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HIGH PRESSURE MECHANICAL PROPERTIES
OF INDIANA LIMESTONE

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HIGH PRESSURE MECHANICAL PROPERTIES OF INDIANA LIMESTONE

ABSTRACT

The pressure-volume relationship, shear strength at failure, and ultrasonic velocities were measured on dry, 50%-, and 100%-saturated Indiana limestone. Quasi-hydrostatic loadings to 3.9 GPa show volume changes of 23% and 15% for the dry and 100% saturated limestone, respectively. Several phase transitions were noted. Hydrostatic loading to 1.4 GPa produces peak compressions of 16%, 11%, and 7%, in the dry, 50%-, and 100%-saturated materials respectively. After unloading to atmospheric pressure, these materials show permanent volume changes of 10%, 7%, and 3%. Bulk moduli are shown to be complicated functions of pressure and water content. Failure envelopes for all three saturations have been determined in compression to 700 MPa. At pressures <25 MPa brittle fracture is universal; plastic flow dominates at all higher pressures. The failure envelope for the dry limestone, although increasing with pressure, possesses a monotonically decreasing slope. Shear stresses range from 10 to 260 MPa. The 50%-saturated limestone shows identical behavior up to 30 MPa confining pressure; at higher pressures, the shear stress increases only slightly. Complete saturation lowers the shear strength of the limestone to the 40- to 80-MPa region over the pressure range investigated. Shear and compressional velocities (1 MHz) were measured at atmospheric pressure over the entire saturation range. In the region from 9% to 100% saturation, V_p increased by 6% while V_s decreased 4%.

INTRODUCTION

The source characteristics of underground nuclear explosions as well as their detection in space and time can often be ambiguous or inaccurate without knowledge of the physical characteristics of the immediate surrounding rock materials. A systematic determination and evaluation of the equation-of-state (EOS) behavior of representative rocks together with theoretical and calculational programs is required in order to develop a predictive capability for detection. The Seismic Evasion program at the Lawrence Livermore Laboratory has had an ongoing effort in support of work in these three areas. During the past several years, many studies presenting the EOS behavior of common rock types have been reported.¹⁻⁴ The purpose of this report is to present similar results on another common rock, porous limestone.

We chose Indiana limestone (Upper Mississippian) quarried near Bedford, Indiana for our study. The measured initial interconnected porosity was $14.0 \pm 0.1\%$ and the

dry density was 2.29 g cm^{-3} . This limestone can be classified as a calcarenite or more closely as a spargenite; that is, a carbonate rock having a grain size ranging from 0.15 to 1 mm, containing <10% quartz, and made up of an intermediate mixture of oolites and microfossil debris cemented with calcite.^{5,6} Total calcium carbonate content of this rock ranges from 90 to 95%, the remaining portion being dolomite.⁶

Three types of EOS measurements were made on the dry, partially saturated, and fully saturated samples of Indiana limestone. These included hydrostatic pressure-volume determinations to 1.4 GPa and quasi-hydrostatic measurements to 3.9 GPa, uniaxial strength measurements at confining pressures ranging to 700 MPa, and ultrasonic velocity data taken at atmospheric pressure. These data are presented in the form of the volume compressions and the limiting failure strength at pressure along with the initial loading moduli. Our results then serve as input for calculational codes to predict the loading-unloading response of porous limestone rock as a function of water content.

The apparatus and experimental techniques used in these types of tests have been described in detail elsewhere.⁷⁻¹⁰ Briefly, the pressure-volume relation to 3.9 GPa was determined in a piston-cylinder device in which piston displacement is measured as a function of piston loading. The pressure transmitting medium is tin, and although the shear strength of this material is low, the stress distribution is nevertheless quasi-hydrostatic. Hydrostatic P-V measurements were made up to 1.4 GPa on strain-gaged, jacketed samples. The shear strength-mean pressure failure envelope was determined from uniaxial stress tests on jacketed samples compressed at a series of pressures ranging up to 700 MPa. In all cases, the axial loads were corrected for the strength of the plastic jacket. The acoustic velocities were determined only at atmospheric pressure by measuring the transit times of both compressional and shear waves at a center frequency of 1 MHz. In these latter measurements, water saturations ranged from 9 to 100%. All stresses are referred to a Cartesian coordinate system with σ_1 , σ_2 , σ_3 , being the maximum, intermediate, and least principal stress, respectively. Compression is taken as positive. Strain rates for the quasi-static, pressure-volume, and the uniaxial stress measurements ranged near 10^{-4} sec^{-1} . All test data were taken at 22-25°C.

EXPERIMENTAL RESULTS

Pressure-Volume

The pressure-volume relationships were determined for Indiana limestone with three different H_2O contents. The dry and 100% saturated limestone samples were compressed to 1.4 GPa hydrostatically and to 3.9 GPa quasi-hydrostatically. The 50% saturated limestone was tested only to 1.4 GPa hydrostatically. For the dry and 100% saturated limestone data, the pressure-volume relationship was constructed to 3.9 GPa from the hydrostatic test data at low pressures (<0.4 GPa) and from the quasi-hydrostatic

tests at high pressures (>1.4 GPa). In the intervening region (0.4 to 1.4 GPa), the hydrostatic curve and the quasi-hydrostatic curve were parallel, but offset slightly. Adjustment of the quasi-hydrostatic curve by $<0.01 V/V_0$ brought the two into good agreement in this region. This adjustment was required to compensate for the inherent inaccuracies stemming from the geometry of the quasi-hydrostatic test (i.e. piston fraction, non-zero shear strength of the confining pressure medium).

The loading and unloading curves to and from 1.4 GPa are shown in Fig. 1 for dry, 50%, and 100% saturated Indiana limestone. Upon initial loading (<0.1 GPa), the slope of the P vs $\Delta V/V_0$ curve is greatest for fully saturated, least for the dry, and intermediate for the 50% saturated material. We believe this to be a consequence of the water inhibiting crack closure. At about 0.1 GPa, in the dry and 50%-saturated limestone, the decrease in bulk modulus indicates the beginning of large-scale pore collapse. For the saturated limestone, the slope of the pressure-volume curve increases smoothly up to a pressure of about 1.15 GPa where there is a phase transition. At about 1.3 GPa this transition is completed and the bulk modulus again increases. Upon unloading along a nearly constant slope, no reversal of the phase transition can be seen until a pressure of about 0.4 GPa is reached. Further unloading to atmospheric pressure indicates a permanent pore collapse of about 3% that is probably the result of incomplete saturation of non-interconnected porosity. The loading-unloading behavior is very reproducible in detail; the curve for the saturated limestone is based on the average of 10 tests. The very large hysteresis (from 0.4-1.15 GPa) shown for this material is unusual and argues against the transition being due to the 980 MPa transition of water.

The loading curves shown in Fig. 1 for the dry (2 tests) and 50% saturated limestone (3 tests) agree well up to a pressure of about 0.4 GPa. At this point the air filled portion of the interconnected porosity has been removed in the 50% saturated limestone and the bulk modulus increases sharply in contrast to that of the dry limestone. Continued loading of the 50% saturated limestone shows that the phase transition observed at 1.15 GPa in the 100% saturated material is just starting at about 1.35 GPa. This phase transition evidently continues upon initial unloading from 1.4 GPa, as indicated by the positive slope of the unloading curve. Following this, the volume decrease is normal with a nearly constant slope down to a pressure of about 0.55 GPa. At this point a similar but less distinct version of the transition reversal observed for the fully saturated limestone occurs. In both cases the hysteresis loops are very similar in magnitude, but less distinct in the 50%-saturated material. When fully unloaded, the 50%-saturated limestone shows a permanent compaction of about 7%.

The dry Indiana limestone shows a normal loading behavior up to the highest pressure — that is, a smooth curve with a monotonically increasing slope from 0.2 to 1.4 GPa. The unloading behavior from 1.4 GPa is normal. Figure 1 shows a permanent volume change of about 10% after the loading-unloading cycle.

In Fig. 2, the continuation of the dry and 100%-saturated pressure-volume curves are shown up to 4.0 and 3.9 GPa, respectively. The two curves shown in this figure

Fig. 1. Hydrostatic P - V/V_0 loading-unloading curves for dry, 50%-saturated and 100%-saturated Indiana limestone.

Fig. 2. P - V/V_0 loading-unloading curves for dry and 100%-saturated Indiana limestone based on both hydrostatic and quasi-hydrostatic data.

are based on two and four tests, respectively. As noted above, the two loading curves up to 1.4 GPa are based on the data as shown in Fig. 1. The unloading curve for the dry limestone is based on the actual data upon unloading from 3.9 GPa. The missing portion of the unloading curve between 3.9 and 2.4 GPa is indicated by a dashed line.* In both the dry and fully saturated limestone, a second phase transition can be seen at about 1.8 to 2.0 GPa. The unloading curve for the dry limestone is smooth, with about 12% total permanent volume decrease after unloading from 3.9 GPa. Due to the complex nature of the unloading of the 100% saturated limestone from 4.0 GPa, it was not possible to report useful data in this region.

All of the various phase transitions shown in Figs. 1 and 2 appear to be reversible upon release of pressure, and thus we have no supporting evidence for the reactions involved in the individual transformations. In Table 1 we list several transitions known

Table 1. Observed phase transitions in the $\text{CaCO}_3\text{-H}_2\text{O}$ system at 25°C.

Pressure, GPa	Reaction	ΔV , %	Reference
0.6 ?	$\text{H}_2\text{O} + \text{CaCO}_3 \rightarrow \text{CaCO}_3 \cdot 6\text{H}_2\text{O}$	20.0	11,12
0.98	Water $\rightarrow$ Ice VI	7.0	14
1.40	Calcite I $\rightarrow$ Calcite II	0.4	13
1.80	Calcite II $\rightarrow$ Calcite III	2.6	13
2.23	Ice VI $\rightarrow$ Ice VII	5.7	14

to occur in the $\text{CaCO}_3\text{-H}_2\text{O}$ system over the pressure range investigated. Additional studies are now underway to correlate the observed P-V behavior with these (or other) transitions.

Values of the bulk modulus for the dry, 50%-, and 100%-saturated limestone have been calculated from Figs. 1 and 2 and plotted vs pressure in Fig. 3. Both loading and unloading data are shown, and as might be expected from inspection of the first two figures, the moduli are quite complicated functions of pressure. The difference between these loading-unloading curves is largely due to the observed hysteresis between loading and unloading, permanent volume decreases, and the kinetics of the various phase transformations. The closing and opening of cracks at low pressure, permanent compaction of gas-filled porosity, and phase transformations all may contribute to the complexity of any single curve. It is obvious that use of a constant bulk modulus or a bulk modulus that is a simple function of pressure, as is usually input to the calculational codes for prediction of rock response, would be incorrect in the case of the Indiana limestone.

* In the quasi-hydrostatic test, the uncertain piston friction upon initial unloading makes interpretation of the PV results difficult in this region.

Fig. 3. Bulk modulus vs pressure for dry, 50%-saturated, and 100%-saturated Indiana limestone (loading and unloading), based on Figs. 1 and 2.

Uniaxial Stress

The deformational behavior of dry, 50%-, and 100%-saturated Indiana limestone was determined from a series of uniaxial compression tests up to 700-MPa confining pressure. The cylindrical test samples were 19 mm in diam by 38 mm long and were plastic-jacketed to exclude the confining pressure fluid during deformation. The raw data were taken in the form of force-displacement traces, converted to differential stress-axial strain curves, and the strength and ductility characteristics correlated for each water content. Tensile strengths for each material type were also determined from the indirect tensile (Brazil) tests. Tensile data were taken on unjacketed samples fractured at atmospheric pressure only.

For all samples, failure from the Brazil tests was characterized by tensile fracture only on a single plane oriented normal to σ_3 . However in the compression tests, two distinct types of failure behavior were noted. At confining pressures (σ_3) $\leq$ 25 MPa, the tests were terminated when the limestone failed by through-going tensile and/or shear fractures at axial strains of $<2\%$. In these samples, the tensile planes were normal to σ_3 , the shear planes being oriented at about 30° to σ_1 . Cohesion of the samples from all Brazil tests as well as for those in compression ($\sigma_3 \leq 25$ MPa) was either lost or poor at best. Behavior in these cases is termed brittle. In tests conducted at confining pressures of 50 to 700 MPa, all samples from each test series could be deformed to at least 15% by either well-distributed micro-fracturing or plastic flow in the calcite with no loss in cohesion.^{15,16} This behavior is termed ductile. For purposes of correlation of strengths for each of the three saturations of this limestone, we arbitrarily placed the transition from brittle fracture to ductile flow at 3 to 5% permanent axial strain and report stresses at that strain. All strength and failure behavior properties are summarized in Tables 2 to 4.

Table 2. Indiana limestone, dry.

Test type	Principal stress, MPa			Shear strength, MPa	Mean pressure, MPa	Behavior
	σ_1	σ_2	σ_3	τ	P_m	
Brazil	16.5	0.1	-5.5	11.0	3.7	Brittle
Brazil	17.7	0.1	-5.9	11.8	3.9	Brittle
Brazil	14.1	0.1	-4.7	9.4	3.1	Brittle
Uniaxial stress	61.6	0.1	0.1	30.8	20.5	Brittle
Uniaxial stress	63.5	0.1	0.1	31.8	21.	Brittle
Uniaxial stress	124.	25.	25.	49.5	58.	Brittle
Uniaxial stress	173.	50.	50.	61.5	90.	Ductile
Uniaxial stress	271.	100.	100.	85.5	157.	Ductile
Uniaxial stress	458.	200.	200.	129.	286.	Ductile
Uniaxial stress	630.	300.	300.	156.	410.	Ductile
Uniaxial stress	763.	400.	400.	181.	521.	Ductile
Uniaxial stress	930.	500.	500.	215.	643.	Ductile
Uniaxial stress	1093.	600.	600.	246.5	764.	Ductile
Uniaxial stress	1223.	700.	700.	261.5	874.	Ductile

Table 3. Indiana limestone, 50% saturated.

Test type	Principal stress, MPa			Shear strength, MPa	Mean pressure, MPa	Behavior
	σ_1	σ_2	σ_3	τ	P_m	
Brazil	9.2	0.1	-3.1	6.2	2.1	Brittle
Brazil	7.5	0.1	-2.5	5.0	1.7	Brittle
Uniaxial stress	39.6	0.1	0.1	19.8	13.2	Brittle
Uniaxial stress	42.5	0.1	0.1	21.2	14.2	Brittle
Uniaxial stress	112.9	25.	25.	44.	54.3	Brittle
Uniaxial stress	168.3	50.	50.	59.	89.	Ductile
Uniaxial stress	271.	100.	100.	86.	157.	Ductile
Uniaxial stress	424.	200.	200.	112.	275.	Ductile
Uniaxial stress	592.	300.	300.	146.	397.	Ductile
Uniaxial stress	691.	400.	400.	146.	497.	Ductile
Uniaxial stress	795.	500.	500.	148.	598.	Ductile
Uniaxial stress	920.	600.	600.	160.	707.	Ductile
Uniaxial stress	1026.	700.	700.	163.	809.	Ductile

Table 4. Indiana limestone, 100% saturated.

Test type	Principal stress, MPa			Shear strength, MPa	Mean pressure, MPa	Behavior
	σ_1	σ_2	σ_3	τ	P_m	
Brazil	9.3	0.1	-3.1	6.2	2.1	Brittle
Brazil	9.3	0.1	-3.1	6.2	2.1	Brittle
Uniaxial stress	37.9	0.1	0.1	19.0	12.6	Brittle
Uniaxial stress	41.5	0.1	0.1	20.8	13.8	Brittle
Uniaxial stress	73.	25.	25.	24.	41.	Brittle
Uniaxial stress	137.	50.	50.	43.5	79.	Ductile
Uniaxial stress	187.	100.	100.	43.5	129.	Ductile
Uniaxial stress	203.	100.	100.	51.5	134.	Ductile
Uniaxial stress	295.	200.	200.	47.5	232.	Ductile
Uniaxial stress	413.	300.	300.	56.5	338.	Ductile
Uniaxial stress	508.	400.	400.	54.	436.	Ductile
Uniaxial stress	617.	500.	500.	58.5	539.	Ductile
Uniaxial stress	738.	600.	600.	69.	646.	Ductile
Uniaxial stress	853.	700.	700.	76.5	751.	Ductile

The failure strengths for each test as defined above are plotted in Fig. 4 for the dry, 50%-, and 100%-saturated Indiana limestone. Also shown is the approximate transition from brittle fracture to ductile flow for each material. The failure envelope

Fig. 4. Shear strength vs mean pressure for dry, 50%-saturated, and 100%-saturated Indiana limestone. Transition from brittle to ductile behavior is indicated.

drawn through the locus of failure strengths for the dry limestone is smooth and well behaved with a monotonically decreasing slope up to a mean pressure of at least 880 MPa. The envelope for the 50% saturated material is virtually identical up to 300 MPa ($P_m = 400$ MPa); at higher pressures the two envelopes diverge strongly, the shear stress for the 50%-saturated limestone increases only slightly up to 800 MPa. We interpret the inflection point near $P_m = 400$ MPa as due to the final crush-up of the initial 50% gas-filled porosity, after which the limestone is fully saturated. At mean pressures in excess of 400 MPa, the pore water pressure (P_p) is thought to be about equal to σ_3 , less the inherent strength of the limestone. Thus the effective confining pressure ($\sigma_3 - P_p$) would remain approximately constant at 300 MPa (2 τ at the inflection point in Fig. 4) for all pressures in excess of 400 MPa. Confirming evidence for this interpretation comes from the P-V behavior of the dry and 50%-saturated limestone shown in Fig. 1. It was pointed out above that the two P-V curves first begin to deviate at about 400 MPa and the 50%-saturated limestone showed a permanent volume decrease of about 7% (about one-half of the initial porosity) upon unloading.

The measured shear strengths defining the failure envelope for the 100%-saturated limestone are considerably lower than those for the dry and 50%-saturated materials and increase only slightly from 80 to 750 MPa mean pressure. Although the shear strengths for the 100%-saturated ($P_m > 80$ MPa) and 50%-saturated ($P_m > 400$ MPa) materials are different by a factor of 2 to 4, the slopes of the failure envelopes are nearly equal in this region. This difference in shear strength is believed to be due to work-hardening in the calcite during the elimination of the gas-filled porosity of the limestone. Ample evidence for work hardening in calcite and calcite-bearing rocks is available from the literature.^{8,15,17,18}

Again, at pressures in excess of approximately 100 MPa, for the fully saturated limestone, we infer that the effective pressure is about constant at 100 MPa. This is similar to, but less than the effective pressure for the 50%-saturated limestone. The difference between the two is likely related to work-hardening in the solid matrix (or absence thereof) for the two different conditions of saturation. The explanation advanced above for the nearly constant 200-MPa differential strength between the two failure envelopes also seems consistent with an identical offset between the 100%- and 50%-saturated P-V curves discussed above for Fig. 1.

The brittle-ductile transitions for the three saturations shown in Fig. 4 are only slightly dependent upon mean pressure; each occurs at 25- to 50-MPa confining pressure (Tables 2 to 4). In the brittle region, the degree of water saturation has little effect upon the shape or position of the failure envelope. Tables 2 to 4 show that the tensile strength of the Indiana limestone is nearly two-fold higher for the dry material than for either the 50%- or 100%-saturated limestone.

Ultrasonic Velocities for Various Water Contents

Ultrasonic velocities were determined by pulse transmission using $Pb(Zr,Ti)O_3$ transducers affixed to samples 19 mm in diam by about 25 mm long. All measurements

were made at atmospheric pressure after waiting at least 24 h for velocity equilibration at each water content. Such measurements have provided evidence of chemical effects on mechanical properties² and provide indications of characteristic microstructures.¹⁹

Velocity data are presented in Fig. 5. Even at the highest water contents obtained, unfilled voids are present since the P-V results (Fig. 1) suggest that approximately 3% of the porosity is unconnected. Compressional velocity (V_p) increases slightly (about 6.1%) up to apparent saturation. Such increases are usually associated with water saturation of microcracks.²⁰ Scatter in the V_p data may be due to non-uniform drying of the specimens. The slight decrease in V_p at apparent saturation is probably an experimental artifact. Most of the 4.1% decrease in shear velocity is due to the bulk density increase of the limestone at apparent saturation. A minor contribution to this decrease in V_s may be due to a lower effective shear modulus as a result of interaction between the solid matrix and the pore water.

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Fig. 5. Compressional and shear wave velocities vs pore water content for Indiana limestone. All data taken at atmospheric pressure.

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