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SEMIANNUAL REPORT

on

THE EFFECTS OF FLUIDS AND CYCLIC LOADING ON THE ELASTIC CONSTANTS OF ROCKS

by

Phillip N. La Mori, Principal Investigator
Phone 614-299-3151 X3415

August, 1971

to

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505 King Avenue
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INTRODUCTION

It has been known since the earliest days of compressibility measurements on rocks(1), i.e., equation of state measurements, that the effects of cracks and voids have a predominant effect on the value of the modulus at low confining pressures. Birch's(2) data suggested that at low pressures cracks and voids were strongly influencing the velocity of sound measured in a large suite of rocks. Ide(3) was the first to point out that there were discrepancies between the values of the elastic moduli of rocks when measured by static and dynamic methods.

More recently, Simmons and Brace(4) compared bulk moduli of the same rock determined by static and dynamic methods at pressures up to 8 kb. They found the dynamic bulk modulus to be larger than the static at low pressures. However, at higher pressures the differences decreased. Simmons and Brace ascribe this difference to cracks which affect static moduli more than they affect the propagation of acoustic waves. La Mori(5) has shown from experiments to 40 kb that the differences in moduli determined by the two methods are in the relationship predicted by theory or within experimental error.

Simmons and Brace compared results from separate static and dynamic tests on different samples from the same rock. This technique is useful but has several disadvantages. One major disadvantage is that even rocks of the highest homogeneity are still prone to local hetrogeneities and therefore it is difficult to make comparisons with absolute certainty. Because of this and the fact that certain effects tend to show up more in one type of experiment than the other, it is difficult to compare interesting, experimental phenomena by the two techniques. The program we are discussing here measures one acoustic velocity and the static volume change simultaneously on the same sample. Thus, local hetrogeneities become more easily recognizable and interesting experimental phenomena can be compared simultaneously. An additional advantage of this technique is that density, required in the equation $C = \rho V^2$, is determined exactly; C = modulus, ρ = density, V = acoustic velocity.

Derivation of the relations between stress and strain in elastic theory is well known and need not be repeated here. For the experiments used in this work, the elastic constants are determined by both dynamic, i.e., ultrasonic velocities, and static methods, i.e., linear strain. For a material which is isotropic, determination of linear strain is sufficient to determine the density and bulk modulus. Additionally, determination of linear strain in two perpendicular directions when the sample is stressed differently along principle stress axes will define all the other elastic constants. For the velocity experiments with an isotropic material, determination of the compressional and shear wave velocities and density are sufficient to define all of the elastic constants.

For materials which are transversely isotropic, such as certain sedimentary and metamorphic rocks, determination of two compressional and two shear wave velocities plus a compressional wave at 45° to the principle axis will define the elastic moduli if the density is known. Similarly with selected strain gage, hydrostatic, and triaxial experiments, it is possible to determine the static elastic moduli. Thus, for most rocks, it is possible to define the elastic response by either simple acoustic or linear strain measurements.

The purpose of this research program is to measure the elastic constants of rocks, simultaneously, by velocity and linear strain measurements in order to understand the differences and similarities between the dynamic and static values. A second goal of this research program is to measure the effects of cyclic loading to different or similar stress levels on the measured values of the elastic constants by static and dynamic means.

La Mori(6) and Stephens(7) have shown that high hydrostatic confining pressure markedly affects the volume of certain rocks when they are returned to one atmosphere. For example, rocks with a porosity greater than a few percent generally show a crush-up or decrease in volume after an excursion to high pressure. Rocks with a low initial porosity generally increase their volume after high pressurization. La Mori (Figure 1) has shown the effects of cyclic pressure on a dense granitic-type rock markedly affect the low pressure volume after each cycle to high pressures. These experiments suggest that a detailed examination at lower pressures would be quite valuable. From a purely practical point of view, rocks which are being excavated by mechanical or explosive means generally are cycled to some pressure regime above ambient before they are removed from the rock mass. Thus, an understanding of how cyclic loading changes the elastic constants of rocks may be useful in either evaluating the effects of certain drilling techniques or devising a superior drilling technique.

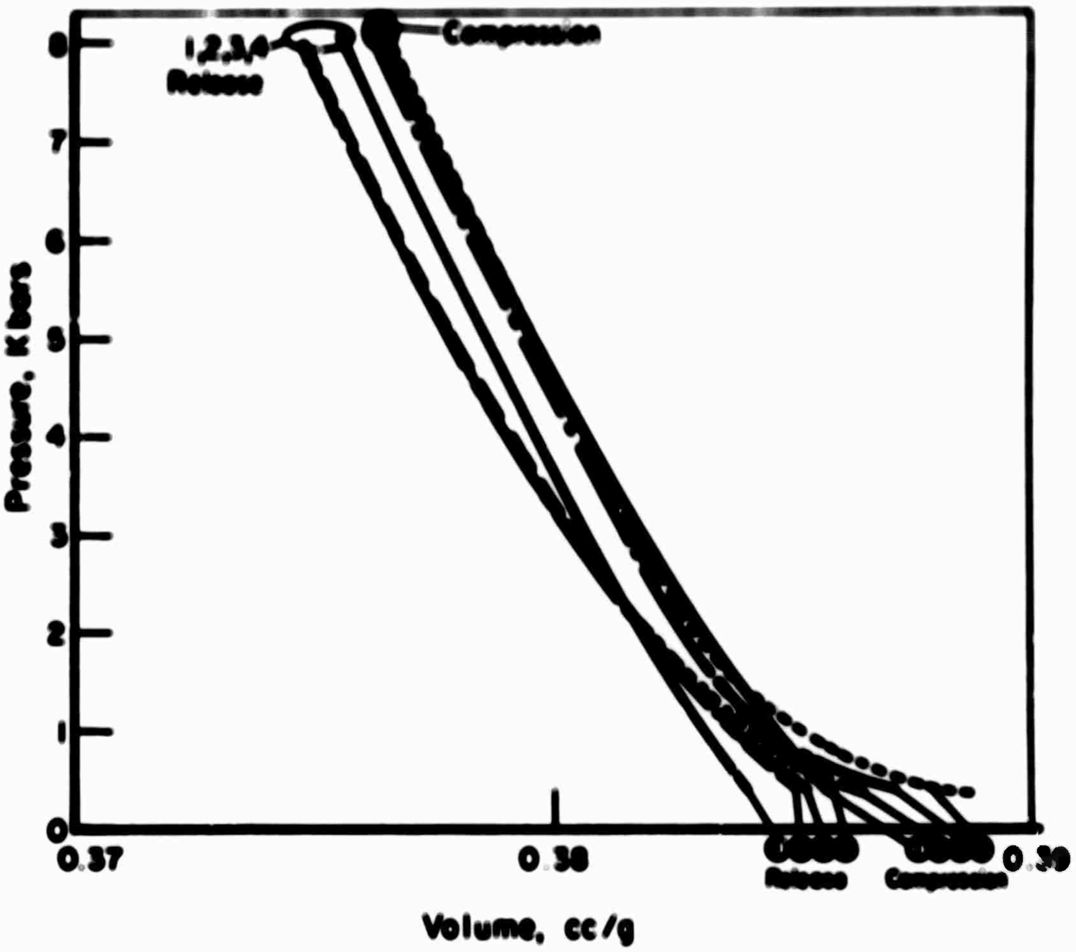


FIGURE 1. LOW-PRESSURE CYCLING DATA FOR DRY LOW-POROSITY GRANITIC ROCK

This program was initiated with the intent of studying three different rocks of widely different porosities and elastic properties which could be expected to be found in medium-to-hard rock excavation problems. Because of the exploratory nature of the program, rocks with idealized chemistry and fabric were selected. Salem limestone which is composed of approximately 100% calcite shell fragments and has a porosity of approximately 10% but has a fairly high compressive strength of 10,000 psi was chosen as a representative medium material. Berea sandstone was chosen as a harder material because of the quartz mineral content and a material which had a high porosity of 18%. Westerly granite was chosen as a representative of very hard, dense, and low porosity rock. This report describes results of tests on Salem limestone and Berea sandstone.

Experimental

The elastic constants of the rocks are measured by two techniques. One technique, referred to as the static or volume method, uses strain gages mounted on the sides of the cylindrical sample. The other technique, referred to as the dynamic or acoustic velocity method, determines the transit time of acoustic waves through the sample. The experiments are made in a standard hydrostatic pressure system with a maximum pressure-generating capability of approximately 9 kb.

Volume Method

In this technique the 25.4 mm diameter x 40.0 mm-long samples are enclosed in an 0.2 mm-thick copper jacket. The virgin samples are placed in the hydrostatic pressure environment and pressurized to about 100 bars. This is necessary in order to test the integrity of the copper jacket as well as to shrink the jacket onto the sample. If the copper jacket does not hold, the fluid will seep into the pores of the rock (detected by noticing a change in the weight of the rock plus the jacket). While this pressurization results in some uncertainty in the very low pressure compressibility measurements, it is required for the present technique.

In the strain gage part of the experiments, two perpendicular strain gages are mounted on the copper-jacketed rock samples (see Figures 2, 3, and 4). By mounting the strain gages axially and circumferentially around the sample, it is possible to measure the linear compressibility in mutually perpendicular directions and thus determine the difference in compressibility of the sample in different directions. Ideally, this should be done on three mutually perpendicular samples but for the present experiments this has not been done.

Two sizes of strain gages have been used: 6.2 mm square and 12.7 mm square. Because of the small grain size and the quality of the specimens used in this research, either one of these strain gages should statistically sample the strain of the rock (neglecting void spots which can appear under the gages randomly). These void spaces are a result of the high porosity in the limestone and sandstone. The experiments to date show that the void spaces under the strain gages do not have a noticeable effect on the accuracy of the experiments except when the gage breaks due to a large amount of strain across the corner of the indentation. Since these gages break anyway, they have no influence on the final results. Gage failure on the limestone and sandstone is approximately 25% but this is unavoidable.

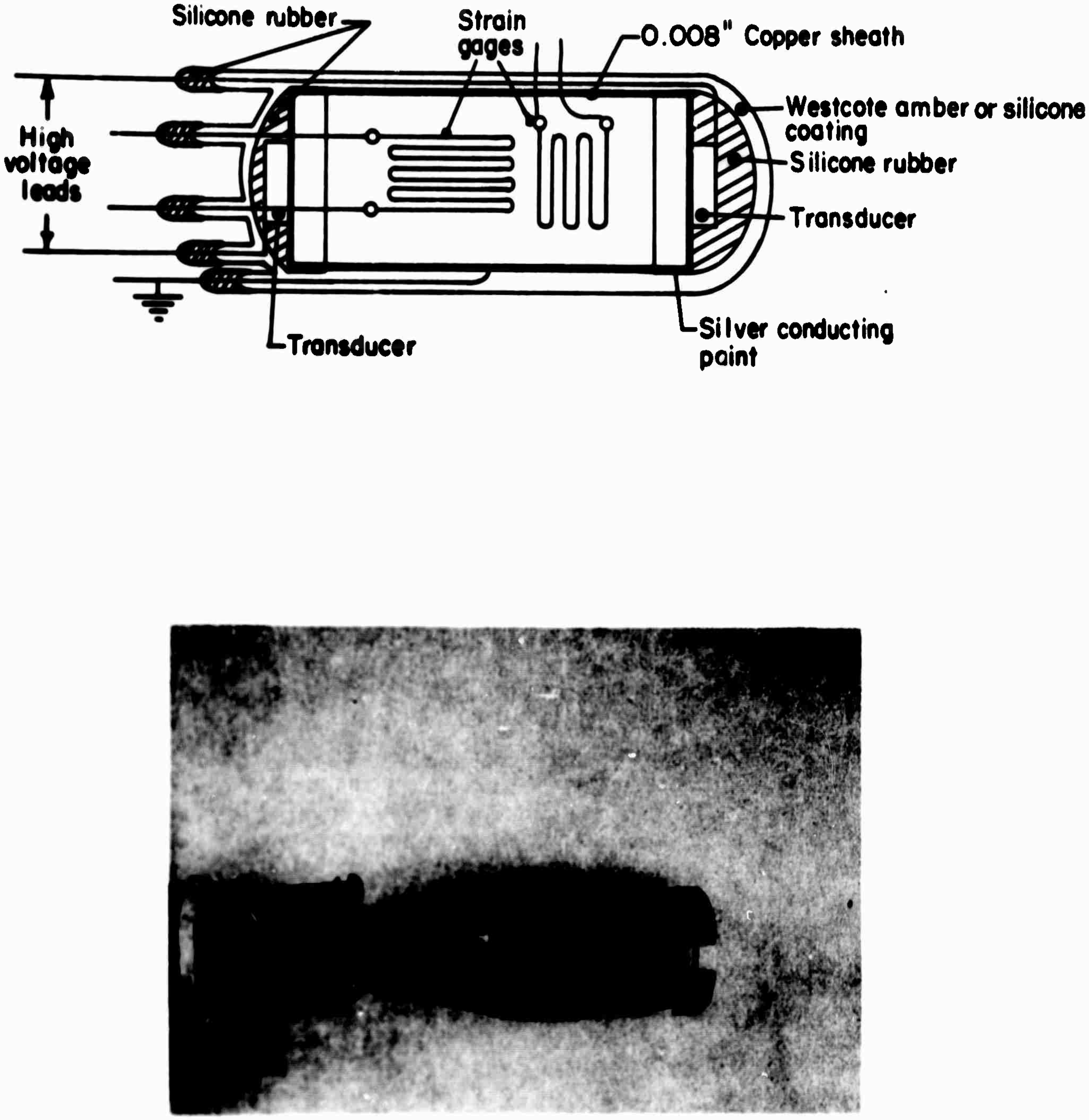


FIGURE 2. OLD SPECIMEN ASSEMBLY FOR ACOUSTIC-VELOCITY AND VOLUME-STRAIN MEASUREMENTS

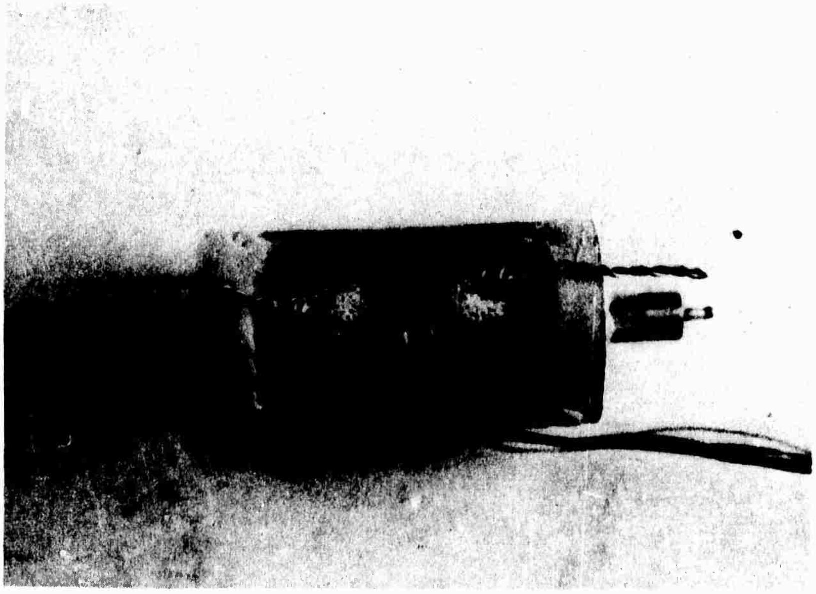


FIGURE 3. SAMPLE ASSEMBLY FOR SIMULTANEOUS ULTRASONIC AND VOLUME MEASUREMENTS ON ROCKS

Polysulfide rubber end seals and seal along copper seam are shown. Braided wire is the ground; round pieces from both ends are the copper electrodes. Radial strain gage is shown. Pockmarks are due to porosity in sample.

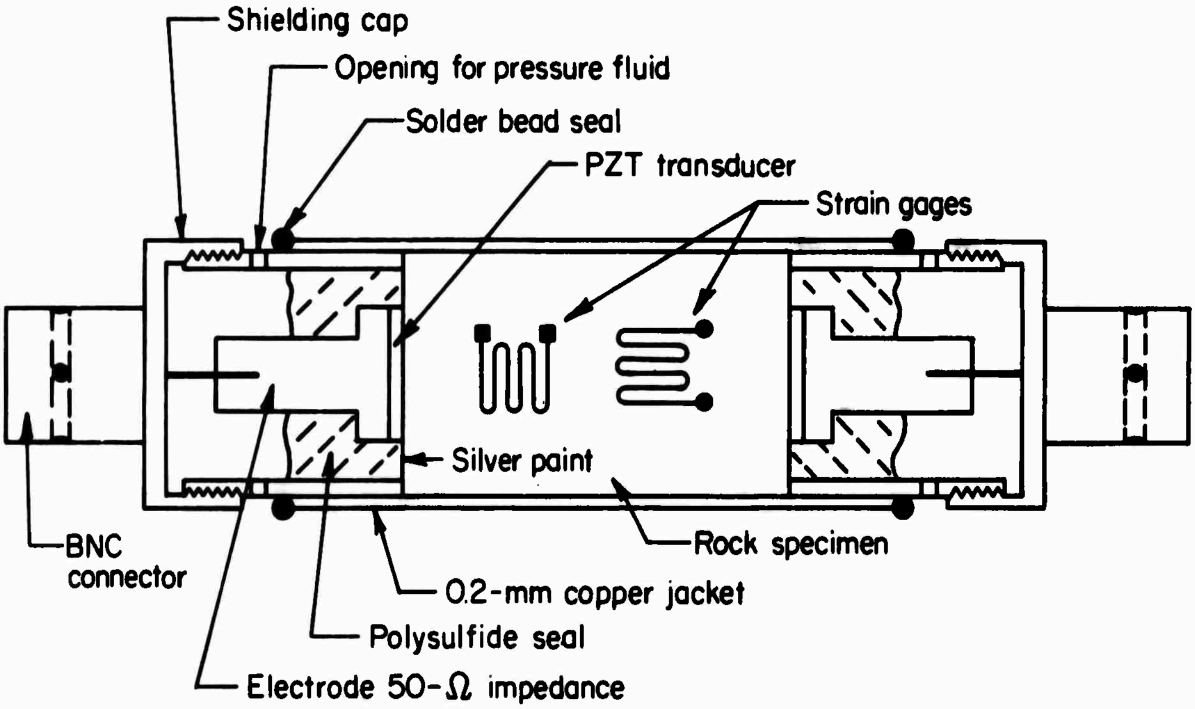


FIGURE 4. SAMPLE ASSEMBLY FOR ULTRASONIC AND VOLUME HIGH PRESSURE EXPERIMENT WITH NO ELECTRIC PICKUP

For these experiments it is necessary to measure the compressibility of the rocks in relation to the compressibility of a known standard, in this case iron. This is necessary in order to correct for the effects of pressure on the gage factor of the strain gages, the effect of the mounting material, and the effect of curvature on the circumferential strain gage. As first pointed out by La Mori(6), this last effect is important. The correction factors for the two gages are: $\frac{\Delta L}{L_0}$ axial strain gage = $- 2.34 \times 10^{-7}$ per bar and the circumferential gage has a value of -3.10×10^{-7} per bar plus 5.41×10^{-12} per bar^2.

Velocity Method

Measurement of acoustic velocity in rock specimens at pressure was first described in detail by Birch(2). In his method, the transit time of sound waves through the specimen was measured. A similar signal was passed through a material of known velocity (delay line) at one atmosphere and the travel time between the two was matched. The velocity of sound in the specimen was calculated from the known velocity of sound in the delay line, the distance of travel in the delay line, and the distance of travel in the specimen.

A similar technique is used in this experiment (see Figure 5). The high-powered pulse oscillator and the oscilloscope are triggered externally. The signal from the pulsed oscillator is sent to one transducer of the specimen and the oscilloscope. The received signal is amplified and sent to the other preamplifier of the oscilloscope. The time-mark generator provides a time base through a second channel on one of the preamplifiers of the oscilloscope. Thus, three traces are observed on the oscilloscope, the oscillator signal, the received signal, and the time-mark pulses (Figure 6).

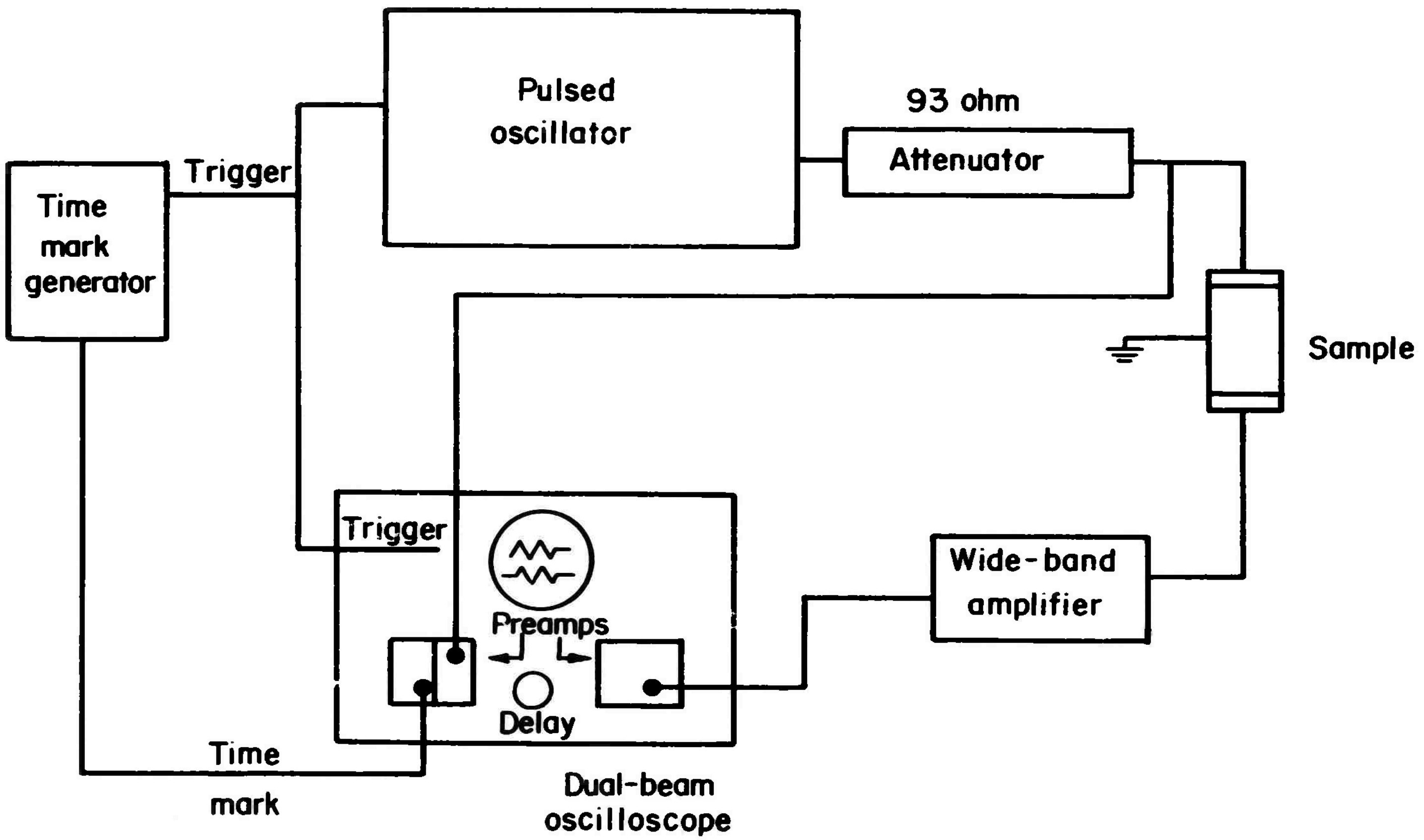


FIGURE 5. SCHEMATIC OF ULTRASONIC MEASUREMENT

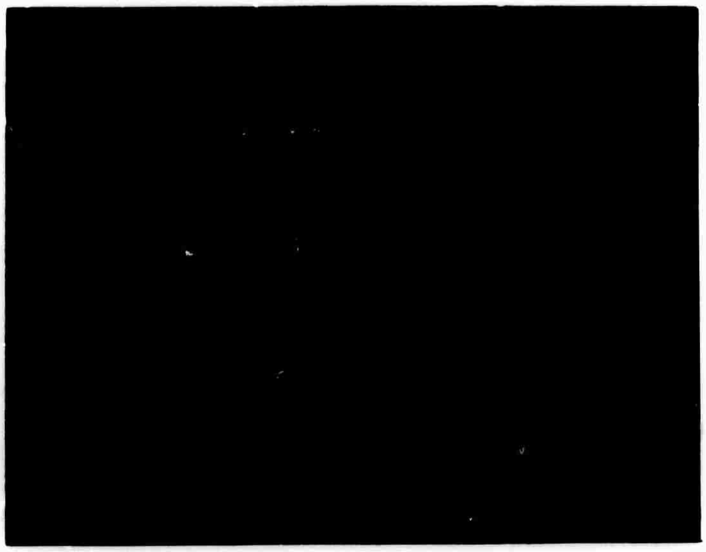


FIGURE 6. OSCILLOSCOPE TRACES OF ULTRASONIC EXPERIMENT IN SALEM LIMESTONE (PRESSURE = 600 BARS)

Top Trace - Initial Pulse
Middle Trace - Received Signal
Bottom Trace - 1 μsec Time Marks

The pulsed oscillator signal also causes an electric signal due to pick-up in the wires that go through the high-pressure head. This electric signal also travels through the wide-band amplifier ; its delay for a sample of zero length has been measured. By using the internal delay in the oscilloscope and the timing marks, we measured the delay difference between the electronic signal and the received pulse to obtain the travel time in this specimen. The delay line in the oscilloscope is only used to divide up the difference between two one-microsecond timing marks. Thus, a few percent error in the oscilloscope delay line results in only a few tenths of a percent error in velocity. Since the velocities determined by travel times are generally accurate to 1%, any error resulting from the oscilloscope delay line is unimportant. A 1% accuracy in velocity leads to accuracies of approximately 2, 6, 8, and 10% in shear modulus, bulk modulus, Poisson's ratio, and Young's modulus, respectively.

Figure 2 shows a schematic of our initial sample configuration. We had used this configuration previously but found it unsatisfactory for the present experiments for two reasons. One reason was that the Westcoat amber had to be applied at 350°F and this was deemed not desirable. Another reason is that both the amber and the silicone react with the pressure fluid over the longer times of the present experiments. A considerable effort was necessary to obtain a more suitable potting material. Figure 3 shows the present sample configuration.

In this configuration we have done away with the complete casting of the copper-jacketed sampled in a plastic material. We have also found that a polysulfide rubber, RTV, material (Eccobond) to be satisfactory in all requirements. The material also has the advantage of having a 900 psi tensile strength so that it transmits hydrostatic pressure quite readily. A special feature of this sample design is the electrodes which have been designed with a fifty ohms impedance to the outside copper jacket. This design materially reduces the electronic pick-up at the sample.

Of special interest is the sample assembly in Figure 4. The sample assembly shown here has no electronic pick-up at the frequencies (one Mega Hertz) of the experiment. At one atmosphere this assembly has given observable pulse-echo signals in Salem limestone. We are, however, still faced with the problem of electric pick-up through the high-pressure leads into the pressure vessel. Until this problem is solved, we will be unable to benefit from using the sample assembly in Figure 4 in the experiments. Thus, the assembly that we use (Figure 3) is the most convenient variant of sample assembly shown in Figure 4.

We are quite excited about this development and experimental technique for it could allow us to make acoustic shear and compression measurements on the same sample in the high-pressure environment. We have, therefore, designed a new high-pressure closure assembly which hopefully will remove any electric pick-up in the sending and received signals. This will be tested out in the remaining period of the contract.

Coupling of the shear-wave transducer to the sample is somewhat of a problem because of the high-pressure environment. Usual coupling by means of cementing the transducer to the specimen often results in a broken transducer after excursion to high pressure. While this generally gives the desired velocity measurements, it is undesirable because of the continued breakage of transducers. We have experimented with several materials and following a suggestion by Schock(8) have found that high viscosity polystyrene resins available from several manufacturers will transmit the shear wave satisfactorily and still not cause transducer failure in the high-pressure environment. Resins with viscosity of approximately 10,000 centipoises have worked satisfactorily.

Results

A large number of partially successful experiments were made during the modifications and advancements in the experimental technique discussed in the above section. These data are in agreement with the more successful experiments obtained from using the sample configuration shown in Figure 3. Results for the compressibility and velocity experiments in Salem limestone and Berea sandstone are given in Figures 7 through 15. These results are plotted in terms of linear strain versus pressure and velocity versus pressure. The final report will present the data in terms of the elastic constants also. However, significant conclusions can be drawn from the data as presented here.

Salem Limestone

The Salem limestone has been cycled to pressures of 1, 5, and 7 kb. In the 1 kb cycle (Figure 7) the limestone acts in a fairly normal manner. The curvature of the compressibility curves are concave upward with the exception of the very highest pressure on the radial strain gage. The axi 1 strain gage shows a slight reverse hysteresis which results in a lengthening of the sample on reduction to zero pressure. This effect is no doubt due to the anisotropy in the sample as the axis is perpendicular to the bedding plane. The radial strain gage shows a slight permanent decrease in radius.

With the exception of the 1 kb datum point, the velocity curve looks quite natural and similar to those reported in literature for other

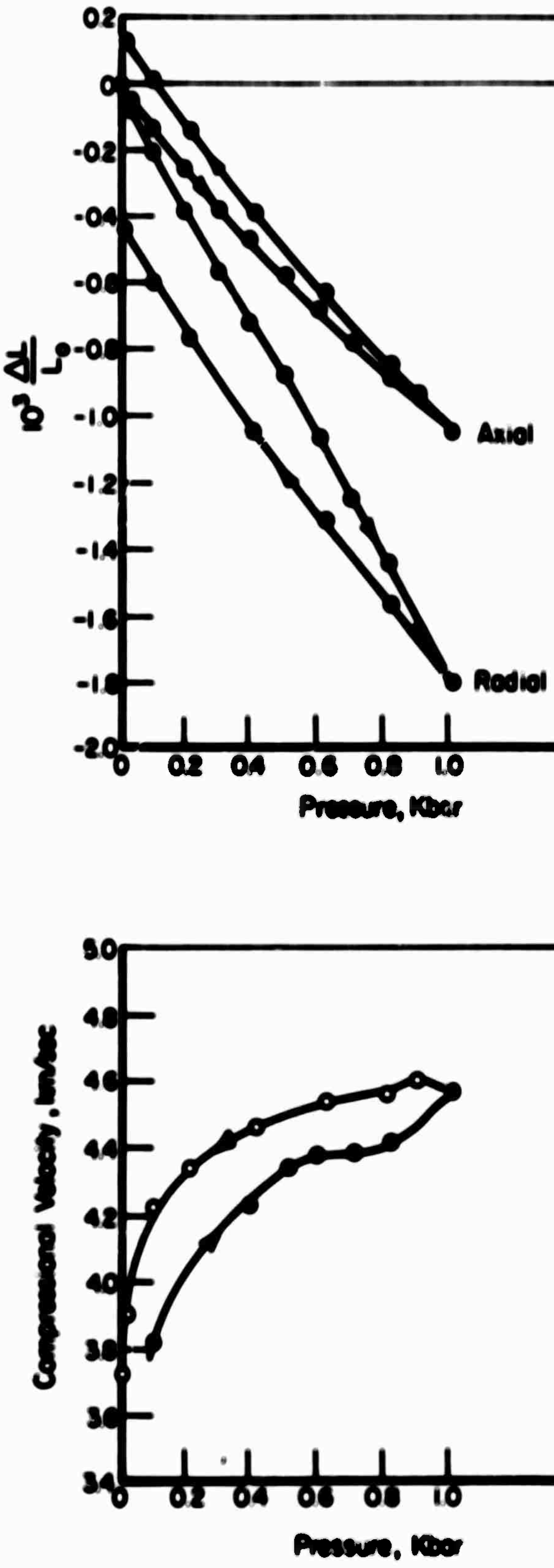


FIGURE 7. SALEM LIMESTONE, DRY (1-KBAR CYCLE)

rocks. There is the large increase in velocity at low pressures due to closing of thin cracks and a positive hysteresis on release of pressure. Understanding of the 1 kb velocity point was not possible until several other experiments and cycles to higher pressure had been made.

Figure 8 shows the 5 kb cycles of Salem limestone. The pressure increasing curve reproduces the decreasing curve of the previous 1 kb cycle within experimental error. The compressibility curves show a rather large increase in compressibility at pressures above 1.5 kb; this suggests that the similar phenomena on the radial gage in the 1 kb cycle was a forerunner of this.

The velocity curve shows essentially a small increase of velocity with pressure except that a bump is noted above 2 kb. The shear wave velocity data which were obtained after this reporting period show a similar effect to the compressional velocity. This means that the volume bulk modulus has decreased markedly while the velocity modulus has remained approximately the same.

The large increase in volume compressibility is caused by crush-up or the crushing mode of failure of the limestone into the pore spaces. Experiments with the polysulfide rubber sealed capsules result in the rubber end pieces in Figure 3 being stretched out after excursion to high pressure due to the air squeezed out from the collapsed pore spaces.

The release curves of both the velocity and volume experiments are markedly different from the compression curve. We see on the volume curve that we have approximately 1.1% axial permanent strain and 1.3% radial permanent strain for a total of permanent volume strain of about 3.7%. The velocity curve suggests rather significant changes in the structure of the sample.

Figure 9 is the 7 kb cycle of the Salem limestone. The compression curve faithfully reproduces the decompression curve of the 5 kb release cycle

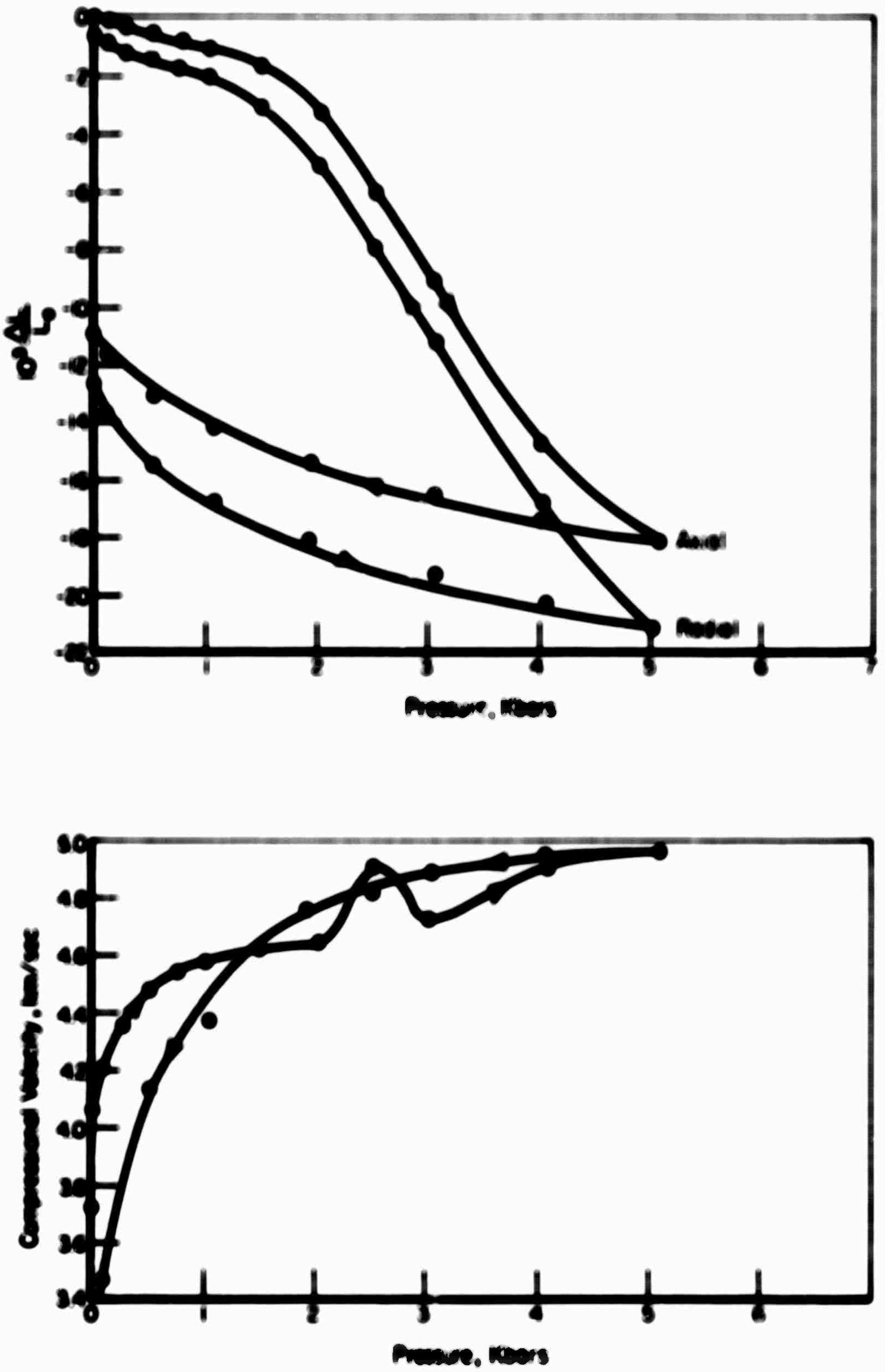


FIGURE 8. SALEM LIMESTONE, DRY (5-KBAR CYCLE)

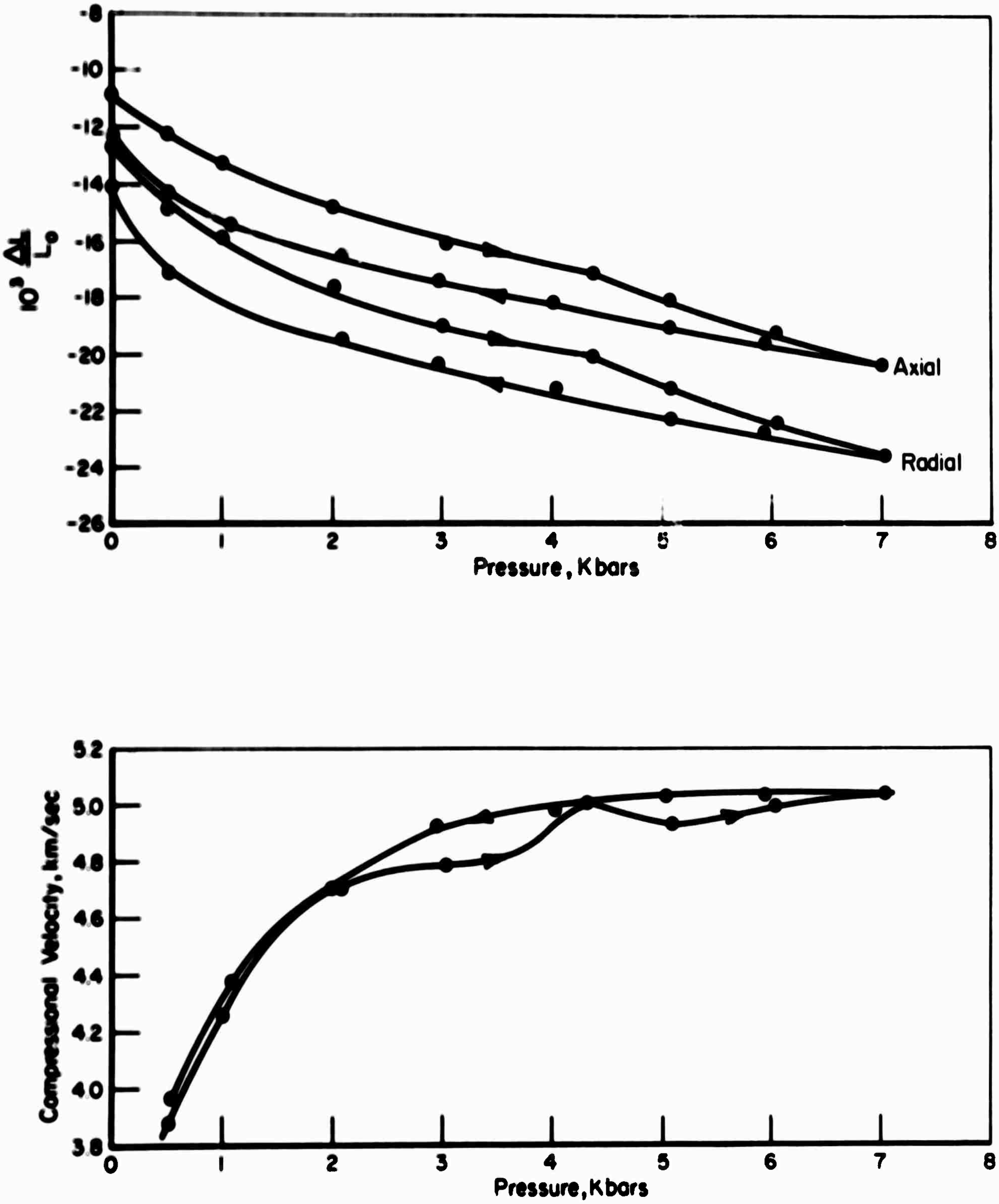


FIGURE 9. SALEM LIMESTONE, DRY (7-KBAR CYCLE)

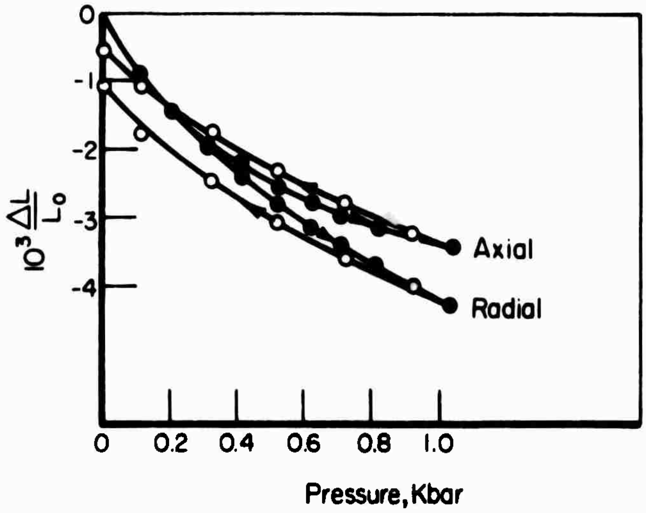
and also shows an increasing compressibility above 5 kb. This increasing compressibility is less than shown in the 5 kb cycle and indicates that many of the pores have now closed and little void space remains. The release volume curve indicates slightly more permanent strain in the sample. The velocity curve shows a similar rise and decrease in pressure near the start of the crushing mode of failure as was noticed in the 1 and 5 kb cycles.

Figures 14 and 15 show some preliminary measurements on water saturated Salem limestone. These data show much less permanent volume strain than dry sample showed and the velocity measurements show little of the structure that existed for the dry samples.

Berea Sandstone

Figure 10 shows the 1 kb cycle for the Berea sandstone. The volume measurements are quite normal with concave upward curves and generally normal hysteresis. A slight amount of permanent deformation is noted in the excursion to 1 kb and release to 0 kb. The velocity experiment is also quite normal with a large increase in velocity at low pressures due to squeezing out of small thin cracks and an abrupt change in slope near 1 kb. The velocity data at 0 and 0.1 kb may be the result of the previous prepressing to 0.1 kb in order to fix copper jacket to the sample.

Figure 11 gives results for the 5 kb cycle. Here interesting phenomena similar to that noticed in the Salem limestone are beginning to develop but at approximately 3 kb higher pressure. There is an abrupt increase in compressibility of the sample at approximately 4.5 kb as the sandstone undergoes the crushing mode of failure. Pressure release from 5.5 kb gives a completely different release than the limestone curve which indicates that this material is reacting in a manner considerably <u>different</u> than the limestone. The limestone



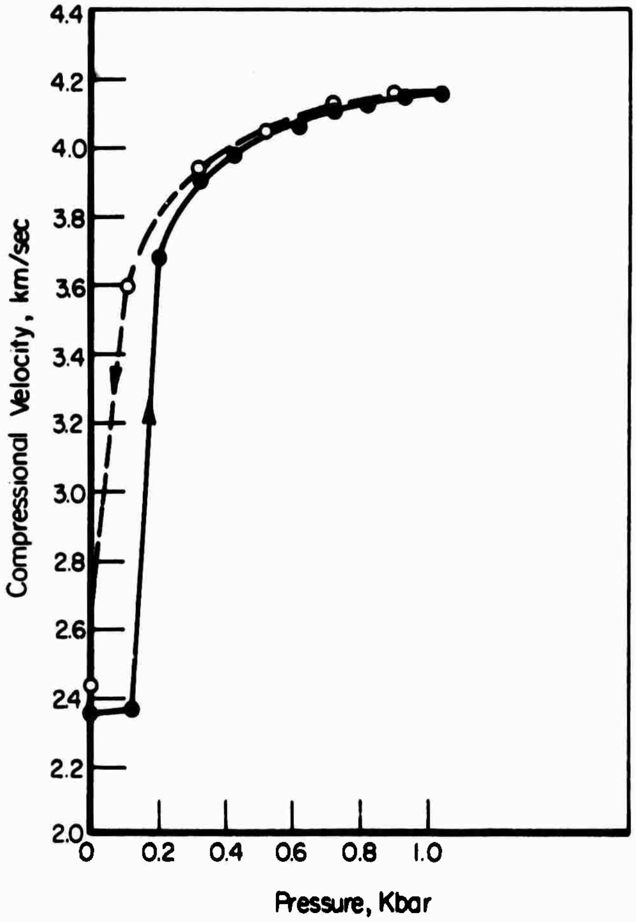


FIGURE 10. BEREA SANDSTONE, DRY (1-KBAR CYCLE)

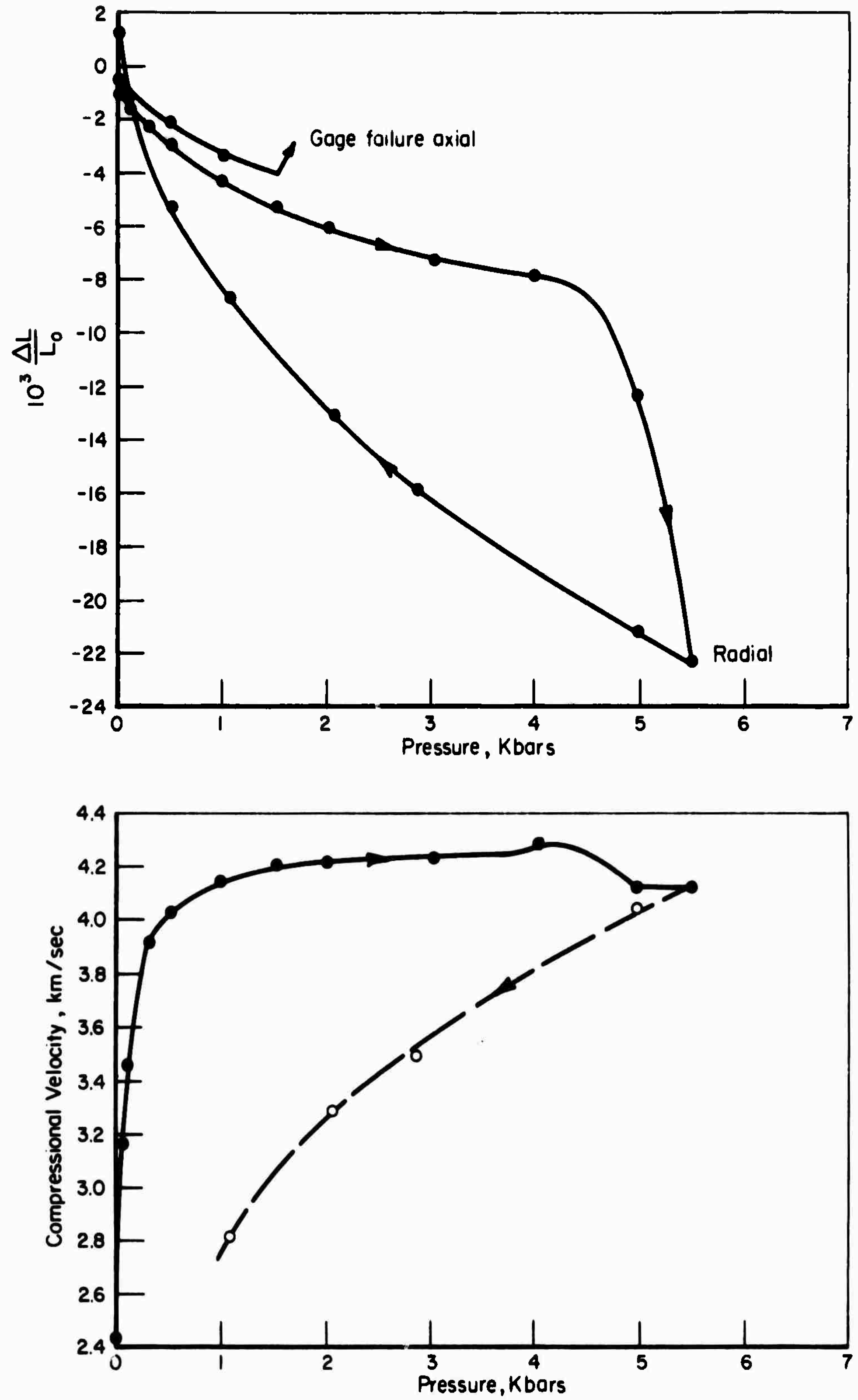


FIGURE 11. BEREA SANDSTONE, DRY (5-KBAR CYCLE)

showed permanent negative deformation, that is the sample decreased in volume. We see in this case that the sandstone actually increases in volume as we release the pressure to zero.

Also shown on this plot is what happens when a strain gage fails. The actual gage failed between the 1.5 and 2.0 kb measurements. The measurements continue in a normal manner and then generally an extremely high resistance or open circuit is noticed. The examination of the gage and the pockmarks on the sample after the experiment showed that the gage broke over the sharp edge of the depression. The axis of the sample was perpendicular to the bedding plane. Therefore, the strain gage broke across the bedding plane. Observation of the samples after the experiment shows that the edges of the depressions are a much sharper parallel in the bedding plane than at right angles to it. All the failures of the strain gages in the sandstone samples occur in this axial mode of failure. The radial strain gages have their wires parallel to the bedding plane and therefore they do not cross the very sharp bends.

The velocity curve for the 5 kb cycles starts out in a very normal manner. We observe, however, a similar bump or knee in the velocity curve associated with the onset of the crushing mode of failure found in the limestone. The velocity release curve is remarkably different from that of the compression curve but is similar to that found in the limestone for the 7 kb cycle. This suggests that crushing the sample has produced large changes in acoustic transmission ability.

Figures 12 and 13 show the 8 kb cycle for the sandstone. Here the crush-up is much less than in the previous 5 kb cycle. The release curve shows some permanent strain quite different from that on the previous cycle. At the present time this has not been examined in sufficient detail for an explanation. The velocity curve shows a small bump in the compression cycle near the crush-up region shown on the volume compression curves.

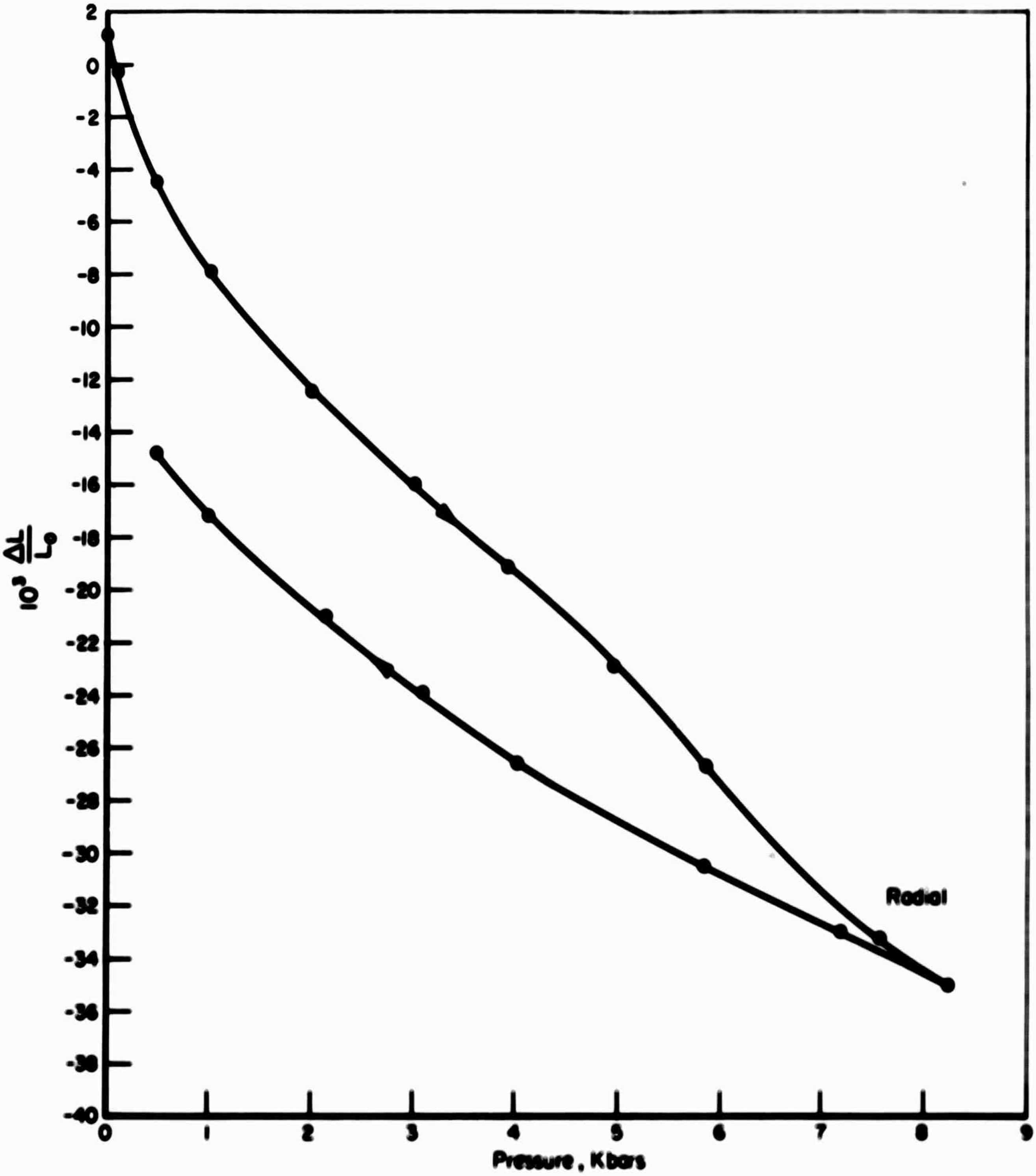


FIGURE 12. BEREA SANDSTONE, DRY (8-KBAR CYCLE)

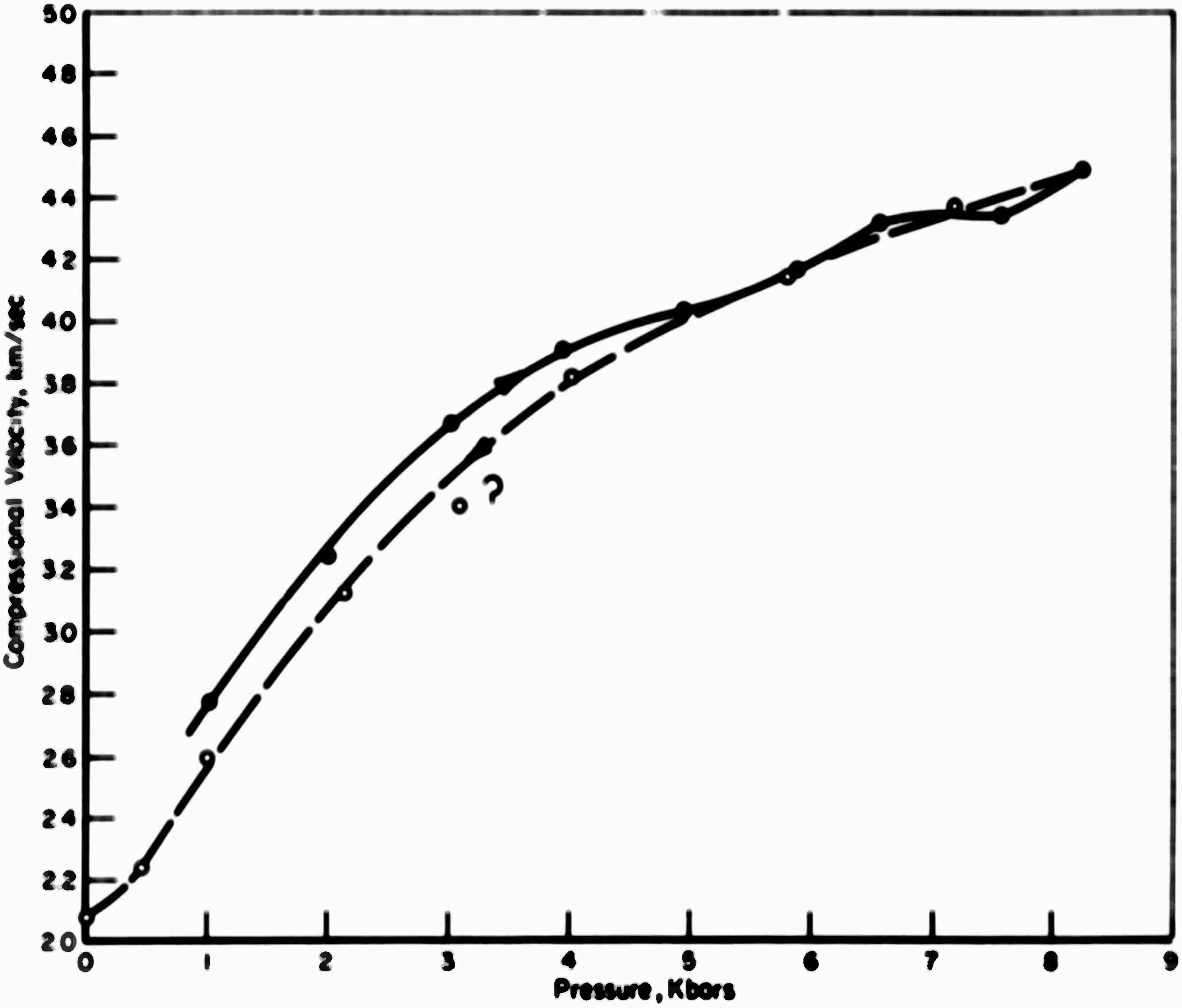


FIGURE 13. BEREA SANDSTONE, DRY (8-KBAR CYCLE)

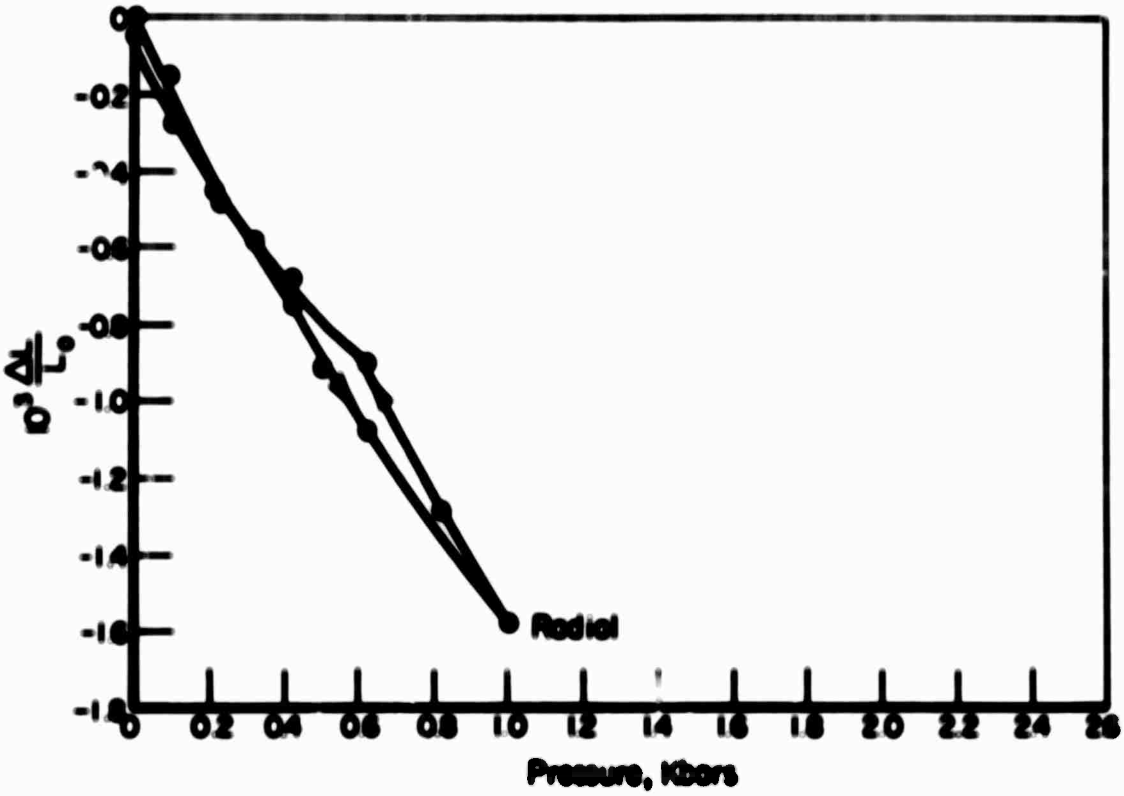


FIGURE 14. SALEM LIMESTONE, WATER-SATURATED (1-KBAR CYCLE)

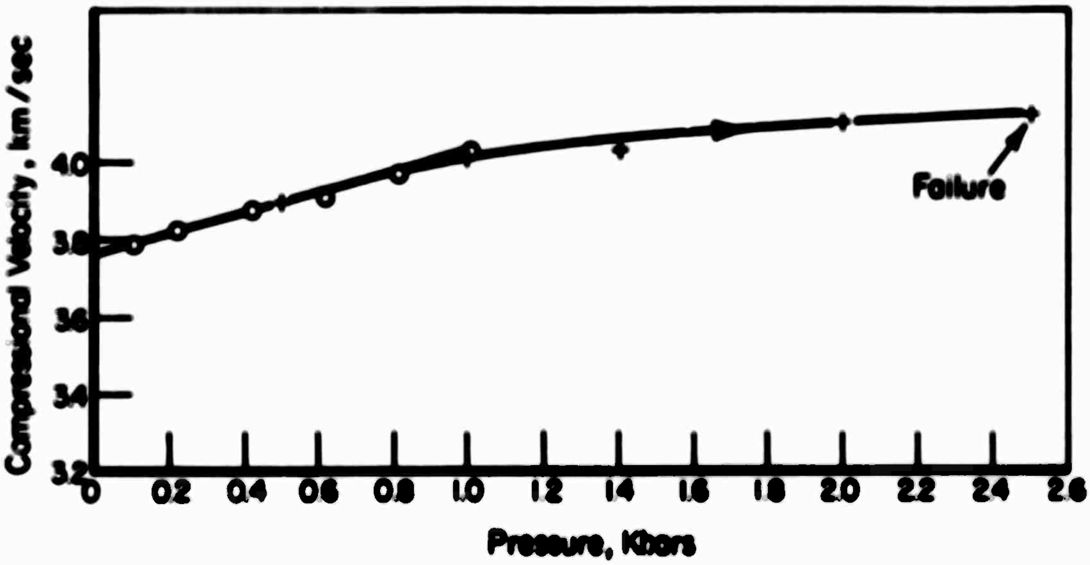


FIGURE 15. SALEM LIMESTONE, WATER SATURATED (1-KBAR AND 3-KBAR CYCLES)

Shear velocity experiments have been made on Berea sandstone after the end of the six month reporting period. These measurements indicate similar velocity features as found in the compressibility experiments.

Discussion

Both of these samples have a large porosity and with sufficiently high pressure develop a crush-up or crushing mode of failure. Both samples show a rather large decrease in bulk modulus as determined by volume techniques during the crush-up occurrence. However, the modulus as determined by velocity experiment remains essentially the same during the crush-up. The remarkable appearance of a knee in the velocity versus pressure curves, which apparently signifies the beginning of the volume crush-up phenomena, appears in all the curves. A similar effect has been noted in other published work of velocity versus pressure curves. However, this was ascribed to unknown causes. To our knowledge, this is the first time that it has been shown to be related to the crush-up in the sample. This evaluation is possible because of the simultaneous measuring of velocity and linear strain of the sample.

These experiments show that of the two extreme crack geometries, i.e., spheres and thin penny-shaped cracks, that the volume compressibility experiments are sensitive to the spherical cracks and insensitive to the thin cracks. Just the opposite occurs for the velocity experiments. At low pressures the thin penny-shaped cracks affect the total acoustic modulus of the sample very significantly. Once they are closed the acoustic transmission path remains relatively unchanged in properties except for small percentage increase due to the decrease in porosity. However, the volume modulus does appear to be very insensitive to the closure of these thin cracks but shows significant decrease when the compressibility increases due to the crushing of the pores.

These experiments show that as the cycling and crush-up continue more and more cracks are produced, many of these of the thin variety. Therefore, after each pressure excursion the average acoustic modulus of the sample increases with difficulty on pressure increase.

These results suggest that the use of an acoustic velocity technique to measure elastic properties in advanced mining systems may not give useful indications of the elastic properties of the rocks. The data show that at low pressures in these dry rocks the acoustic velocity is almost entirely dependent upon the location, orientation and the amount of small thin cracks. We would expect that a volume type experiment would more nearly relate the elastic properties of the rock to that encountered by the drill bit. These results also suggest that the relationships between velocity and mineral fabric in a rock may be more complex than a direct relationship.

Future studies on these materials will include some triaxial experiments to measure the volume elastic constants at higher confining pressures and thus obtain all the volume elastic constants. However, because of the pressure limitation of the present apparatus we have been unable to completely compress the sample and remove all of the porosity. Therefore, we plan to make an additional experiment to 15 to 20 kb on each sample in our piston-cylinder apparatus. This will obtain a complete volume pressure curve which extends through the crushing mode of failure.

Petrographic Studies

Detailed petrographic analysis of the specimens has not as yet been made. Thin sections have been obtained and have been studied to some extent. Figures 16 and 17 show the thin sections under crossed nicols for the limestone and the sandstone both before and after pressure cycling. Figure 16a,

50X

a. Crossed-Nicols, Unpressurized

50X

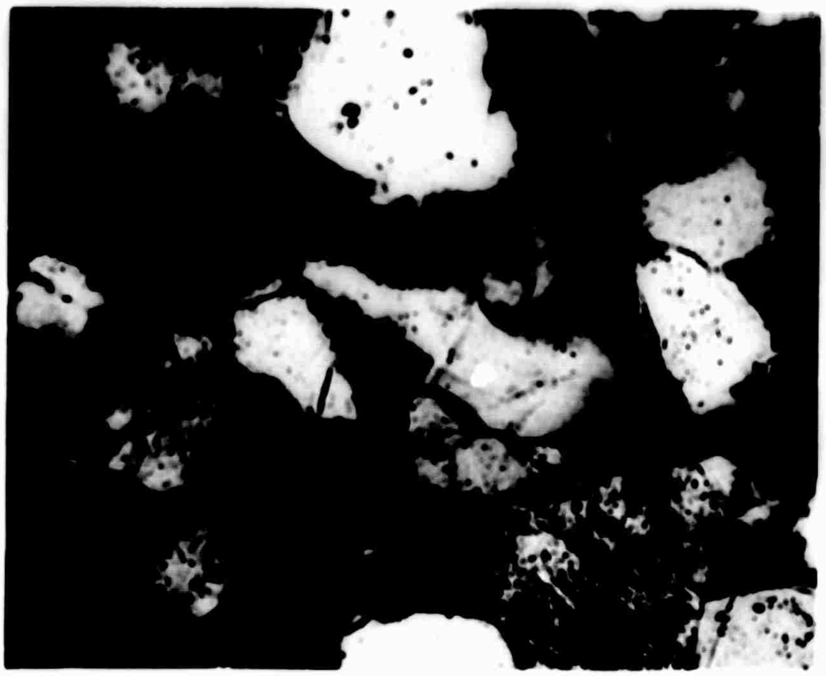
b. Crossed-Nicols, 7 Kb

FIGURE 16. SALEM LIMESTONE - THIN SECTIONS

NOT REPRODUCIBLE

150X

a. Crossed-Nicols, Unpressurized



155X

b. Crossed-Nicols, 8 Kb

FIGURE 17. BEREA SANDSTONE - THIN SECTIONS

the Salem limestone, shows many dark regions. These regions correspond to void spaces in the thin section or porosity in the sample. We see that in Figure 16b the porosity is much less after pressurization. Many of the grains of this sample are made up of very fine polycrystalline fragments which are too small to be examined as single crystals in the optical microscope. However, in many cases, some of these fragments are really mosaics of crystals of slightly different orientations. In the unpressurized sample we notice certain regions of typical calcite cleavage cracks. This is widespread throughout the unpressurized samples. These cleavages are almost totally destroyed in the pressurized samples and only very fine thin shadows remain to indicate their former presence.

Most of the dark areas in Figure 17a indicate pore spaces in the Berea sandstone. Certain of the quartz grains are at or near extinction and a few of the dark spots indicate iron-oxide. The large quartz grains are relatively strain free, some show undulose extinction, but few show cracking. The individual quartz grains are also separated to a great extent in the unpressed state.

Figure 17b shows a section of quartz taken after pressure of 8 kb. Here the quartz grains are in intimate contact and detailed petrographic examination indicates that none of the black areas shown are caused by voids between the grains. Quartz grains are pushed very close together and many have been cracked and fractured into hundreds of smaller pieces. In the center of this figure note a pointed grain pushing into another grain with cracks extending from the tip. The sections show considerably more undulose extinction of the quartz grains than the virgin sample as well as a large amount of cracking and what appears to be deformation lamella. On removal from the copper jacket, the sandstone almost crumbles apart. This occurs in spite of the fact that the quartz grains are pushed very close together. Apparently, the excursion to high pressure, even though it has crushed the grains and forced them together, has destroyed the cohesion of the sample.

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