ARMA 15- 517

SAND2015-2187C

Gas flow measurements of consolidating crushed salt

S.J. Bauer, S.T. Broome, F.D. Hansen,
Sandia National Laboratories, Albuquerque, NM, USA

B. Lampe, M. Mills, and J. Stormont
University of New Mexico, Albuquerque, NM, USA

This paper was prepared for presentation at the 49th US Rock Mechanics / Geomechanics Symposium held in San Francisco, CA, USA, 28 June-1 July 2015.

ABSTRACT: Crushed salt is being considered as a backfill material in the event of a salt repository for high level nuclear waste. The thermal-mechanical-hydrological properties of crushed salt as it reconsolidates in response to pressure and temperature changes are therefore important. An experimental system to measure gas flow through consolidating crushed salt at elevated temperature and pressure has been developed and tested. An experiment completed at 250°C, and hydrostatic pressures to 20 MPa, compacted a crushed salt sample from ~40 percent porosity to near zero porosity. For this consolidation history, apparent permeability decreased from greater than 10^{-12} m^2 to ~10^{-22} m^2.

1. INTRODUCTION

It is important to improve the understanding of the coupled thermal-mechanical-hydrologic response of granular (or crushed) salt used as a seal material for shafts, drifts, and boreholes in mined salt repositories. Granular salt consolidation is being investigated through an integrated program of laboratory measurements, observations, and constitutive model development and evaluation. In particular, we are focused on measuring the deformation and corresponding permeability reduction of granular salt as it consolidates to fractional densities greater than 0.90. Behavior at high fractional densities is of principal importance to repository applications because this is when the permeability of granular salt is expected to decrease to a condition comparable to intact salt, that is, it becomes nearly impermeable. An extension of an existing constitutive model is being developed using these data to allow prediction of changing properties as granular salt consolidates.

Rock salt has been considered as a potential disposal medium, because salt is essentially impermeable. The very existence of salt millions of years after deposition is proof that water has not flowed through the formation. The undisturbed formation permeability of salt is essentially too low to measure using traditional hydrologic and reservoir engineering methods. In undisturbed and healed salt, brine is not able to flow at rates that would lead to significant radionuclide mobilization and transport. In terms of disposal, one of the most important features of salt as an isolation medium is its ability to heal previously damaged areas. Damage recovery is often referred to as “healing” of fractures. The healing mechanisms include microfracture closure and bonding of fracture surfaces. Evidence for healing of fractures in salt has been obtained in laboratory experiments and through observations of natural analogs. Fracture healing can restore the low permeability of intact rock salt. The consolidation of granular rock salt is envisioned to occur under similar mechanisms to healing.

After waste emplacement and backfilling, the natural salt host rock creeps under the effect of temperature and ambient stress conditions. As a result, backfill will progressively reconsolidate. Determination of the thermal-hydraulic-mechanical constitutive properties, their inter-relationships, and operative deformation mechanisms of consolidating crushed salt are objectives of this study.

Over the years, salt reconsolidation has been a topic of great interest to international salt repository studies [1, 2]. A preponderance of these studies has been at room

temperature, with a few tests at elevated temperatures up to 100°C. Today there is a renewed national and international interest in salt reconsolidation at elevated temperature, particularly as applied to disposal of heat-generating nuclear waste. There are recent experimental studies in the U.S. and abroad [3] where crushed salt reconsolidation has been accompanied by measurement of its changing permeability.

The work described here is focused on measuring gas flow through consolidating crushed salt at elevated temperatures. The fluid flow measurement system is designed and assembled for samples undergoing hydrostatic and/or shear stress conditions at elevated temperature and pressure [4]. For the high flow rates expected at large porosities, gas flow is measured using a constant head (or pressure) technique with an inert gas as the permeant. For anticipated low flow rates at low porosities, gas flow is measured using a helium mass spectrometer. Several experimental conditions are of interest in our study: temperatures ranging from lab ambient to 250°C; dry crushed salt and dry crushed salt with specified weight percent water added; porosities initiating at ~40 percent and decreasing to values representative of intact salt; and hydrostatic and deviatoric stress conditions consistent with excavations and ultimate closure of a potential salt repository at approximately 700 meters depth. This paper reports on the initial experiment at 250°C.

2. MATERIALS AND METHODS

2.1 *Sample Material and Preparation*

Samples are assembled from run-of-mine salt from the Waste Isolation Pilot Plant (WIPP) in New Mexico. This salt is mined from the WIPP radioactive waste disposal horizon and represents bulk mineralogy of the formation, including 1.0–1.6% water-insoluble impurities. X-ray diffraction analysis of this material shows it closely resembles previous analyses showing up to 5% water-soluble and insoluble impurities, including quartz, gypsum, and clays [5]. Water content of this salt, held predominantly in hydrous minerals, negative crystals, and grain boundaries, is on the order of 0.45 weight per cent [6].

This work builds on previous experimental efforts of Broome et al. [4]. Figure 1 shows a typical Sandia-designed creep testing system modified for the specifics of this work. The test system consists of a load frame that generates the axial force by reacting against a hydraulic cylinder located at the base and a pressure vessel that houses the test specimens. The reaction frame system is capable of applying loads of up to 0.44 MN. The pressure vessel is rated to 69 MPa and is equipped with electrical band heaters capable of maintaining test temperatures up to approximately 250°C. Silicon oil is used as the confining medium. Fluid pressures are adjusted using an air-assisted pump and can be maintained constant using a dilatometer system that either injects or withdraws oil from the vessel. Vessel pressures are measured by a pressure transducer plumbed into the hydraulic line leading from the vessel to the dilatometer. Axial loads applied by the hydraulic cylinder are measured by a load cell located directly above the hydraulic cylinder in line with the axial push-rod that extends into the pressure vessel and applies axial load to the ends of the specimen. Small friction forces in the seals are calibrated and factored into the data reduction and analysis. Test temperature is recorded by two thermocouples, one located near the top of the pressure vessel and other near the vessel midheight. Sample temperature (250°C in this test) for this apparatus is determined by system calibration.

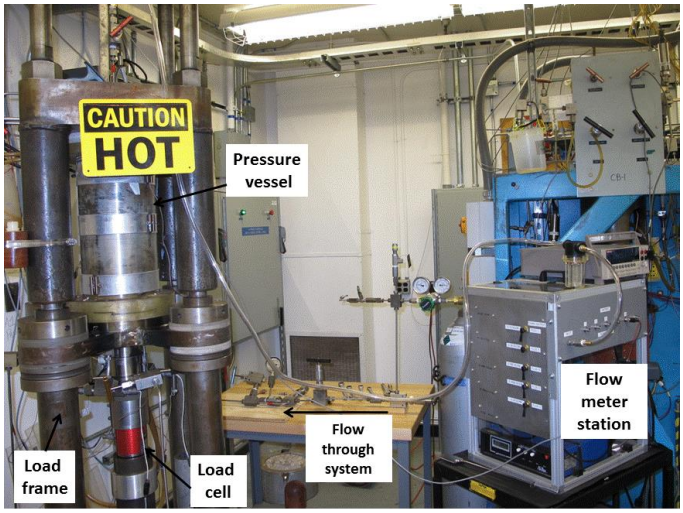

Figure 1.Creep load frame and flow through system at Sandia National Laboratories.

The specimen assembly (Figure 2) consists of a right circular cylinder of unconsolidated granular salt, nominally 8.5 cm in diameter and 16.5 cm in length, constrained vertically between specially machined aluminum end caps. Machining the outer diameter of the end caps enables a metal-metal seal of an outer lead jacket to the end cap. Each end cap is vented to the flow system during the test. Beveled face plates are placed between the end caps and salt to accommodate diametral change of the granular salt and allow space for the inner lead jacket. A porous metal frit, shown in the figure as porous felt metal, is placed on either end to allow fluid migration and venting across the entire specimen surface area. The outer lead jacket, a soldered lead tube (0.094 cm thick), encases an inner copper (0.013 cm thick) sheath, which covers the inner lead sheath (0.094 cm thick) and laterally contains the entire assemble from top

to bottom, and isolates the inner jacketing and the end caps from the confining medium. The lead and copper assemble is highly malleable and conforms to the shape of the specimen when hydrostatic pressure is applied. Sandwiching of jacketing materials was found to prevent jacket leaks during large volume changes of the sample.

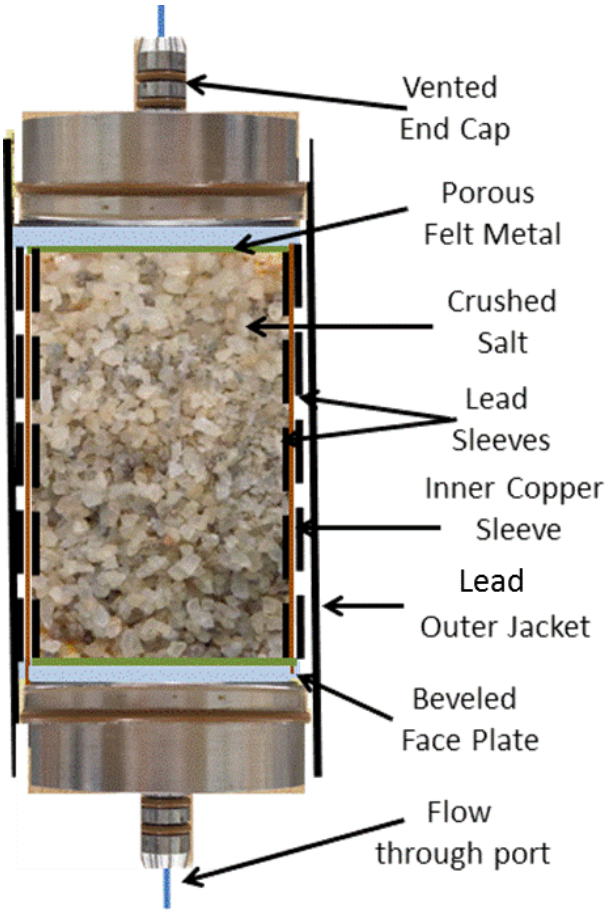

Fig. 2. Schematic of Test Arrangement

Deformations of the specimen are recorded from axial LVDT's mounted the outside the pressure vessel and using dilatometry. The two methods are used together to determine dimensional changes and volume strain of the sample. Typically, LVDT's are used during relatively short term, large dimensional change situations (when temperature or pressure was ramped up), and dilatometry is used during long time periods, for example at constant pressure.

Data collected in the experimental study included force, pressure, temperature, displacement, and volume change. Typically, these data are acquired using electronic transducers in which the electrical output is proportional to the change in the measured variable. In all cases, the constants of proportionality were determined through careful calibration using standards traceable to the National Institute for Standards and Technology.

The gas flow measurement system is designed and assembled to flow through samples undergoing hydrostatic and/or shear stress conditions at elevated temperature and pressure [4]. For anticipated high flow rate conditions (high porosity, up to 900 sccm down to 0.1 sccm), gas flow is measured using a constant head (or pressure) technique with an inert gas as the permeant; this was accomplished with a flow meter panel, wherein an array of flow meters is used with specific ranges of operation. For anticipated low flow rate conditions, (low porosity; to 10^{-8} sccm) gas flow is measured using a technique in which a helium mass spectrometer is used to detect flow [7]. Gas supply flow is supplied upstream from a regulated tank of ultra-dry research grade nitrogen or helium gas (<1ppm H_2O). Upstream pressure for all measurements is 124 kPa, and downstream pressure is atmospheric room pressure (83 kPa). The test specimen is placed between two metal platens having central ports that permit the permeant (helium) to enter and exit the specimen. Highly-permeable porous felt metal disks are placed in the interface between the platens and specimen to distribute the helium pressure across the full cross-section of the specimen. The specimen dimensions are tracked with displacement gages and dilatometry during deformation, allowing updated specimen dimensions to be used in subsequent permeability calculations. Gas flow is measured at specific reconsolidated states.

For both flow measurement methods, apparent permeability of the sample, k, is calculated from Darcy's law for steady-state flow:

$$k = \frac{Q\mu L}{A\Delta P} \qquad (1)$$

where k is the permeability (m^2)

Q_X is the flow rate in the axial direction of the specimen (m^3/s)

μ is the gas viscosity (Pa sec)

ΔP is the pressure differential measured across the ends of the specimen (Pa)

L is the length of the specimen (m), and

A is the cross-sectional area perpendicular to the axis of the specimen (m^2).

For the initial flow while the specimen was still very permeable, periodic permeability measurements using nitrogen gas were made to capture potential permeability changes in response to consolidation. After flow was established through the specimen, flow continued for five to ten minutes to ensure a consistent measurement and was then stopped. It is unlikely these relatively short time measurements dehydrated the specimens;

however, the nitrogen gas used was extremely dry so as not to add water to the specimen. For mass spectrometry test periods, reagent grade helium was used as the permeant and more time was needed to reach flow equilibrium.

2.2 Sample Loading

The stress and temperature history is given in Figure 3. An initial axial stress of approximately 0.2-0.3 MPa was applied to the sample followed by a confining pressure of 0.5 to 0.6 MPa under room temperature. This condition was maintained for about 30 minutes. The small differential axial stress was applied to ensure the end caps remained in contact with the actuator piston. Next confining pressure was increased to approximately 1.5 MPa while maintaining the small 0.2 to 0.3 MPa differential axial stress. After about 16 hours, temperature was increased to 50°C. The sample remained at this condition for approximately 120 hours. Then temperature was increased to 250°C, over about a 10 hour time span. After attaining 250°C for 10 to 12 hours, confining pressure was ramped up to 20 MPa in ~15 minutes (while maintaining the same small differential stress). These conditions were maintained for approximately 290 hours. Displacements and volume changes were continuously recorded.

During the entire test the sample was allowed to drain/vent into the gas flow lines; and gas flow measurements were periodically made. Gas was flowed for just the amount of time it took to make the measurement.

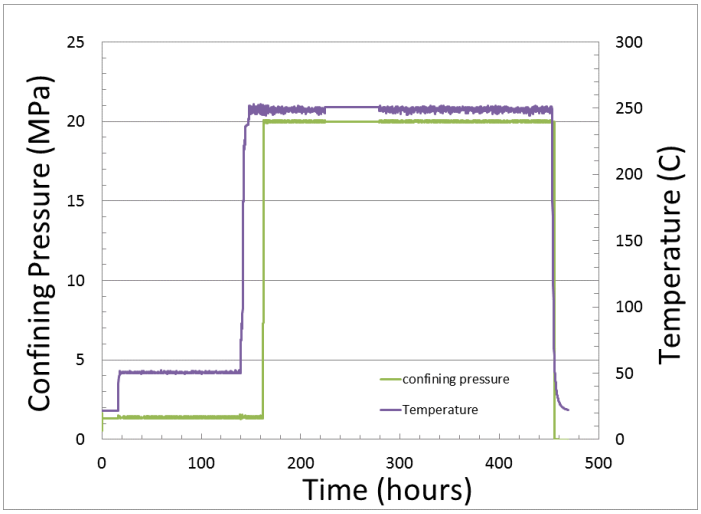

Fig. 3. Confining pressure and temperature versus time

3. EXPERIMENTAL RESULTS AND OBSERVATIONS

During the heating phase from 50°C to 250°C, steam and bubbling brine was observed in the clear tubing connected to the downstream side of the sample. There was no observable moisture after about 24 hours. The sample, once removed at the conclusion of the test, was observed to have deformed somewhat uniformly into a compacted cylinder (Figure 4). The malleable jacketing deforms rather easily to accommodate the initially rough surface of the crushed salt and it maintained its integrity for the duration of the consolidation process. Note the significant dimpling of the lead jacket, caused by the presence of local voids and undulations in the granulated salt surface coupled with malleability of the lead/copper jacketing material.

The consolidated salt retained a rather cylindrical shape upon compaction, perhaps indicating uniform stresses and small end friction constraint.

Macroscopically, the post-test sample appears to be fully reconsolidated, with little to no evident porosity (Figure 5). In low magnification reflected light, grain boundaries appear to be fully joined and intimately contacted to nearest neighbor grains (Figure 6). Some grain boundaries are clean and free of impurities, while some are decorated with such (Figure 7). The deformed sample is generally free of residual brine, except for a few small negative crystals as shown in Figure 8.

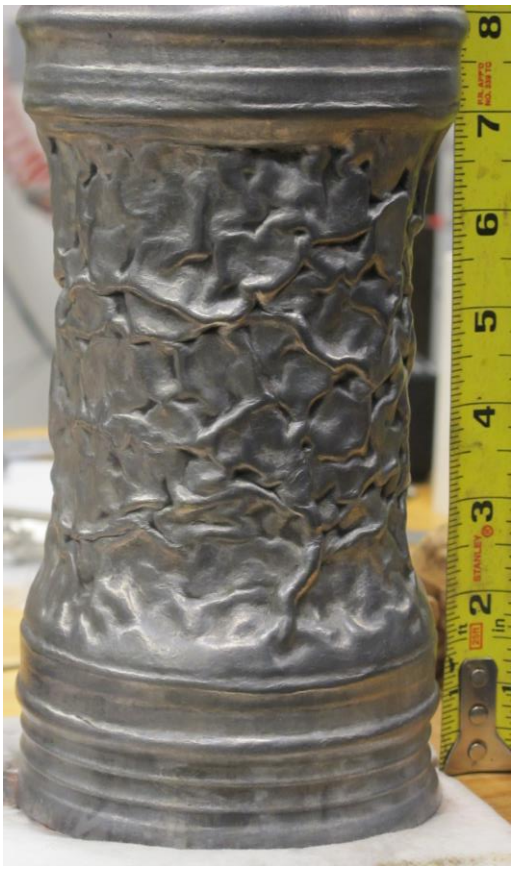

Fig. 4 Deformed sample

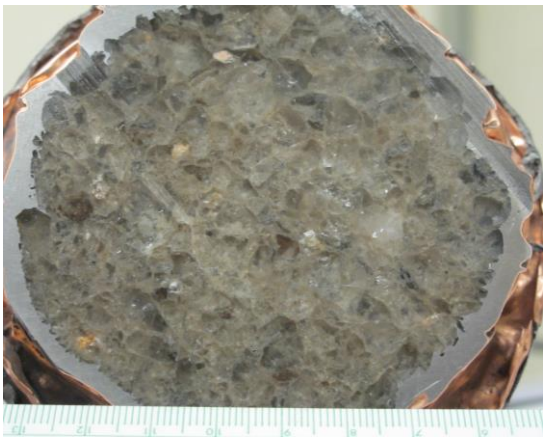

Fig. 5. Diametral slice of sample

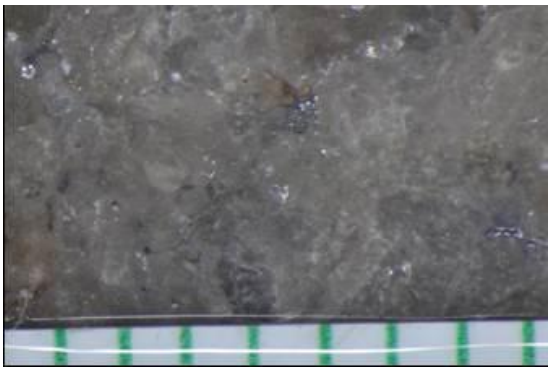

Fig. 6. Typical texture of deformed sample, 1 mm scale

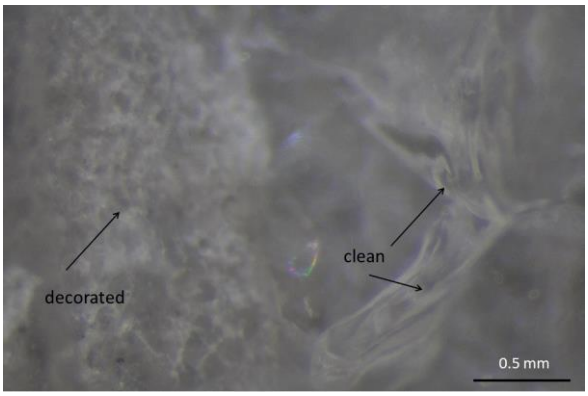

Fig. 7. Clean and decorated grain boundaries

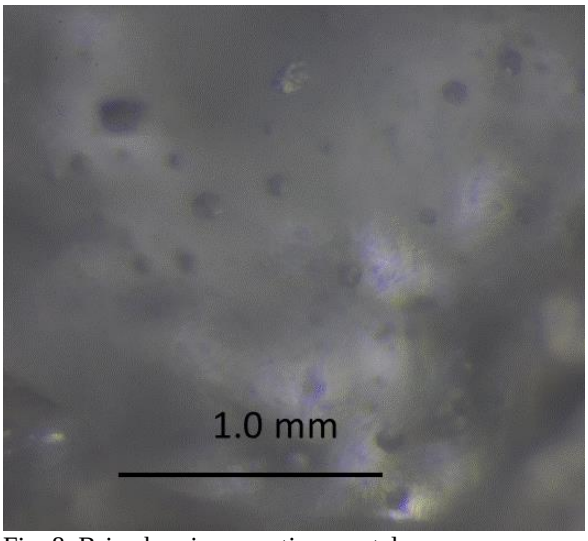

Fig. 8. Brine bearing negative crystals

The imposed temperature and confining pressure history, along with the measured volume strain, is given in Figure 9. Deformation of the sample begins with application of initial seating and confining condition. The sample is quite susceptible to compaction at this point, as it had an initial porosity of over 40%. The increase in pressure to approximately 1.5 MPa, with a small 0.2 to 0.3 MPa differential axial stress and 50°C for approximately 120 hours resulted in about 10% volume strain. The temperature increase to 250°C over about a 10 hour time span resulted in an additional 17 to 18% volume strain. These amounts of strain are consistent with Broome et al. [4]. The confining pressure ramp up to 20 MPa added another ~12% volume strain; from that time forward (an additional 290 hours), the sample compacted only an additional 0.5 to 1% volume strain.

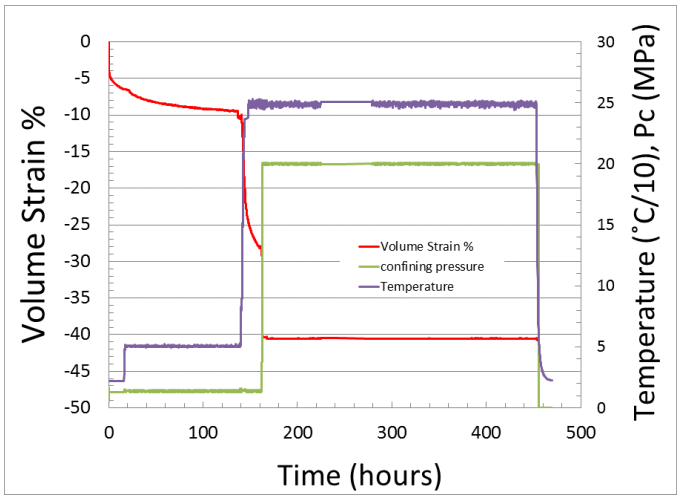

Fig. 9 Volume strain, temperature, and pressure versus time

The periodic gas flow measurements allowed us to estimate apparent permeabilities of the samples during the deformation (Figure 10). At porosities, greater than 0.25, the apparent permeability is in the 10^{-12} m^2 to 10^{-13} m^2 range; this is likely the upper limit of permeability measurement limit as restricted by the high pressure tubing used in our flow through system. For porosities of 0.15 to 0.2, the apparent permeability is observed to decrease to 10^{-13} m^2 to 10^{-14} m^2 range. With application of high temperature and high confining pressure, consolidation happens rather quickly and the apparent permeability decreases from 10^{-15} m^2 to 10^{-22} m^2.

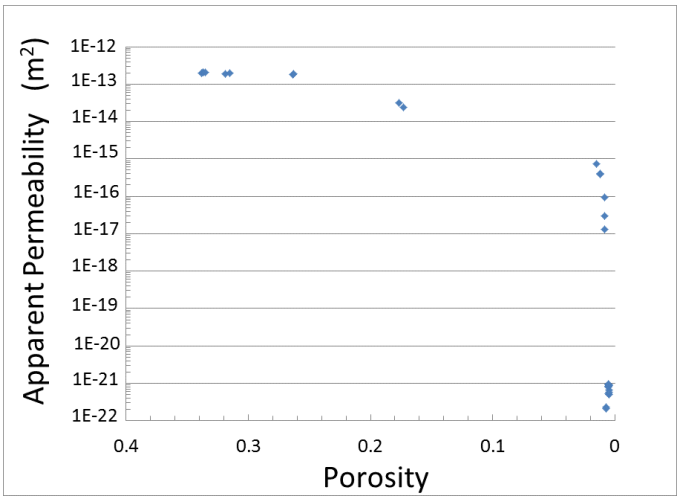

Fig. 10. Porosity versus apparent permeability

The apparent permeability is plotted versus fraction of theoretical density in Figure 11 (considering the empirically determined grain density of this rock salt as 2.145 g/cc). There is some uncertainty in the absolute values of the porosity and fractional density in the narrow range where the permeability decreases dramatically due to measurement uncertainties and assumptions. Planned measurements of porosity using observational methods will be useful in confirming the values in Figures 10 and 11.

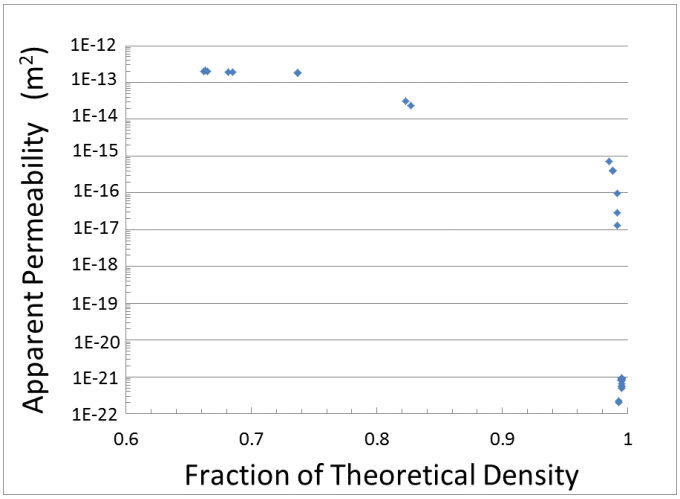

Fig. 11 Fraction of Theoretical Density versus Apparent Permeability

4. DISCUSSION AND CONCLUSIONS

We developed and demonstrated a high temperature gas flow-through system used during reconsolidation of crushed salt and to determine permeability as crushed salt is reconsolidated.

Results are reported for a single experiment run to 250°C and 20 MPa of confining pressure resulting in fully compacted granular salt. Apparent permeability decreases dramatically to very low values in the porosity range of 0.02 to near 0, or theoretical densities of 0.995 to 1. This means that for greater porosities, and although low, there is sufficient pore interconnectivity to facilitate at least some gas flow. Apparent permeabilities for clean healed domal salt, porosity in the range 0.01 to near 0, are in the range of 10^{-21} m^2 to 10^{-22} m^2.

The presence of brine in this rock salt and its escape from the consolidating system has been previously observed and noted [4]. Its impact on the gas flow measurements is unknown. It appears that there is enough brine left in the sample at the end (although sparse) to facilitate solution/precipitation and maturity of a near zero porosity recompacted rock.

5. ACKNOWLEDGEMENTS

Sandia National Laboratories is a multi-program laboratory managed and operated by Sandia Corporation, a wholly owned subsidiary of Lockheed Martin Corporation, for the U.S. Department of Energy's National Nuclear Security Administration under contract DE-AC04-94AL85000. Partial support for this work was provided through the Department of Energy's Nuclear Energy University Program Grant DE-NE0000733. SAND2015-ZZZZ C

6. REFERENCES

1. Aubertin, M. and H. R. Hardy. 1996. *The Mechanical Behavior of Salt, Proceedings of the Fourth Conference*. Montreal Canada. Trans Tech Publications. ISBN 0-87849-103-1.
2. Wallner, M., K-H Lux, W. Minkley, and H. R. Hardy. 2007. *The Mechanical Behavior of Salt, Proceedings of the Sixth Conference*. Hannover Germany. Balkema. ISBN 13: 978-415-444398
3. Kröhn, K-P., C-L Zhang, O. Czaikowski, D. Stührenberg, U. Heemann. 2015. The compaction behaviour of salt backfill as a THM-process *The Mechanical Behavior of Salt, Proceedings of the Eighth Conference*. Rapid City S.D. USA.
4. Broome, S.T., S.J. Bauer, and F.D. Hansen. 2014. Reconsolidation of Crushed Salt to 250°C Under Hydrostatic and Shear Stress Conditions, *48th US Rock Mechanics / Geomechanics Symposium*. Minneapolis, MN, USA.
5. Stein, C. 1985. Mineralogy in the Waste Isolation Pilot Plant (WIPP) facility stratigraphic horizon. *SAND85–0321*. Sandia National Laboratories, Albuquerque, NM, USA.
6. Bauer, S., A. Urquhart, S. Broome, F. Hansen. 2014. Experimental Determinations of Thermophysical Properties of Reconsolidated Crushed Salt, *48th US Rock Mechanics / Geomechanics Symposium*. Minneapolis, MN, USA.
7. Bauer, S.J., M.Y. Lee, and W.P. Gardner. 2015. Helium-Mass-Spectrometry-Permeameter for the measurement of permeability of low permeability rock with application to triaxial deformation conditions, *49th US Rock Mechanics Symposium* San Francisco, CA, USA.