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METHOD FOR ROCK PROPERTY DETERMINATION IN ULTRAHIGH VACUUM

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REFERENCE: Podnieks, Egons R. and Chamberlain, Peter G., "Method for Rock Property Determination in Ultrahigh Vacuum", ASTM/IES/AIAA 5th Proceedings of Space Simulation Conference, Gaithersberg, Maryland, Sept. 14-16, 1970.

ABSTRACT: A special ultrahigh vacuum system was designed for determining deformational and strength properties of simulated lunar rocks. The system provides a vacuum of 10^{-9} to 10^{-10} torr (10^{-7} to 10^{-9} N/m^2) depending on the specimen size and rock type. The vacuum was measured by various types of gages and also by ion pump current measurements. The partial pressure measurements of the various gas components were obtained by a quadrupole residual gas analyzer. The vacuum chamber has two stainless steel bellow-type feedthroughs for loading and a spring mechanism to compensate for the atmospheric pressure effect on the rock specimen. The uniaxial load was applied to the specimen by a servo-controlled hydraulic testing machine. Specimens of tholeiitic basalt, dacite, and semiwelded tuff were used. Special procedures in specimen preparation and preconditioning were developed. During the pumpdown period, prolonged roughing and an initial bakeout produced significantly low final pressures in the chamber. Test data presented include a typical load-deformation curve from rock specimen in ultrahigh vacuum, outgassing characteristics during loading in terms of pressure variation, and changes in the composition of gases being released by the specimen.

KEY WORDS: rock mechanics, space environment simulation, ultrahigh vacuum, pressure measurements, outgassing, mass spectroscopy, mechanical properties, anisotropy, compression tests.

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Since 1965 the Bureau of Mines has been engaged in NASA-sponsored multidisciplinary research leading to the use of extraterrestrial resources. As a part of this broad program, rock failure processes and the strength and elastic properties of rock in simulated lunar pressure environment have been investigated. To facilitate these studies a special ultrahigh vacuum system has been designed that can be readily moved in and out of a hydraulic testing machine, to permit axial loading of a rock specimen through a set of bellow feedthroughs. This vacuum system is equipped with a variety of pumps incorporating both momentum transfer and capture types. With this system an ultrahigh vacuum of 10^{-9} to 10^{-10} torr (10^{-7} to 10^{-8} N/m^2) can be obtained in 2 days depending on the rock type and specimen size. During the axial loading of a specimen, the rock deformation behavior is determined and compared with simultaneously obtained outgassing data. Total pressure rise in the chamber and the partial pressure measurements of the outgassing components, made by a quadrupole residual gas analyzer, are correlated with the deformational and fracture characteristics of the test specimen.

EXPERIMENTAL EQUIPMENT

The vacuum chamber consists of a 14-in. (35.5 cm) stainless steel cylinder 26-in. (66 cm) long equipped with ports for attaching pumps, gages and other accesories (Fig. 1). The purpose of this chamber is to provide an ultrahigh vacuum environment for rock testing in simulated lunar pressure environment. Since the first phase of this research covers only the effect of vacuum on rock strength, a relatively compact test chamber is quite satisfactory.

The pumping system (Fig. 2) consists of a 400 l/sec ($0.4 m^3/sec$) ion pump supplemented by titanium sublimation and cryopumping. The ion pump is directly attached to the chamber by a 6-in. (15.8 cm) port. The sublimator deposits titanium film directly on the wall of the test chamber and onto a chilled copper coil with the test specimen and the gaging protected by proper baffling. The cryopumping is accomplished by flowing liquid nitrogen (LN_2) through a coil of copper tubing lining the chamber near the test specimen. The initial pumpdown of the system is performed by three cryosorption pumps supplemented by a mechanical carbon vane pump.

The vacuum conditions in the chamber are first monitored by thermocouple and alphatron gages mounted on the roughing manifold. After the roughing cycle the vacuum measurements are obtained directly from inside the chamber by a nude ionization gage and/or a cold cathode (Kreisman) gage and also by ion pump current measurements. A quadrupole residual gas analyzer is used intermittently first for leak detection and second for giving partial pressure measurements of the various gas components originally present in the system or being released by the rock specimen during the test.

The vacuum chamber is specially adapted for compression testing of rock specimens in vacuum (Figs. 3 and 4). Both of the axial loading platens are connencted with the chamber by stainless steel bellows to allow platen movement during the test and also to provide a positive bakeable vacuum seal. In addition the platens are spring mounted to compensate for the force applied to them by the pressure differential during the pumpdown of the system. The rock sample is surrounded by a metal screen to prevent rock particles that spall away during the compression test from damaging other components in the chamber. The load applied to the specimen is measured by a load cell located underneath the specimen and by a second load cell exterior to the vacuum chamber. The specimen deformation is measured by a strain gage - cantilever beam type extensometer which indicates the deflection between the platens.

The vacuum chamber assembly is mounted on wheels and is moved into position in the compression testing machine when a test is to be made (Fig. 5). A servo-controlled hydraulic testing machine is used to apply the axial load to the specimen at a controlled load or deformation rate (Fig. 6). During the compression tests an X-Y recorder handles load-deformation data, a strip chart recorder monitors pressure changes in the vacuum chamber, and an oscilloscope camera documents mass spectrometer traces at various stages of specimen deformation.

EXPERIMENTAL TECHNIQUES

Rock Selection

The rock materials chosen for this program were earth-formed rocks assumed to simulate lunar rocks. In establishing the rock standards for lunar research the following criteria were used for rock selection (1)[2]: 1) they should represent lunar rocks on the basis of lithology, texture, and composition, 2) they should meet specific requirements for the particular research likely to be performed on the rocks, 3) they should be uniform in composition and texture and 4) they should be convenient to collect in sufficient quantity. Following these criteria the Bureau of Mines selected fourteen simulated lunar rocks covering a broad range of possible lunar rocks (2). From this suite of fourteen rocks three were first tested in this program: 1) a tholeiitic basalt (from N. E. of

2 "The numbers in parentheses refer to the list of references appended to this paper."

Madras, Oregon) to simulate rock from large basaltic flows; 2) a dacite (from West of Bend, Oregon) to simulate an extrusive rock with a composition intermediate between rhyolite and basalt; and 3) a semiwelded tuff (from Bend, Oregon) to simulate possible ash flows on the Moon. These three rocks represent a wide range of properties: bulk density - 1.15 to 2.84 g/cc (1150 to 2840 kg/m^3), Young's modulus - 0.3 to 10.3 x 10^6 psi (2.1 to 71 GN/m^2) and compressive strength - 850 to 53,000 psi (5.9 to 370 MN/m^2).

Rock Specimen Preparation and Preconditioning

Preparing rock specimens for compression testing requires special attention because of the time consuming preparation and preconditioning processes involved. Since most rocks are anisotropic and nonhomogeneous, i.e., their properties vary with direction and location within a rock mass (3), their properties have to be measured and analyzed with respect to fixed reference directions. At the Twin Cities Mining Research Center a standard procedure has been developed for obtaining oriented rock specimens. If possible, rock blocks are oriented with respect to in situ principal geologic and geographic directions. From each block a 3- or 4-in. (7.6- or 10.1 cm) spherical specimen is prepared maintaining reference directions with respect to the source block throughout the process (4). Longitudinal pulse velocity measurements are performed on these spheres in many diametrical directions. These measurements are plotted on equal area nets and contoured, resulting in velocity symmetry patterns that provide information on the orientation of rock fabric features, e.g., elongated vesicles, which control other property anisotropy as well (3). From the velocity symmetry patterns meaningful directions in which to measure the compression properties are selected, and since the sphere orientation is carried throughout the process, it is possible to prepare oriented specimens from the source block. Cores were drilled from the source block in the selected directions with a diamond impregnated bit, and cut to size with a diamond-blade saw. For these tests cylindrical specimens were used with a length to diameter ratio of 2:1. The tholeiitic basalt and dacite specimens were 1-in. dia. (2.54 cm), whereas the semiwelded tuff specimens were 2-in. (5.08 cm) in diameter because of large inclusions in the rock. The dimensional tolerances being formalized into ASTM Standards were used in the preparation of rock specimens (5). The ends were cut and surface ground perpendicular to the axis of the specimen within 0.25°, the diametrical variations were held within 0.005 in. (.1270 mm), and the end roughness within 0.001 in. (.0254 mm). During the cutting, drilling and grinding operations the only coolant was water. Before being placed in the vacuum chamber, the rock specimens were dried in a vacuum oven for one week at 135°C and about 50 x 10^{-3} torr (6.66 N/m^2)

pressure. After being cooled to room temperature, the specimens were backfilled with dry air so that they could be transferred to desiccator for storage under moderate vacuum.

Test Procedure

The preconditioned rock sample was placed in the vacuum chamber while the chamber was in the testing machine to assure proper alignment, preloading and checkout of interior instrumentation. While the chamber is open to the atmosphere, dry nitrogen gas was forced through it to minimize moisture contamination. After the specimen was properly mounted on the test platens, the system was closed and the rough pumpdown started by a carbon vane vacuum pump. At that time the pressure differential across the platens forced the bellows to extend until both platens contacted the specimen and created a preload on the specimen. This preload was held to the desired value by the compensating springs. With the specimen secured between the platens, the vacuum chamber was moved out of the testing machine for the pumpdown cycle.

The initial rough pumpdown of about 15 minutes by the carbon vane pump reduced the chamber pressure to approx. 25 torr (3340 N/m^2). Thermostatically controlled electrical band heaters encircling the exterior of the chamber were then turned on and final roughing by cryo-sorption pump was initiated and continued for 4-hr. During this pumpdown phase the chamber pressure was monitored with an alphatron gage located in the manifold section of the vacuum system. After the 4-hr. roughing, when the pressure in the chamber is in the middle of 10^{-4} torr (high 10^{-2} N/m^2) region, the manifold was closed and the ion and the sublimation pumps were started. During the following pumpdown ion and cold cathode (Kreisman) gages were used to monitor the chamber pressure. A 24-hr. bakeout was initiated when the ion pump was turned on. This bakeout was thermostatically controlled at 135°C and was automatically interrupted if pressure in the chamber rose above the 10^{-5} torr level. During the bakeout the system was checked for leaks with a mass spectrometer using helim gas. After bakeout the system was cooled for 24-hr. period before the rock specimen was tested. Figure 7 shows typical pumpdown curves for the empty chamber and for chamber containing specimen of basalt, tuff and dacite.

After the desired vacuum has been reached, usually in the low 10^{-9} or 10^{-10} torr (low 10^{-7} or 10^{-8} N/m^2) range, the vacuum chamber was positioned in the testing machine and the pretesting alignments were made. Shortly before the start of the compression test, the cryoshroud was cooled off with LN_2 resulting in a 0.5 - 1.0 decade pressure drop within the test chamber. The rock specimen was compression-tested to complete failure at a constant deformation rate of 1 x 10^{-5} cm/cm/sec.

During the tests the load applied to the specimen and the deformation of the specimen were recorded on an X-Y plotter (Fig. 8). The chamber pressure variation (Fig. 9) as well as partial pressure changes indicated by the mass spectrometer traces (Fig. 10) were recorded simultaneously with the load-deformation data.

RESULTS AND CONCLUSIONS

Applying vacuum technology to rocks requires a modification of the conventional methods of simulating a space environment. The problems of space technology usually deal with man-made materials that are specifically designed to meet the extraterrestrial environment requirements. In this program, however, natural rock material, being studied under simulated lunar vacuum conditions, cannot be chosen by its adaptability to vacuum technology. This factor requires special attention to establish valid preconditioning methods in order to approach conditions in the rock specimen (that have been selected because of the likelihood of their occurrence on the Moon) that are similar to those in actual lunar rocks.

Previous studies at the Twin Cities Mining Research Center of vacuum and moisture effects on rocks (5,6,7) have helped to establish specimen preconditioning requirements for various rocks and specimen sizes. These studies have shown that is is nearly impossible to obtain complete equilibrium between the vacuum environment surrounding the rock specimen and the entire volume of the specimen. Equally evident from the experiment is the effect of preconditioning treatment on the pumpdown and bakeout procedures. Since terrestrial rocks normally contain gases and moisture either in pores or interstitially, the preconditioning treatment and the initial rough pumping have to be prolonged enough to remove the most of the gas load before the chamber is sealed for high vacuum pumping with ion and sublimation pumps. Because of the rock structure, the bakeout process requires caution. To avoid causing irreversible structural changes in the rock, bakeout temperatures should be kept to the minimum required to discharge water vapor.

As the rock undergoes deformation during the compression test, cracks are formed and existing cracks either opened or closed. This formation of new surfaces and opening of interior cavities causes a very distinct increase in pressure (Fig. 9) which continues until the specimen fails (compressive strength). During the post-failure loading when mostly redistribution of broken rock particles takes place, few new fracture surfaces are formed and the pressure tends to drop because the outgassing rate falls below the pumping capacity of the system. The outgassing during a compression test depends greatly on the rock type. Rocks with interconnected pores tend to release a smaller amount of gasses because of the ease with which they are removed during preconditioning and pumpdown. Whereas relatively dense rocks with interstitially held gas loads tend to release larger amounts during the deformation and failure process (5). Mass spectrometer analyses indicate that for the three rock types used the principal outgassing components were: water vapor, nitrogen, hydrogen, and carbon dioxide (Fig. 10). The principal increase in outgassing during the compressive loading cycles was contributed by water vapor and nitrogen.

The prime objective of this presentation is to describe the problems associated with research in rock properties in simulated space vacuum environment that were encountered in our study on the compression properties of simulated lunar rock in ultrahigh vacuum. The main emphasis is directed toward obtaining the deformation and strength properties of rock under carefully controlled conditions of vacuum. These studies also have shown that the analysis of outgassing characteristics of rock in vacuum gives highly significant data on the basic mechanism of rock deformation and failure.

ACKNOWLEDGEMENTS

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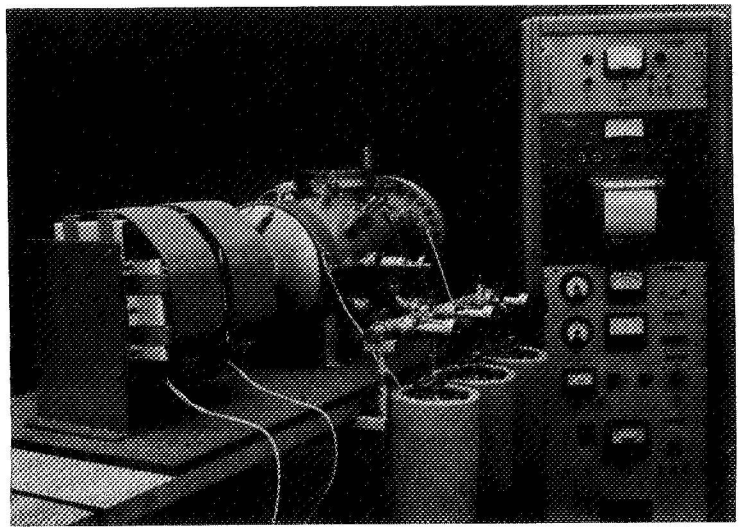


Fig. 1--Ultrahigh Vacuum System.

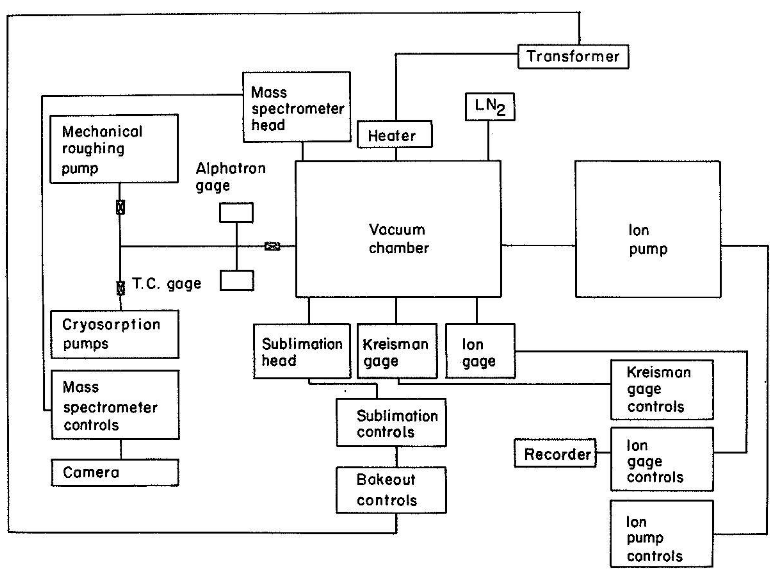


Fig. 2--Ultrahigh Vacuum System.

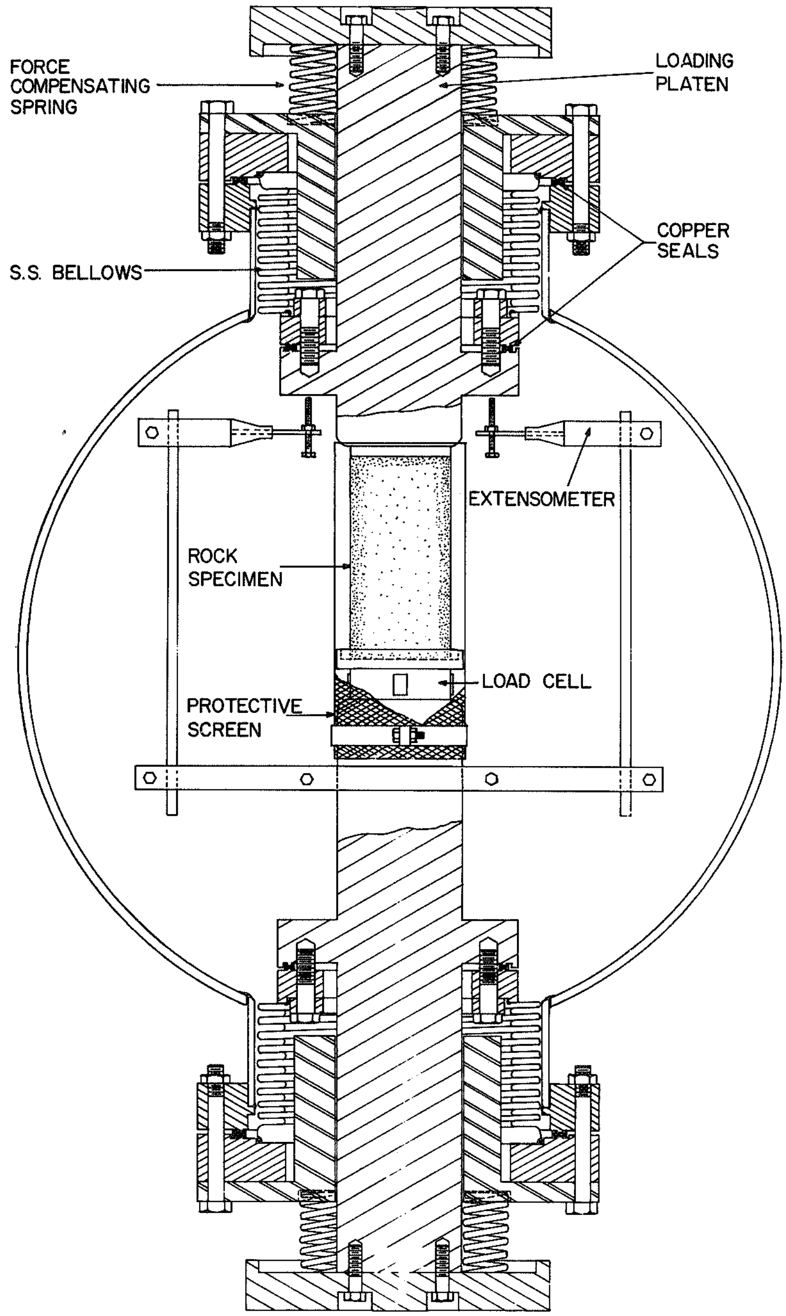


Fig. 3--Adaptation of Vacuum Chamber for Compression Tests.

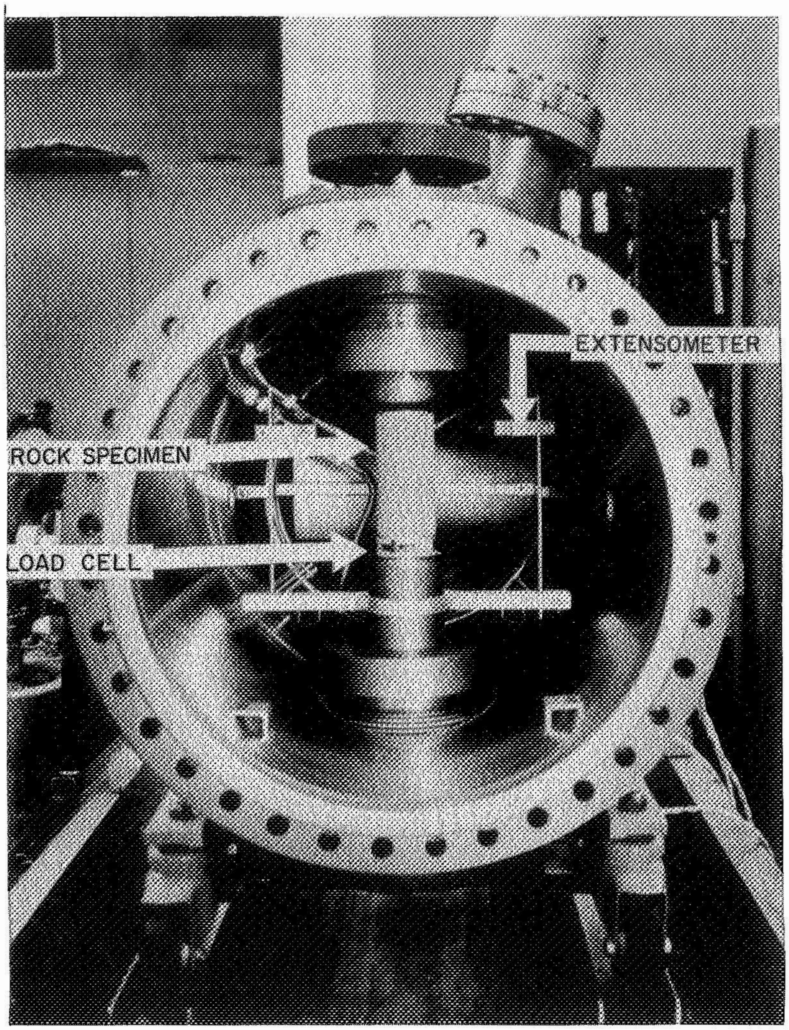


Fig. 4--Specimen in Position Within Vacuum Chamber.

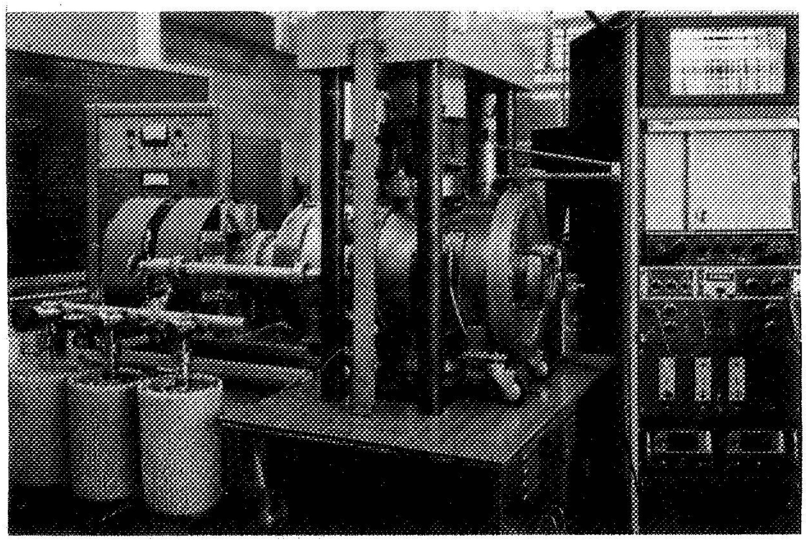


Fig. 5--Vacuum Chamber Positioned in Compression Testing Machine

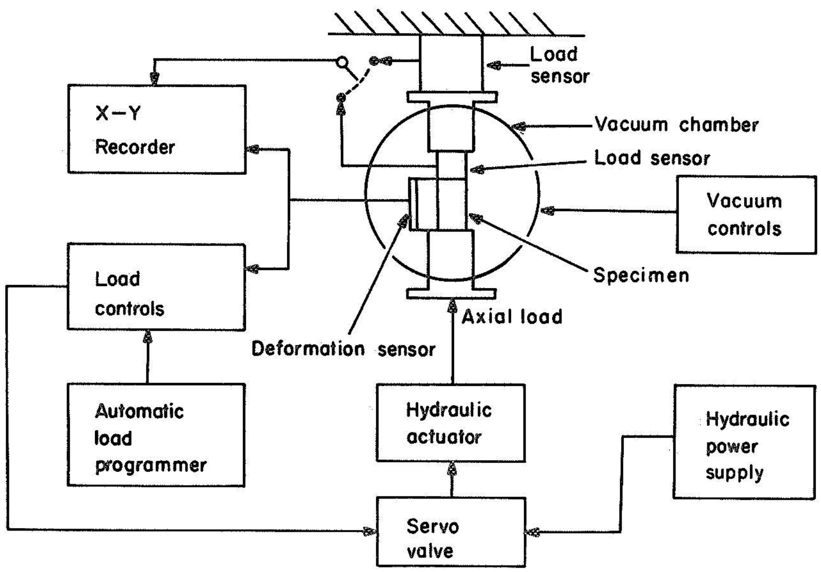


Fig. 6--Compression Testing System.

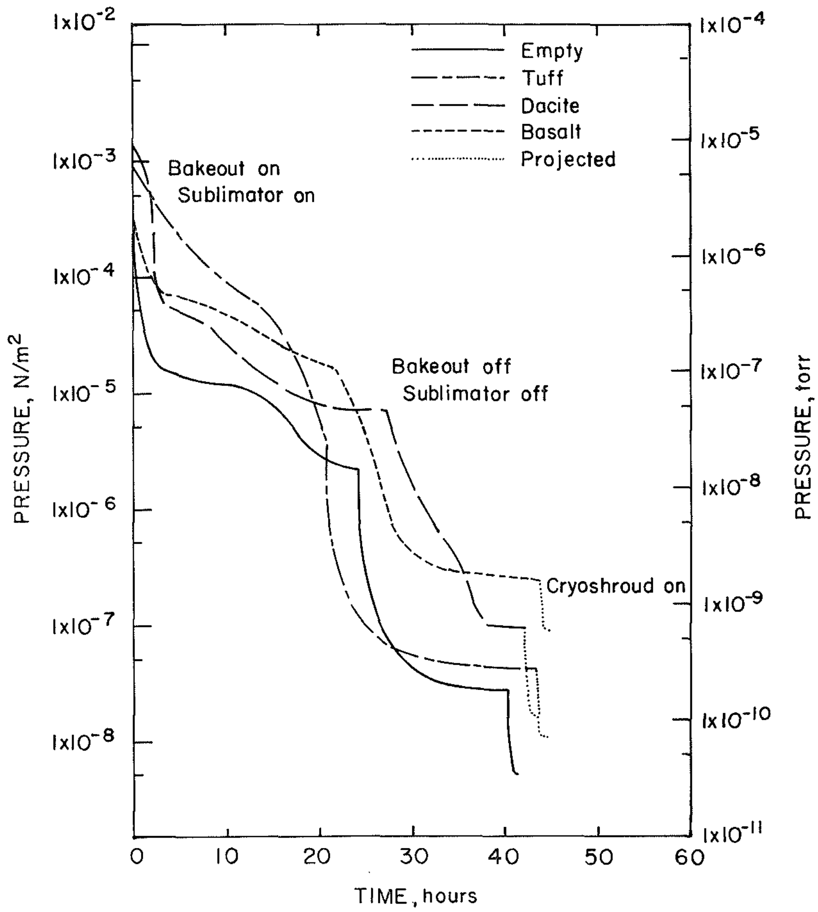


Fig. 7--Typical Pumpdown Curves.

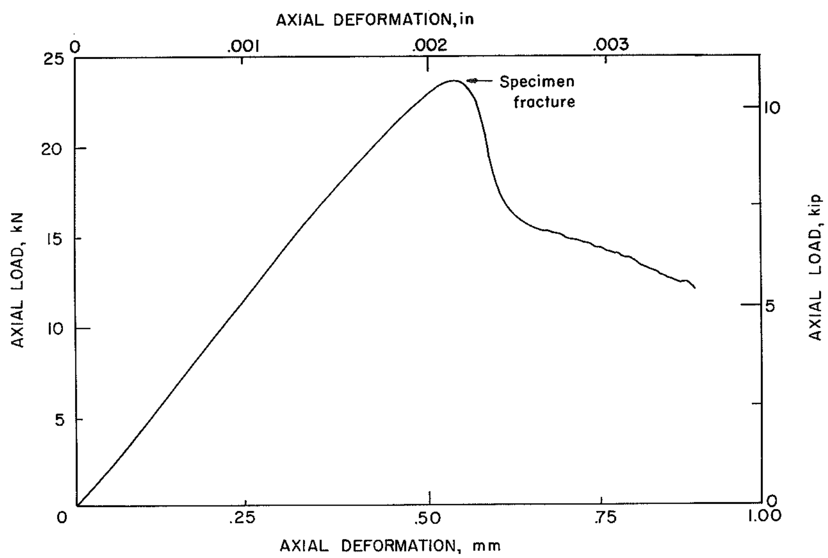


Fig. 8--Typical Load - Deformation Curve for Tuff.

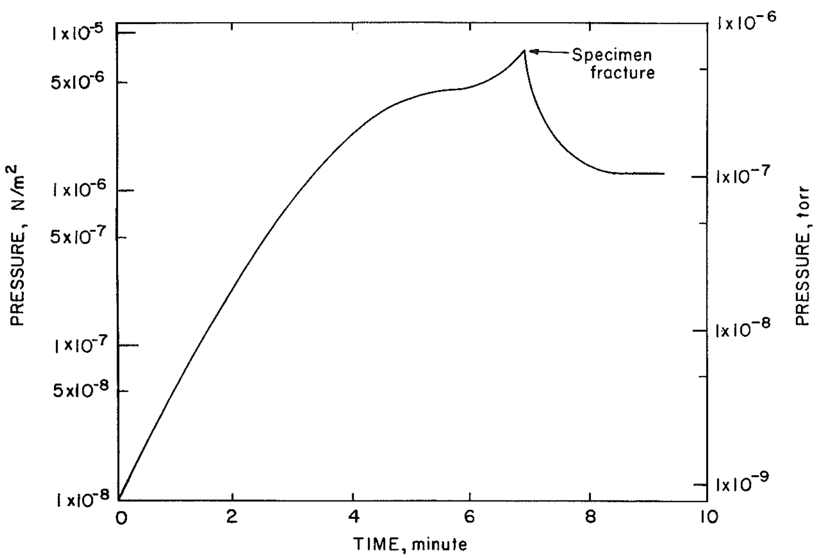
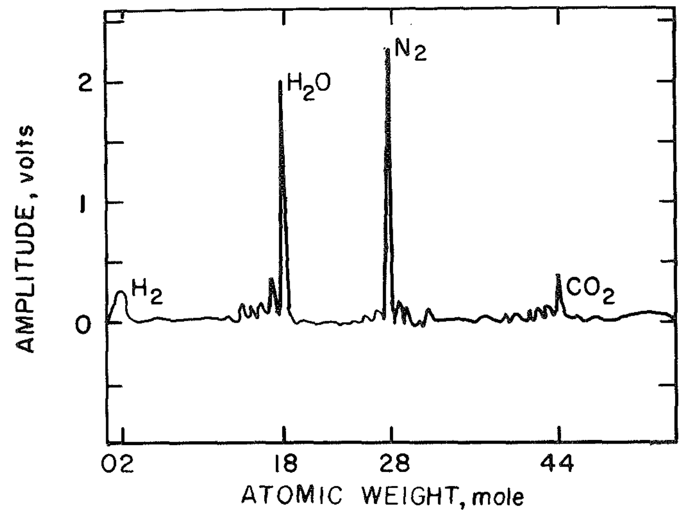
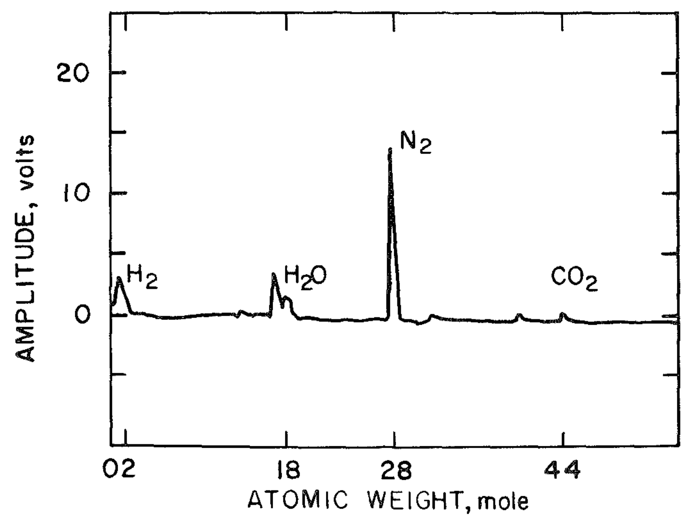


Fig. 9--Chamber Pressure During Compression Test on Tuff.



(a) - At Initiation of Compressive Loading.



(b) - At Specimen Fracture.

Fig. 10--Mass Spectrometer Traces for Tuff During Compression Test.