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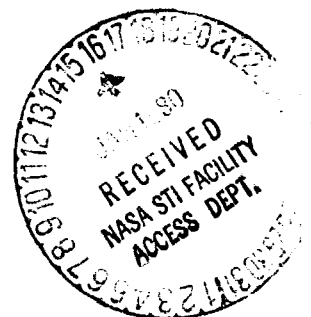
FINAL TECHNICAL REPORT

NASA GRANT NSG 9039

TITLE: X-RAY DIFFRACTION STUDIES OF SHOCKED LUNAR ANALOGS

SUBMITTED BY:

Dr. Robert E. Hanss  
Principal Investigator  
St. Mary's University  
San Antonio, Texas 78284



TO:

Dr. Friedrich Hörz  
NASA Technical Officer  
Lyndon B. Johnson Space Center  
Houston, Texas 77058

DATE:

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## Final Report: X-RAY DIFFRACTION STUDIES OF SHOCKED LUNAR ANALOGS

### Abstract

This research focuses on x-ray diffraction experiments on shocked rock and mineral analogs of particular significance to lunar geology. It utilizes materials naturally-shocked by meteorite impact (Ries, Charlevoix, and Brent craters), nuclear-shocked (Piledriver event), or artificially-shocked in the Johnson Space Center 20mm flat-plate accelerator. Four areas were outlined for investigation: powder diffractometer studies of shocked single-crystal silicate minerals (quartz, orthoclase, oligoclase, pyroxene), powder diffractometer studies of shocked polycrystalline monomineralic samples (dunite), Debye-Scherrer studies of single grains of shocked granodiorite, and powder diffractometer studies of shocked whole-rock samples. The principal goal of the research is the rapid, quantitative interpretation of peak shock pressures experienced by materials found in lunar or terrestrial impact structures.

Experiments on shocked single-crystal quartz revealed a correlation between shock pressure and a diffractometer parameter: the peak-height/half-width ratio. This parameter, approximately linear with shock pressure, was also determined for orthoclase, oligoclase, and pyroxene (enstatite). Shocked polycrystalline samples of dunite revealed a similar relationship. X-ray sample preparation techniques were developed to increase the reproducibility of results.

Debye-Scherrer studies of rotated single grains of shocked materials were based on the shock-pressure measurement technique developed by Hörz and Quaide. Measurement of shock pressures for several hundred quartz and feldspar grains of Piledriver samples indicates that these nuclear-shocked materials did not experience shock pressures greater than 20 GPa. These samples are compared with accelerator-shocked samples of the Climax (Nevada) granodiorite. The accelerator-shocked samples reveal a narrower pressure distribution range than nuclear or meteorite-shocked materials. Several hundred grains from specific Ries crater samples were investigated by the Debye-Scherrer method, and pressure distribution curves of these samples are presented. This data is compared with shock stages determined by petrographic studies.

Powder diffractometer studies of whole-rock samples of shocked materials utilized quartz and feldspar data to estimate shock pressure. Initially this technique yielded fair agreement with optical data on Ries samples, and it was proposed as a way to evaluate rapidly the in-situ stress level around a terrestrial impact structure--the Charlevoix crater in Quebec. Studies of 37 coded samples were begun. Shock pressure parameters were found to be dependent upon quantities of quartz and feldspar present, so an attempt was made to determine quartz-feldspar percentages by comparison with unshocked standardized mixtures of these two minerals. Compositional variation, plus identification problems for some of the coded samples, leaves the Charlevoix crater pressure estimates less precise than originally hoped for.

A proposed Tertiary impact crater near Uvalde, Texas was investigated for shock effects by both powder diffractometry and Debye-Scherrer techniques. Quartzitic samples revealed shock effects to be 0 or less than 5 GPa.

## Title: X-RAY DIFFRACTION STUDIES OF SHOCKED LUNAR ANALOGS

### INTRODUCTION

The ensuing paper comprises the Final Technical Report of NASA Grant NSG-9039. The time period of the original grant and two renewal grants extends from January 1976 to December 1979. During this total grant period intensive investigations of shocked lunar analogs through x-ray diffractometry have been conducted. The abstracts of project proposals and summary progress reports are contained herein in chronological order. Pertinent publications, presentations, or related field work are included with each project proposal.

### PERSONNEL AND EQUIPMENT

#### A. Personnel Summary

The principal investigator, Robert E. Hanss, was engaged in directing and implementing the overall project for 25% of his time during 27 months of the project, and 50% of his time during approximately eight months (summers). Research assistants--undergraduates who normally worked 15 hours/week--included: Charles Galindo, a student majoring in geology, who worked from January, 1976 until his graduation in December, 1978, and employment with Monarch Logging Company; Bruce R. Montague, a physics and computer science major, who worked from January, 1976 through his graduation in May, 1978, and continued as a voluntary consultant and equipment repair person during his current employment as a computer specialist at Kelly Air Force Base in San Antonio; Mark K. Davis, a physics major, who joined the research in 1977, and continued until its completion in December, 1979, while working as a laboratory instructor in physics at

St. Mary's University; Lionel Lopez, a geology-engineering major, who worked briefly in the spring of 1978, and Frank Rodriguez, a geology major, who worked from January, 1979, until his employment with Northrup Services at Johnson Space Center in August, 1979. All personnel participated in the formulation and writing of research results for publication; Montague and Davis authored individual research abstracts or presentations of their own. All research assistants and the principal investigator participated in the Lunar and Planetary Science Conferences each year in Houston, and occasional other conferences.

#### B. Equipment Summary

X-ray diffraction equipment available at St. Mary's University before the project began consisted principally of a G.E. XRD-5 diffractometer and Debye-Scherrer camera. During the project, this equipment was upgraded for shocked materials studies by the addition of a curved-crystal graphite monochromator, a sealed-gas (xenon) proportional counter tube, a fine-focus Cu-target x-ray diffraction tube (replaced twice during the course of research), radiation safety-shutters, a Polaroid Laue camera, and an additional Debye-Scherrer camera. Support facilities added consist principally of a dental x-ray developing tank, a binocular microscope for sample mounting, and a Mettler analytical balance for sample weighing.

The 20mm flat-plate accelerator at Johnson Space Center and target preparation facilities were utilized for preparation of all artificially-shocked samples. During May, 1976, x-ray diffractometer facilities at JSC were also extensively used for this project. An additional Debye-Scherrer camera was loaned from JSC in 1977 and returned six months later upon acquisition of our own camera.

## A RESEARCH PROGRAM FOR X-RAY DIFFRACTION STUDIES

### OF SHOCKED LUNAR ANALOGS

October, 1975

#### Abstract

We propose to perform x-ray diffraction experiments to determine the shock effects in rock and mineral analogs of particular significance to lunar geology. These materials consist of naturally-shocked samples from terrestrial impact structures and samples artificially-shocked at known peak pressures at the 20 mm flat plate accelerator located at Johnson Space Center. The pressure range (20 to 1300 kilobars) at this facility is sufficient to model the entire range of meteorite impact effects in any rock-like materials, from elastic brittle fracture through plastic deformation, solid state vitrification, selective partial melting, total rock melting, and possibly to vaporization. The shocked samples are recovered 100%. X-ray diffraction investigation of these samples reveals the degree of induced lattice disorder as a function of peak pressure. Calibration standards for peak pressure in single grains have been developed for a few minerals in particular pressure ranges. This quantification of peak pressures will be refined for quartz and feldspars, extended to other silicate minerals of lunar significance, and applied by powder diffractometer techniques. The determination of the distribution of peak shock pressures within phaneritic rock materials will be done by means of Debye-Scherrer rotated grain patterns.

The proposed experiments include a) powder diffractometer (line-broadening) studies of accelerator-shocked single-crystal pyroxene, b) powder diffractometer studies of accelerator-shocked monomineralic, polycrystalline rocks, e.g., anorthosite, c) Debye-Scherrer rotated-grain studies of naturally-shocked granitic minerals from the Ries Crater, Germany, and d) powder diffractometer studies of naturally-shocked whole-rock samples of suevite from the Ries Crater, Germany.

A continuation of the present project will extend these x-ray diffraction studies to a) shocked particulate, monomineralic powders (selected grain sizes and controlled porosities) and b) shocked particulate multiphase assemblages, e.g., plagioclase-pyroxene-olivine analogs of lunar soils.

The results of these experiments will assist in developing a set of diffractometer standards for rapid, quantitative interpretation of peak shock pressures experienced by silicate minerals found in lunar or terrestrial impact structures. They will also provide data on the distribution of peak shock pressures and shock wave energy transfer in crater materials.

## B. Progress Report Summary - June, 1976

### 1) Studies of shocked single-crystal quartz

X-ray diffractometer studies were made for single-crystal quartz samples shocked 0-31.0 GPa at the JSC flat-plate accelerator. An internal standard (5-micron aluminum oxide) was mixed with the shocked quartz. Sample mounting procedures were tested to develop maximum reproducible pattern intensities for the extremely small amounts (10 mg or less) of sample available. The diffraction patterns were compared for both Philips and G.E. diffractometers, and a method developed to correlate the results. Graphs of the shape of the  $26.6^\circ$  two-theta quartz diffraction maxima as a function of peak shock pressure are shown in the appendix (2).

### 2) Studies of shocked granodiorite

Samples of the Climax granodiorite from the Nevada Test Site were studied to compare the effects of nuclear shock (the 1962 Piledriver event) with the shock effects of the JSC flat-plate accelerator. Debye-Scherrer studies of 350 single grains of nuclear-shocked quartz and feldspar were made, and the peak shock pressures were estimated using the calibration standards developed by Hörz and Quaide (1). Ten samples of unshocked granodiorite were then subjected to shock pressures 5.1-48.2 GPa and the recovered samples prepared for Debye-Scherrer and diffractometer studies. Since the quantity of accelerator shocked material was much less than the available nuclear-shocked material, only about 150 single-grain studies of the JSC-shocked material could be made. These Debye-Scherrer patterns were also evaluated for peak shock pressure using the standards of Hörz and Quaide (1).

The finer-sized material from the accelerator-shocked samples was used for diffractometer studies, as was finely-ground nuclear-shocked material. Diffractometer scans of these whole-rock samples were evaluated



for peak shock pressures using the line-broadening characteristics observed in the single-crystal quartz studies. The varying quantities of quartz and feldspar present in such whole-rock samples makes the evaluation of peak shock pressures somewhat imprecise, but the general characteristics of these patterns permit an approximate determination.

The tentative conclusion of our data, both Debye-Scherrer and diffractometer, is that the nuclear-shocked samples did not experience shock pressures exceeding 20.0 GPa. Graphs of the distribution of peak shock pressure in nuclear-shocked and accelerator-shocked Piledriver samples are shown in the appendix (2), along with summaries of the diffractometer determinations on whole rock samples.

### 3) Studies of naturally-shocked Ries samples

Forty naturally-shocked rock samples collected from the Ries (Germany) impact structure were prepared for diffractometer scans. Although the varying mineral amounts in whole rock samples (as in the case of the granodiorite described previously) causes some uncertainty in peak shock pressure estimates, a rough correlation was found between the peak amplitude-half width ratios for the  $26.6^\circ$  two-theta quartz maxima and the degree of shock estimated by optical petrography and field relationships. The correlation of x-ray diffraction determinations of shock pressure with optical and field classification of samples is shown in the appendix (2).

### 4) References

- (1) Hörz, F. and Quaide, W.L. (1973) Debye-Scherrer Investigations of Experimentally Shocked Silicates. The Moon 6, p. 45-82.
- (2) Hanss, R.E. (1976) NSG 9039 Progress Report: X-Ray Diffraction Studies of Shocked Lunar Analogs.

### C. Publications and Presentations

A presentation entitled "X-Ray Diffraction Studies of Shocked Lunar Analogs" was accepted and delivered to the 1976 Annual Denver X-Ray Conference in August of that year. The entire article presented at the Denver Conference was published forthwith in the 1977 issue of Advances in X-Ray Analysis, Volume 20, p. 337-344. Authors were R.E. Hanss, B.R. Montague, and C. Galindo.

A RESEARCH PROGRAM FOR X-RAY DIFFRACTION STUDIES  
OF SHOCKED LUNAR ANALOGS

December, 1976

Abstract

We propose to perform x-ray diffraction experiments on shocked rock and mineral analogs of particular significance to lunar geology. Shock pressures ranging from 20 to 1300 kilobars can be generated in the 20 mm flat plate accelerator facility located at Johnson Space Center. This pressure range is sufficient to model the entire range of meteorite impact effects in any rock-like materials, from elastic brittle fracture through plastic deformation, solid state vitrification, selective partial melting, total rock melting, and possibly to vaporization. The shocked samples are recovered 100%. X-ray diffraction investigation of these samples reveals the degree of induced lattice disorder as a function of peak pressure. Calibration standards for peak pressure in single grains have been developed for a few minerals in particular pressure ranges. This quantification of peak pressures will be refined for quartz and feldspars, extended to other silicate minerals of lunar significance, and applied by powder diffractometer techniques. The determination of the distribution of peak shock pressures within phaneritic rock materials will be done by means of Debye-Scherrer rotated grain patterns.

The proposed experiments include a) powder diffractometer (line-broadening) studies of shocked single-crystal quartz, feldspars, and pyroxene, b) powder diffractometer studies of shocked monomineralic, polycrystalline rocks, e.g., anorthosite, c) Debye-Scherrer rotated-grain studies of granodiorite samples shocked by the Piledriver nuclear test (1962) and in the flat-plate accelerator, and d) powder diffractometer studies of naturally-shocked and accelerator-shocked samples of basalt from the Lonar impact crater, India.

A continuation of the present project will extend these x-ray diffraction studies to a) shocked particulate, monomineralic powders (selected grain sizes and controlled porosities) and b) shocked particulate multiphase assemblages, e.g., plagioclase-pyroxene-olivine analogs of lunar soils.

The results of these experiments will permit rapid, quantitative interpretation of the peak shock pressures experienced by materials found in lunar or terrestrial impact structures. This will assist in compiling the shock history of the lunar rocks and regolith, the earth's lithosphere, and meteorites.

## B. Progress Report Summary - June, 1977

### 1) Single Crystal Studies -- Quartz

An important mineral found in many terrestrial impact craters is quartz. This mineral is uncommon in lunar materials, although its polymorphs tridymite and cristobalite occur in many lunar samples. Because of its importance in crustal materials of the earth and because Debye-Scherrer x-ray studies of this mineral had already been carried out by Hörz and Quaide (2) and others, we determined to design a diffractometer technique to measure shock impact effects in powdered samples of quartz. The quartz samples were sections cut from single crystals and shocked normal to the (0001) plane at pressure intervals from 0 - 31.0 GPa. All shocked specimens were prepared in the 20mm flat-plate accelerator at Johnson Space Center.

The recovered shocked samples usually consisted of 10-50 mg of clean material. The samples were then ground to approximately 50 microns (-325 mesh), and then mixed with 5-micron aluminum oxide as an internal standard. Aluminum oxide was chosen for this purpose because its diffraction pattern does not interfere with that of quartz in the two-theta regions studied. Details of the sample mounting procedure and x-ray operating conditions are given in the paper by Hanss, Montague and Galindo (1).

Our diffractometer studies revealed a general decrease in the height of diffraction maxima with increasing shock pressure, until at 310 kb only faint remnants of the major peaks are distinguishable. Quantitative studies of the major diffraction peaks ( $26.6^\circ$  and  $20.8^\circ$  two-theta) were done by comparing the peak amplitude/half-width ratios for various shock pressures.

Table 1 (3) presents two sets of data for the quartz (101) diffraction peak ( $26.6^\circ$  two-theta). Figure 1 (3) shows these data -- peak height/half-

width plotted as a function of shock pressure. The peak height/half-width ratios are normalized to a set aluminum oxide value to correct for variations in sample quantities, and then are plotted as a percentage of the ratio for the unshocked (0 GPa) sample. We hope that this procedure will enable this technique to be useful to other investigators, by freeing it from dependency on particular x-ray operating conditions or the need to have a set of calibrated shock standards.

Extrapolation of our data indicates that peak height/half-width ratios for the (101) and (100) quartz diffraction maxima should decrease to zero value at approximately 32.0 GPa shock pressure. These are the major front-reflection (low angle, high intensity) diffraction peaks for quartz. Evidence from Debye-Scherrer and diffractometer studies of shocked granodiorite (Part 4 of this report), however, indicates that remnants of these diffraction maxima can persist to shock levels as high as 39.4 GPa. No quartz diffraction maxima of any kind were observed at shock levels above 39.4 GPa.

The mechanism of structural deformation in the mineral which produces the diffraction peak changes is described by Hörz and Quaide (2). In general, the long-range order is first diminished (causing high angle back-reflections to disappear), then short-range order also is destroyed, producing an x-ray amorphous diaplectic glass.

The effect of shock impact direction -- here normal to the (0001) plane, but of unknown crystallographic orientation in the shocked granodiorite -- on the deformation of the sample has not been studied by x-ray diffraction.

## 2) Single crystal Studies--Orthoclase

The second mineral we investigated for shock effects in x-ray diffractometer patterns was orthoclase, a potassium feldspar abundant in

terrestrial granitic rocks, but somewhat rare in lunar samples. The samples were cut from single crystals of clear Madagascar orthoclase, and were shocked normal to the (001) plane. Sample preparation procedures, x-ray and aluminum oxide internal standards were the same as those described earlier for quartz. Quantitative studies were made of the variation of the peak height/half-width ratio for the (002) diffraction maxima with varying shock pressure. Table 2 (3) presents the data obtained for this measurement; Figure 2 (3) shows the peak height/half-width plotted as a function of shock pressure.

As for quartz, the peak height/half-width ratio decreases with increasing shock pressure; it reaches zero near 29.7 GPa (no observable diffraction maxima), presumably because the sample has been entirely converted to amorphous diaplectic glass (maskelynite). Evidence from Debye-Scherrer and diffractometer patterns of shocked granodiorite (see Part 4 of this report) does indicate a few diffuse feldspar diffraction maxima in samples shocked up to 35.0 GPa. Hörz and Quaide (2) also show faint oligoclase diffraction peaks in samples shocked to 34.0 GPa. The granodiorite evidence may be due to oligoclase; distinction between orthoclase and oligoclase diffraction patterns is difficult for highly shocked samples.

### 3) Nuclear-shocked Granodiorite

Samples of granodiorite from the Climax stock, Nye County, Nevada, were studied to compare the effects of shock from the 61 kT Piledriver nuclear test (1962) with shock effects produced at the 20mm flat-plate accelerator at Johnson Space Center. This rock is a medium-grained (plutonic) multiphase material; modal analyses indicate an average composition as follows: quartz-28%, potash feldspar-16%, plagioclase feldspar-45%, and biotite-9%. For the Piledriver event, a set of six shocked samples (at varying radial distances from the detonation center)

and also an unusually complete set of stress and acceleration data was available (I.Y. Borg, 1972).

Debye-Scherrer studies of 350 single grains of nuclear-shocked quartz and feldspars were made, and the peak shock pressures were estimated using the calibration standards developed by Hörz and Quaide (2). Figure 3 (3) shows the maximum, minimum and mean values for the estimated shock pressures of each sample (50-75 grains) plotted as a function of the reported stress-gage peak shock pressure.

In addition to the Debye-Scherrer studies, diffractometer scans were made of finely ground whole-rock Piledriver-shocked samples. These were evaluated for peak shock pressures using the line-broadening characteristics observed in the single crystal quartz and orthoclase studies. The varying quantities of quartz and feldspars present in such whole-rock samples makes the evaluation of peak shock pressures somewhat imprecise, but the general characteristics of these patterns indicate shock pressures comparable to the average Debye-Scherrer values for the samples. Figure 4 (3) shows representative diffractometer patterns (25-30° two-theta) for nuclear-shocked specimens.

The conclusion of our data, both Debye-Scherrer and diffractometer, is that the nuclear-shocked samples did not experience shock pressures exceeding 20.0 GPa.

#### 4) Accelerator-shocked Granodiorite

In order to evaluate the behavior of Climax granodiorite under shock stresses similar to those of the quartz-feldspar Debye-Scherrer standards developed by Hörz and Quaide, thirteen samples of unshocked granodiorite (discs 6mm dia. x 1mm thick) were subjected to shock pressures 5.1-48.2 GPa at the JSC flat-plate accelerator. The recovered samples were prepared for Debye-Scherrer and diffractometer studies. Since the quantity of accelerator-shocked material was much less than the available nuclear-shocked material,

only about 170 single-grain studies of the JSC-shocked material could be made. These Debye-Scherrer patterns were also evaluated for peak shock pressure using the standards of Hörz and Quaide.

Figure 5 (3) shows the maximum, minimum and mean values for the estimated shock pressures of each sample (4-25 grains) plotted as a function of the accelerator peak shock pressure, computed from projectile velocity and equations of state. Shock pressures seem fairly well-defined, with only one sample (accelerator peak shock pressure 14.4 GPa) showing a Debye-Scherrer range of more than 10.0 GPa. For all samples of accelerator peak shock pressure less than 35.0 GPa, the average range of estimated Debye-Scherrer shock values is 5.2 GPa. These shock pressure estimates include both quartz and feldspar grains; at some shock levels, the Debye-Scherrer standard patterns do not permit more exact definition of shock value than about 5.0 GPa. Debye-Scherrer standard patterns were not established by Hörz and Quaide for shock levels greater than 31.0 GPa in quartz and 34.0 GPa in feldspar (oligoclase), therefore the accelerator shock patterns obtained for pressures of 35.0, 39.4, 45.8, and 48.2 GPa were not quantitatively estimated, but will be described here.

Sample 394 (10 grains, shocked to accelerator peak pressures of 35.0 GPa shows no visible quartz or feldspar. In general, the patterns resemble those of glass, with a few faint lines caused by undetermined minerals. Sample 393 (19 grains, shocked to 39.4 GPa) does show the two major lines ( $26.6^\circ$ ,  $20.8^\circ$ ) of quartz in 13 grains, and an apparent feldspar line ( $27-28^\circ$ ) in one grain. Sample 387 (10 grains, shocked to 45.8 GPa) shows no visible quartz or feldspar, but some faint lines at  $22^\circ$ ,  $24^\circ$ ,  $44^\circ$ , etc., possibly due to an accessory ferromagnesian mineral (some grains appear dark). Sample 386 (8 grains, shocked to 48.2 GPa) reveals only a faint powder pattern (not quartz or feldspar) in three grains; again this may be due to an



accessory ferromagnesian mineral.

Figure 6 (3) displays representative diffractometer patterns ( $25-30^\circ$  two-theta) for the whole-rock samples of Climax granodiorite shocked in the JSC flat-plate accelerator. These peaks cannot be treated as quantitatively as the single-crystal quartz and orthoclase, but qualitatively they reveal shock levels equivalent to the Debye-Scherrer patterns of the same samples.

#### 5) Preliminary work with Ries shocked materials

The Ries impact structure in Germany has been extensively studied in its mineralogy and petrology, and systems of classification have been developed for the shock effects in crystalline rocks of this locality. We prepared 40 Ries whole-rock samples for diffractometer scans to investigate the utility of this method for preliminary determination of shock pressures. Although the varying amounts of each mineral in the whole rock samples causes some uncertainty in peak shock pressure estimates, a rough correlation was found between the peak height/half-width ratios for the  $26.6^\circ$  two-theta quartz maxima and the degree of shock estimated by optical petrography and field relationships. Figure 7 (3) shows the correlation of the x-ray data with the optical petrography classification of the samples.

#### 6) Conclusions and recommendations for further investigation

This research on shocked lunar analogs by x-ray diffraction methods has revealed the usefulness of the diffractometer for rapid determination of shock level and has developed a procedure for quantitative determination of peak shock pressures. Debye-Scherrer studies of single grains showed good agreement with diffractometer scans. Study of the Climax granodiorite provided a correlation between the peak shock pressures developed during the Piledriver event and those produced at the JSC flat-plate accelerator.

Other lunar analogs, especially plagioclase feldspar, pyroxene and olivine need diffractometer calibrations for shock pressures. These same

minerals need Debye-Scherrer single-grain standards similar to those developed by Hörz and Quaide, but for higher shock pressures. Studies of the range of peak shock pressures developed within a rock sample are necessary for learning how a shock wave transverses a multiphase, polycrystalline rock material.

## 7) References

- (1) Hanss, R.E., Montague, B.R., and Galindo, C. (1977) X-Ray Diffraction Studies of Shocked Lunar Analogs. *Advances in X-Ray Analysis* 20, 337-344.
- (2) Hörz, F. and Quaide, W.L. (1973) Debye-Scherrer Investigations of Experimentally-Shocked Silicates. *The Moon* 6, 45-82.
- (3) Hanss, R.E. (1977) NSG-9039 Progress Report: X-Ray Diffraction Studies of Shocked Lunar Analogs.

## C. Progress Report Summary - February, 1978

### 1) Diffractometer studies of shocked materials

a. Quartz - We continued to investigate the effects of shock loading on single crystals of quartz. Samples previously studied for shock effects on the  $20.8^\circ$  and  $26.6^\circ$  two-theta peaks were re-used for detailed study of higher two-theta angle peaks. Because diffraction peak intensities for higher two-theta angles are usually less than 10% of the  $26.6^\circ$  intensity, measurement in this area is difficult and less conclusive. Moreover, some loss of powdered material occurred handling and remounting the samples.

b. Orthoclase - No additional studies were done on this mineral during the reporting period (June 1-December 31, 1977).

c. Oligoclase - Single-crystal oligoclase (Muscwa Lake, Ontario) was shock-loaded normal to the (001) crystallographic plane, at pressures ranging from 9.9-48.9 GPa. Peak-height/half-width ratios were measured for the  $27.8^\circ$  ( $\bar{2}20$ ) diffraction peak. Peak profiles and a plot of peak-height/half-width ratios vs. shock pressure are shown in the appendix (1).

d. Pyroxene - Single-crystal enstatite (Bamble, Norway) was shock loaded normal to the (210) crystallographic plane, at pressures ranging from 20.1-60.4 GPa. Peak-height/half-width ratios were measured for the  $28.0^\circ$  (420, 221) diffraction peak. Peak profiles and a plot of peak-height/half-width ratios vs. shock pressure are shown in the appendix (1).

e. Dunite - Polycrystalline olivine samples (Twin Sisters, Washington, dunite) were shock loaded from 21.2-59.8 GPa. The  $36.5^\circ$  (112) diffraction peak for each shocked sample was measured to obtain the peak-height/half-width ratio. These values were normalized against the  $47.0^\circ$  (220)  $\text{CaF}_2$  peak height, which material was used as an internal standard with the dunite. Peak profiles and a plot of peak-height/half-width ratios vs. shock pressure are shown in the appendix (1).

f. Effect of sample size on peak-height/half-width ratio - Shocked pyroxene (enstatite) samples mounted on glass without an internal standard were compared by x-ray fluorescence analysis for total iron content. Normalization of measured peak-height/half-width ratio according to Fe 6.403 keV fluorescence intensity produced little change from the original peak-height/half-width values for samples originally weighed to be 10 mg.

Unshocked quartz samples varying in weight from 2.5-28.0 mg were prepared for diffractometer study. The corresponding x-ray patterns were measured to determine peak-height/half-width ratios for the  $26.6^\circ$  two-theta peak. Very little correlation seems to occur directly between the measured ratio and the sample weight. Apparently, for diffractometer samples of such small size, sample surface geometry is of greater importance than sample weight. Data for these studies is given in the appendix (1).

## 2) Debye-Scherrer Single Grain Studies

a. Ries Crater, Germany - Four samples -- Otting 604, Zipplingen 352, Aumuhle 174, and Otting 680 -- (all granitic clasts from suevite) were

prepared for single-grain studies. 200 grains of Aumühle 174 were photographed, and 50-70 grains of each of the other samples. Cumulative pressure distribution graphs of each sample were prepared. These are shown in the appendix (1). Separation of the pressure values for quartz and for feldspar seems to indicate higher pressures appear for the feldspar grains, but more evidence is needed to state this definitively.

Coesite samples were recently (1/78) obtained from Dr. Jorg Arndt of the Mineralogisch-Petrographisches Institut (Tübingen, Germany). These were x-rayed to obtain standard patterns for comparison with uncertain mineral lines noted in Otting 680 -- the highest shock pressure. This work is not complete.

b. JSC Accelerator-Shocked Climax (Nevada) Granodiorite - Additional grains of accelerator-shocked granodiorite were prepared for Debye-Scherrer studies to increase the rather small sample size of previous studies (reported June, 1977). Cumulative pressure distribution graphs for accelerator-shocked samples are shown in the appendix (1).

Comparison of the accelerator-shocked grains with the naturally-shocked Ries samples presents some interesting conclusions. The Ries samples display a much wider range of shock pressures within each sample. The median Debye-Scherrer shock pressures for accelerator-shocked samples agree well with impact velocity pressure calculations. The median Debye-Scherrer shock pressures for the Ries samples are significantly lower than those traditionally quoted from microscopic observations; this is because such optical studies are based on the highest shocked species per any given thin-section and especially because optical characterization does not necessarily include the weak to modestly-shocked components. It is therefore suggested that detailed shock histories of naturally shocked specimens may only be developed if a statistically-significant number of randomly

selected grains is investigated.

### 3) References

- (1) Hanss, R.E. (1978) NSG-9039 Progress Report: X-Ray Diffraction Studies of Shocked Lunar Analogs.

### D. Publications and Presentations

An abstract entitled "X-Ray Diffraction Studies of Shocked Materials" was submitted and published in Lunar Science VIII (Abstracts of Papers Submitted to the Eighth Lunar Sci. Conf.), 1977, p. 395-397. The Lunar Science Inst., Houston, Tx. was the publisher, authors were R.E. Hanss, B.R. Montague, C. Galindo, and F. Hörz.

X-Ray Diffraction Studies to Evaluate In-Situ Stress Level Around  
Terrestrial Impact Craters - February, 1978

ABSTRACT

We propose to perform x-ray diffraction investigations on shocked mineral assemblages from a terrestrial impact crater to test the suitability of x-ray methods for rapid, quantitative evaluation of meteorite-induced stresses. Such studies are important to evaluate recently developed x-ray techniques as important tools in assessing shock pressures developed during meteoroid bombardment of the Moon, Mercury, and Mars as well as Earth. Accurate radial profiles of peak shock pressure are needed to develop adequate models of energy transfer during meteorite impact.

X-ray diffraction data has been obtained for quartz, orthoclase, plagioclase, pyroxene, and olivine (lunar analogs) recovered from controlled shock experiments performed at the Johnson Space Center flat-plate ballistic accelerator. Debye-Scherrer rotated single-grain studies and complementary powder diffractometer investigations have provided a set of shock-level standards for the estimation of the stress level experienced by naturally-shocked specimens of these minerals. A set of line broadening vs. shock pressure curves has been developed by the above investigators to facilitate rapid comparison of the average stress experienced by mineral separates or by whole rock samples containing the above minerals. Preliminary investigation of naturally-shocked materials has been carried out on granitic clasts from the Ries Crater (Germany) and the range of shock pressures developed within individual samples has been determined.

The proposed research is directed toward the development of a radial pressure profile across a known terrestrial crater by x-ray diffractometry and Debye-Scherrer techniques. The Charlevoix, Quebec, impact structure is selected as the most suitable crater for this investigation. The crater is relatively large (50 km in diameter), accessible, and has gneiss units containing abundant quartz and feldspar in the central uplift. Shock pressure contours have been established for this structure by optical studies of quartz deformation, and a set of coded samples for this structure is already in hand.

The investigation includes: a) x-ray diffraction analysis of the initial Charlevoix samples (100) and determination of shock levels for each sample; b) comparison of x-ray data with optical data; c) visit to the Charlevoix sample locations, and collection of additional samples needed to complete the radial profile; d) x-ray investigation of the additional set of samples and determination of shock level data; and e) preparation of the research findings for publication. The total research time for which support is requested is one year.

## B. Progress Report Summary - February, 1979

### 1) Debye-Scherrer Studies of Shocked Materials

A total of 100 good quality Debye-Scherrer films were prepared of the Otting 604 samples from the Ries structure, Germany. Shock pressures were estimated using our own standards prepared from accelerator-shocked samples. These standards are quite similar in general shock characteristics to those developed by Hörz and Quaide, but have density characteristics similar to other films prepared on the G.E. XRD-5 equipment in use in our laboratory.

The cumulative pressure distribution graph is shown in the appendix (Figure 1) (1).

### 2) Diffractometer Studies of Shocked Materials

The principal goal of the present research project is to test the x-ray diffractometer technique as a rapid means of determining shock pressures on a series of samples from the Charlevoix, Quebec, impact structure, and to prepare a radial pressure profile across such a crater. However, in the early stages of the project (late spring 1978) our diffractometer studies of Ries crater samples and Piledriver nuclear event samples revealed a complication that threatened to impinge on the reliability of shock pressure determination for whole-rock samples. Hence, we embarked on a rather meticulous review of previous diffractometer data and an investigation of the reproducibility characteristics of our diffractometer technique. This research is outlined below.

a. Reproducibility: In an attempt to improve reproducibility of the diffractometer peak-height/half-width ratio (our shock pressure parameter), we increased the size of our diffractometer samples from 5 mg to 25 mg. We formed this sample into a uniform layer within a uniform area approximately  $1.2 \text{ cm}^2$ , on a glass slide approximately 1.4 cm wide. We also selected the -400 mesh fraction from the Pitchford grinding mill rather than our previous

work with 200-300 mesh fractions. Samples were leveled and inspected microscopically for uniform topography. With these precautions we found that slides prepared with different portions of the same sample (Charlevoix 6 was selected for this study) yielded peak-height/half width ratios within  $\pm 20$  counts per second/degree two-theta of the mean value. This reproducibility ( $\pm 10\%$  for this sample) we considered very acceptable and somewhat better than values commonly reported in literature on quantitative analysis by x-ray diffraction.

b. Determination of the Percentage of Quartz-Feldspar by Diffractometry

To independently determine one of the two variables affecting PH/HW, we decided to utilize the unshocked quartz-feldspar mixtures to provide standard patterns for the estimation of quartz content in the Charlevoix crater samples. Diffractometer patterns (used in the interaction effects PH/HW measurements) provided visual standards for 0-15-30-45-60-100% quartz included in either a) microcline, b) oligoclase, or c) equal microcline-oligoclase matrix. We compared each Charlevoix sample with the appropriate standards, according to whether it showed a  $27.4^\circ$  two-theta microcline peak, a  $28.0^\circ$  oligoclase peak, or both. Comparing the relative peak amplitude of quartz and the feldspar(s) was our method to estimate percentage of quartz present. This involves an assumption that in the Charlevoix samples, the quartz and feldspars are shocked approximately equally, causing similar shock diminishment in the peak amplitude of each. The percentage estimates of quartz are given in Table 1 (1).

c. Determination of Average Shock Pressure for Charlevoix Samples

A graph of PH/HW vs shock pressure is shown in Figure 2 (1). The sloping lines project linear decrease in PH/HW as a function of shock pressure for varying quartz content. Intercepts on the vertical (PH/HW) axis are PH/HW values for unshocked quartz in the standards prepared for



the quartz-feldspar interaction studies described above. The common intercept on the horizontal (pressure) axis is 32.0 GPa, a value determined from studies of shocked single-crystal quartz.

d. Interaction of Quartz and Feldspar in Whole Rock Mixtures

We had previously found that peak height/half-width ratios (PH/HW) for accelerator-shocked, single-crystal standards of quartz, orthoclase, oligoclase, enstatite and olivine all seemed to decrease linearly with increasing shock pressure (Hanss et al., X-ray diffractometer studies of shocked materials, in Proc. Lunar Planet. Sci. Conf. 9th, 2773-2787 (1978). Yet we noted that this PH/HW variation was not solely dependent upon shock pressure but also was a function of certain sample characteristics including the quantity present for the component under study. In other words, the amount of quartz present in a whole rock mixture of quartz and feldspar makes a difference in the PH/HW. We prepared a series of 25 mg samples of varying proportions of unshocked quartz, microcline, and oligoclase (the principal components of the Charlevoix granitic samples) and carefully measured the PH/HW for each sample. The results of this study showed that a given quantity of quartz (e.g. 15%) mixed with microcline or oligoclase or both had a definite PH/HW, and PH/HW increased linearly with increasing percentage of the quartz component. Measurements of the feldspar PH/HW yielded similar results.

We conclude that whole rock samples of uniform weight, cross-sectional area, grain-size, and surface topography, the PH/HW is a linear function of two independent variables: the percentage of the component being studied (e.g. quartz) and the average shock pressure experienced by the sample.

Using the estimated quartz percentage and the measured PH/HW for quartz (Table 1) (1) each Charlevoix sample is plotted in Figure 2 (1) and the estimated shock pressure determined. These pressures are also reported

in Table 1 (1).

Since the determination of quartz percentage is done visually, we allow  $\pm 5\%$  error to be reasonable here. This error on the graph yields approximately  $\pm 5$  GPa in the shock pressure rating. (The amount of error is less for high quartz content or for high shock pressures.)

e. Verification of Results

Shock pressures in the Charlevoix samples have been optically estimated based on planar deformation features in the quartz and potassium feldspars by M.R. Dence, P.B. Robertson, R.A.F. Grieve, etc. of the Canadian Department of Energy, Mines and Resources. Our samples, however, were coded to prevent bias in our diffractometer study. The results were forwarded to the Canadian Department of Energy, Mines and Resources for verification or comment about our pressure estimates.

We did also utilize three shocked samples from the Ries Crater, Germany, for which we have previously done extensive Debye-Scherrer studies of shock pressure. Our diffractometer determinations, based on quartz content estimates, are given in Table 2, along with Debye-Scherrer average pressures. Agreement with Debye-Scherrer average pressures is excellent ( $\pm 1$  GPa) for two of the samples, and fair (5 GPa) for the third sample.

3) References

- (1) Hanss, R.E. (1979) NSG-9039 Progress Report: X-Ray Diffraction Studies to Evaluate In-Situ Stress Level Around Terrestrial Impact Craters.

C. Publications and Presentations

The following abstracts and article were published, and the presentation given in 1978:

Hanss, R.E., Montague, B.R., Davis, M.K., Galindo, C.P., and Horz, F.  
(1978) Pressure Distribution in Naturally and Experimentally Shocked

Granodiorites (Abstract). Lunar and Planetary Science IX, p. 451-453,  
Lunar and Planetary Inst., Houston.

Hanss, R.E., Montague, B.R., Davis, M.K., Galindo, C.P., and Horz, F.  
(1978) X-Ray Diffractometer Studies of Shocked Materials. Proc. Lunar and  
Planetary Science Conf. IX, p. 2773-2787, Pergamon Press.

Montague, B.R. (1978) Development of an X-Ray Diffractometer Technique  
(Abstract). Meteoritics, The Meteoritical Society.

A presentation entitled "X-Ray Investigations of Materials Shocked by  
Meteorite Impact Cratering" was given by M.K. Davis to the 1978 Southwest  
Undergraduate Research Conference in San Antonio, Texas. An abstract was  
distributed at the Conference. Davis received the Reuben L. Kahn Award  
for undergraduate research excellence.

#### D. Final Progress Report - December, 1979

##### 1) Verification Schemes of the Charlevoix Impact Crater Diffractometer Shock Pressure Estimates

###### a. Verification Scheme - Optical Shock Pressure Estimations

As stated in NSG-9039 Progress Report for June, 1979, we were awaiting  
verification of our diffractometer pressure estimates for the Charlevoix  
crater samples. The verification procedure relies on comparison of  
diffractometer shock pressure estimates with optical shock effects (planar  
deformation features) observed in the quartz and potassium feldspar. In  
late June, 1979, the optical shock pressure estimates for the Charlevoix  
samples arrived through the courtesy of Blythe Robertson, Canadian Depart-  
ment of Energy, Mines, and Resources. Of the total 37 coded Charlevoix  
samples for which we obtained diffractometer shock estimates, only 21  
samples could be identified and 17 of these were assigned an optical shock  
pressure estimate, while the other four samples were observed to have no

quartz. The comparison of shock pressure characteristics through diffractometer and optical methods is given in Table 1.

b. Analysis

From Table 1, it becomes apparent that a majority of the optical (microscope) shock pressure estimates fall into the rather broad ranges of the diffractometer estimates. Examples of these are C-1, C-2, C-3, etc. These are, however, nine diffractometer shock estimates which are not in agreement with the optical data. For instance, C-7, a diffractometer shock value has been assigned from 0-10 GPa whereas the optical data indicates there is no quartz at all in the sample. Furthermore, the identification of several samples is only tentative, and comparison of some samples, such as C-7, is doubtful. On the other hand, samples such as C-32 indicate that the diffractometer shock pressure range is not in agreement with the optical shock estimate.

c. Verification Scheme: Debye-Scherrer Correlation with Diffractometer Shock Pressure Estimations, and Analysis

Due to the number of incompatible optical and diffractometer shock pressure values exhibited in Table 1, and the sizeable group of diffractometer shock pressure estimates for which no corresponding optical value could be obtained, it was decided to utilize the Debye-Scherrer rotated single-grain technique of Horz and Quaide (The Moon 6 (1973), 45-82) to obtain additional shock values for several quartz grains from each Charlevoix sample. Variance among the several Debye-Scherrer values for each sample, however, indicates that a larger number of grains is necessary if significant results are to be obtained.

d. Concluding Remarks on Verification

Pending the completion of the optical shock pressure estimate

identifications and the confirmation of those samples tentatively identified, the verification process has halted with those samples in Table 1 for which optical shock values have been assigned. Utilizing the Debye-Scherrer method of investigation will require considerably more work to be done. Additional optical data or sample identification may still be forthcoming. Results of optical and diffractometer data compared favorably in a majority of values, however. In a number of cases, even closer agreement is expected if the quartz content of the samples can be more closely estimated.

## 2) Continuing Research

Research continued on diffractometer investigations of samples from the Brent impact crater (Ontario) and the Piledriver Nuclear Event (Nevada). All peak height/half-width ratios have been calculated for the  $26.6^\circ$  two-theta quartz diffraction maxima for the total of 15 Brent crater samples and a total of seven samples from the Piledriver Nuclear Event. All diffractometer samples of Brent and Piledriver were prepared according to the improved technique presented in the June, 1979 Progress Report.

Table 1

Correlation between shock pressure determined by diffractometer (x-ray)  
and optical (microscope) techniques on quartz from Charlevoix.

\*Bracketed samples denote tentative identity of sample.

<u>Sample No.</u>			
NASA			
C-1	MBP 8-67	3-13 GPa	5.0 GPa
2	MBP 130-69D	4-14	5.0
3	MBP 136-69A	2-12	12
4	MBP 12-67A	0-8	5.0
5	MBP 134-69	0-5	5.0
7	(MBP 135-69A)*	0-10	no quartz
8	MBP 127-69	13-23	not measured, est. 12-14
11	(MBP 39-67A)	20-30	7
12	(MBP 93-67)	3-13	no quartz
15	MBP 54-67A	8-18	5.0
18	(MBP 51-67A)	no quartz	22.5
20	MBP 3-67	24-34	no quartz
23	(MBP 98-67B)	no quartz	no quartz
24	MBP 83-68	7-17	not measured, est. 10-12
25	(MBP 86-68)	9-19	10
31	MBP 71-68A	2-12	10.5
32	MBP 102-68A	0-7	16
33	MBP 25-67B	3-13	14
34	MBP 120-69	2-12	20
35	MBP 30-67	0-9	5.0
36	MBP 49-67	0-5	5.0

### 3) Related Research: Field Study

#### PRELIMINARY REPORT ON X-RAY STUDIES OF CARRIZO SANDSTONE FROM PROPOSED TERTIARY IMPACT CRATER IN ZAVALA COUNTY, TEXAS

An area of deformation in Eocene clastic sediments 20 km south of Uvalde, Texas, has been suggested to be an impact crater by W.F. Wilson and D.H. Wilson (Geology v. 7, pp. 144-146, March, 1979). Samples of suspected shocked material from the rim area and central area were collected on February 3 and May 5, 1979, and were studied by x-ray analysis techniques in May, 1979 at St. Mary's University.

Three samples were studied: sample U10 from the southwest rim area, and samples U3 and U4--both from the central area. These samples were collected from outcrops of strata or several large blocks rather than loose float. Sample U10 was a durable, sugar-white medium-to-coarse grained sandstone (quartz arenite); samples U3 and U4 were well-cemented dark-brown quartz sandstones with hematite cement.

25 mg samples of -400 mesh powder were prepared from each whole rock specimen. X-ray diffractometer patterns using Cu K-alpha radiation were prepared of each whole-rock sample, and the peak height/width-at-half-height ratios were determined for the quartz  $26.6^\circ$  two-theta peak. These values were then compared with standards prepared from accelerator-shocked quartz specimens from Johnson Space Center. The ratios and the estimated shock pressures are as follows:

U3 - 870 cps/ $^\circ$ two-theta	-- ( 40% quartz)	-- $\leq 5$ GPa
U4 - 673	( 30% quartz)	-- = 0 GPa
U10- 3126	( 100% quartz)	-- = 0 GPa

Since the percentage of quartz is an important factor in the interpretation of the peak height/half-width ratios, we prepared a concentrate of quartz grains from sample U4 by hand-picking the hematite-free grains microscopically and using this sample for a second x-ray pattern. The U4 concentrate (100%

quartz) yielded a ratio value of 2609--interpreted as 0 GPa shock value.

As an independent confirmation, Debye-Scherrer studies of rotated single grains of hematite-free quartz were done. The resulting film patterns were compared with film standards prepared with accelerator-shocked samples, using the method of Hörz and Quaide. The results were as follows:

U3-1: = 0 GPa; U3-2:  $\leq$  5 GPa; U4-1: = 0 GPa; U4-2: = 0 GPa.

Hence, it appears from the samples thus far studied that shock levels experienced by the samples are not more than 5 GPa, and probably simply unshocked.

An attempt will be made to obtain samples from an oil exploration well drilled in the central part of the area; however, the upper 121 m of the 356 m well was not logged.

#### OVERALL PROJECT EVALUATION AND FUTURE DIRECTIONS

##### A. Main Discoveries and Benefits

This research stressed the utilization of x-ray diffraction as an adjunct to microscopic study and field methods for determination of the peak pressures experienced by terrestrial or lunar shocked materials. It reemphasized the value of the Hörz-Quaide (Debye-Scherrer) technique and introduced the feasibility of a more rapid technique using the powder diffractometer. Diffractometer shock pressure standards were published for quartz, orthoclase, oligoclase, enstatite, and olivine. An x-ray sample preparation technique was presented to standardize diffractometer measurements of shock pressure. The abstracts, articles and presentations made during the course of the research served to communicate its results to the scientific community. The laboratory facilities and experience developed during this research are a lasting benefit to the Department of Geology at St. Mary's University and to the research assistants who participated in



this work. A number of interesting research problems have been initiated and will be continued by the personnel during the time ahead.

#### B. Limits and Suggestions

The powder diffractometer technique relies on the comparison between the behavior of shocked standards and the unknown sample. The parameters for shocked standards are rather dependent upon the operating conditions at hand and the sample geometry; hence exact duplication of the data from one laboratory to another is unlikely, although relative results can be expected. The shocked standards are produced in very small quantities and are costly in time and materials to prepare (approximately \$100-\$200/sample). The diffractometer specimens are fragile and not readily transported without damage. However, our data indicates that the diffractometer parameter (peak-height/half-width ratio) behaves as a shock-pressure function of this same parameter for unshocked material--which is not costly and can be used to establish local operating conditions.

The attempt to utilize whole-rock specimens leaves several problems to be solved: an alternative method of compositional determination, and determination of x-ray absorption effects of one component upon the other(s). A simpler approach to the Charlevoix problem would be to attempt to locate monomineralic samples, such as quartzites, to establish some pressure contours for the area.

#### C. Acknowledgements

The principal investigator and research assistants benefited greatly from the continued guidance and support during these past four years by the NASA-JSC technical officer, Dr. Fred Hörz. He provided most of the research samples, extensive literature, use of shock facilities, and much editorial assistance in our publications. We also express our thanks to Rand Schaal

and Tommy Thompson of Lockheed Electronics Company, who prepared the shocked samples for us; to Drs. Michael Dence and Blythe Robertson of the Canada Department of Energy, Mines and Resources who encouraged and assisted us to undertake the Charlevoix research; and to many others at Johnson Space Center who provided discussion and encouragement. Finally, we thank the National Aeronautics and Space Administration which provided the support for this research under NASA Grant NSG-9039.