature of 100°C and the short bake-out time used in the tests,

TABLE 1. - Contact electrification of minerals in vacuum and in air

Mineral	Initial atmospheric pass[1]	First pass under vacuum[2]			Second pass under vacuum[3]		Pass after sample returned to atmosphere[4]		
	Potential (volts)	Pressure (10^{-7} torr)	Potential (volts)	Average particle charge coulombs (10^{-14})	Pressure (10^{-8} torr)	Potential (volts)	Weight (grams)	Potential (volts)	Potential[5] (volts)
Microcline	-11.0	5.5	-29	1.3	7.6	-34	1.574	-8.5	-10.8
Olivine	+6.0	4.9	+6.3	0.21	7.8	+8.0	1.526	+10.0	+13.0
Hornblende	-15.5	4.2	-40	1.4	6.1	-26	1.659	-15.0	-18.0
Quartz	-14.0	4.0	-42	1.8	7.5	-36	1.867	-12.0	-12.9

[1] Initial weight of sample = 2.000 grams. Size range is minus 65- plus 150-mesh. For microcline, olivine, hornblende, and quartz, relative humidities were 49, 54, 42, and 56 percent, and temperatures 26, 26, 25, and 29 °C, respectively.

[2] First pass conducted with sample at room temperature and no intervening increase in temperature.

[3] Second pass conducted at room temperature but with an intervening 18-hour sample temperature of 102±2 °C.

[4] For microcline, olivine, hornblende, and quartz, relative humidities were 46, 45, 38, and 52 percent, and temperatures 26, 26, 27, and 30 °C, respectively.

[5] Calculated for 2 grams.

these changes could only result from the desorption of high vapor pressure substances including water. The higher vacuum is not considered an influential factor since it only increases the mean free path in the gas phase.

Status of Manuscripts

Mineral Separation in a Vacuum, by F. Fraas, is under preparation as a journal article.