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Experimental Deformation of Salt in Cyclic Loading

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Compressed air energy storage in geologic media has been proposed to help "firm" renewable energy sources, for example wind and solar, by providing a means to store energy when excess energy was available, and to provide an energy source during non productive renewable energy time periods. Such a storage media may experience hourly (perhaps small) pressure swings within a geologic storage media. This implies that the storage "container", for example, a salt cavern, may experience small irregular pressure cycling.

An initial study has been undertaken wherein room temperature confined rock salt specimens (a Gulf Coast domal salt 99% halite) have been cyclicly stressed. The rock salt was first characterized by developing a dilatancy "strength" criterion. Then, samples confined at 3000 psi were cycled (in triaxial compression) between 25-30% and 50-60% of the dilatant strength. Samples were cycled up to the 50-60% load, held at constant stress for ~ 3 hours, then cycled down to the 25-30% load, and then again held for 3 hours. Samples experienced about four load cycles per day; tests ran from 12 days to about 60 days, resulting in about 40 to 240 load cycles on different samples.

During tests, axial and radial displacements were recorded. For all tests, it was found that Young's Modulus determined from unloading cycles decreased with increasing axial strain, load cycle, and time after an initial period of small change wherein the modulus remained the same or increased. Using a dilatancy criterion of the volume strain changing from compaction to dilation, the samples are also observed to appear to dilate at these low stress levels. These strain measurements, from this limited study, imply that the samples were cracking at these low cyclicly-applied differential stresses well below the dilation criterion curve. The recording of acoustic emissions during testing and presence of microfactures in deformed samples observed using rhodemine dyed epoxy impregnation confirmed the source of the dilatant behavior.

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Introduction

The U.S. administration's energy and environment agenda calls for substantial, near-term increases in renewable generation. At the same time, system operators, DOE and other stakeholders are recognizing that the previously existing "reserve capacity" in some energy regions and subregions has already been committed to support interconnection of the "first wave" of intermittent renewable generation. In those regions, the "critical" levels of remaining reserve capacity have already started to slow or preclude the interconnection of the desired "next wave" of intermittent renewable generation. As a result, developers of new intermittent renewable generation are being required to demonstrate the availability of sufficient "back-up" generation (typically gas-fired) to firm deliveries of power from their project before they are allowed to interconnect.

Early penetration of renewable energy resources, onshore wind and solar, into the U.S. electrical grid have not yet suffered from their single major weakness, the penetration, at scale of renewables won't be accomplished without bulk storage. The weakness is the variability of wind and solar: Where the wind is not blowing or the sun is not shining, the grid could fail at these points of generation.

Solar power is a predictably intermittent energy source, meaning that although solar power is not available at all times, we can predict generally when it will and will not be available (on a daily basis). Some technologies, such as solar thermal concentrators have an element of thermal storage, such as molten salts. These store spare solar energy in the form of heat which is made available overnight or during periods that solar power is not available to produce electricity. On a day with intermittent clouds passing, for example, a large photovoltaic array solar power generation is unpredictable.

Electricity generated from wind power can be highly variable at several different timescales: from hour to hour, daily, and seasonally. Annual variation also exists, but is not as significant. Related to variability is the short-term (hourly or daily) predictability of wind plant output. Like other electricity sources, wind energy must be "scheduled". Wind power forecasting methods are used, but predictability of wind plant output remains low for short-term operation. The first generations of wind power systems have been able to depend on existing peaker capacity to even out their variability, but this is neither a scalable economic solution nor a CO_2 sensible solution.

Because instantaneous electrical generation and consumption must remain in balance to maintain grid stability, this variability can present substantial challenges to incorporating large amounts of renewable power into a grid system. Intermittency and the non-dispatchable nature of renewable energy production can raise costs for regulation, incremental operating reserve, and (at high penetration levels) could require an increase in the already existing energy demand management, load shedding, or storage solutions or system interconnections. At low levels of renewable penetration, fluctuations in load and allowance for failure of large generating units requires reserve capacity that can also regulate for variability of renewable generation.

Renewable power can be supplemented by storage or replaced by other power stations during low renewable generation periods. Transmission networks must already cope with outages of generation plant and daily changes in electrical demand. Systems with large renewable components may need more spinning reserve, that is plants operating at less than full load (Wikipedia, 2010).

Grid scale energy storage can store energy developed by renewables and release it when needed. Stored energy could increase the economic value of renewable energy since it can be shifted to displace higher cost generation during peak demand periods. Storage can provide reliability without additional transmission costs. There will be generation, transmission, and distribution applications for different storage technologies, to support the integration of renewables for both grid performance and grid efficiency.

The addition of utility scale, bulk energy storage will be required to realize the potential of the emerging smart grid and facilitate the integration of additional intermittent renewable generation. CAES facilities utilizing the geographically dispersed U.S. salt structures currently perhaps offer the greatest potential for additional bulk energy storage.

We are in the process of developing an experimental and analysis program to understand the potential for high frequency pressure cycling in underground storage media that may be required to support integration of intermittent renewable generation into the grid. The calculated geomechanical stresses on salt caverns will in part be used to design tests to determine the geomechanical response of cyclically-deformed rocksalt. The observed experimental results (damage, dilatancy, healing, creep response) will then be used to refine constitutive models for subsequent numerical analyses and assess the results of those analyses. The analysis results will help define acceptable pressure load cycling rates and perhaps provide insight towards engineering guidelines for the operation/design of caverns for use in this high frequency manner.

Here we report on the initial results from an *in progress* experimental program designed to study the effect of cyclic differential stress changes on the mechanical properties of rock salt. The differential stress regime was chosen to be relatively low in order to discover if small stress changes would have any impact on the mechanical properties of the rock salt.

If cavern pressurizations/depressurizations are to load follow a renewable in situations described above in an unbuffered manner (buffering may be achieved with another smaller capacity energy storage medium), changes in cavern pressure could be abrupt and repetitious. If a cavern were to be operated at a relatively high average pressure (for example 0.6 to 0.9 of the casing shoe pressure) the salt stress state condition should remain in a non-dilatational state. Our desire was to evaluate the scenario wherein rock salt was subjected to cyclic loads at differential stresses below the dilation stress state.

The effect of cyclic loading on the elasticity and strengths of geologic materials has long been recognized (for example, Haimson, 1974). Cyclic loading coupled with time dependent deformation of a semibrittle material like salt has also been studied, (for example: Thoms and Gehle, 1982, Matei and Cristescu, 2000; Allemandou, and

Dusseault, 1996, and other researchers). This work intends to extend the results of these previous studies.

Experimental Approach

A Gulf Coast domal salt, (approximately 99% halite) was used in this study. The crystal size ranged from about ¼" to ½" with a density of about 2.15-2.16 g/cc, and the average UCS for this rock salt is approximately 3500 psi. Room dry samples were first mechanically characterized for its dilation "strength" response (Figure 1) through a series of triaxial compression tests over a range of confining pressures. The dilation strength was determined as the stress state at which a change in direction of the volume strain versus differential stress curve was observed wherein the behavior changes from compaction to dilation. A curve was fit to this experimental data. Using this empirical curve as a guide, and selecting (for this test series) 3000 psi as a confining pressure, stress states appropriate to 50% and 60% of the differential stress dilation "strength" were determined (Figure 1).

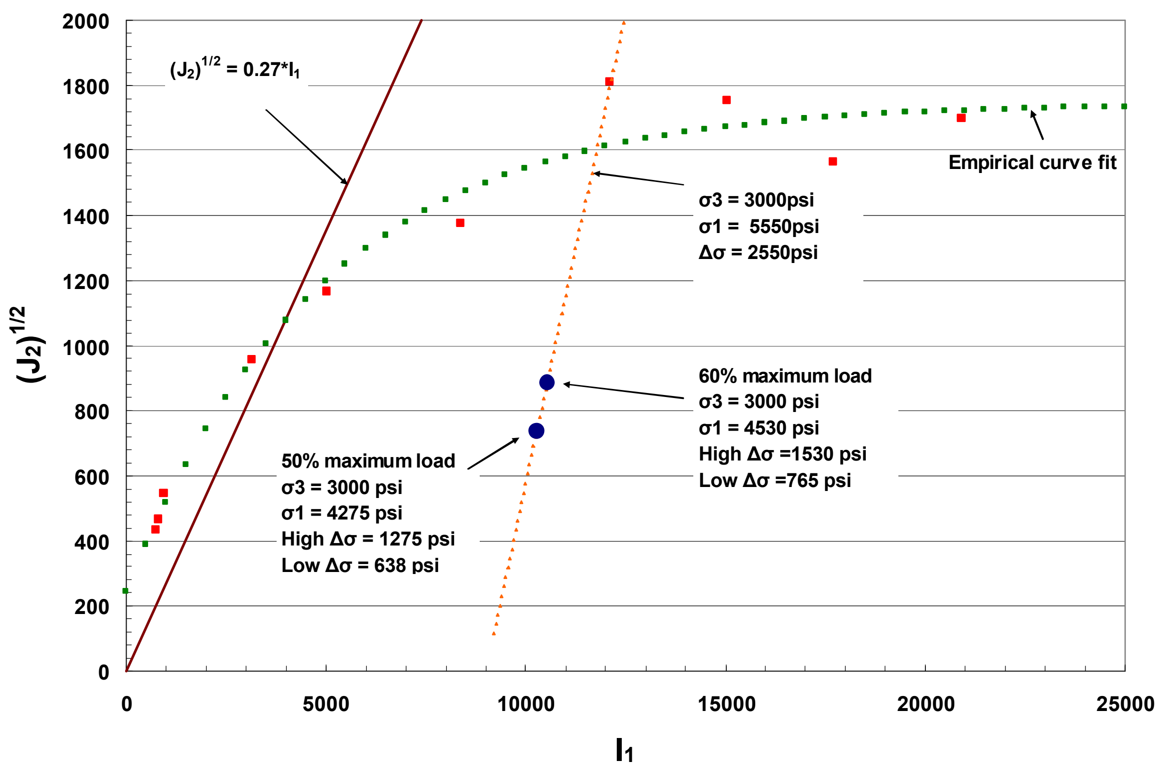

Figure 1. Dilatant behavior of salt determined from quasi-static tests and stress states for tests conducted as part of this study.

Using this mechanical characterization, stress state cycling limits were chosen to test the salt. Then, room dry and oven dried samples of field core (right circular cylinders nominally 4" in diameter and 8" long) were confined at 3000 psi for 24 hours and were cycled (in triaxial compression) between 25-30% and 50-60% of the dilatant strength. Samples were cycled up, held at constant stress for ~ 160 minutes, then cycled down, and then again held for ~ 160 minutes. Samples experienced about four load cycles per day; tests ran from 12 days to about 60 days, resulting in about 40 to 240 load cycles on different samples. The load cycles are intended to be used to elucidate potential effects of load cycling of salt at confining pressure. The load cycles are not intended to represent load cycles imposed by renewable energy resources.

The samples were cyclicly loaded and unloaded for the test duration. (Note: our test durations where constrained by other competing uses of the experimental testing apparatus, and were not stopped because some interesting phenomenon was observed). Each sample was ramped up to the desired upper load at ~$2x10^{-6}$ s^{-1} loading rate (Samples were in load control). Samples were then held at the constant differential stress for approximately 160 minutes, and then unloaded at ~$2x10^{-6}$ s^{-1} unloading rate to the desired lower differential stress and held there for approximately 160 minutes. Data collection rates were 120 seconds during the "hold' portions of the test, and 5 seconds during the load ramp periods; the 5 second data rates began one minute before initiating and after ending a ramp period. The load cycling was repeated for the duration of each experiment. Samples were unloaded to hydrostatic, and subsequently returned to ambient pressure. During each experiment, axial force and axial displacements were recorded; for tests 3 and 4 radial displacements were recorded as well. In test 4, acoustic emissions were recorded for a portion of the test. Thus far, four experiments have been completed of about 12, 23, 32, and 62 days duration (Table 1). Tests 1 and 2 samples were off the lab shelf samples, thus they were room dry. Tests 3 and 4 samples were oven dried for 24 hours at 100^{o}C. The water content of all test samples is unknown.

Table 1. Test matrix

Test #	dryness	σ_D max (psi)	σ_D min (psi)	Duration (days)	E_i (msi)	E_{max} (msi)	E_{min} (msi)	
1	room	1596	800	61.9	4.85	5.02	3.92	
2	room	1273	637	12.0	4.87	4.90	4.41	
3	Dried at 100^{o}C	1275	638	32.0	4.87	5.10	3.95	
4	Dried at 100^{o}C	1532	770	22.9	4.58	4.71	4.36	

The computer-controlled servohydraulic testing system used to conduct the room-temperature (77°F) quasi-static triaxial compression tests on the domal rock salt specimens described in Table 1 is shown in Figure 2. The system is comprised of an SBEL pressure vessel assembly and an MTS Systems reaction frame. During testing, the pressure vessel housing the test specimen was hydraulically connected to a pressure intensifier capable of inducing pressures up to 30,000 psi using Isopar® as the pressurizing medium. The reaction frame is equipped with a moveable cross-head to accommodate various sizes of pressure vessel assemblies and is capable of applying axial loads up to 220,000 pounds through a hydraulic actuator located in the base of the frame. Vessel pressures were measured by a pressure transducer plumbed directly into the hard line that supplies the pressure to the vessel. It is located ~ 10 ft. from the vessel pressure. Axial loads were measured by a load cell located outside the pressure vessel.

Each test specimen was jacketed in a ~1/16-inch-thick impervious heat shrink membrane to prevent the confining pressure fluid from contacting and/or entering the pore space of the specimen when it was placed under pressure in the pressure vessel. The jackets were sealed to hardened steel end caps above and below the specimen with 304 stainless steel wire. The ends of the specimens are lubricated to the end caps with a 50:50 mixture of Vaseline and stearic acid.

Test specimens were instrumented with electronic deformation transducers before they were placed in the pressure vessel assembly. Radial deformation was measured using two linear variable differential transformers (LVDTs) oriented at 90^{o} to each other near the specimen mid height. Axial deformation was measured by two linear variable differential transformers (LVDTs) mounted to the specimen endcaps. Displacement measurements are averaged for strain calculations. During unload cycles, the axial stress versus strain response permitted determination of Young's modulus.

Figure 2. Testing system used to conduct quasi-static triaxial compression tests.

Setup of the quasi-static triaxial compression tests included placing the jacketed, instrumented specimen assembly into the pressure vessel, connecting instrumentation leads to feed-throughs in the pressure vessel and mounting the pressure vessel assembly into the reaction frame (Figure 2). The actuator in the base of the frame was then advanced gradually raising the pressure vessel assembly into position for the test. A small axial preload was applied to the specimen through a push-rod that extended through the top of the pressure vessel and was in direct contact with the crosshead load cell on one end and the specimen on the other. Then, initiation of the test was turned over to the test system controllers which automatically increased the confining pressure and axial stress to ensure the specimen was loaded to the correct hydrostatic confining stress. Once the test system had stabilized at the target pressure for 24 hours, the cyclic loading portion of the test was initiated. (Note: Test 1 was subjected to extended time periods of high and low "holds" early in the test, while we were developing the experimental method; Test 4 was overloaded by about 70 psi at the start of the test for about 700 seconds.

Results

An example of the test load changes and axial and volume strain measurements through time are shown in Figures 3, 4, and 5 for Test 3. At a confining pressure of 3000 psi, the sample was cycled between 1275 psi and 638 psi for about 32 days. Axial strain tends to increase with time and volume strain increases and then begins to decrease with time. There are almost rhythmic minor periodic oscillations in axial and volume strain with time as compared to a "smooth curve". This perhaps reflects small temperature variations in the lab and instrumentation, and thus recorded data. We recorded lab/vessel temperature during the tests, and it varied about a degree C.

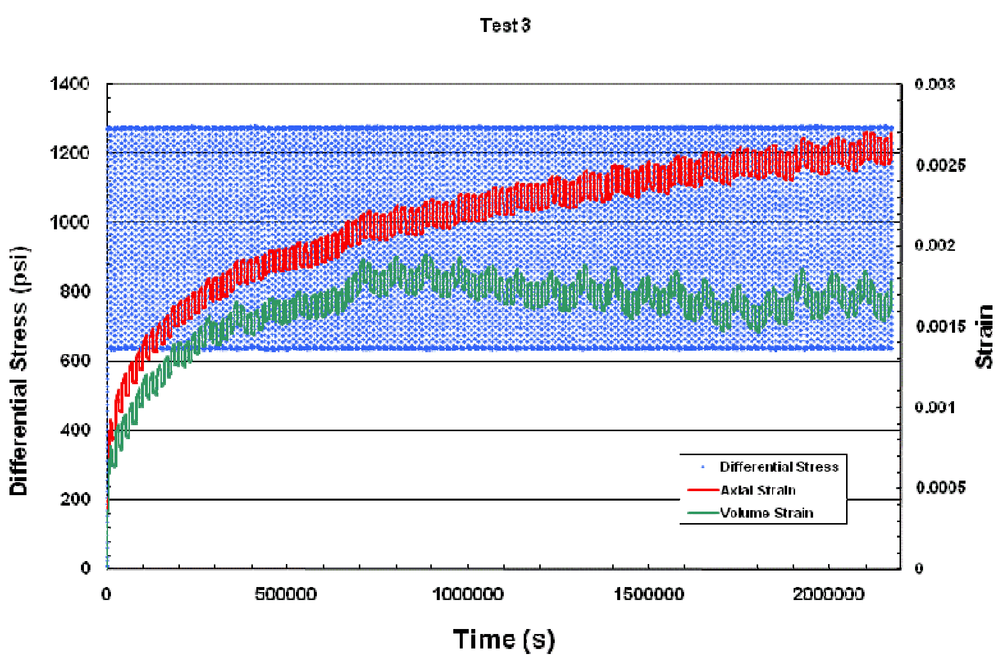

Figure 3. Differential stress, axial and volume strain versus time, Test 3.

In Figure 4, differential stress versus axial strain is plotted for Test 3. When the sample is first loaded to the maximum load, it strains by creeping until it is unloaded. Upon unload to the minimum load, the sample continues to creep (shorten) with time. With each load cycle, the amount of creep shortening decreases, however, the sample does continue to shorten.

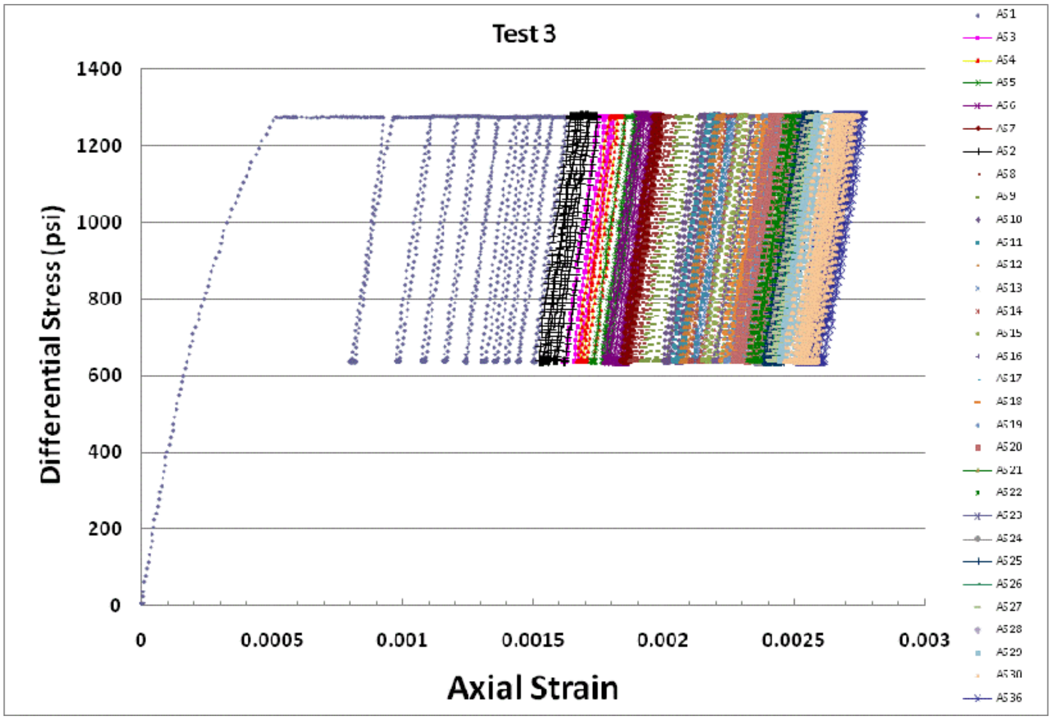

Figure 4. Differential stress versus axial strain, Test 3.

In Figure 5, differential stress versus volume strain is plotted for Test 3. When the sample is first loaded to the maximum load, it strains by compaction creep until it is unloaded. Upon unload to the minimum load, the sample continues to compact with hold time. With each load cycle, the amount of creep by compaction decreases until it appears that the sample stops compacting and cycles between dilation and compaction for successive load cycles, trending toward dilation. At the end of the test, the sample unloads from a point of lower volume strain than the minimum compacted volume achieved, implying the sample has dilated.

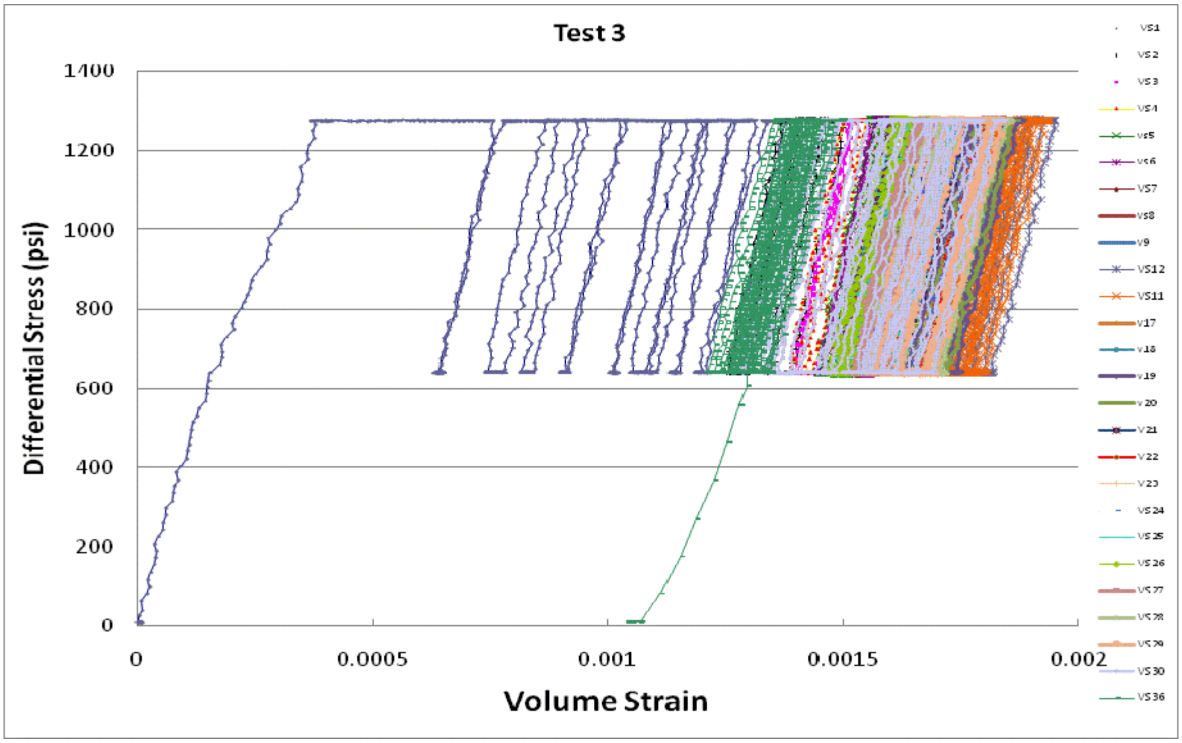

Figure 5. Differential stress versus volume strain, Test 3.

For Test 4 we were able to utilize an acoustic emission recording system for two portions of the test. We have not fully analyzed this data, and we recognize that we may need to improve our acoustic emission measurement technique, but we want to report that acoustic emissions were recorded. During this test it appears that we recorded emissions during the compactive deformation and on into when the sample could have been dilating.

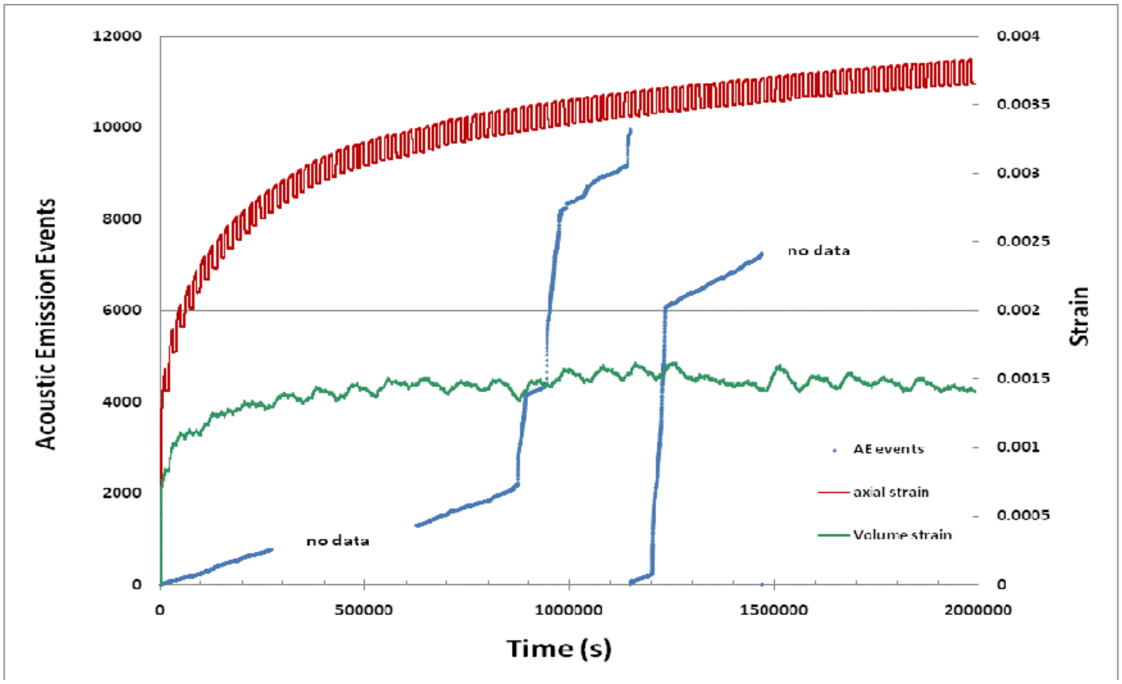

Figure 6. Acoustic emission events and strain versus test time for Test 4.

Young's Modulus (YM) was calculated for each unload cycle for each test, and is plotted versus test time in Figure 7. Generally, it appears that for all tests, YM increases or remains the same for the early portion of the test. Then YM is observed to decrease for all tests with increasing time, load cycle, and axial strain. For Test 1, YM decreases, then increases, then decreases. The general decrease in YM for all tests may imply that microfractures are forming with successive load cycle to cause the sample to become "softer".

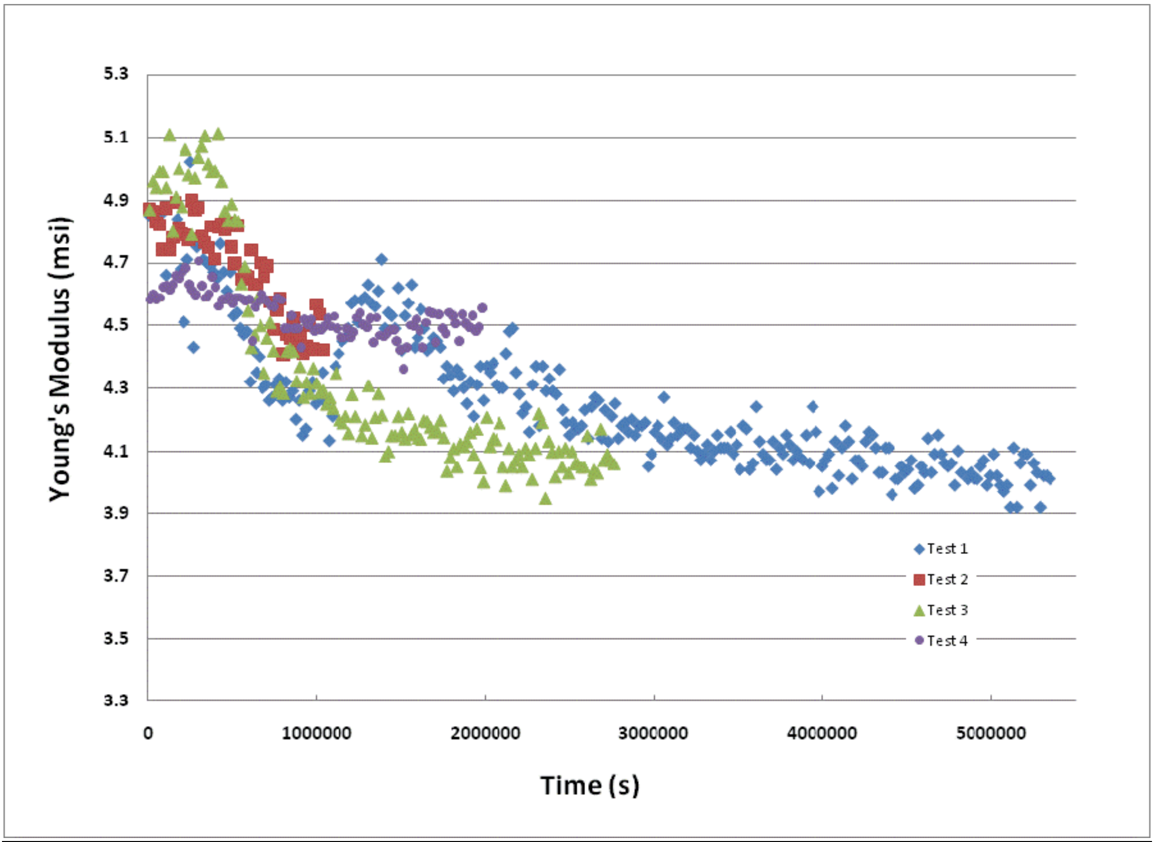

Figure 7. Young's Modulus versus time, all tests.

In Figure 8, examples of "thick sections" of rhodemine dye impregnated samples are presented. Load direction is vertical, the viewed area is approximately 2" wide by 3" tall. The presence of cracks within these samples implies some microfracturing may have occurred during the experiment; observations comparing these samples to undeformed samples are planned. The crack details have not been quantified, nor have further details of the microstructure (i.e. dislocation densities); this is planned for future work.

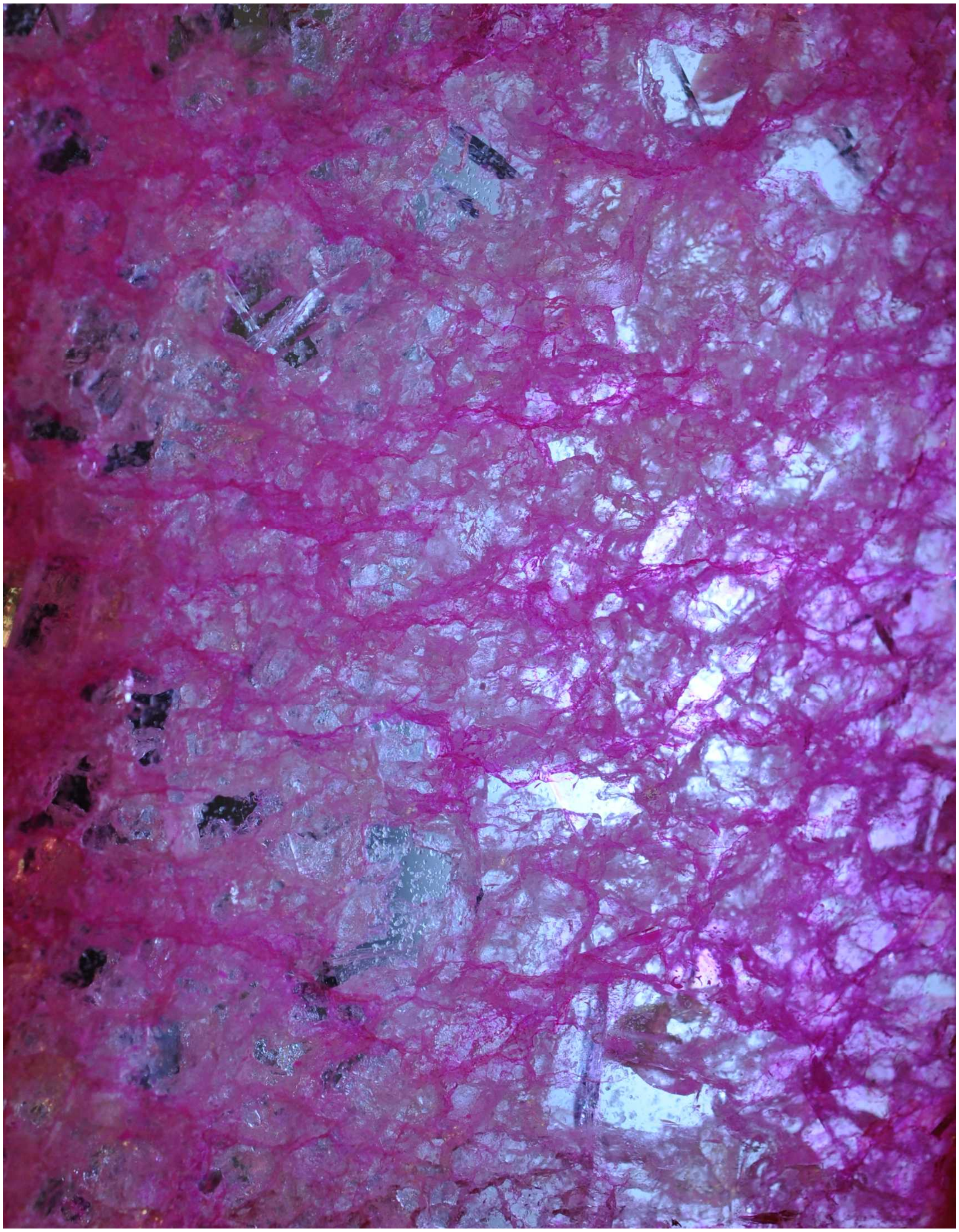

Figure 8. Images of deformed samples: Test 1, Test 2.
Note to reviewer-these photos will be replaced with better ones-the edges of the sample look redder because of poor lighting in the picture.

Discussion, Comments, Conclusions

We have presented preliminary results of four confined cyclic loading tests on a domal salt. The samples were subjected to cyclic differential stresses well below what had been determined to be the dilatancy "strength". It appears, from these few tests, and these preliminary analyses, that microfractures are being induced. The evidence presented includes the observed changes in Young's Modulus, the transition from compactive to dilation behavior during the tests, the recording of acoustic emissions (part of one test), and the observation of microfractures in deformed samples.

The decrease in YM with increasing load cycle upon unconfined rock salt leading to failure below the short term unconfined compressive strength has recently been documented (Fuenkajorn and Phueakphum, 2009). In their much greater loading frequencies 0.001 Hz to 0.03 Hz a 10 % decrease in YM was observed and 30% decrease in strength. For their test conditions, it is likely that brittle processes (microfracture) dominated the deformation.

In uniaxial stress stepped creep tests Matei and Cristescu (2000) observed the dynamic velocities (V_p, V_s) and elastic property parameters to first increase, remain the same, and then decrease depending on the stress state domain (compressibility, transition, dilatancy).

Mechanistically, we offer the following explanation of our results, considering that a polycrystalline aggregate is being deformed. At room temperature and 3000 psi confining pressure we would expect both plastic and brittle deformation mechanisms to be operative. Early in the tests the constant or increasing YM may reflect hardening or compression of the material. Compression implies closure of pre-existent microfractures. Hardening implies dislocations have moved to pileup positions. Each and both of the mechanisms are possible. At room temperature one may not expect a great deal of climb from pileups. During continued cycles of deformation, dislocations are continually pushed to pile up positions, and at times locked into these positions. Further cycles eventually lead to microcrack formation. The deformation process may be systematic, but depends on local crystallographic orientation relative to the locally imposed stress and strain field in the polycrystalline aggregate, thus the manifested deformation in terms of the decrease in modulus is locally abrupt in terms of cracks forming, but perhaps less abrupt and/or gradual in terms of the decrease in modulus observed. This explanation is consistent with the small oscillatory transition in volumetric strain observed, the acoustic emissions recorded, and the observed (but not as yet quantified) presence of microcracks. The semi-brittle deformation observed is consistent with the work of Fuenkajorn and Phueakphum (2009) and Matei and Cristescu (2000), and others not cited.

Shortcomings of our work include the fact that although we used, for each experiment, the same rock salt, each had, perhaps, different moisture content, and inherent damage from the field coring process. This could have influenced the results. Also, no measures to "anneal" the samples, or condition them as has been recommended (Allemandou and Dusseault, 1993) were employed. The presence of pre-existing cracks in an unknown way most likely effected the measured results within each sample, and could be a cause of sample to sample test result variability. Finally, the displacements/strains recorded during these tests are small, however the experimental results are somewhat consistent/repeatable from test to test, the results are consistent with evaluating similar phenomena identified by previous workers using a different approach, and the measurement magnitudes are similar to those of previous researchers (for example (Allemandou and Dusseault, 1993).

The presence of water and thus its effect in our tests is an unknown; the presence of water and its effect on salt cavern behavior may be a similarly enigmatic. It is generally believed that domal salts are dry, owing to the deep burial and opportunity for water to be squeezed out during the natural deformation. Some bedded salts, not having experienced similar burial nor deformation may still have water present, at least at grain boundaries. Perhaps our samples had some water in grain boundaries? Because water is used to leach caverns and there is opportunity for bedded salts to contain water, it may be acceptable that our samples could have had some water in them.

The results presented are preliminary because the experimental methods are still being developed. Unfortunately, tests are of long duration thus they rely on extended use of equipment and uniform experimental conditions for the test duration. Perhaps the experimental study could be shortened with a slight increase in temperature (this would result in less accurate displacement measurements), or smaller grain size starting material (this would necessitate a re-characterization of a starting material).

An implication may be that the dilatancy criteria used to determine operational limits of salt caverns should be revisited, especially if and how salt caverns will be used to support renewable energy resources. If salt strengths are lowered due to frequent small pressure change cycling, this could be detrimental to cavern longevity. Perhaps operational characteristics of such caverns need be adjusted to allow for crack healing. Perhaps at elevated temperatures commensurate with the geologic thermal gradients, and or ground warming due to gas compression, more ductile deformation mechanisms will be present and brittle processes will be eschewed.

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