Final Report

PREPARATION OF SIMULATED LUNAR SAMPLES

By: P. S. DeCARLI and R. W. GATES

Prepared for:

NASA MANNED SPACECRAFT CENTER
GEOLOGY AND GEOCHEMISTRY BRANCH
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I. INTRODUCTION AND SUMMARY

This program has been concerned with the development of techniques for shock loading and recovering mineral samples under simulated lunar conditions. Program objectives were as follows: The mineral samples must be sealed in evacuated containers which are then shocked to sufficiently high pressures to lithify the samples. The containers must remain vacuum tight on release of pressure. In addition, the container must be designed so that it is possible to conduct the operations of loading and sealing the containers and of post-shock opening of the containers with simple tools in a vacuum glove chamber.

During the course of this program, three basic sample container systems were designed, tested, modified, and retested. Two of these systems did not yield satisfactory results. The third system worked very well, permitting recovery of vacuum-tight sample containers after shock loading to pressures as high as 300 kbar. The successful system employs an essentially one-dimensional shock propagation geometry that permits reasonably accurate characterization of the pressure history of the sample.

Shock-lithified samples prepared in the course of this program have been sent to Dr. W. R. Greenwood, NASA Manned Spacecraft Center. Some of these samples were shock lithified in low-pressure atmospheres ($\sim 10^{-2}$ Torr) of helium and of helium and argon. A further study of these samples should permit evaluation of the possibility that shock wave gas implantation occurred within the lunar regolith.

II. EXPERIMENTAL STUDIES

A. Sample Material

The sample material used in all of the experimental work is Vacaville basalt, obtained through the courtesy of the United States Geological Survey, Menlo Park, California. This rock is approximately 53% andesine (An 45-An 53), 31% augite, and 16% accessory minerals. The rock was comminuted by ball milling until all of the particles would pass through a 40 mesh sieve.

B. Cylindrical Shock Geometry

The cylindrical shock geometry used in the initial experiments is shown in Fig. 1. Sample containers were made from 1/2-inch-I.D. soft copper tubing with a 0.040-inch-thick wall. A copper plug was silver-soldered in one end of the tube. The tube was loaded with powdered basalt and evacuated. Pumping was continued for several hours to permit the sample to outgas and then the tube was crimped to form a vacuum-tight seal. It is expected that the residual gas pressure in the sample tube was sufficiently low that test results would not be affected. Sealed sample containers were placed in a helium atmosphere for 12 to 16 hours and then placed in a vacuum system connected to a helium leak detector. The failure to detect helium in these tests was considered to be adequate indication of the vacuum tightness of the sample containers. The sample containers were centered in lengths of 1-inch-I.D. steel pipe which was then filled with molten Wood's metal. The pipe was wrapped with explosive which was detonated at one end, producing a cylindrically converging shock wave that traveled down the cylinder.

Three experiments of this general type were performed. In each case, the sample container and most of the sample were recovered. The fragments of recovered sample could be described as "shock lithified." However, the sample containers were ruptured, and it is suspected that the ruptures were caused by jets originating in the sample cavity. These jets could have been the result of a nonuniform sample density within the tube.

On the basis of past experience with converging shock recovery experiments we conclude that unbroken sample containers could be recovered if the

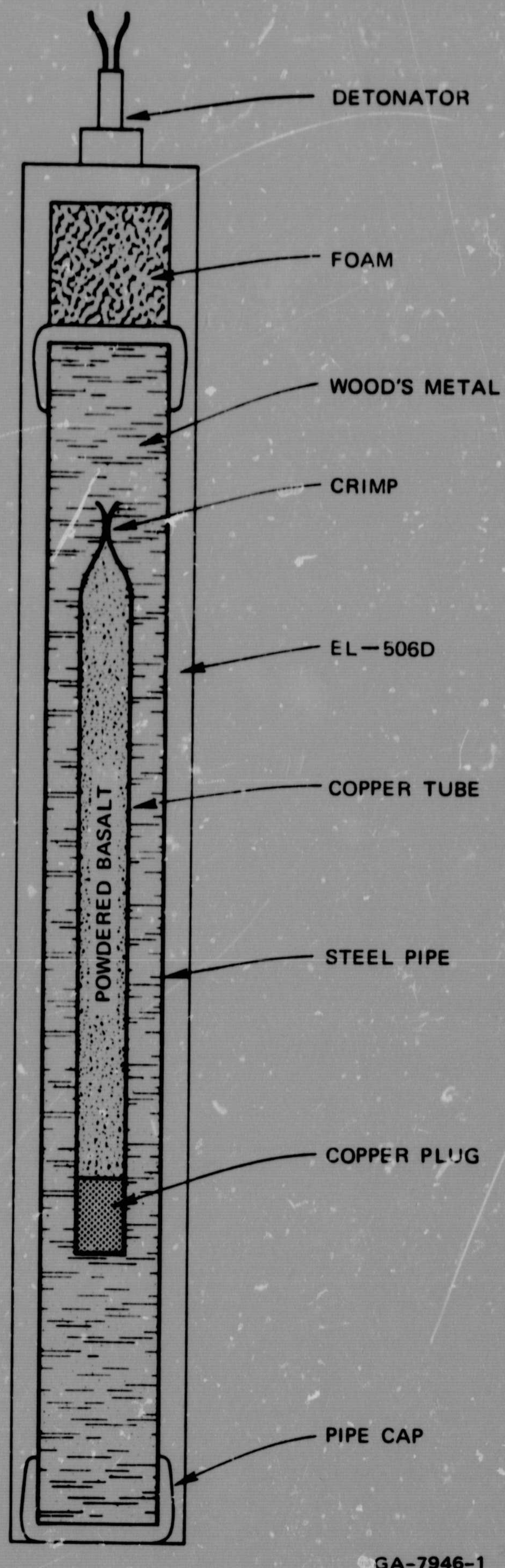

FIGURE 1 CYLINDRICAL SHOCK GEOMETRY

sample were packed to uniform density, if the container were cylindrically symmetrical, and if the container had thicker walls. These requirements appear to be incompatible with the requirement that the sample container be loaded and also opened after shock within a vacuum glove chamber. Furthermore, it is virtually impossible to accurately characterize the shock parameters for a porous sample shock loaded in the converging cylindrical shock geometry. We therefore abandoned the cylindrical geometry.

C. Quasi-Plane Wave Geometry

A second system, shown in Fig. 2, was designed and tested. This system was intended to combine the advantages of crimp sealing a copper tube with the benefits of an approximately characterizable shock geometry. The sample was placed in a copper tube which was evacuated and crimp sealed. The tube was then flattened in a press. It was necessary to reinforce the crimp seal by silver soldering to permit flattening. The container was leak tested and then placed in a groove in a steel block. Type metal was cast in the groove to fill the gaps between copper container and steel plate. The surface was then planed and a steel cover plate was bolted on.

The first sample container of this type was shocked to 100 kbar and recovered. The sample container was cracked and no longer vacuum tight. However, the damage was not severe, and it was hoped that the damage could be averted by using a redesigned momentum trapping system.

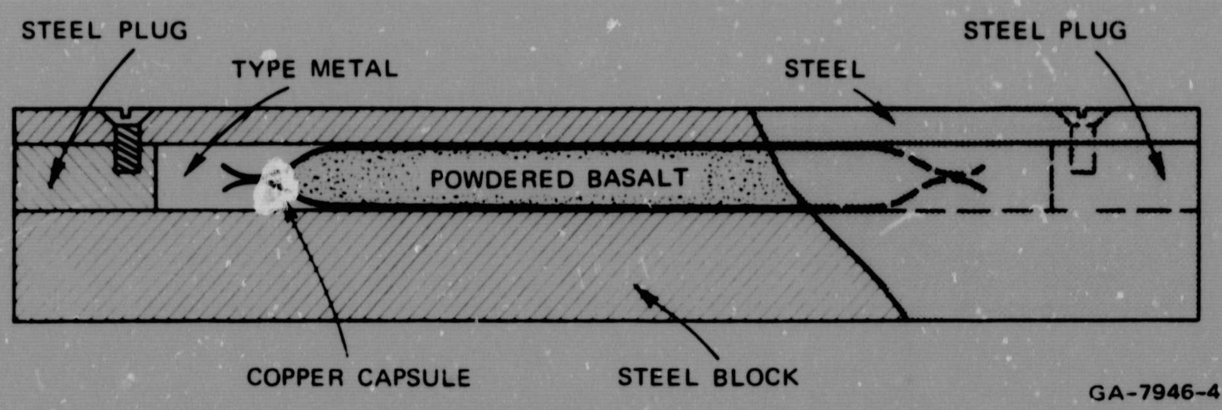

FIGURE 2 QUASI-PLANE WAVE SYSTEM

Post-shock examination of the damaged sample container indicated that the probable cause of failure was the interaction of rarefactions originating at the interfaces between the low-shock-impedance-type metal and the high-shock-impedance copper container. A search was made for alloys with low melting points which more closely matched the shock impedance of copper. None were found. However, it was noted that dental amalgam is an excellent shock impedance match to copper.

One experimental assembly was made up with the substitution of dental amalgam for the type metal used in the first experiment. This assembly was shock loaded to 100 kbar and successfully recovered. The recovered sample container was in excellent condition, but it was no longer vacuum tight. The vacuum leak did not appear to be the result of shock wave damage but rather the result of corrosion cracking attributable to the presence of mercury in the amalgam. This modified geometry would probably be satisfactory if a protective coating were used to prevent attack of the copper sample container by mercury.

The second approach taken to improve the quasi-plane wave geometry was to fit the sample container very closely to the momentum traps so that a filler material would not be needed. Cavities that very nearly conformed to the shape of the sample container were machined in stainless steel momentum trap halves. The momentum trap halves were pressed into good contact with the sample container and secured with bolts. Two such assemblies were built and shock loaded to 100 kbar. Both assemblies were recovered in good condition, but the momentum trap halves were firmly bonded together. In the course of splitting the momentum trap halves with rather vigorous hammer and chisel work, the sample containers were broken. It was observed that shock wave welding had taken place between the copper sample container and the stainless steel momentum trap halves. It appeared that it would be rather difficult to split apart the momentum trap halves in a vacuum glove box, but this geometry would be usable if shock wave welding could be prevented. In view of the successful results obtained with the plane wave geometry (described below), the quasi-plane geometry was shelved.

D. Plane Wave Geometry

The sample container developed for the initial plane wave shock loading experiments is shown in Fig. 3. The rock powder was placed in a cup machined from soft aluminum (alloy 1100, 99% pure aluminum). The sample cavity was closed by a soft aluminum piston which was backed up by a hard aluminum (alloy 2024-T4) piston. An indium ring was placed between the aluminum pistons to serve as a seal. Matching steps were machined in the pistons to minimize the area of indium that must be compressed to form the seal. The aluminum sample container fit into a mild steel cup which in turn fit into a mild steel ring. The steel cup, the steel ring, and a steel anvil (Fig. 4) comprised a momentum trap, which prevented destruction of the sample container by tensile interactions of rarefactions originating at free surfaces. The steel cup performed an additional duty in preventing deformation of the soft aluminum cup as the pistons were pressed in to compress the sample and to form the indium seal.

In the absence of a vacuum glove box, a simple vacuum chamber was jury-rigged to permit loading and sealing the sample containers under vacuum. The aluminum cup, fitted into the steel cup, was loaded with 30 grams of basalt and placed in the vacuum chamber. An alignment jig held the soft aluminum piston, the indium ring, and the hard aluminum piston above and well clear of the mouth of the cup. The vacuum chamber was sealed and pumped down overnight to thoroughly outgas the basalt. A plunger, working through a vacuum seal, was used to push the pistons and seal into the cup. A force of about 20 tons was then applied to the 2-inch-diameter pistons to compress the sample (to a density of about 1.9 g/cc) and to seal the sample cavity. Subsequent helium leak testing indicated that the indium (and possibly the soft aluminum) had flowed sufficiently to effect a vacuum tight seal of the sample cavity.

Two sample assemblies of this type were shock loaded to 100 kbar and successfully recovered. After machining off the steel cup to remove a source of virtual leak, the aluminum capsules were helium leak tested and found tight.

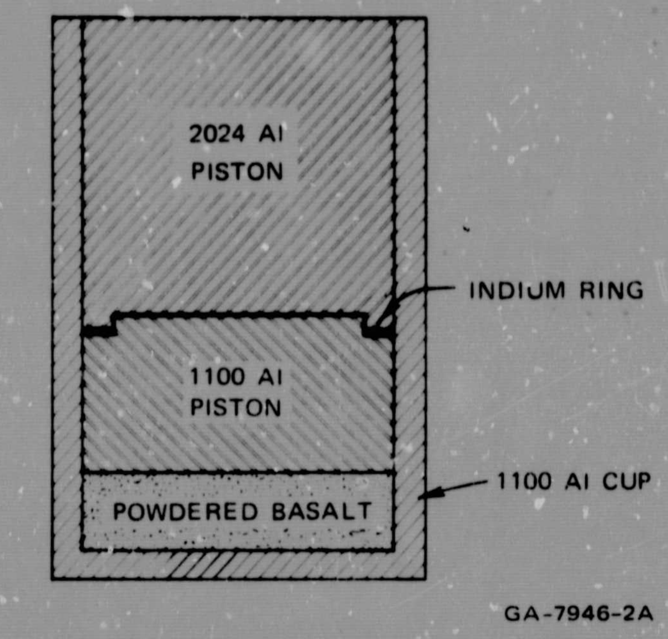

FIGURE 3 SOFT ALUMINUM CONTAINER

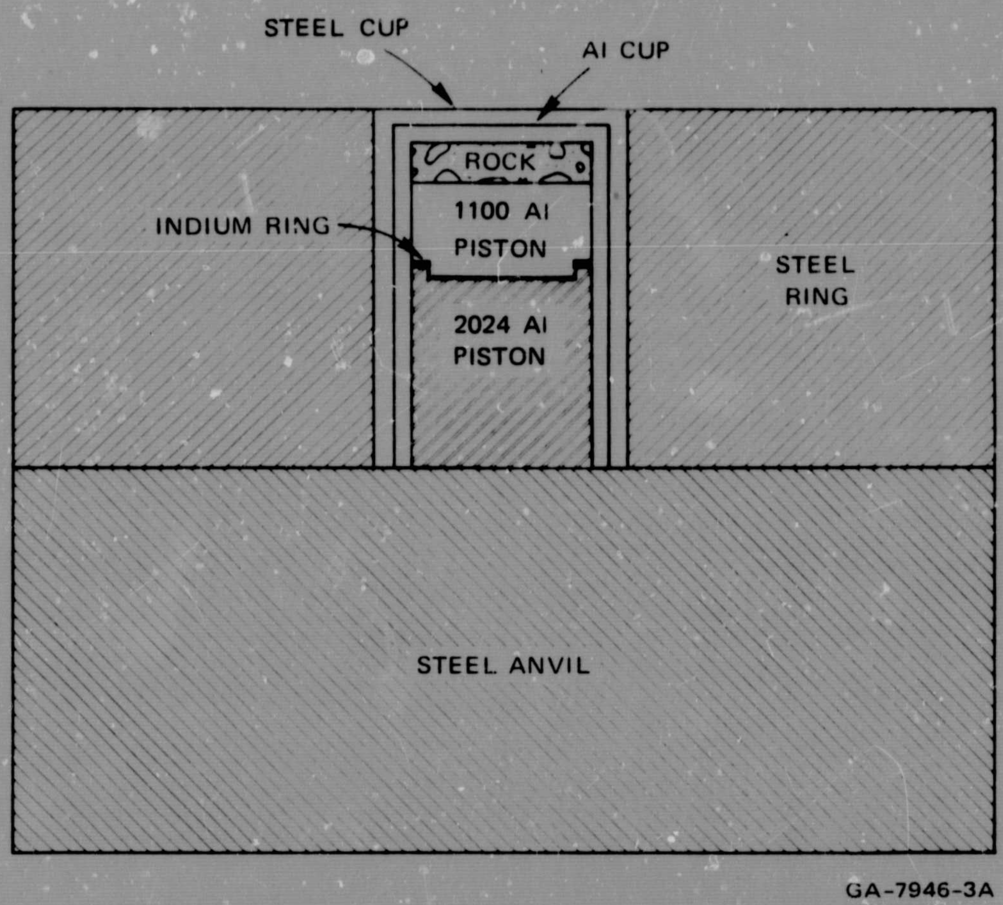

FIGURE 4 PLANE WAVE GEOMETRY CONTAINER AND MOMENTUM TRAP

Although some shock wave hardening of the aluminum container had occurred, it was relatively simple to cut open the container with a hammer and chisel. We judge that this operation could be easily performed in a vacuum glove box, if it were desirable to do so. However, it would be awkward to load the containers in a glove box because of the large force needed to compress the indium seal.

The sample container design was modified by the addition of an O-ring groove to the outer hard aluminum piston. The O-ring serves as a temporary seal that can be made when it is desirable to load the sample in a vacuum glove chamber. The container can then be removed from the vacuum chamber and placed in a hydraulic press for application of the necessary force to compact the sample and the indium sealing ring. The indium seal effectively isolates the sample from the O-ring and from the decomposition products to be expected as a result of shocking the O-ring.

Four plane wave geometry assemblies of the O-ring type were loaded and tested. One assembly was shocked to 100 kbar and was vacuum tight before and after shock loading. Two assemblies were shocked to 200 kbar and were vacuum tight before and after shock loading. The fourth assembly was shock loaded to 300 kbar and was vacuum tight before and after shock loading. The amount of deformation of the container shocked to 300 kbar was very large; the diameter increase was nearly 50%. We therefore consider that the present system is probably marginal for recovery of samples shocked to pressures near or above 300 kbar. However, we would expect routine recovery of vacuum tight containers shocked to pressures in the vicinity of 200 kbar.

These last four tests of the plane wave geometry were also intended as gas implantation experiments. After the container and sample had been outgassed within the vacuum chamber to a residual pressure less than 10^{-3} Torr, helium was admitted to raise the pressure to approximately 10^{-2} Torr. The container was then closed and removed from the vacuum chamber. The container was then subjected to a helium leak test, to determine whether the O-ring seal was effective. The container was next placed in a hydraulic press for further compaction of the sample and the indium sealing ring.

Five shock-lithified samples shock loaded in plane wave geometry were sent to NASA Manned Spacecraft Center. (See Table I).

Table I

SAMPLES SENT TO NASA MSC

(All Shock Loaded in Plane Wave Geometry)

Peak Pressure on Container (Kbar)	Container Atmosphere	Loading Chamber Pressure (Torr)
100	Vacuum	10^{-3}
100 (unopened sample container)	Helium	10^{-2}
200	Helium	10^{-2}
200	Helium	10^{-2}
300	Helium + Argon	2×10^{-2}

III. DISCUSSION

All of the samples shock loaded to pressures of 100 kbar could be described as shock lithified, although they are rather friable. The samples shocked to 200 kbar are much stronger, but the sample shocked to 300 kbar is the least well bonded of any of the samples. It is entirely possible that the large permanent deformation of the container shocked to 300 kbar can account for the poor bonding. This deformation, which took place during release of pressure, could have resulted in the breaking up of bonded material.

For the plane wave geometry experiments the pressure histories of the samples can be estimated with reasonable accuracy, for the input pressure pulses were sufficiently long ($> 5\ \mu$sec) to permit the sample to come to pressure equilibrium with the aluminum sample container. The actual pressures in the samples are believed to be within 10% of the nominal values of 100, 200, and 300 kbar. Since the sample attains pressure equilibrium with the container, via a series of shock reflections, the pressure estimates do not require knowledge of the Hugoniot equation of state of the sample material. The pressure uncertainties are due primarily to uncertainties in the velocities of the explosively accelerated flyer plates that impacted the sample containers.

For accurate estimation of shock temperature and residual temperature (immediately after pressure release) of the samples, one would require accurate knowledge of the Hugoniot equation of state and the release adiabats of the sample material. Unfortunately, the necessary data do not exist. However, we have made a few rough approximations to permit crude estimations of the residual temperatures of the shock-lithified specimens. The Hugoniot of the porous sample was approximated on the basis of the assumptions that the material is compacted to full density by pressures of 50 kbar or less, and that at higher pressures the compression curve of the material corresponds to the compression curve for the solid rock. The release adiabats of the sample were approximated by the compression curve of the solid rock. Using these data, the path (in the pressure-volume plane) of the material during a shock compression-pressure release cycle could be estimated. The area enclosed by the path is equated with the internal energy increase of the sample. This energy increase is assumed to be entirely thermal; phase transitions and other possible effects are neglected.

For the samples shocked to 100 kbar, the estimated temperature immediately on release of pressure is $400^{\circ}C \pm 150^{\circ}$. For the 200 kbar samples, we estimate $700^{\circ}C \pm 250^{\circ}$. For the 300 kbar sample, we estimate $900^{\circ}C \pm 350^{\circ}$.