

Final Report

**Improving the understanding of the coupled thermal-
mechanical-hydrologic behavior of consolidating granular
salt**

Prepared by:

John Stormont (PI) jcstorm@unm.edu

Brandon Lampe

Melissa Mills

Laxmi Paneru

Timothy Lynn

Aayush Piya

Department of Civil Engineering
University of New Mexico
Albuquerque, NM 87131

Performed under:

NEUP project 13-4834

Contract DE-NE0000733

DUNS Number: 868853094

TPOC: Robert Mackinnon

FPOC: Prasad Nair

September 9, 2017

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Executive Summary

The goal of this project was to improve the understanding of key aspects of the coupled thermal-mechanical-hydrologic response of granular (or crushed) salt used as a seal material for shafts, drifts, and boreholes in mined repositories in salt. To achieve this goal, we conducted laboratory investigations of consolidation of granular salt and developed a constitutive model to describe this behavior.

We consolidated granular salt under pressures and temperatures conditions relevant to nuclear waste repositories. We measured the consolidation (pore volume reduction) and corresponding changes in hydrologic and thermal properties of granular rock salt at high fractional densities (or low porosity), which is the critical range when granular salt is expected to achieve functionality as a seal material. We documented the role of temperature and pressure on consolidation. In addition, we produced data that clearly reveals that pore pressure affects consolidation. We developed expressions for permeability and thermal properties changes during consolidation to lower porosities ($<1\%$) than had been measured previously. Our results indicate that, for these test conditions, the pore network remains sufficiently connected so that granular salt is much more permeable than intact salt until porosities of less than 2% .

Microstructural observations on consolidated samples allowed us to interpret deformation mechanisms as a function of moisture, temperature and stress conditions. The role of small amounts of moisture, whether natural or added, was confirmed as accelerating consolidation due to pressure solution.

A constitutive model for granular salt consolidation was developed that includes the influence of pore pressure on viscous deformation. Existing constitutive models do not include this capability as an explicit feature, although the behavior has been experimentally documented. The model is shown to capture the dependence of viscous behavior on pore pressure as well as account for temperature effects and hardening due to porosity reduction from consolidation. The developed model also has the attribute of requiring many fewer parameters than other existing constitutive models for granular salt consolidation.

1 Overview of project

This report provides accomplishments of the NEUP project entitled “Improving the understanding of the coupled thermal-mechanical-hydrologic behavior of consolidating granular salt.” The report is the final project report.

1.1 Report organization

This report is organized as follows:

- Section 1 - This section contains an overview of the accomplishments of the project.
- Section 2 – A brief introduction of the project is given in this section.
- Section 3 - Consolidation testing accomplishments are described.
- Section 4 – Post-consolidation testing accomplishments are given in this section.
- Section 5 - This section contains a description of accomplishments involving microstructural observations.
- Section 6 – The accomplishments related to constitutive model evaluation and development are given in this section.

1.2 Goals and objectives

The overall goal of this project is to improve the understanding of key aspects of the coupled thermal-mechanical-hydrologic response of granular (or crushed) salt used as a seal material for shafts, drifts, and boreholes in mined repositories in salt. Achieving this goal will result in greater confidence in granular salt consolidation as a principal strategy for permanent isolation.

The objectives of the project to achieve this goal are:

1. Measure the consolidation (pore volume reduction) and corresponding changes in hydrologic and thermal properties of granular rock salt at high fractional densities (or low porosity), which is the critical a range of conditions expected for nuclear waste repository considerations.
2. Interpret deformation mechanisms as a function of moisture, temperature and stress conditions from microstructural observations on consolidated samples.

3. Extend existing constitutive model to include coupled thermal-mechanical-hydrologic behavior of consolidating granular salt so that it can be incorporated into existing numerical models for predicting repository performance.

1.3 Summary of Accomplishments

A brief summary of accomplishments to achieve each objective are given below. A complete and detailed description of project accomplishments is given in the body of the report.

Objective 1 – We successfully conducted consolidation measurements on granular salt under high pressures and temperatures with concurrent gas permeability measurements. We have conducted tests at 90 to 250°C under hydrostatic stresses up to 38 MPa. Some tests involved maintaining pore pressures up to 22 MPa. The testing capability leveraged the unique facilities and experience of the SNL Geomechanics Lab. The consolidation tests go beyond previous test conditions, and are focused principally on achieving a high fractional density condition. These tests include concurrent permeability measurements which provide important data to assess the thermal-mechanical-hydrological coupling of salt consolidation.

Test results reveal that the hydrostatic creep consolidation test response is strongly dependent on temperature. All consolidation tests conducted at 250 °C resulted in a final condition with a low porosity (<0.01) and high fractional density (>0.99). This final condition is independent of the type of salt, whether water added or not, and whether the sample was drained or undrained. Tests at 175 °C and 90 °C consolidate to low porosities, but at a slower rate. The tests also demonstrated that water accelerates consolidation. This water can be “natural” or added.

Experimental evidence indicates that pore pressure affects consolidation. Multi-stage hydrostatic creep consolidation tests reveal that the volumetric strain rate (or rate of fractional density change) is responsive to pore pressure changes in addition to confining pressure. These findings may be understood in terms of a local stress at the particle scale that varies from the macroscopic state of stress applied to the bulk sample. Based on a recognition of a local shear stress when interpreting these test results, pore pressure was observed to influence the shear stress within a bulk crushed-salt sample.

The permeability and porosity data obtained from tests concurrent with consolidation and those from tests after consolidation were consistent: permeability is a strong function of porosity, and is independent of the stress or temperature conditions that produced the porosity. There was no evidence that samples consolidated with 1% additional moisture have a systematically lower permeability than those without added water. Both data sets indicate that rock salt remains conductive until a very low porosity is achieved. Our results suggest that, for these test conditions, the pore network remains sufficiently connected so that granular salt is much more permeable than intact salt until porosities of less than 0.02. We concluded that intrinsic permeability can be reasonably expressed solely as a function of porosity to porosities of less than 2%, or fractional densities of greater than 0.98. These data were fit to a power law expression of the form $k = k_0 \phi^x$, and the resulting fit is similar to expressions reported by others from tests at greater porosities.

Thermal conductivity and specific heat of intact salt crystal, laboratory consolidated salt and in situ consolidated salt at various temperatures were measured. Results indicate that thermal conductivity decreases with an increase in temperature and porosity. Specific heat increases with an increase in temperature. At lower temperatures, specific heat decreases with an increase in porosity. At higher temperatures porosity dependence is not apparent. Expressions for thermal properties as a function of temperature and porosity were developed.

Objective 2 – We have developed the ability to observe microstructure in deformed granular rock salt by means of optical microscopy and SEM. This capability involves sample preparation, microscope use, and the ability to relate images to deformation mechanisms and characteristics within the salt. In addition, we developed the capability to measure porosity and grain size using imaging techniques.

We found that any sample with additional moisture appears to have a higher level of cohesion at grain boundaries and more instances of pressure solution processes when compared to no moisture samples having copious amounts of mechanically ground surfaces. At higher temperatures (250°C), samples experience recrystallization, apparent in etched thin sections under reflected light.

Objective 3 – We developed a constitutive model for granular salt consolidation to address limitations of existing constitutive models. A principal shortcoming of existing constitutive models was that the influence of pore pressure on viscous deformation was not an explicit feature, although this behavior has been experimentally documented. An additional shortcoming of existing models was the large number of material parameters included in the models (some have more than 20 material parameters). With this large number of material parameters, ascertaining the physical role a particular parameter has on the material deformation is very difficult. Additionally, when fitting parameters to existing models, we found equivalent model responses could be obtained with varying sets of parameter values, that is, the response of is non-unique. Because of this, relating an experimentally observed characteristic to a material parameter was impossible.

The constitutive model presented here was developed to achieve the following goals: (1) an accurate prediction of experimentally observed consolidation at conditions representative of a subsurface repository, (2) utilization of basic poromechanics principals to predict material deformation, and (3) limiting the number of material-specific parameters that would need to be “fit” to experimental data. Unlike most existing constitutive models for intact and granular salt, the development of this model does not rely upon an accurate description of presumed micromechanisms; rather, the model described herein is based on a basic understanding of mechanics of porous media. Additionally, material parameters included in the model can be fit to experimentally measured bulk volumetric deformations of granular salt. The model is shown to capture the dependence of viscous behavior on pore pressure as well as account for temperature effects and hardening due to porosity reduction from consolidation. The developed model also has the attribute of requiring many fewer parameters than other existing constitutive models for granular salt consolidation.

1.4 Project problems and challenges

A significant problem that the project encountered was difficulties in conducting the consolidation testing. In particular, we had numerous jacket failures during the consolidation testing that slowed our progress but did not prevent us from successfully completing our planned tests. These problems set the project nominally 6 months behind the initial proposed schedule.

Another issue has been numerical implementation of the numerical model to implement a constitutive model that includes the role of pore pressure. The challenges were a result of the large amount of strain experienced during the consolidation tests.

1.5 Departures from original plan

The original plan involved applying triaxial stresses in addition to hydrostatic stress conditions during consolidation testing. We did not conduct triaxial tests beyond some preliminary testing because: (1) the control of the axial load for the triaxial stress conditions was very difficult to maintain with the level of accuracy needed for these tests, (2) the permeability measurements during consolidation were more straightforward under the configuration used for hydrostatic stress conditions, and (3) we judged that the hydrostatic tests were more important because the degree to which significant triaxial stresses are expected under field conditions is not certain.

Another significant departure from the original plan concerns the constitutive model for granular salt consolidation. In the original plan, we planned to modify the model of Callahan. However, we found that the Callahan model was extremely difficult to work with because of the large number of parameters associated with it. Parameter fitting of consolidation data to the Callahan model suggested that equivalent model responses could be obtained with different sets of parameters. Further, the Callahan model was originally developed to be consistent with and represent consolidation mechanisms. However, it was not possible to associate parameter values with actual mechanisms. Finally, because of the extremely complicated and involved nature of the model, it was not possible to include pore pressure in a straightforward manner. Consequently, we opted to develop a very simple model that has no deformation mechanism specific interpretation associated with it.

1.6 Training and professional development

There has been much training of personnel on this project. The consolidation testing was principally being conducted at Sandia National Laboratories Geomechanics Laboratory. The first priority for UNM personnel to work in this laboratory was to undergo comprehensive safety training. Once safety trained, there was considerable instruction and guidance from SNL personnel on the fabrication of samples, the use of the creep load frame, and data acquisition and

control system. This instruction represents a significant benefit to the project participants as they learn to conduct experiments in this world-class laboratory.

The microstructural observations involve the use of sophisticated and specialized equipment (e.g., scanning electron microscopes) along with specific sample preparation procedures. UNM personnel have undergone considerable training on these equipment and procedures. In addition, students were being instructed by SNL personnel on how to interpret deformation mechanisms from these observations and images.

Project personnel also received training on ABAQUS software, a general purpose finite element code and LABVIEW, software for laboratory data acquisition.

UNM personnel have been involved in review and development of laboratory safety procedures for activities in the UNM geotechnical laboratories.

The following graduate students were supported on this project. All of the students were in the Civil Engineering Department at the University of New Mexico. Their degrees and completion dates are given below:

- Laxmi Paneru – MS, 2016
- Melissa Mills – MS, 2016
- Aayush Piya – MEng, 2017
- Brandon Lampe – PhD, 2017 (anticipated December, 2017)
- Timothy Lynn – MS, 2017 (anticipated December, 2017)

1.7 Collaborations

This was a collaborative project between the University of New Mexico (UNM) and Sandia National Laboratories (SNL). Stephen Bauer was the principal collaborator at SNL. UNM directed the project, including overseeing graduate students working on the project. Existing facilities at SNL were used for the salt consolidation testing, and SNL personnel are providing direction on experimental methods.

There was also collaboration with Mechanical Engineering faculty from UNM and Washington State University regarding constitutive model development.

1.8 Publications

Milestone reports

Stormont, J.C., B. Lampe, T. Lynn and A. Piya (2014) Update on the existing constitutive model for granular salt consolidation, Report prepared by the University of New Mexico for the Department of Energy, under Contract DE-NE0000733, NEUP 13-4834 Improving the understanding of the coupled thermal-mechanical-hydrologic behavior of consolidating granular salt, October 31, 2014.

Stormont, J.C., B. Lampe, M. Mills, L. Paneru, and T. Lynn (2015a) The role of moisture in the consolidation of granular salt. Report prepared by the University of New Mexico for the Department of Energy, under Contract DE-NE0000733, NEUP 13-4834 Improving the understanding of the coupled thermal-mechanical-hydrologic behavior of consolidating granular salt, May, 2015.

Stormont, J.C., B. Lampe, M. Mills, L. Paneru, and T. Lynn (2015b) Observations of permeability changes from granular salt consolidation. Report prepared by the University of New Mexico for the Department of Energy, under Contract DE-NE0000733, NEUP 13-4834 Improving the understanding of the coupled thermal-mechanical-hydrologic behavior of consolidating granular salt, July 15, 2015.

Stormont, J.C., B. Lampe, M. Mills, L. Paneru, T. Lynn and A. Piya (2016a) Assessment of preconsolidation to achieve high fractional densities. Report prepared by the University of New Mexico for the Department of Energy, under Contract DE-NE0000733, NEUP 13-4834 Improving the understanding of the coupled thermal-mechanical-hydrologic behavior of consolidating granular salt, March 15, 2016.

Stormont, J.C., B. Lampe, L. Paneru, T. Lynn and A. Piya (2017a) Coupled behavior at high fractional densities, Report prepared by the University of New Mexico for the Department of Energy, under Contract DE-NE0000733, NEUP 13-4834 Improving the understanding of the coupled thermal-mechanical-hydrologic behavior of consolidating granular salt, March 31, 2017.

Stormont, J.C., B. Lampe, T. Lynn and A. Piya (2017b) Constitutive model for granular salt consolidation, Report prepared by the University of New Mexico for the Department of Energy, under Contract DE-NE0000733, NEUP 13-4834 Improving the understanding of the coupled thermal-mechanical-hydrologic behavior of consolidating granular salt, May 15, 2017.

Annual reports

Stormont, J.C., B. Lampe, M. Mills, L. Paneru, and T. Lynn (2015c) Annual progress report for the period January 15, 2014 to January 15, 2015. Report prepared by the University of New Mexico for the Department of Energy, under Contract DE-NE0000733, NEUP 13-4834 Improving the understanding of the coupled thermal-mechanical-hydrologic behavior of consolidating granular salt, May 18, 2015.

Stormont, J.C., B. Lampe, M. Mills, L. Paneru, T. Lynn, and A. Piay (2016b) Annual progress report for the period January 15, 2015 to January 15, 2016. Report prepared by the University of New Mexico for the Department of Energy, under Contract DE-NE0000733, NEUP 13-4834 Improving the understanding of the coupled thermal-mechanical-hydrologic behavior of consolidating granular salt, April 13, 2016.

Stormont, J.C., B. Lampe, M. Mills, L. Paneru, T. Lynn, and A. Piay (2017c) Annual progress report for the period January 15, 2016 to January 15, 2017. Report prepared by the University of New Mexico for the Department of Energy, under Contract DE-NE0000733, NEUP 13-4834 Improving the understanding of the coupled thermal-mechanical-hydrologic behavior of consolidating granular salt, May 3, 2017.

Papers (completed)

Bauer, S.J., S.T. Broome, F.D. Hansen, B. Lampe, M. Mills, and J. Stormont, 2015b, Gas flow measurements of consolidating crushed salt, *49th US Rock Mechanics/Geomechanics Symposium*, San Francisco, CA, USA.

Mills, M., F. Hansen, S. Bauer, and J. Stormont, 2015, “Microscopic evidence of grain boundary moisture during granular salt reconsolidation,” *Annual Meeting of the American Geophysical Union*, San Francisco, December.

Stormont, J.C., Lampe, B.L., Lynn, T.D., and Bauer, S.J., 2017d, Gas permeability of granular salt during consolidation, *51st US Rock Mechanics/Geomechanics Symposium*, San Francisco, CA, USA.

Lampe, B.C., Stormont, J.C., Lynn, T.D, Bauer, S.J., 2017a, Influence of Pore Pressure on the Reconsolidation of Crushed Salt Under Hydrostatic Creep Conditions, *51st US Rock Mechanics/Geomechanics Symposium*, San Francisco, CA, USA.

Lampe, B.C., Stormont, J.C., Lynn, T.D, Bauer, S.J., 2017b, Potential Influence of Granular Salt on Cavern Stability, *SMRI Spring 2017 Technical Conference* 24 – 25 April 2017, Albuquerque, New Mexico, USA

Papers (in preparation)

Paneru, L., Bauer, S.J., and Stormont, J.C., Thermal properties of consolidated granular salt, *Rock Mechanics and Rock Engineering*. In revision.

Mills, M., F. Hansen, S. Bauer, and J. Stormont, Micromechanical Processes in Consolidated Granular Salt, *Tectonophysics*, In review.

Lampe, B., Stormont, J., Schreyer, H. Constitutive model for shear behavior under hydrostatic conditions, *International Journal of Rock Mechanics and Mining Science*. In preparation.

Lampe, B., Stormont, J., Bauer, S.J. Granular salt consolidation including pore pressure, *International Journal of Rock Mechanics and Mining Science*. In preparation.

2 Introduction

This report provides the final report for the NEUP project “Improving the understanding of the coupled thermal-mechanical-hydrologic behavior of consolidating granular salt.”

The goal of this project is to improve the understanding of key aspects of the coupled thermal-mechanical-hydrologic response of granular (or crushed) salt used as a seal material for shafts, drifts, and boreholes in mined repositories in salt. The project is organized into three tasks to accomplish this goal: laboratory measurements of granular salt consolidation (Task 1), microstructural observations on consolidated samples (Task 2), and constitutive model development and evaluation (Task 3). Task 1 involves laboratory measurements of salt consolidation along with thermal properties and permeability measurements conducted under a range of temperatures and stresses expected for potential mined repositories in salt. Testing focused on the role of moisture, temperature and stress state on the hydrologic (permeability) and thermal properties of consolidating granular salt at high fractional densities. Task 2 consists of microstructural observations made on samples after they have been consolidated to interpret deformation mechanisms and evaluate the ability of the constitutive model to predict operative mechanisms under different conditions. Task 3 concerns the development of the coupled thermal-mechanical-hydrologic constitutive model for granular salt consolidation. The measurements and observations in Tasks 1 and 2 were used to develop a thermal-mechanical constitutive model. Accomplishments and status from each of these efforts is reported in subsequent sections of this report.

An important aspect of this project was the collaboration with Sandia National Laboratories (SNL). The consolidation testing was conducted at SNL’s Geomechanics Laboratory. In addition, some of the microscopy work was conducted at SNL. Moreover, SNL personnel were involved in the training and instruction of UNM students working on this project. The use of SNL facilities and the collaboration with their personnel provided an extraordinary opportunity to leverage their unique capabilities and experiences.

Overall, the achieved progress met the planned work and achievements.

3 Consolidation testing

A principal focus of the project was conducting consolidation tests on granular salt under a range of conditions (temperature, moisture, stress). This testing provides the data for the evaluation and parameterization of constitutive models of granular salt consolidation and permeability changes. In addition, sub-samples from post-test consolidation tests have been used for thermal property measurement, porosity measurements, permeability measurements and microstructural observations.

The consolidation tests were conducted at the Sandia National Laboratories Geomechanics Laboratory, and utilized with some modifications of methods and equipment previously developed for salt consolidation testing. The test methodology for the consolidation testing was largely developed in the first two years of the project and modified in the third year. In order for UNM personnel to be able to work in the SNL laboratory, they first had to undergo comprehensive safety training. Additional work was conducted at the UNM Geotechnical Laboratory.

The tests were conducted in three groups or series (Table 3.1). Each series was targeted toward obtaining data that addresses a somewhat different objective. Project milestone reports include description of and data from consolidation tests as given in Table 3.1.

Table 3.1 – Summary of consolidation test series.

Series	Principal factor being investigated	Dates	Associated project milestone reports
1	The role of moisture	11/14 to 04/15	Stormont et al., 2015a Stormont et al., 2015b
2	Preconsolidation	06/15 to 08/15	Stormont et al., 2016a
3	Pore pressure effects	10/15 to 2/17	

Results from testing of post-consolidated samples, including thermal properties, permeability and microstructural observations are described in Chapter 4.

3.1 Methods and materials

3.1.1 Materials processing

Two types of granular salt have been procured for consolidation testing. One source is the Waste Isolation Pilot Plant (WIPP) facility in Southeastern New Mexico. This salt is from a bedded salt formation. The other source is the Avery Island mine in Louisiana, which is a domal salt formation.

Upon receipt at the Geotechnical Laboratory at UNM, sample sizes of 1700 g were created for each salt type. Particles larger than 9.5 mm were excluded from the samples. Some salt had to be dried in response to crusting during times with high humidity. The particle size distribution of each sample was measured and recorded. Samples were stored in sealed metal storage containers, to be subsequently transferred to the SNL laboratory as needed. There are 40 individual sample lots that have been prepared in this manner.

A procedure was developed to add about 1% water to granular salt. The goal of the procedure is to ensure that the additional water is well distributed throughout the granular salt mass. The procedure developed utilized the concrete curing room in UNM's Civil Engineering Department, which is kept at 22 °C and 100% humidity. Samples of granular salt were oven dried for 24 hours at 110°C before beginning the wetting process. Samples were then separated onto four trays lined with release film, weighed, and placed inside a plastic container which was moved into the concrete curing room. The container was fitted with two small battery powered fans to circulate the air. Through numerous trials, it was determined that to achieve a 1% water content for 1,000 grams sample, 45 minutes was required for Avery Island salt and 40 minutes for WIPP salt. To produce salt samples large enough to be used in constructing samples for subsequent consolidation tests, nominally 1,700 grams of salt was distributed evenly over the four trays, and the dry weights of each of the four trays was recorded. The wetting container was then placed in the curing room for the assumed necessary amount of time. The container was then removed and each tray was weighed and the moist weights were recorded. The initial dry weight of the salt, tray, and film were compared to the moist weights to establish an estimated water content and determine if additional time in the curing room was necessary. The estimated water content was generally 0.1-0.2% higher than the actual water content. Once the target estimated water content

was achieved the contents of each of the four trays was placed inside a freezer bag and sealed. Four control samples of 25-30 grams were removed from the bag and placed in a moisture can, weighed and oven dried to determine the water content for the entire sample.

3.1.2 Sample construction

Samples were constructed in “jackets” to provide confinement and isolation from the confining pressure fluid. Because these tests were conducted at elevated temperatures, special jacketing materials were needed for some tests. During the initial Stage 1 testing, there were two basic configurations. For tests above 100 °C, the samples were prepared with an inner lead sheet, a copper sheet and an outer lead jacket. This system was developed in order to maintain confinement while the sample experienced very large deformations under high temperatures (Broome et al., 2014). For samples tested below 100 °C, the samples were prepared with an inner copper sheet, a lead sheet, and a viton outer jacket. For all types of samples, porous disks, chamfered end pieces and end caps on both ends complete the assembly. Figure 3.1 is a general schematic of a prepared sample.

Viton jackets were not used on any samples beginning with Test Series 2 because they are not compatible with the preconsolidation test stage. Viton cannot be used for tests that have to be removed from the test frame after pre-consolidation as it loses continuous contact with the sample (Figure 3.2). The initial tests in Test Series 2 used two lead sheets with an inner copper sheet. Subsequently, all samples have been prepared with a single lead sheet and two copper sheets.

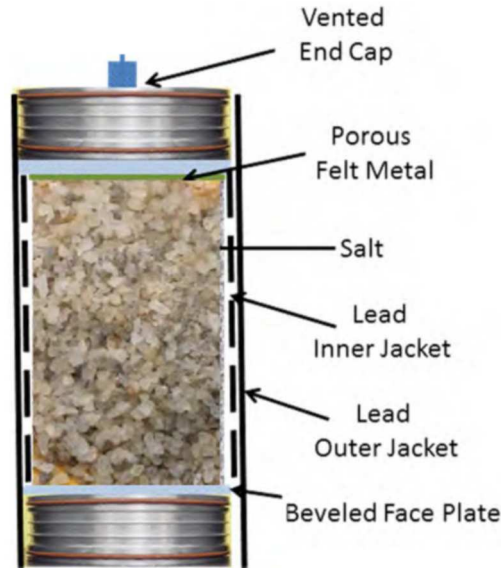


Figure 3.1 - – Generalized schematic of granular salt samples prepared for consolidation testing (Broome et al., 2014) at temperatures up to 250 °C. Different combinations of copper and lead have been used as jacketing material. All samples tested included a vented bottom end cap on the bottom similar to that shown for the top.



Figure 3.2 - Viton jacket after initial consolidation showing how it wrinkled and lost complete contact with the sides of the granular salt sample.

Beginning with Test Series 2, samples were modestly compacted as the granular salt was being emplaced in the jacket system. Samples are constructed with four lifts of salt, each receiving ten

blows from a 1.54 kg hammer. Each blow is nominally 12 to 30 cm of fall with the 1.54 kg hammer. This compaction gives a sample with an average initial fractional density of 0.70.

A test of a blank lead jacket assembly revealed that the lead jacket readily deformed at ambient temperature under as little as 35 kPa. It is concluded that the lead provides no significant resistance to consolidation and can be used for all tests if desired.

The initial tests in Test Series 2 used two lead sheets with an inner copper sheet. Subsequently, all samples have been prepared with a single lead sheet and two copper sheets.

3.1.3 Consolidation testing

3.1.3.1 *Mechanical system*

The system for conducting the consolidation tests includes a creep load frame and pressure vessel to apply the axial and confining pressures, respectively. A photograph of the creep load frame with the pressure vessel loaded in it is given in Figure 3.3. The pressure vessel has band heaters on its outside to allow the sample temperature to be elevated. Thermocouples are used to measure the confining fluid pressure. The confining pressure was controlled by a dilatometer and/or a pump that allowed its pressure to be monitored and adjusted.



Figure 3.3 – Photograph of creep load frame in the SNL Geomechanics Laboratory with pressure vessel (steel cylinder directly behind caution sign).

A data acquisition and control system was used to monitor and record confining pressures, axial load, axial displacements, lateral displacements and temperature (Figure 3.4). The data acquisition system was modified to allow some data for permeability measurement to be recorded (gas pressure and flow rate).



Figure 3.4 – Photograph of data acquisition and control system.

3.1.3.2 Analysis of consolidation data

The majority of the experimental data associated with these tests were collected and recorded by the data acquisition system. In some tests, certain parameters may not have been recorded due to instrument failure, instruments exceeding their range, problems with the data acquisition system, or other factors.

A principal parameter of interest is the sample's fractional density or porosity. The porosity and fractional density are related one-to-one as shown below:

$$\phi = 1 - \rho_f \quad (3.1)$$

where ϕ is the porosity and ρ_f is the fractional density of the specimen. The porosity is determined from the volumetric strain experienced during consolidation which is estimated from the changing sample dimensions during the test. Because the volume change of non-salt components was negligible, all volume changes were that of the salt specimen. Additionally, the deformation of granular (or intact) salt under a hydrostatic stress is isotropic, therefore the same deformation occurred in all three spatial dimensions.

In Test Series 1, the LVDT data were used to interpret axial strain. When Schuler gages were used to measure lateral deformation, their data could be used to obtain lateral strain. Volumetric strain can be obtained from the dilatometer or pump data used to control the confining pressure. Because these were intended to be hydrostatic tests, the assumption of homogeneous deformation is generally reasonable. In this way, measures of linear strain can be used to interpret lateral strain and vice versa. This assumption was necessary in cases where there were difficulties with a particular measurement, such as the Schuler gages deforming beyond their calibrated range. Because the dilatometer system uses a small volume to allow for precise measurements of small volumetric strains, another issue was the difficulty of measuring relatively large and rapid volumetric strains during heating or pressure increases.

The issues described above led to changes in the test conduct and data collection beginning with Test Series 2. An initial consolidation stage (or “preconsolidation”) was included in the test procedure for all tests beginning with Test Series 2 (described in Section 3.2). Additional measurements were made of pre- and post-test sample dimensions to confirm consolidation data. We determined the as-built sample fractional density from external measurements of the sample. The mass and volume of all non-salt components used to encase the specimen was determined prior to sample construction. After construction, the mass was measured and the height and width of the sample was measured. The net as-built fractional density was determined by subtracting the known contribution of the non-salt components to the mass and volume. After the initial consolidation stage, we determined the fractional density from the fluid immersion technique and in some cases from external measurements as well.

In Test Series 2 and 3, the volumetric deformation was calculated from the measured radial deformation. Radial deformation was determined using a pair of Schuler gages on two orthogonal planes at the sample’s mid-height. Because the axial piston was not used, LVDTs could not be used. All volumetric deformation measured by the Schuler gages was attributed to changes in pore volume, and from this a change in pore volume along with the initial porosity, the porosity was determined throughout the test. Additionally, because the salt was consolidated under constant stress, no elastic deformation occurred once at the desired confining pressure. As the sample consolidated, its volume decreased and additional confining fluid was metered into the test frame to maintain the confining pressure. The volume of metered fluid was equivalent to

the volume change of the sample at constant pressure and therefore equivalent to the change in pore volume. Then, the volume of fluid needed to maintain a constant confining pressure equaled the change in pore volume, and from this a change in porosity was also calculated. This second method of porosity calculation provided quality assurance for the transient salt porosity measurement. After the consolidation test, fluid immersion and external measurements were made on the sample to obtain estimates of fractional density of the entire sample.

With some tests in Test Series 2, the interpreted fractional density history during the consolidation test was adjusted in order to be consistent with fractional densities found on the samples post-test. The adjustment was accomplished by small changes in sample dimensions and/or in the fluid compressibility. These adjustments are reported elsewhere (Stormont et al., 2016a). For Test Series 3, the porosity of the samples was measured after consolidation using a commercial device (Corelok). These measurements were made on the entire sample, the central core and/or end pieces. The fractional density from these measurements were compared to those interpreted from the final measured volumetric strain during the consolidation test. The volumetric strain data were adjusted to so as the final porosity during the consolidation test was equal to that measured using the Corelok device. In Test Series 3, the volume strain data were interpreted from the Schuler gage data, either as an average of the two gages or from a single gage is one went off-line during the test.

3.1.4 Permeability system

Some samples prepared without additional water were subjected to gas permeability measurements concurrent with the consolidation test. Because the permeability through consolidating salt is expected to decrease over many orders of magnitude as the porosity changes from around 40% to <1%, different measurement systems were used.

Flow bench - The flow bench is a portable self-contained unit that includes a suite of flowmeters connected in parallel along with a power supply/signal conditioning unit and a data display (Figure 3.5). The flowmeters each have a different range: 0-10, 0-50, 0-500, 0-4000, and 0-20,000 SCCM. In this way, the flow bench effectively can measure flowrates from 20 SLM to about 1 SCCM. The flow bench is intended for steady state flow tests.



Figure 3.5 - Flow bench used to measure steady state flow rates during constant pressure gas flow tests.

Gas is supplied from a gas cylinder through a regulator at a constant pressure to the upstream vent line on the sample. The downstream vent line is connected to the flow bench. The appropriate flowmeter is selected by the following procedure. Beginning with the highest capacity flowmeter, the displayed flow is observed until it stabilizes. If the flow is less than about 10% of the full range of the meter, the next lower range flowmeter is selected and process is repeated until the flow falls within the range of the selected meter (nominally within 10% and 90% of full range of the flowmeter). Flows below about 1 SCCM cannot be measured with the flow bench.

Differential pressure permeameter - The differential pressure (DP) permeameter is intended to measure flows below the range of the flow bench. The permeameter consists of an upstream volume (connected to the upstream of the sample) and a downstream volume (connected to the downstream side of the sample). A differential pressure gage measures the pressure difference between the upstream and downstream volumes. The upstream side includes a separate reservoir that can be included in the upstream volume. The DP permeameter is shown in Figure 3.6.

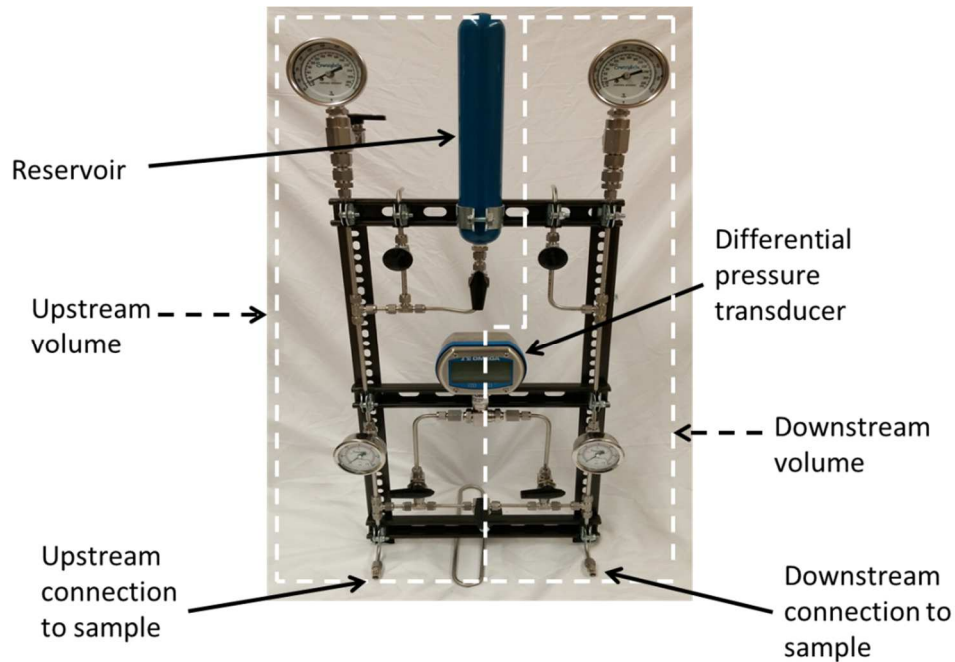


Figure 3.6 – Differential pressure permeameter. Dashed separate the upstream and downstream volumes of permeameter.

The DP permeameter can be utilized in steady state flow tests. In this configuration, the upstream volume is pressurized by means of an external gas supply (pressurized gas cylinder) and then isolated from the gas supply. The downstream is maintained at a constant pressure (typically atmospheric pressure). The pressure in the upstream volume will decrease in response to gas movement through the sample. For sufficiently small flows through the sample, the flow rate can be interpreted from the known upstream volume and its rate of pressure decrease.

The DP permeameter can also be utilized in transient flow tests. In these tests, the upstream and downstream pressures are first equilibrated. The upstream pressure is then increased by nominally 10%, and the decay of the pressure difference is monitored with time as gas flows from the upstream to the downstream. This technique can be used to interpret permeabilities in the range of 10^{-21} m^2 .

The DP permeameter was used in some tests in Test Series 2. This permeameter was also used to conduct permeability testing on post-consolidated samples in the UNM Geotechnical Laboratory.

Helium leak detector permeability system - For extremely low flow rates and corresponding permeabilities, a helium leak detection permeability system (Bauer et al., 2015a) was used. This technique relies on the ability of a mass spectrometer to detect the arrival of helium at very low rates. Helium is supplied to the upstream side of the sample, and a vacuum is pulled on the downstream side. The gas pulled from the sample is run through the mass spectrometer which has been calibrated to helium so that flow rates can be interpreted directly from the mass spectrometer response.

Constant pressure flow tests conducted with the flow bench directly provide a flow rate that can be used with Darcy's law to interpret permeability. Pressure fall-off tests are quasi-steady-state tests conducted with the differential permeameter system. In these tests, the flow rate from the upstream volume of the permeameter is interpreted from the pressure change with time.

Transient testing was not conducted with the DP permeameter. Data to develop the Klinkenberg correction were not obtained in the testing done during consolidation.

3.2 Test Conduct

3.2.1 Test Series 1

Test series 1 was directed at understanding the role of moisture on consolidation and subsequent properties. Table 3.2 provides a summary of tests included in this test series.

Table 3.2 – Summary of consolidation tests included in Test Series 1.

Sample label	Salt type	Temp	Stress conditions	Moisture added	Frame	Dates	Comments
UNM-WP-HY-250-01	WIPP	250	Hydrostatic 20 MPa	No	A2	11-18-14 12-1-14	
UNM-WP-HY-250-02	WIPP	250	Hydrostatic 20 MP	No – not vented	A2	1-22-15 2-4-15	
UNM-WP-HY-90-01	WIPP	90	Hydrostatic 20 MPa	No	A3	12-18-14 4-9-15	
UNM-WP-HY-90-02	WIPP	90	Hydrostatic 20 MPa	1%		4-22-15 5-8-15	
UNM-AI-HY-250-01	AI	250	Hydrostatic 20 MPa	No	A2	3-16-15 3-23-15	
UNM-AI-HY-250-02	AI	250	Hydrostatic 20 MPa	1%		4-1-15 4-9-15	

The tests involved in Test Series 1 were conducted in a generally similar manner. After the sample was placed in the test frame, 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. The axial stress was adjusted to maintain a small differential stress to ensure that the end caps remained in contact with the actuator piston. Note that in these tests, the axial stress and lateral stress are controlled separately; the axial stress is generated from a hydraulic ram which applies a load to the top and bottom of the sample, whereas the lateral stress is generated by pressurizing silicone oil that surrounds the sample. Next, confining pressure was increased to the range of 1 to 2 MPa while maintaining the small 0.2 to 0.3 MPa differential axial stress. The temperature was then increased to either an intermediate value or the desired final value. Finally, at some time after reaching the final temperature, the confining pressure and axial stresses were increased to achieve the target mean stress (20 MPa). As the stress conditions were intended to be hydrostatic, the goal was to control confining pressure and axial stress so they were as similar as possible. In the case of samples without additional moisture and were drained, gas permeability measurements were periodically made as the samples experienced consolidation

from the stress and temperature conditions. Tests durations depended on the test conditions and objectives. Tests were ended by first reducing the temperature and then reducing the mean stress.

3.2.2 Test Series 2

Ten tests focused on hydrostatic consolidation were conducted using the improved test methods described previously. Test conditions are described in Table 3.3. Six of ten tests were completed with the initial consolidation stage and the subsequent hydrostatic consolidation (creep) stages. Four of the tests failed during the initial consolidation testing; most of these failures were jacket failures.

Table 3.3 – Summary of consolidation tests included in Test Series 2.

Sample label	Salt type	Temp	Stress conditions	Moisture added	Frame	Dates (2015)	Comments
UNM-WP-HY-175-01	WIPP	175	Hydrostatic 20 MPa	No	A2	6/17 - 6/19	Possible jacket leak
UNM-WP-HY-90-03	WIPP	90	Hydrostatic 20 MPa	1%	A3	6/25 – 6/26	Jacket failure during ic
UNM-WP-HY-90-04	WIPP	90	Hydrostatic 20 MPa	1%	A3	7/02 - 7/07	
UNM-WP-HY-175-02	WIPP	175	Hydrostatic 20 MPa	No	A2	6/26 – 7/8	Failed after ic
UNM-WP-HY-90-05	WIPP	90	Hydrostatic 20 MPa	1%	A3	7/16	Jacket failure during ic
UNM-WP-HY-175-03	WIPP	175	Hydrostatic 20 MPa	1%	A2	7/15- 7/17	Possible jacket leak
UNM-WP-90-06	WIPP	90	Hydrostatic 20 MPa	1%	A2	7/23	Jacket failure during ic
UNM-WP-HY-90-07	WIPP	90	Hydrostatic 20 MPa	1%	A3	7/24 – 8/17	
UNM-WP-175-04	WIPP	175	Hydrostatic 20 MPa	No	A3	7/24 – 8/31	
UNM-WP-HY-90-08	WIPP	90	Hydrostatic 40 MPa	No	A3	7/29 – 8/7	

The test procedure for Test Series 2 was substantially different than that for prior tests. A principal difference was the conduct of a “preconsolidation” stage that preceded the hydrostatic

creep test. This initial testing stage was performed to reduce the porosity or increase the fractional density of the sample prior to the hydrostatic creep test. The jacketed sample was suspended from the top of the frame, and did not contact the axial piston at all. The confining fluid was silicone oil. The hydrostatic stress varied from 19 to 38 MPa. No deformation was measured during this stage, and the temperature was the ambient temperature (nominally 27 °C). The loading was accomplished in 5-10 minutes time, after which the sample was rapidly unloaded. The sample was removed from the test frame, and its volume was obtained by fluid immersion.

The hydrostatic creep test was considered to start after the preconsolidation stage. The test sample was again suspended from the top of the frame, and did not contact the axial piston at all. In order to accomplish this and allow for optional gas flow testing during consolidation, a long nipple arrangement connected to the sample's bottom end piece was fabricated and used. The long nipple is 24 cm long and allows the sample to travel with deformation while still maintaining the seal at the bottom of the frame. Schuler gages were applied to the sample to measure lateral deformation during creep.

The confining fluid (silicone oil) was first brought to temperature while the pressure vessel was vented to avoid applying a stress to the sample during this step. Next, the vessel was shut-in and the confining pressure was increased to the target pressure by means of a pump. The volume of oil necessary to reach and maintain the confining pressure was recorded, along with the response of the Schuler gages along with the confining fluid temperature.

3.2.3 Test Series 3

Fourteen hydrostatic creep consolidation tests were performed during Test Series 3 (Table 3.4). The principal purpose of these tests was to investigate how pore pressure affects consolidation. Many of these tests were multi-stage tests, meaning that during the test conduct the pore pressure and sometime confining stress were changed. The majority of the tests were conducted at 175 C, and none of these tests included added water. Five of the tests failed before producing any useful data, whereas two tests failed after providing some useful data. One test included concurrent permeability testing, however, a leak in the gas line precluded collecting useful data.

Table 3.4 – Summary of consolidation tests included in Test Series 3

Sample label	Salt type	Temp	Stress conditions (max values shown for multi-stage tests)	Moisture added	Frame	Dates (2016 unless noted)	Comments
UNM-WP-HY-90-09	WIPP	90	Hydrostatic 38 MPa conf 8 MPa pore	No	A2	10/16 - 10/31/15	Burst disk failure
UNM-WP-HY-175-05	WIPP	175	Hydrostatic 38 MPa conf	No	A3	11/10- 11/14/15	Jacket failure during heating
UNM-WP-HY-175-06	WIPP	175	Hydrostatic 38 MPa conf 8 MPa pore	No	A3	11/22- 11/30/15	Leak in confining pressure led to failure
UNM-WP-HY-175-07	WIPP	175	Hydrostatic 22 MPa conf	No	A2	12/21- 12/28/15	Jacket leak
UNM-WP-HY-175-08	WIPP	175	Hydrostatic 20 MPa conf	No	A3	2/4-2/5	Failed
UNM-WP-HY-175-09	WIPP	175	Hydrostatic 38 MPa Conf 8 MPa Pore	No	A3	4/6-6/15	Concurrent perm testing
UNM-WP-HY-175-10	WIPP	175	Hydrostatic 30 MPa Conf 10 MPa Pore	No	A3	7/26-8/1	Leak in pore pressure line

UNM-WP-HY-175-11	WIPP	175	Hydrostatic 30 MPa Conf 25 MPa Pore	No	A2	8/9-8/18	
UNM-WP-250-03	WIPP	250	Hydrostatic 38 MPa conf	No	A3	8/17-8/26	
UNM-WP-HY-175-12	WIPP	175	Hydrostatic 20 MPa Conf 18 MPa Pore	No	A2	8/29-9/8	
UNM-WP-HY-175-13	WIPP	175	Hydrostatic 30 MPa Conf 22.5 MPa Pore	No	A2	9/28-10/26	
UNM-WP-175-14	WIPP	175	Hydrostatic 20 MPa Conf 15 MPa Pore	No	A2	11/21	Failed
UNM-WP-175-15	WIPP	175	Hydrostatic 20 MPa Conf 0-15 MPa Pore	No	A2	12/20-1/12/17	
UNM-WP-175-16	WIPP	175	Hydrostatic 20 MPa conf	No	A2	2/3/17-2/21/17	

The multistage creep test procedure was developed to test samples at varying confining pressures along with the introduction of pore pressure to monitor how changes in effective stress affect the consolidation process. The sample preparation and the test frame configuration remain largely the same as previous hydrostatic tests allowing gas flow through the sample with an important addition; after the sample is installed in the frame the pore gas line is checked with a vacuum

pump to ensure that the system connections will hold a gas pressure. The initial consolidation of samples in Test Series 3 uses helium as a confining fluid. This allows for the salt samples to be reused given a minor failure during initial consolidation; in contrast, if hydraulic oil is used as the confining fluid during the initial consolidation, a leak into the sample will irrevocably alter the sample.

For multi-stage tests, fluid pressures were increased at a rate of 4 MPa/min until the desired creep test pressures were achieved. Samples were typically allowed to consolidate for nominally 24 hours or more to allow a constant consolidation rate to develop. The confining stress and pore pressure changes in the multi-stage tests are shown in the data for these tests in the results section of this chapter.

During Test Series 3, a sample failed at the interface of the aluminum endcap and the steel treads of the screw that connects the nipple to the endcap. The threaded connection was abandoned for a bolt that passes through the chamfered plate and endcap into the nipple providing a much stronger connection. This change however required slight changes in the sample preparation as the chamfered plate, endcap, and nipple act as a single assembly.

Throughout the test series modifications were made to the pore gas line adding instrumentation and additional safety measures. The pore gas assembly consists of valves to isolate the sample from the gas supply and atmosphere, thermocouples to monitor the pore gas temperature entering and leaving the sample, burst discs to further protect the pore gas line from contamination in the event of a jacket leak, and a pressure transducer to monitor the pore gas pressure. A Teflon block was designed and machined to partially enclose the downstream pore gas assembly in order to protect the test frames actuator (which is not in use) from the impact of the downstream assembly, as well as to direct the assembly and any fluid discharged in a specified direction in the event of a failure. The pore gas system is shown in Figure 3.7, independent of the confining pressure cell and load frame.



Figure 3.7 – Pore gas line assembly showing current configuration

3.3 Test results and discussion

3.3.1 Test Series 1

In this section, some of the data obtained during the Test Series 1 consolidation testing and concurrent gas permeability testing are presented and discussed. Detailed results from consolidation testing in this test series are given in previous milestone reports (Stormont et al., 2015a, 2015b).

3.3.1.1 *Results*

In Figures 3.8 through 3.13, the fractional density increase with time is given for the samples tested in Test Series 1 along with the mean stress and temperature history. All samples significantly consolidated (that is, the fractional density increased) in response to increases in mean stress and temperature.

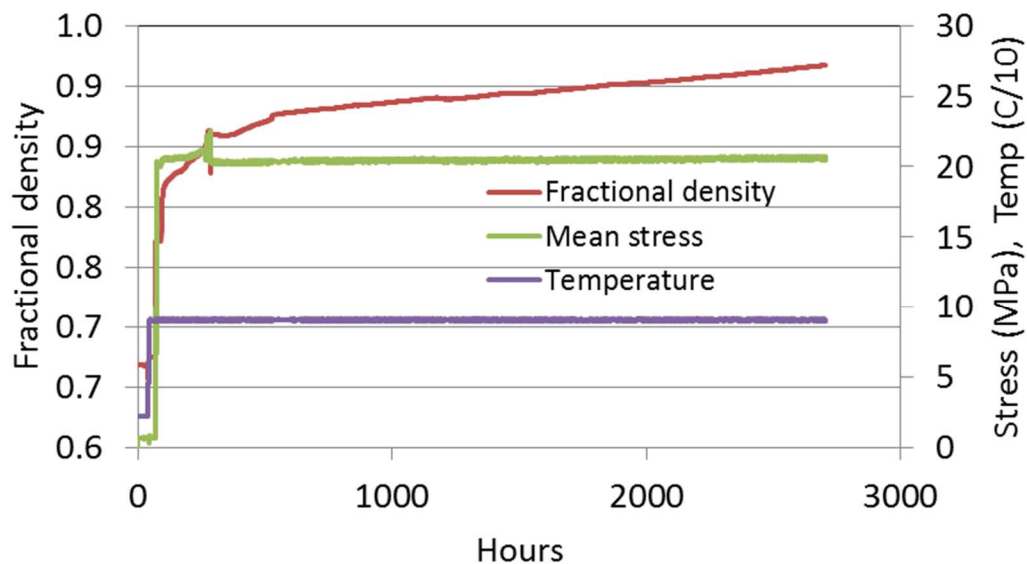


Figure 3.8 – Summary of consolidation test for UNM_WP_HY_90_01.

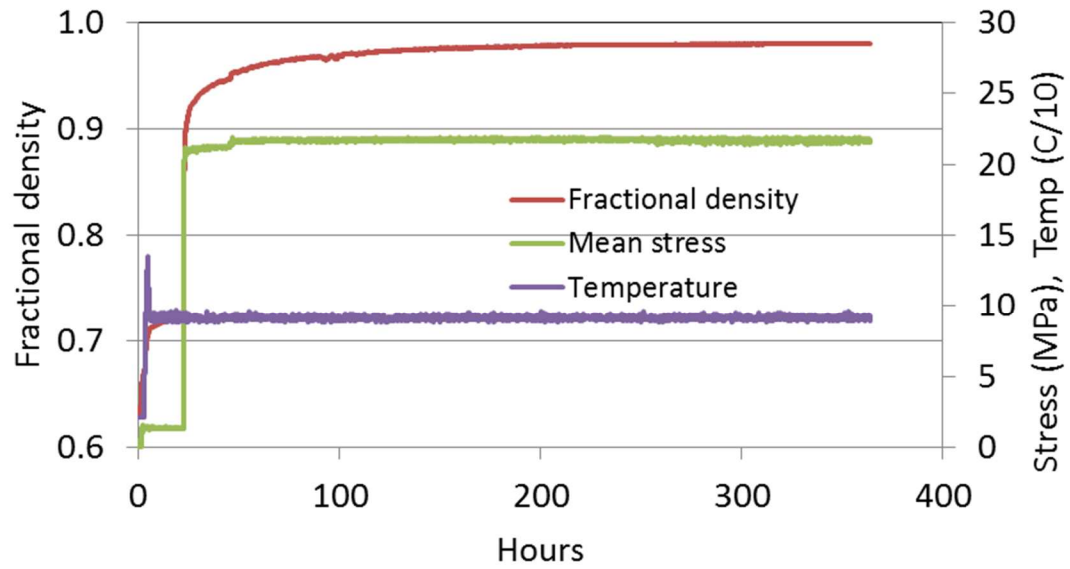


Figure 3.9 – Summary of consolidation test for UNM_WP_HY_90_02.

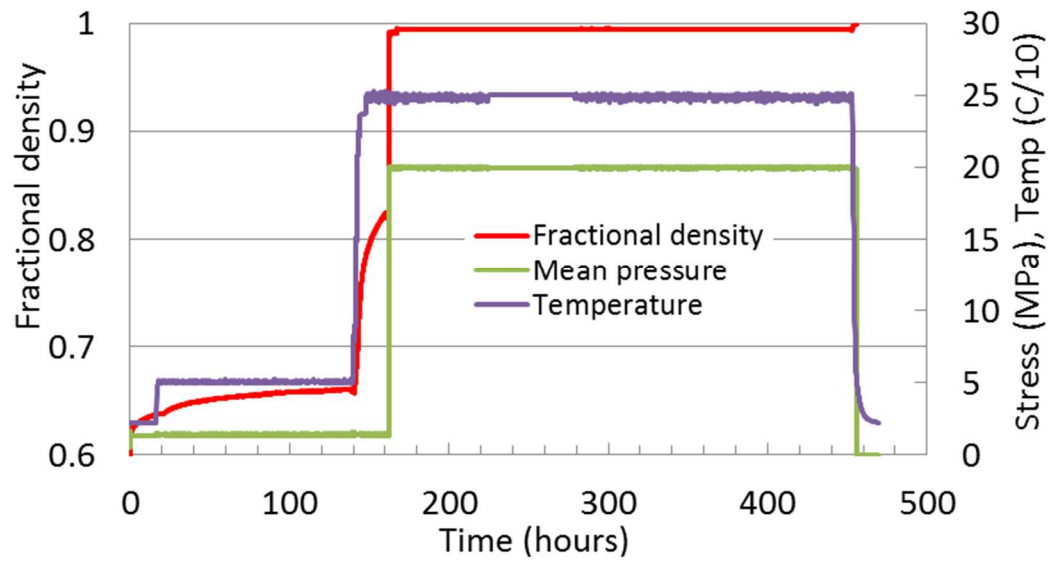


Figure 3.10 – Summary of consolidation test for UNM_WP_HY_250_01.

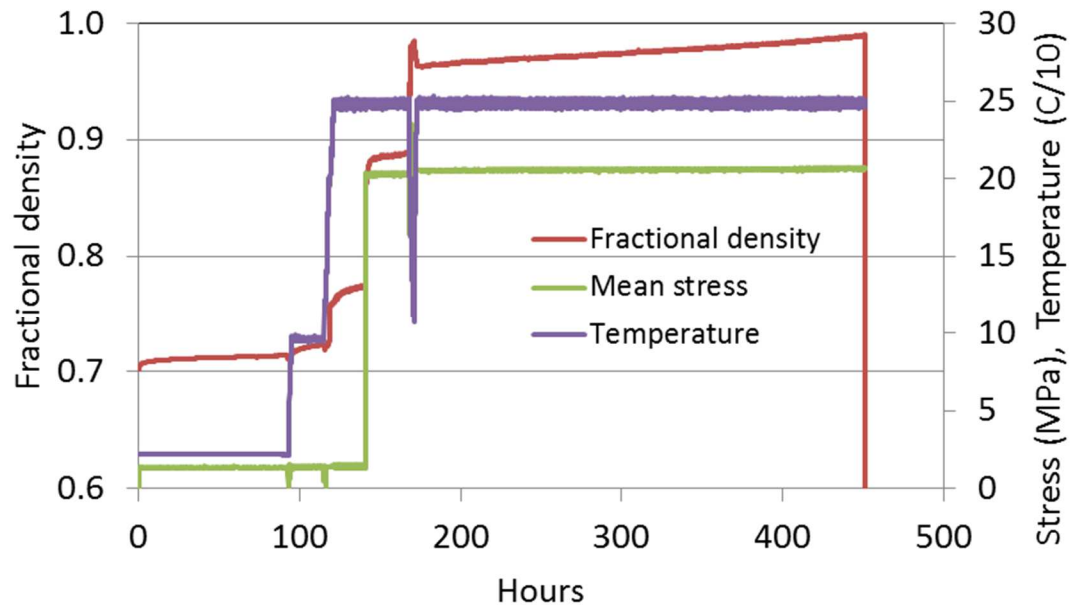


Figure 3.11 – Summary of consolidation test for UNM_WP_HY_250_02.

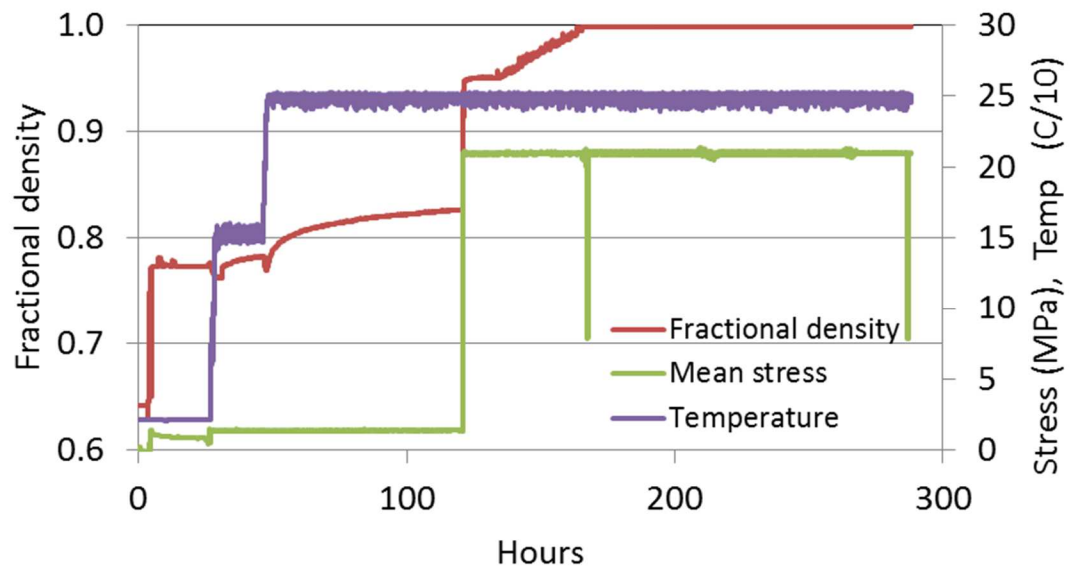


Figure 3.12 – Summary of consolidation test for UNM_AI_HY_250_01.

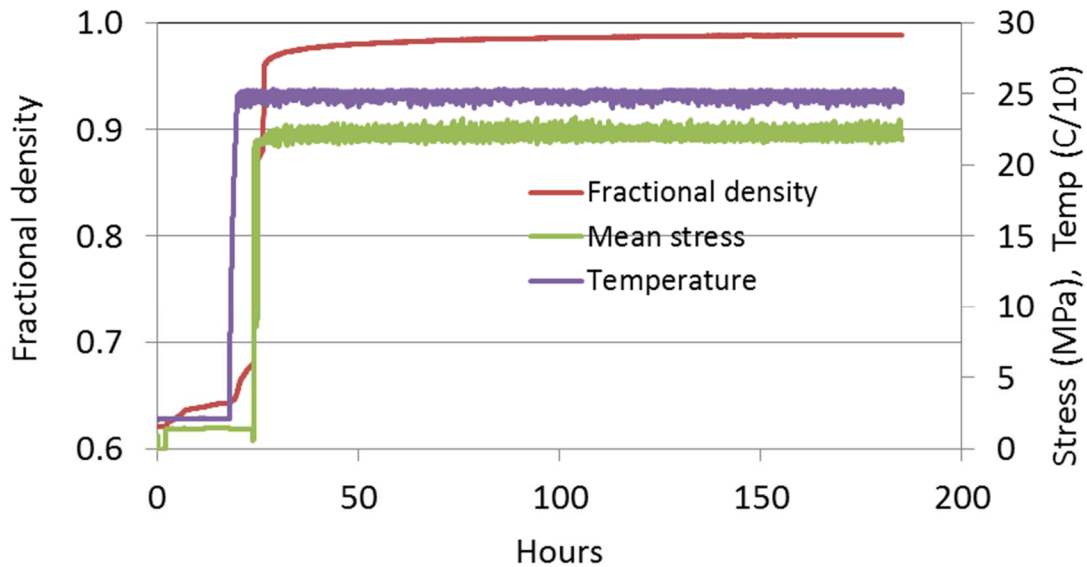


Figure 3.13 – Summary of consolidation test for UNM_AI_HY_250_02.

3.3.1.2 *Discussion*

Drained vs. undrained conditions - Comparison of results from UNM_WP_HY_250_02 (which were undrained) with those from UNM_WP_HY_250_01 (drained) provide an indication of the significance of drained vs. undrained conditions. Both tests produced a low porosity, low permeability salt mass although with a somewhat different response. It is important to note that the two tests had different initial conditions: the initial porosity of the undrained sample was only about 0.30, whereas the drained sample was 0.40. The less dense initial state of UNM_WP_HY_250_01 may be why it experienced much more volumetric strain in response to the initial loading. The undrained sample also experienced much less deformation during the increase of temperature while under a modest mean stress. This result may be a consequence of a reduced effective stress from the increased pore pressure that develops upon heating. The amount of volume strain experienced upon increase in the mean stress to 20 MPa was about equivalent between the drained and undrained cases. However, the samples had much different porosities immediately after the mean stress was increased to 20 MPa: <0.01 and 0.11 for the drained and undrained tests, respectively. Also, the undrained sample had a pore pressure at

this time of about 3 MPa whereas the drained sample had no pore pressure. At the conclusion of the test on the undrained sample, gas flow through the salt was not detectable.

Impact of added moisture on consolidation - The impact of added moisture at high temperature can be assessed by comparing the results of UNM_AI_HY_250_02 (1% added moisture – see Figure 3.13) with those of UNM_AI_HY_250_01 (no water added – see Figure 3.12). The fractional densities for the two tests are given together in Figure 3.14, although direct comparison of the two tests is somewhat difficult because they were conducted differently. Both achieved high fractional densities although they achieved that condition in somewhat different manner. Once the mean stress was increase to 20 MPa on the sample with added water, the consolidation occurred rapidly to a value >0.96 . In contrast, increasing the mean stress to 20 MPa for the sample without added water increased the fractional density to <0.95 . The sample without added water had a significant permeability after the mean stress increase; however, it decreased significantly the sample deformed to fractional densities >0.99 .

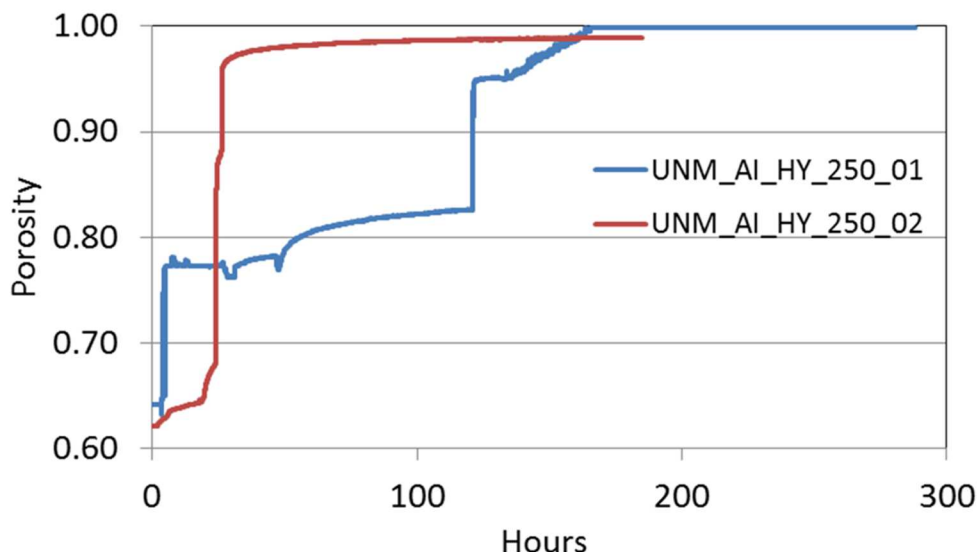


Figure 3.14 – Comparison of fractional density for hydrostatic tests conducted at 20 MPa and 250 C on domal salt with (UNM_AI_HY_250_02) and without (UNM_AI_HY_250_01) additional water.

The results of UNM_WP_HY_90_02 (1% added water) compared to UNM_WP_HY_90_01 (no added water) indicate a much more rapid consolidation is achieved when 1% water is added.

With water, the volume strain after achieving the test conditions (20 MPa mean pressure, 90 °C)

is about 35% in contrast to 25% for the test without water. Moreover, once the test conditions are achieved, the fractional density rapidly approaches 1 for the sample tested with water whereas the fractional density is much lower in the sample without additional water. The fractional density for both tests is shown in Figure 3.15.

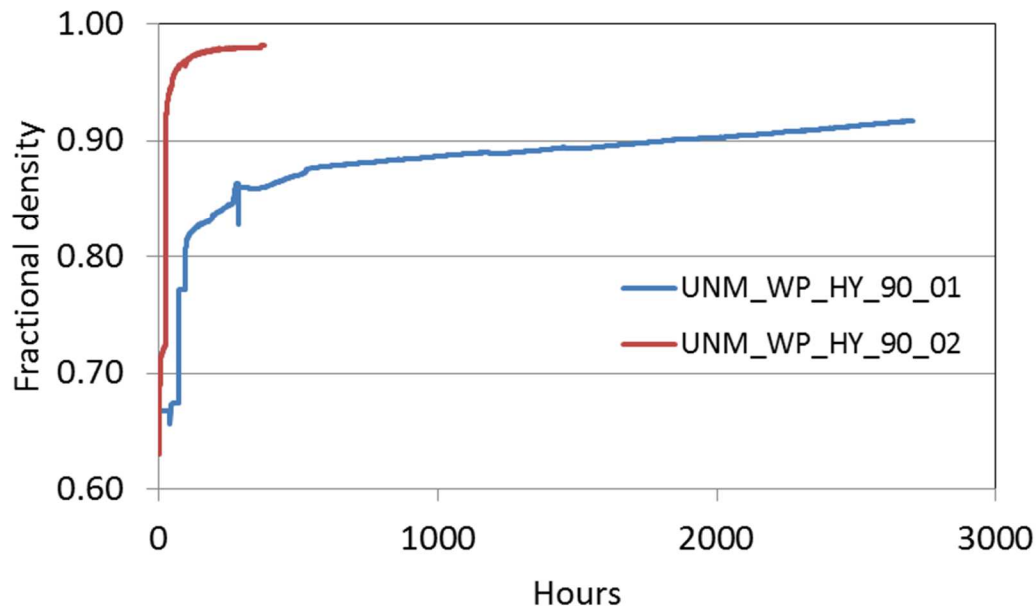


Figure 3.15 – Comparison of fractional density for hydrostatic tests conducted at 20 MPa and 90 °C. UNM_WP_HY_90_02 included 1% added water, UNM_WP_HY_90_01 did not include added water.

3.3.2 Test Series 2

In this section, some of the data obtained during the Test Series 2 consolidation testing are presented and discussed. Detailed results from consolidation testing are given in a previous milestone report (Stormont et al., 2016a).

3.3.2.1 *Results*

In Figures 3.16 and 3.17, the fractional density decrease with time is given for the samples tested in Test Series 2. Results are presented together for tests conducted at 90 °C and 175 °C. All tests were hydrostatic creep consolidation tests on WIPP samples. Thus, a short hand nomenclature is used to denote a sample; e.g., UNM_HY_WP_90_07 is given as simply 90_07, etc.

Data from the three tests at 90 °C are given together in Figure 3.16. The effect of the hydrostatic stress is evident; sample 90_08 was consolidated at 38 MPa whereas the other two samples were consolidated at 20 MPa. For the test on sample 90_07, the Schuler gages were lost after about 1 day. The confining fluid was subsequently used to measure volumetric deformation beginning on day 4 through the end of this test.

Data from the three tests at 175 °C are given together in Figure 3.17. The drop in the fractional density at about day for sample 175_03 is believed to be an electrical issue with the Schuler gages during this time period. The fractional density for sample 175_04 is derived from the fluid volume change; the fluctuations in this response were due to temperature variations caused by intermittent heater performance.

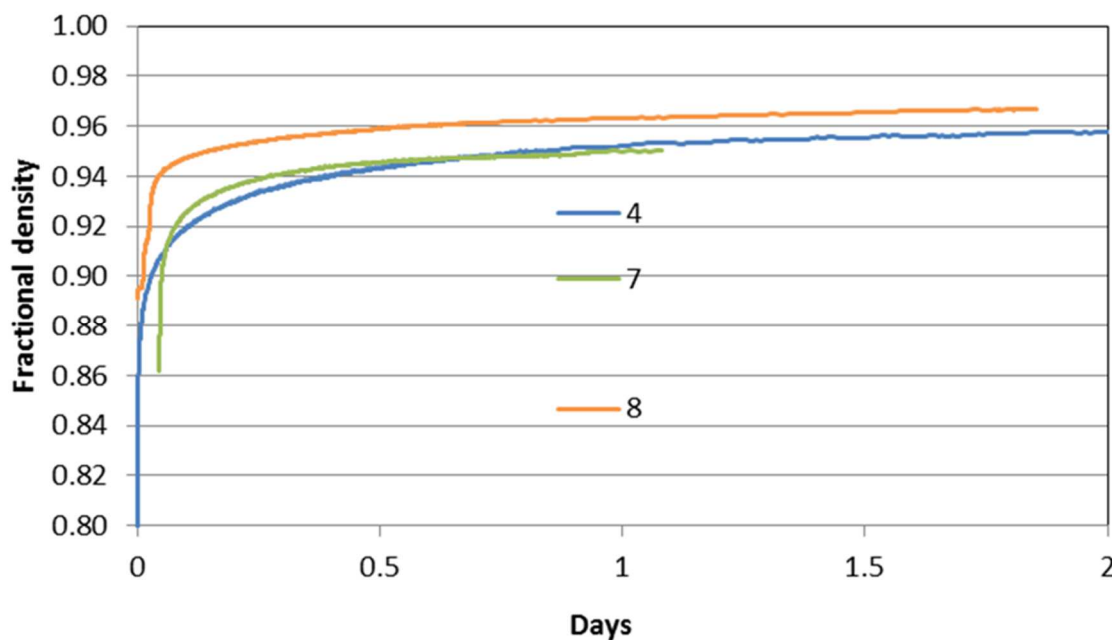


Figure 3.16 – Creep consolidation data for tests conducted at 90 °C for 2 days as part of Test Series 2.

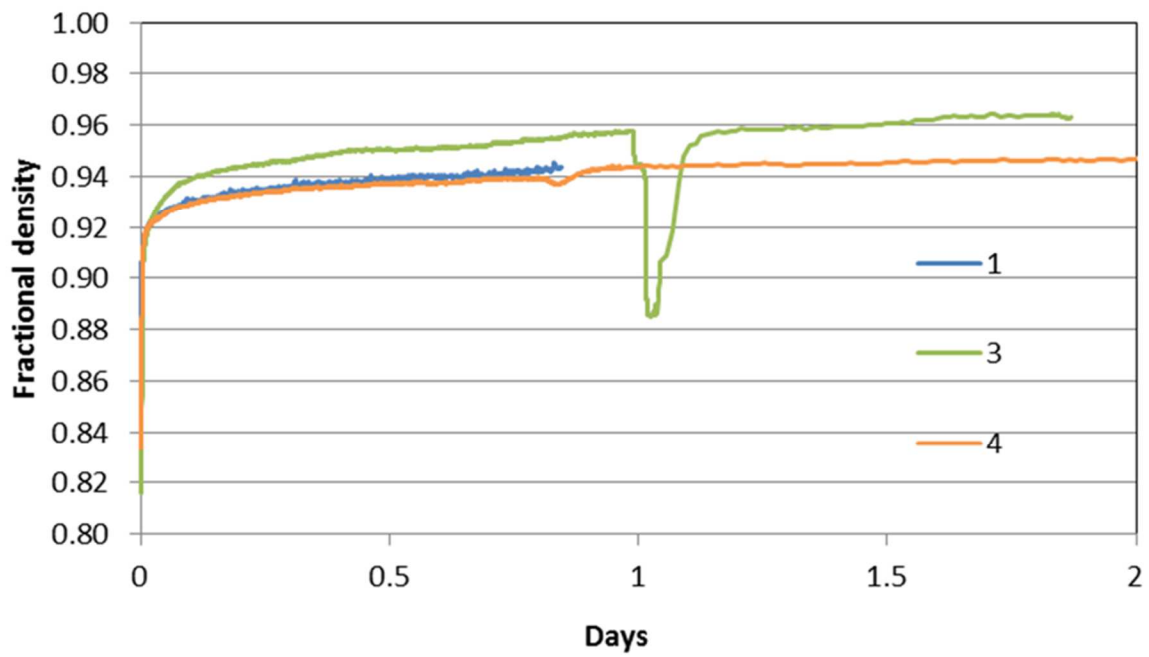


Figure 3.17 – Creep consolidation data for tests conducted at 175 °C for 2 days as part of Test Series 2.

3.3.2.2 Discussion

In Figure 3.18, the fractional density vs. time during consolidation tests at 90 °C and 20 MPa are given. All three samples were prepared with 1% additional moisture. One of these tests (90-02) was conducted with the previous test procedures which did not include a separate preconsolidation stage. The results are compared to tests 90-04 and 90-07 which both used an initial preconsolidation stage. Results from 02 and 07 compare very well. Results from 02 and 04 compare less well, but these tests are of much different duration.

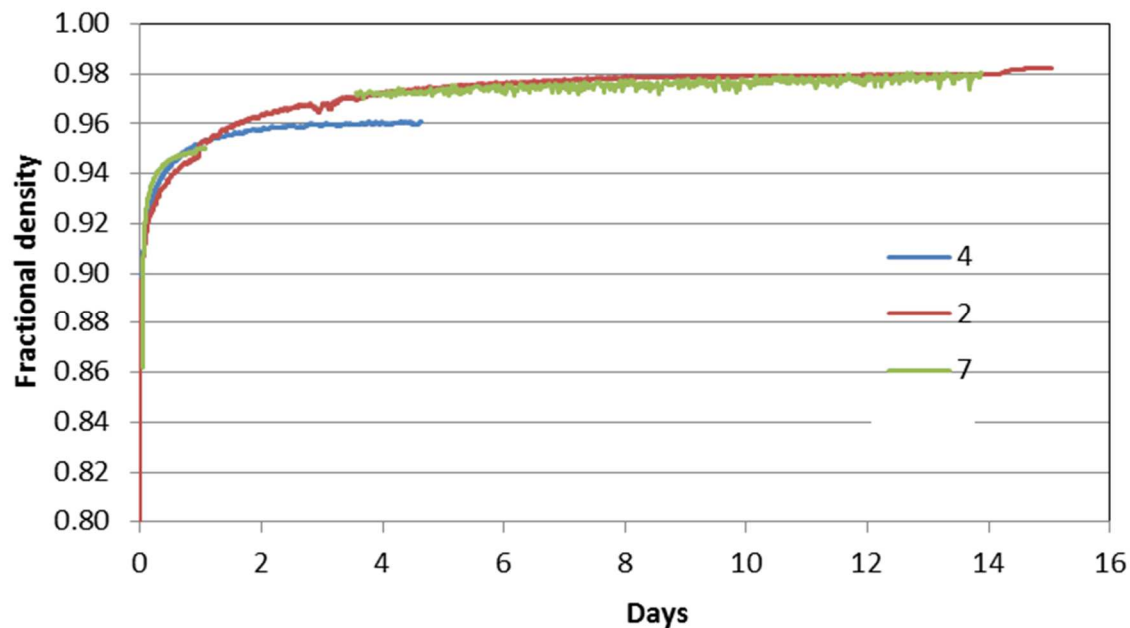


Figure 3.18 – Comparison of consolidation data from tests with an initial consolidation stage (4, 7) and without (2). These tests were conducted at 90 °C and 20 MPa. All samples had 1% added moisture.

Table 3.5 compares thermal and transport properties for these three samples obtained from tests on post-consolidated sub-samples (described in Section 4). While 90_07 and 90_02 had very similar porosities, their permeability values are different by more than two orders of magnitude. Thermal conductivity values are slightly lower for 90_02.

Table 3.5 – Comparison of post-consolidation properties from three samples consolidated at 90°C and 20 MPa with 1 % water.

Sample	Porosity (%)	Permeability (m ²)	Thermal conductivity (W/mK)
90_02	1.49	8×10^{-20}	4.78
90_04	1.44	2×10^{-17}	5.09
90_07	2.98	5×10^{-16}	4.81

Subsequent image analysis revealed that 90_02 had some limited residual canals/pores presumably from moisture within the sample (Figure 3.19). Similar structures were not found in 90_04 or 90_07.

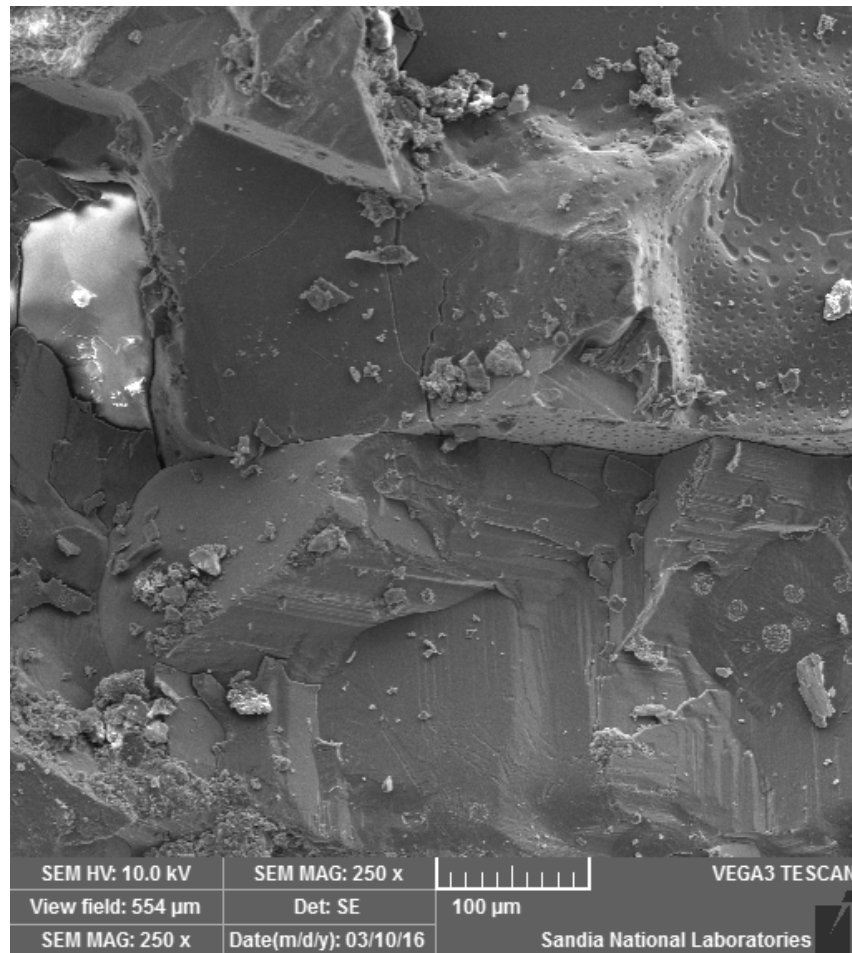


Figure 3.19 – Image of 90_02 showing residual moisture canals/pores (right hand side of image).

The investigation has revealed some differences in measured and observed response of samples consolidated with an initial consolidation stage compared to a single sample consolidated without such as stage. Any differences are most likely principally attributable to the variation in these samples, and not necessarily related to the consolidation test procedures.

With the initial consolidation stage, samples can be prepared with a fractional density in the range of 0.8 to nearly 0.9 using hydrostatic stresses between 20 and 38 MPa. The initial consolidation is consistent with a range for compacted in place densities, depending on the particular placement method. Thus, the initial consolidation stage serves as a reasonable method for preparing samples for subsequent consolidation testing to evaluate as placed granular salt.

Creep consolidation data were obtained for six samples that had an initial consolidation stage and then were subsequently consolidated under hydrostatic stress and elevated temperature. All but one test was conducted with 20 MPa hydrostatic stress, the exception was tested at 38 MPa and 90 °C, and did not have water added. The remaining samples tested at 90 °C had water added; the samples tested at 175 °C did not have water added. The data produced from these tests are consistent with expected behavior, and results in final porosities generally below 5%. Post-consolidation testing of porosity, thermal properties and permeability provide data sets that reveal the degree to which consolidation affects thermal and hydrologic properties.

The measured response during subsequent creep consolidation test was compared for samples with and without an initial consolidation stage. While there were some differences between these samples, the data is very limited and the source of these differences is not obvious.

3.3.3 Test Series 3

In this section, data obtained during the Test Series 3 consolidation testing are presented and discussed.

3.3.3.1 *Results*

Multi-stage hydrostatic creep test data are given in Figure 3.20 and 3.21 for tests UNM_WP_HY_90_09 and UNM_WP_HY_175_06, respectively. These tests both “failed” in that the tests were terminated due to jacket failure and loss of confinement. However, prior to failure, both tests generated important data. Additional multi-stage tests were conducted to completion (UNM_WP_HY_175_9, 10, 11, 12, 13, and 15) are given in Figures 3.22 through 3.27. The response to changes in pore pressure are not always clearly apparent in the volume strain data in all of these test results; however, as described in the subsequent discussion, the strain rates are clearly affected. In addition to the multi-stage tests, tests with no pore pressure were also conducted in this test series (UNM_WP_HY_175_16, UNM_WP_HY_250_03) and are given in Figures 3.28 and 3.29.

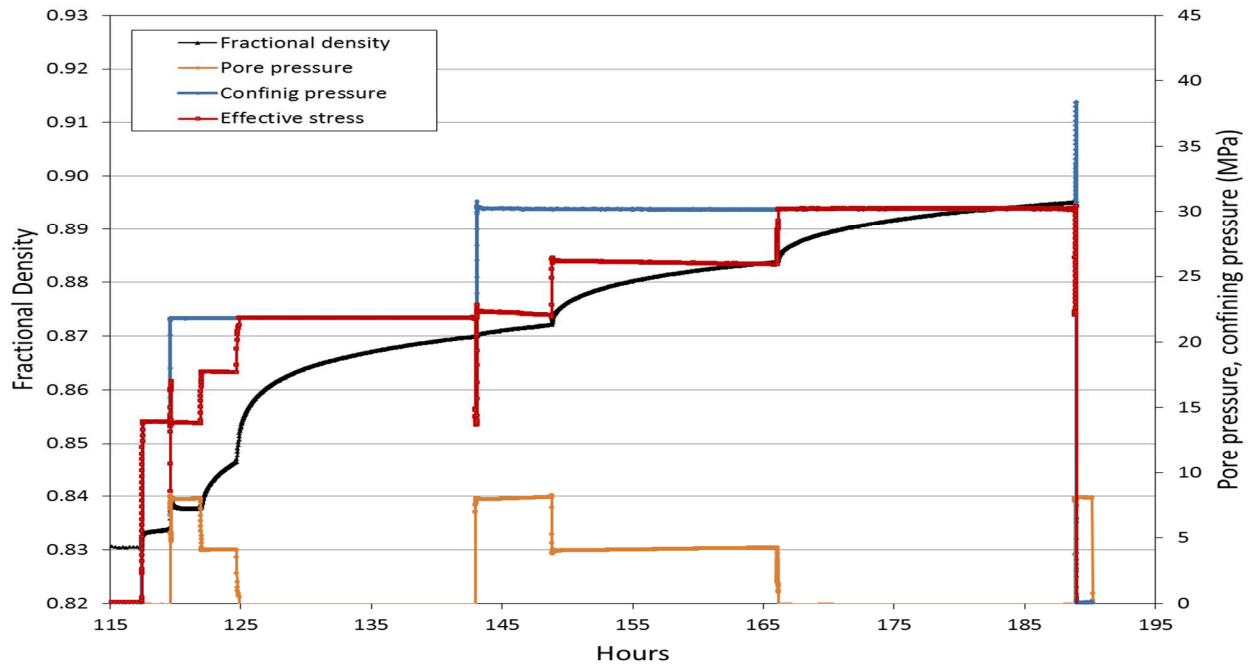


Figure 3.20 – Confining stress, pore pressure, effective stress and fractional density during multi-stage hydrostatic creep test for UNM_WP_HY_90_09. This test was conducted at 90 °C.

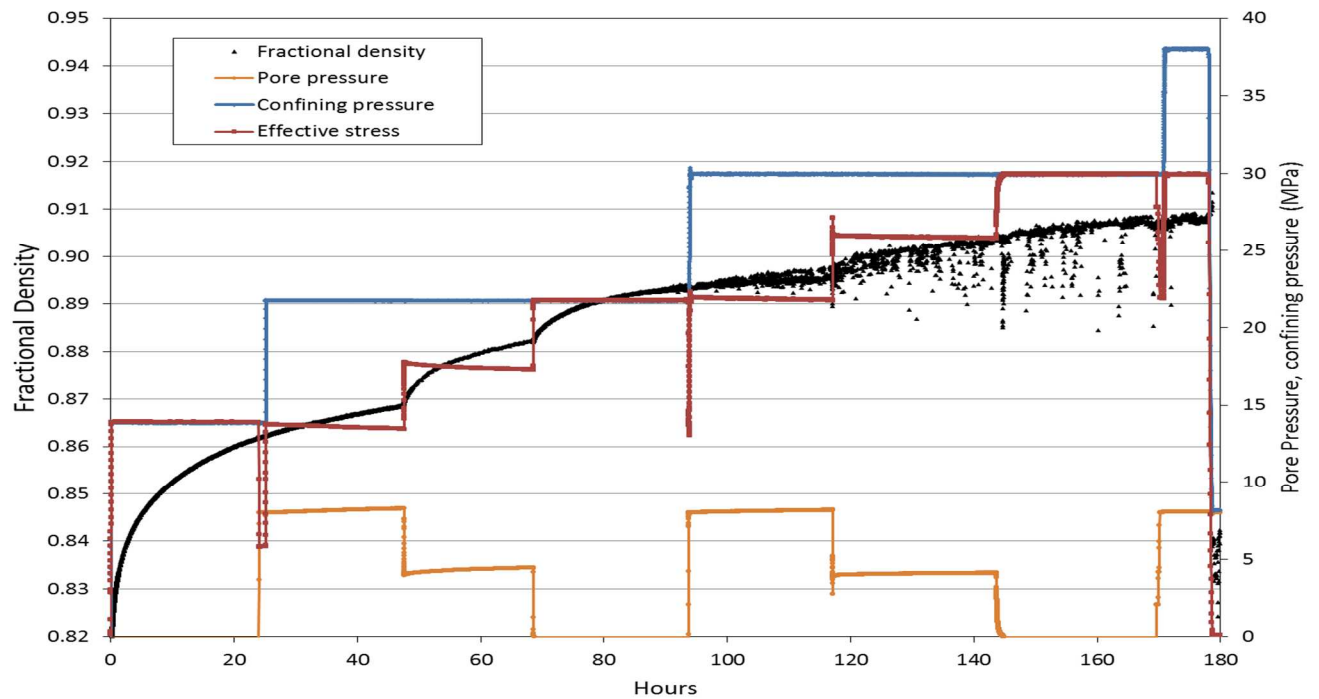


Figure 3.21 – Confining stress, pore pressure, effective stress and fractional density during multi-stage hydrostatic creep test for UNM_WP_HY_175_06.

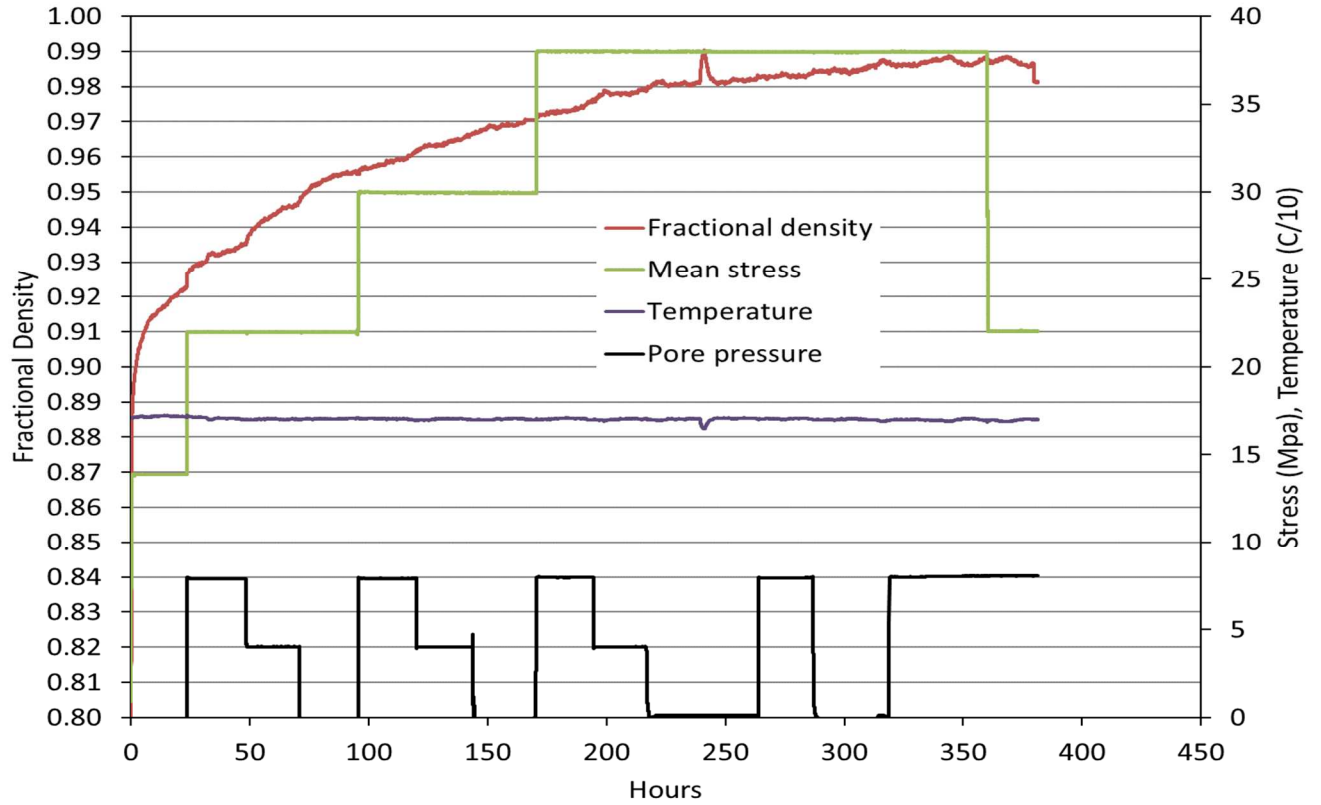


Figure 3.22 – Results from UNM_WP_HY_175_09.

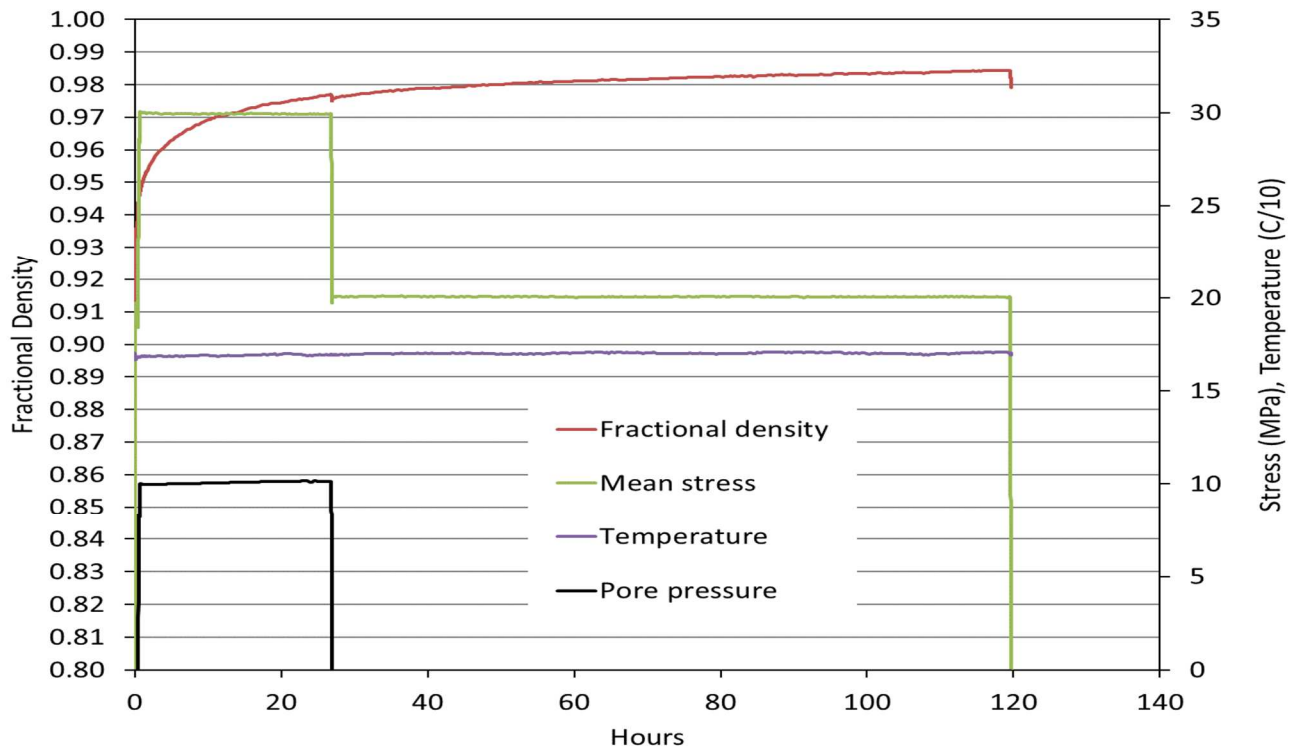


Figure 3.23 – Results from UNM_WP_HY_175_10.

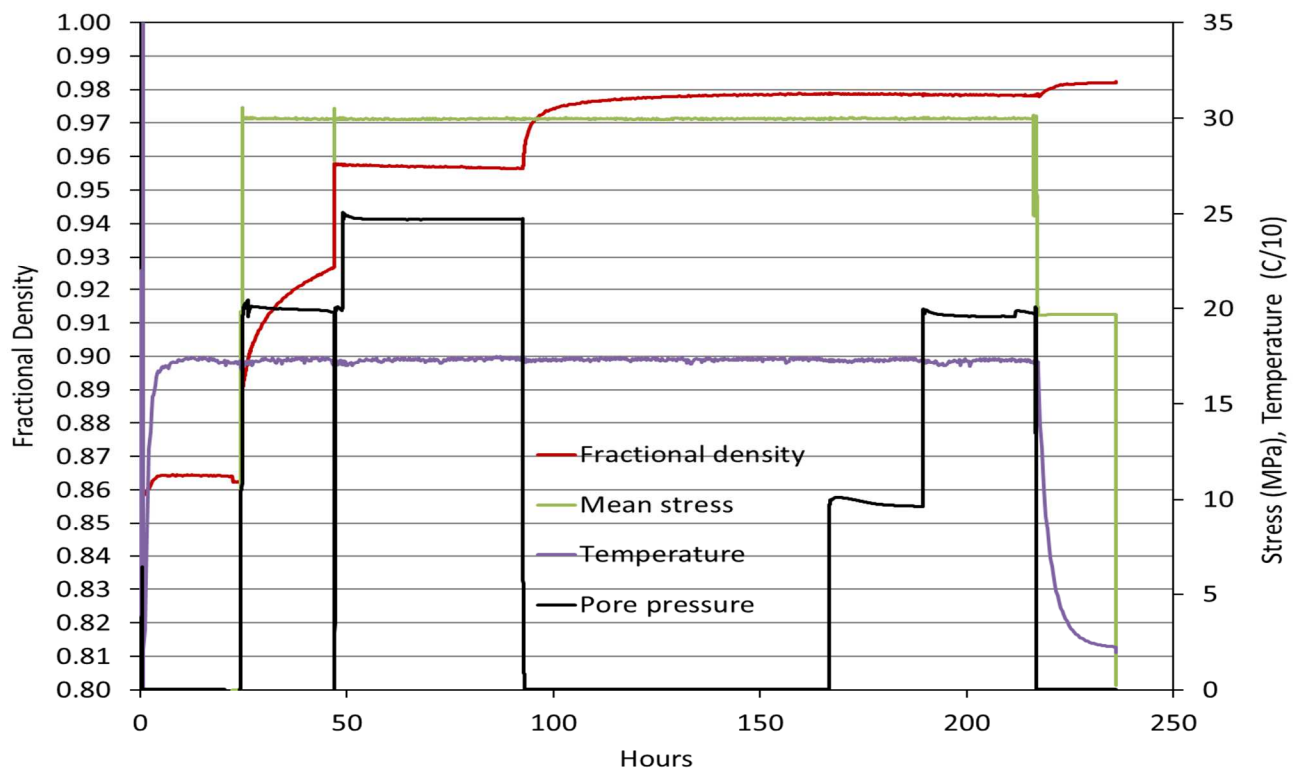


Figure 3.24 – Results from UNM_WP_HY_175_11.

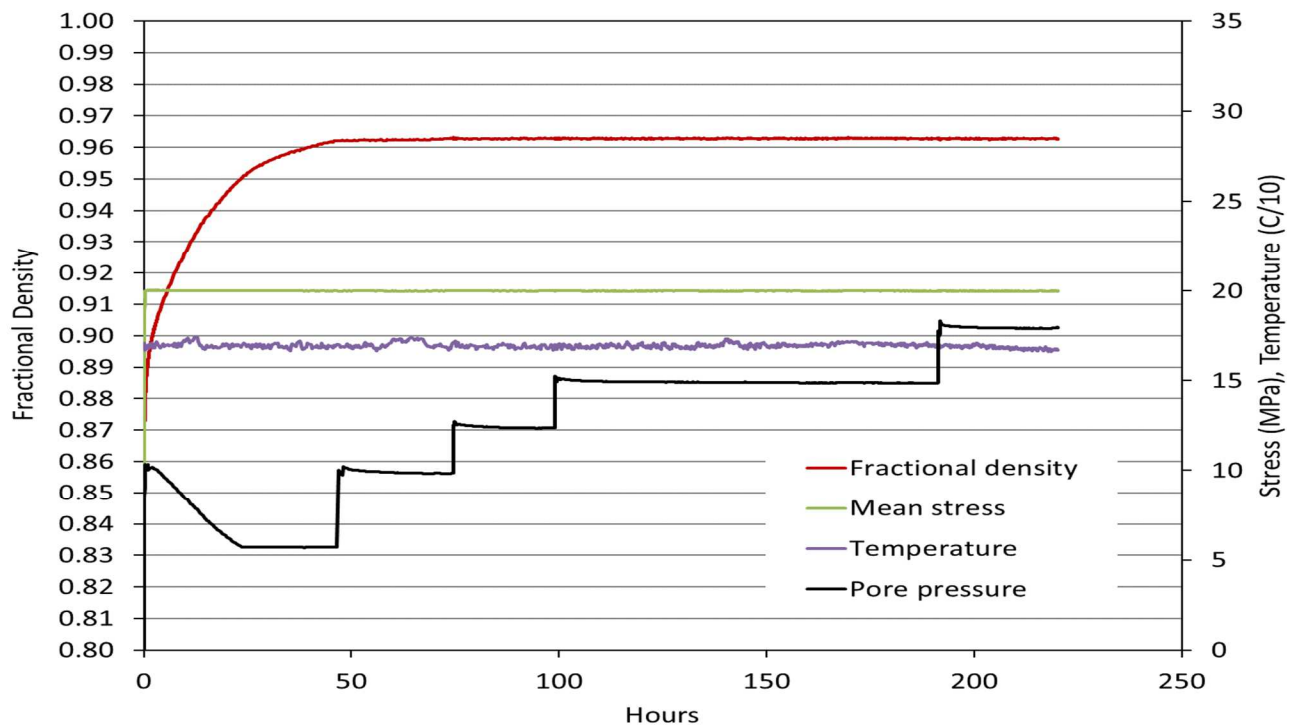


Figure 3.25 – Results from UNM_WP_HY_175_12.

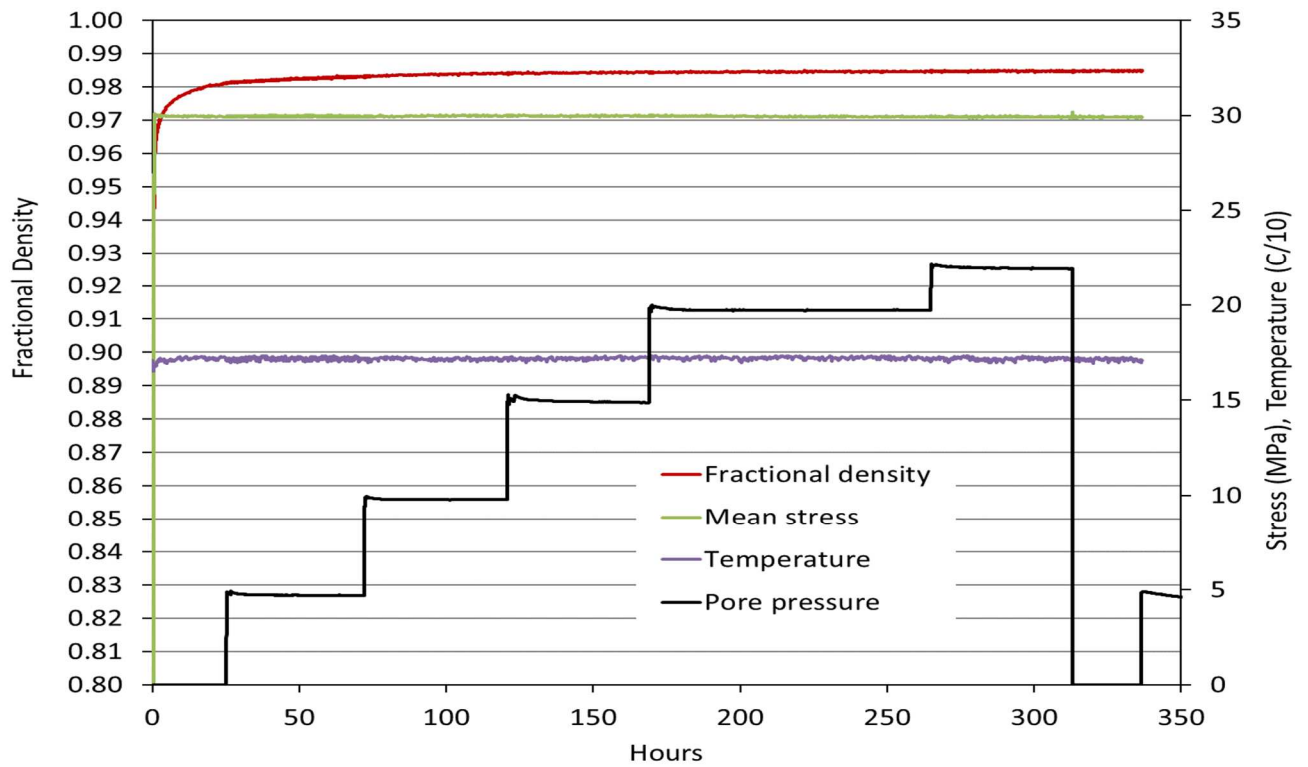


Figure 3.26 – Results from UNM_WP_HY_175_13.

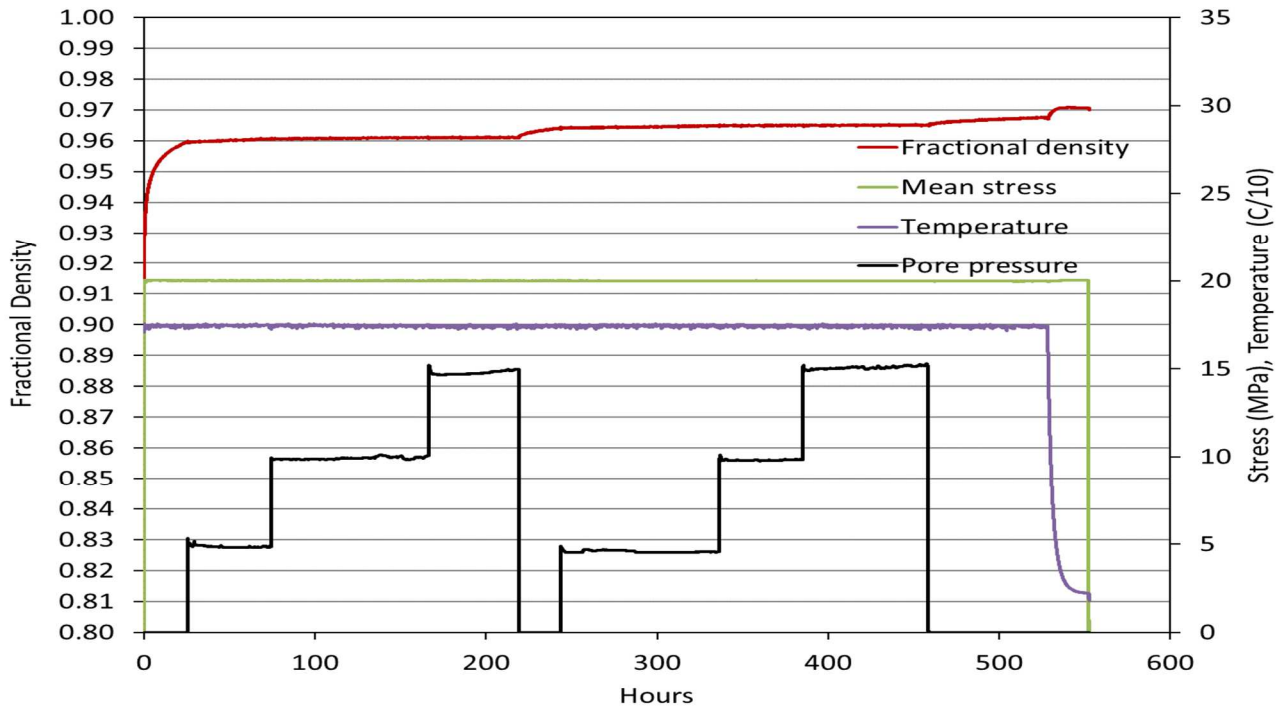


Figure 3.27 - Results from UNM_WP_HY_175_15.

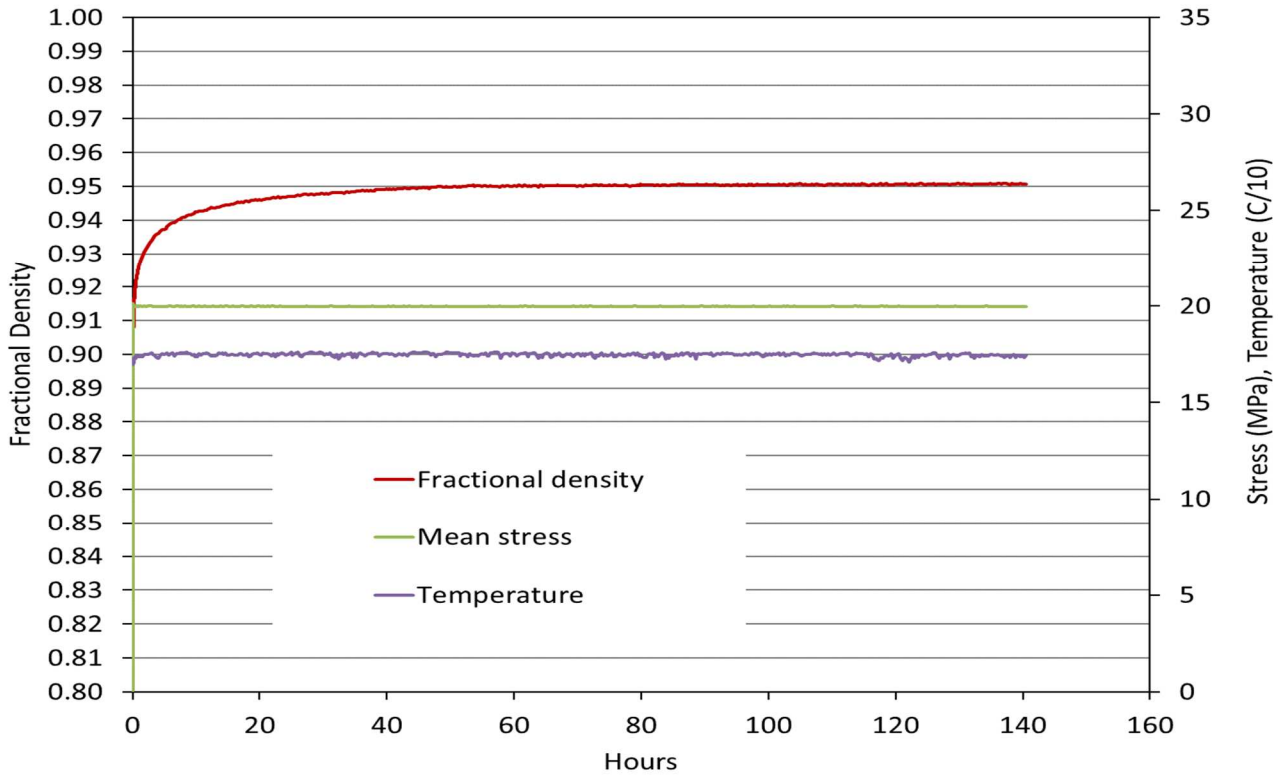


Figure 3.28 - Results from UNM_WP_HY_175_16.

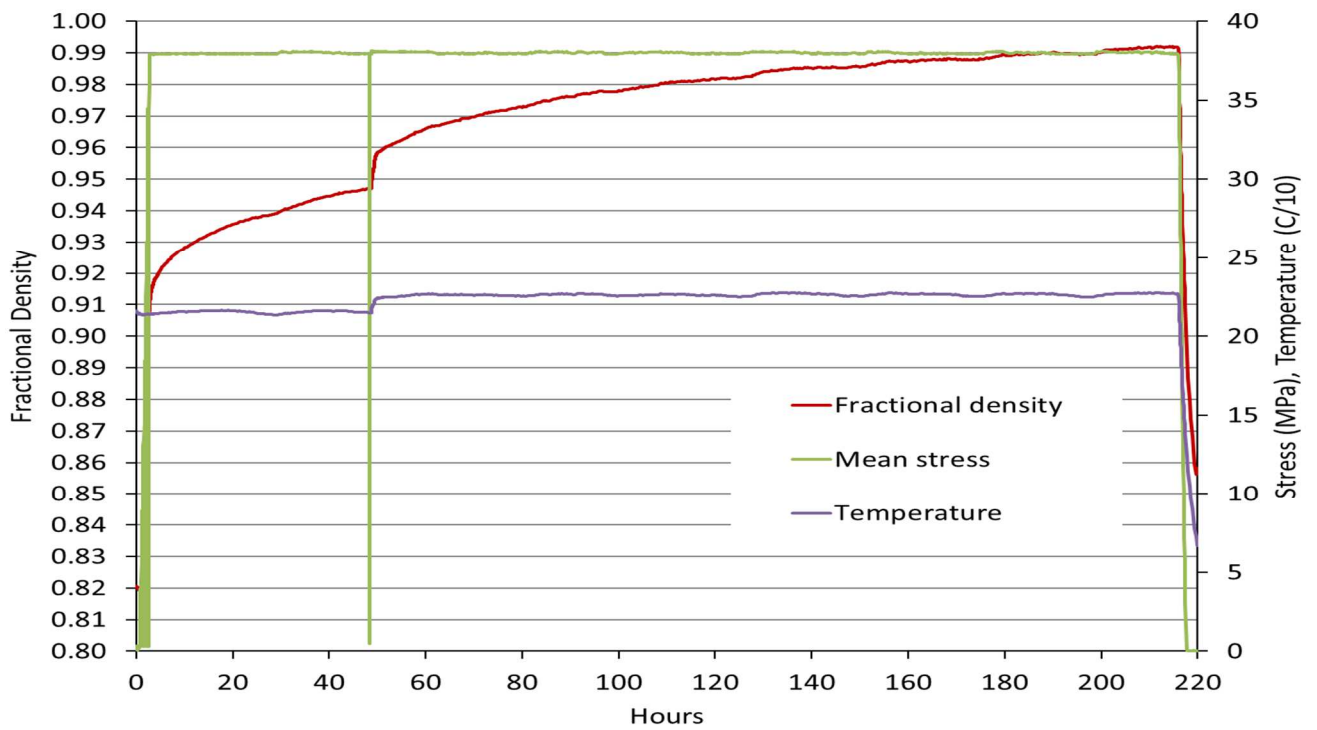


Figure 3.29 - Results from UNM_WP_HY_250_03.

3.3.3.2 *Discussion*

We have found no reported experiments on granular salt which were directed at evaluation of the influence of pore gas pressure on its deformation under constant stress (creep). Presumably, this is because the common understanding is that pore pressure has little influence on the visco-plastic deformation of a material under a constant stress. The influence of a pore liquid pressure on the creep deformation of wet-crushed salt was experimentally evaluated by Spiers et al. (1988), where they concluded that the presence of brine allowed for crushed salt to deform by grain boundary diffusional pressure solution, but this phenomenon would not occur in dry-crushed salt.

Studies have been performed to determine the influence of pore gas pressure on intact and dilated-intact (damaged) salt. McTigue (1986) recognized the influence of pore pressure on the elastic deformation of intact salt and determined a set of poroelastic parameters for intact salt at the WIPP. Kansy (2007) evaluated the influence of pore pressure on the elastic deformation and permeability of dilated-intact salt, and he concluded that a pore pressure applied with either a gas or chemically nonreactive liquid influenced the pore volume of the dilated salt. He also recognized that this change in pore volume resulted in variable permeability and values of the poroelastic Biot parameter.

While conducting gas permeability measurements concurrent with consolidation, we noticed an apparent creep consolidation rate response to changes in pore gas pressure. Based on these preliminary observations, consolidation tests with variable gas pore pressure were conducted to provide data to investigate the effect gas pore pressure has on consolidation.

We evaluated results from three tests in detail: UNM_WP_HY_175-10, 11 and 13. Here, only the response during initial loading is given and described, not subsequent test stages where there were additional changes in the confining pressure and/or pore pressure. Deformations during increases in confining pressure or pore pressure were observed to be both elastic and plastic; however, only the creep deformations during constant pressure conditions are considered here. For clarity, P_c refers to the macroscopic fluid pressure applied to the outer surface of the sample, or the confining pressure. P_p refers to the macroscopic gas pressure in the interconnected pore volume of the bulk sample, or the pore pressure. P_d is the difference between P_c and P_p . During the pressure increase, P_c and P_p were independently controlled such that P_c was increased until the

desired value of P_d was achieved. Then, P_c and P_p were increased uniformly (holding P_d constant) until the desired pressures for the creep tests were achieved. A summary of the creep test conditions is presented in Table 3.6. Note that we use abbreviated sample names in the following discussion for compactness. The porosity measurements following the preconditioning (φ_{pc}) of each sample (via caliper and fluid immersion) are presented in Table 3.1 along with the porosity values measured at the start of the creep test (φ_{crp}^0), after the increase of pressure.

Table 3.6: Summary of creep test conditions.

Abbreviated sample name	Test/Sample (UNM_WP_HY)	P_c MPa	P_p MPa	P_d MPa	φ_{pc} %	φ_{crp}^0 %
A	175-13	30	0	30	21.8	10.9
B	175-10	30	10	20	21.3	11.8
C	175-11	30	20	10	21.0	17.8

The results presented here are focused on identifying the role of P_p on the volumetric plastic strain rate $\dot{\epsilon}_v^p$; therefore, a substantial effort was taken to keep other parameters that influence the test results constant. That is, the water content, particle size, temperature, and P_c were nearly constant between all tests and their respective influences will not be considered here. The section will provide a precise definition of the measured strains, and following this, creep test results will be presented along with a discussion on the observed influence of pore pressure during these creep tests.

Deformations presented below were all observed during a state of constant P_c and P_p , and following the tests, when the samples were unloaded, these deformations have remained indefinitely; therefore, all deformations described in this paper are considered plastic. Deformation of the samples are quantified in terms of a scalar metric describing the bulk volume change of the sample. This metric is the plastic volumetric strain (ϵ_v^p), which is considered positive in compression (decreasing volume) and defined in terms of the natural logarithm as shown in Eqn. 3.2, where V and V_0 represent the current and preconditioned bulk sample volumes, respectively. The plastic volumetric strain rate ($\dot{\epsilon}_v^p$) is primarily considered in this report and it is defined as the

ratio of incremental plastic change in volume strain ($\Delta\epsilon_v^p$) to the incremental change in time (Δt), Eqn. 3.3.

$$\epsilon_v^p = -\ln\left(\frac{V}{V_0}\right) \quad (3.2)$$

$$\dot{\epsilon}_v^p = \frac{\Delta\epsilon_v^p}{\Delta t} \quad (3.3)$$

The deformation of intact salt under creep conditions is assumed to be the result of ductile crystalline plasticity, which is an isochoric process. Accordingly, the volume of the solid particles within the bulk sample was assumed constant during the periods of constant pressure. Therefore, measured bulk volume changes during a creep tests were assumed to result solely from changes in the samples connected pore volume, which is considered below in terms of the bulk sample porosity (ϕ). This assumption is consistent with the observed evidence that solid salt particles plastically flow into the sample void space, which results in a reduced ϕ and permeability (Brodsky, 1994). Fig. 3.30 illustrates the change of ϕ as the samples reconsolidate with test duration for each of the three creep tests. These results all display a similar trend of decreasing ϕ over the creep test duration.

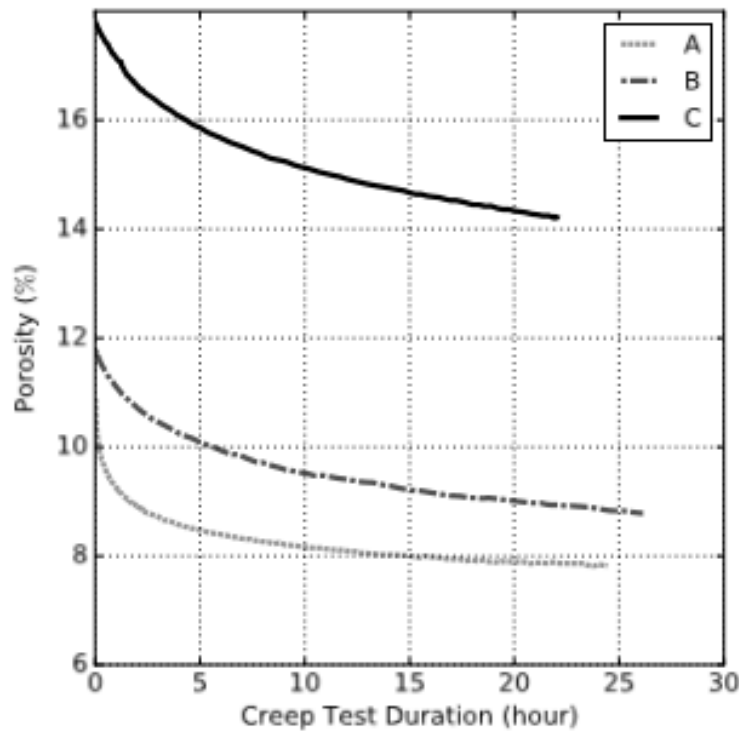


Fig. 3.30 - Porosity versus creep test duration.

The variation in φ_{crp}^0 is believed to result solely from the application of P_p during loading (prior to the creep test). All samples had a similar porosity following the preconditioning (φ_{pc}), all were loaded at the same rate of 4 MPa/min to a P_c of 30 MPa, but each had a different P_p . These results along with observations presented by Brodsky (1994, pg.7) and others, where the deformation of crushed salt was observed to be strongly influenced by the magnitude of the mean stress, indicate the deformations observed during the pressure increase may be understood with the use of a Terzaghi-type effective stress law as was done by Spiers and Brzesowsky (1993) and Olivella and Gens (2002), which under hydrostatic loading necessitates the consideration of only the P_d . This observation is presented here only to provide insight into the cause for variation of φ_{crp}^0 between the three tests. The focus of the study reported here is on the creep test results; therefore, no additional discussion of the poroelastic effects during the loading stage of these tests will be provided.

Fig. 3.31 illustrates the plastic volumetric strain rate ($\dot{\epsilon}_v^p$) decreasing with time for all samples, where strain rates range from $3 \cdot 10^{-5}$ down to $4 \cdot 10^{-8} \text{ sec}^{-1}$. These trends of a decreasing $\dot{\epsilon}_v^p$ with time are similar to those for decreasing φ with time, shown in Fig. 3.1. These observed trends agree well with those presented in Brodsky (1994) and Callahan (1999), where the authors described how reconsolidated crushed-salt samples showed a greater $\dot{\epsilon}_v^p$ with greater φ and the converse of a reduced $\dot{\epsilon}_v^p$ with reduced φ . Trends observed in these three tests agree with the premise that $\dot{\epsilon}_v^p$ will decrease to zero as the φ approaches zero, in which case the sample density would equal that of intact salt. The creep test results presented here were all performed with noticeably different magnitudes of P_d ; however, the influence of P_d is not obvious when the deformation is related to the test duration. Rather, the influence of P_d is much more apparent when the $\dot{\epsilon}_v^p$ is shown with respect to φ , as in Fig. 3.32.

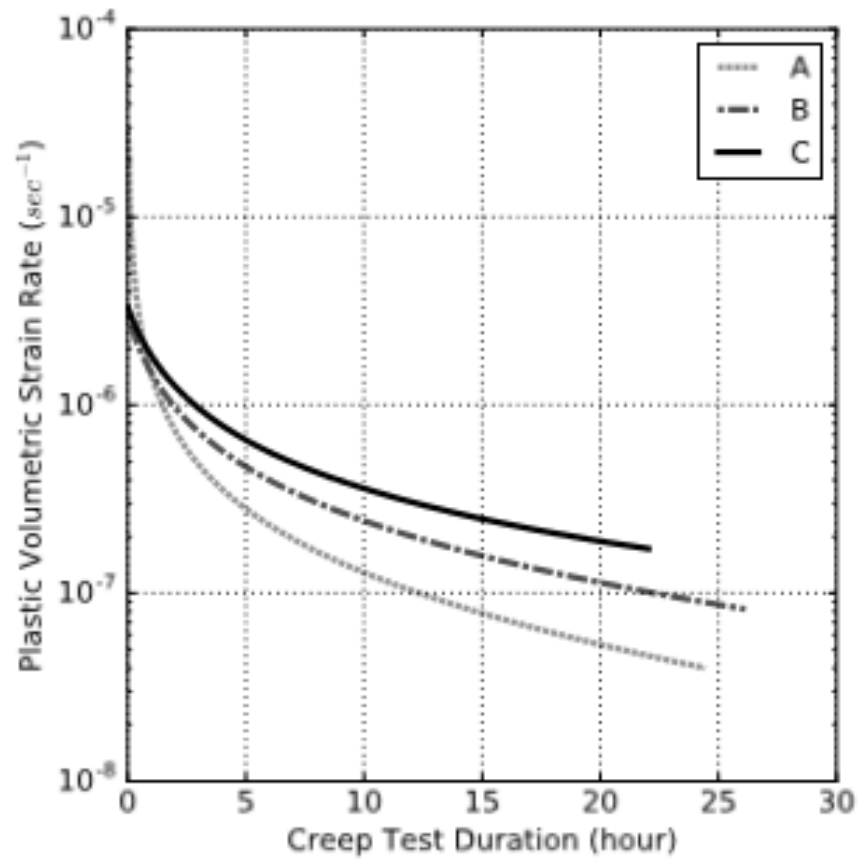


Fig. 3.31 - Volumetric strain rate versus creep test duration.

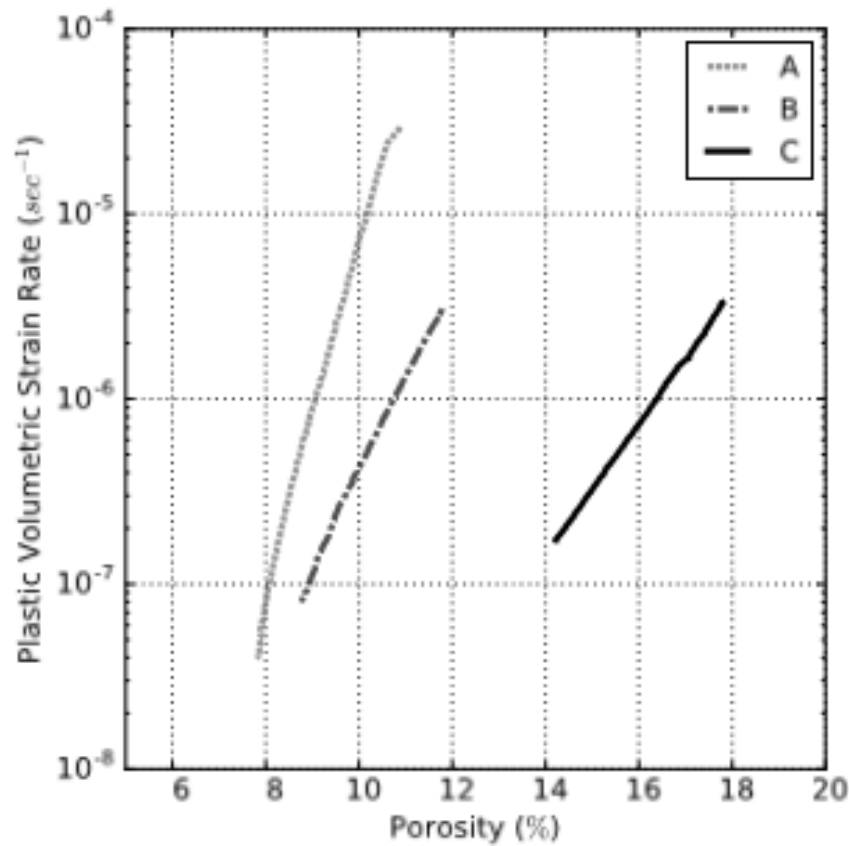


Fig. 3.32 - Volumetric strain rate versus sample porosity.

Fig. 3.32 provides direct the comparison of the $\dot{\epsilon}_v^p$ as a function of ϕ . These results show a trend that is not consistent with the understanding that $\dot{\epsilon}_v^p$ depends only on ϕ and P_c . Here, samples with a wide range of ϕ all exhibit a similar $\dot{\epsilon}_v^p$, e.g., the three samples ϕ values vary by over 7%, but they all exhibit a similar $\dot{\epsilon}_v^p$.

Table 3.7 presents the ϕ for the three samples when their respective $\dot{\epsilon}_v^p$ is 10^{-6} sec^{-1} . Alternatively, consider the $\dot{\epsilon}_v^p$ of Samples A and B when they are both at a ϕ of 10%; their values of $\dot{\epsilon}_v^p$ differ by over an order of magnitude. These observations are consistent with the understanding that the $\dot{\epsilon}_v^p$ is directly influenced by the P_d . In this case, it is reasonable for Sample A to deform at a greater rate than Sample B when both have the same porosity because Sample A is subjected to a greater P_d . These results indicate the $\dot{\epsilon}_v^p$ is influenced by both the ϕ and P_d . To rephrase this, these experimental results show the $\dot{\epsilon}_v^p$ and the affiliated $\dot{\epsilon}_v^p$ of the bulk crushed-salt samples are influenced by the P_p .

Table 3.7: Comparison of porosity (ϕ) at a constant plastic volumetric strain rate ($\dot{\epsilon}_v^p$) from Fig. 3.32 along with the pressure difference (Pd).

Test/Sample	ϕ %	$\dot{\epsilon}_v^p$ sec^{-1}	Pd MPa
A	9.1	10^{-6}	30
B	10.7	10^{-6}	20
C	16.4	10^{-6}	10

Results shown here do not contradict observations made by other researchers regarding the deformation of crushed salt; however, no experimental results were found that were directly comparable. In general, the influence of P_p observed during these experiments does agree with the statement made by Brodsky (1994, pg. 75) regarding drained creep tests on crushed salt: “Results of the hydrostatic consolidation tests show that consolidation rate increases with increasing P_c .” Although $P_p = 0$ during the tests referred to by Brodsky, an increased $\dot{\epsilon}_v^p$ resulting from an increased P_c is consistent with the results presented here. That is, under drained conditions, an increase of P_c is a direct increase in P_d , and the results presented here show that the $\dot{\epsilon}_v^p$ increases with an increased P_d . Results from the creep tests given here bring into question the assumption of a homogeneous stress state throughout a sample, which is a fundamental assumption needed to relate experimental results from the laboratory to theoretical models used to predict the deformations of complex structures. If experimentalists claim to have tested a material under hydrostatic conditions, but the true test conditions include the influence of a local shear, such a misunderstanding will lead to an incorrect interpretation of experimental data and may have adverse effects on the development of constitutive models. The constitutive model described by Callahan (1999) utilizes a pair of ad hoc effective stress definitions (in the non-associative flow rule) that appear to have been developed in order to capture the influence of local shear stresses within a bulk sample of crushed salt. That is to say, the model predicts a nonzero $\dot{\epsilon}_v^p$ under hydrostatic creep conditions but no mention was given to the concept of a local stress or as what causes the crushed-salt sample to undergo a permanent change of volume under such conditions. Experimental observations made here, regarding the influence of P_p on shear stress, appear to be

in contradiction with the constitutive model developed by Olivella and Gens, as their model for crushed-salt allows for P_p to only influence the normal components of the stress tensor. The following was taken from the article presented by Olivella and Gens (2002, pg. 733):

“It does not matter if effective or total stress is used here because deviatoric stresses do not depend on P_p and this law(dislocation creep) does not contain volumetric contribution.”

For context, this statement was made with regard to the influence of P_p on a mechanism used to describe the plastic deformation simulated to occur over a fraction of a solid salt particle within a bulk crushed-salt sample. However, the above statement appears inconsistent under conditions where $P_c = P_p$, which would result in a hydrostatic state of stress at the scale of both the local salt particles and the macroscopic bulk sample, and the shear stress throughout the sample would be diminished to zero as a result of $P_d = 0$. To further elucidate our understanding of the experimental results presented here, the application of P_p during hydrostatic creep tests appears to strongly influence the local shear stress within a porous crushed-salt sample. The local shear stresses do not directly cause a change in volume; rather, the local shear stresses cause local shear strains within the individual salt particles. These shear strains, within the salt particles, result in the salt particles plastically deforming in an isochoric manner into the pore space. This plastic flow of salt particles into the pore space then causes a decreased pore volume and hence a decrease of the sample's bulk volume. As P_p is increased, the associated P_d is decreased, and the magnitude of local shear stresses are also reduced. The reduced local shear stresses result in a reduced $\dot{\epsilon}_v^p$. Alternatively, when $P_p = 0$ and $P_d = P_c$, the local shear is greatest and the $\dot{\epsilon}_v^p$ is also greatest. The influence of P_p on the $\dot{\epsilon}_v^p$ of crushed salt is believed to decrease as the ϕ and associated permeability approaches zero.

3.3.4 Permeability test during consolidation

In this section, gas permeability results are given for seven samples in which gas flow was periodically measured during consolidation. The sample names and consolidation conditions are described in Table 3.8. The consolidation data are presented in previous sections in this report. Tests were only conducted on samples that did not have added water to avoid removing any of this water with the gas flow. We note that although no water was added to these samples prior to consolidation, the samples themselves contained some moisture within negative crystals and associated hydrous minerals. The moisture can be liberated from the samples during the

consolidation tests, and therefore the deformation that occurs during consolidation can include processes that involve pressure solutioning. Thus, the consolidation conditions were not necessarily completely dry. The role of moisture on consolidation is described in a previous project report (Stormont et al., 2015).

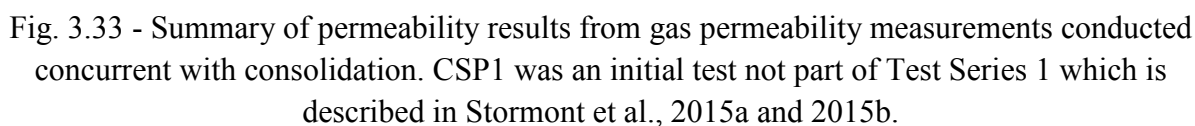
Table 3.8 – Summary of consolidation tests which included concurrent gas flow measurements.

Sample Name	Temperature (°C)	Hydrostatic Stress (MPa)	Duration (days)	Final porosity
CSP1	90	20 to 38	12	0.033
UNM_WP_HY_250_1	250	20	13	0.006
UNM_WP_HY_90_1	90	20	112	0.083
UNM_AI_HY_250_1	250	20	8	0.011
UNM_WP_HY_175_1	175	20	3	0.056
UNM_WP_HY_175_3	175	20	3	0.019
UNM_WP_HY_90_8	90	40	10	0.030

3.3.4.1 *Results*

Permeabilities interpreted during consolidation tests are given in Fig. 3.33 as a function of porosity. Note that abbreviated sample names are used for compactness. The porosity was interpreted from the measured strain data as described previously in this report. Although these tests were conducted at different temperatures, pressures, durations and salt types, there was a general behavior; the permeability steadily decreased from the initial relatively large value. For most of these tests, beyond some “critical porosity” the permeability decreased at an increasing rate with continued consolidation. While the general response appears consistent between tests, permeability values can vary by up to 2 orders of magnitude at the same porosity. These differences could be due to measurement uncertainties and/or variability between samples. These data do not reveal a distinct influence of stress level, temperature or duration on permeability independent of porosity. For example, samples consolidated under same conditions (W175-01 and

A very low permeability ($<10^{-20}$ m²) was achieved from only one sample (W250_1). The permeability of this sample decreased abruptly after its porosity decreased below 0.01. These results suggest a very low porosity will have to be reached before the permeability of consolidating granular salt will be comparable to intact salt for conditions considered here.



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$$k = k_o \phi^x \quad (3.4)$$

where k is the permeability, k_o is a reference permeability, ϕ is porosity, and x is an empirical exponent. A better fit for most data could be developed if the exponent x was chosen to increase as the sample porosity decreased. In general, the values of x at larger porosities (>0.05) were in the range of 2 to 4; for most samples, x increased to more than 10 at the smallest porosities achieved.

3.3.4.2 *Discussion*

The data in Fig. 3.33 suggest that the general permeability-porosity response consists of different regions as shown in Fig. 3.34. This type of permeability-porosity behavior has been observed for other materials, including calcite, quartz, and sandstone (e.g., Zhu et al., 1995). The three regions are described below:

1. Region I – a gradual decrease in permeability as the porosity decreases. In this stage, the porosity remains largely connected and involved in the flow. The changes in permeability are principally due to reduction in the dimensions of the porosity (e.g., pore diameter). The permeability-porosity response in this region may be described by a power law with an exponent near 3.
2. Region II - at porosities below a critical value, the permeability decrease with porosity is accelerated. The decrease in permeability is due to the loss of connectivity as some pores completely close. Isolated porosity may develop. The data in this study suggest the critical porosity is in the range of 0.02 to 0.05. The exponent in a power law may be much greater than 3. The exponent was greater than 10 for most individual data sets at the lowest porosities.
3. Region III- below a percolation threshold, there is no measurable permeability. In this region, there is no significant continuously connected pore network. This percolation or porosity threshold corresponds to the porosity at which consolidation processes produce a complete loss of pore connectivity. There are insufficient data in this data set to confirm that this condition is ever reached, and if it is, to provide a reliable estimate of the percolation threshold.

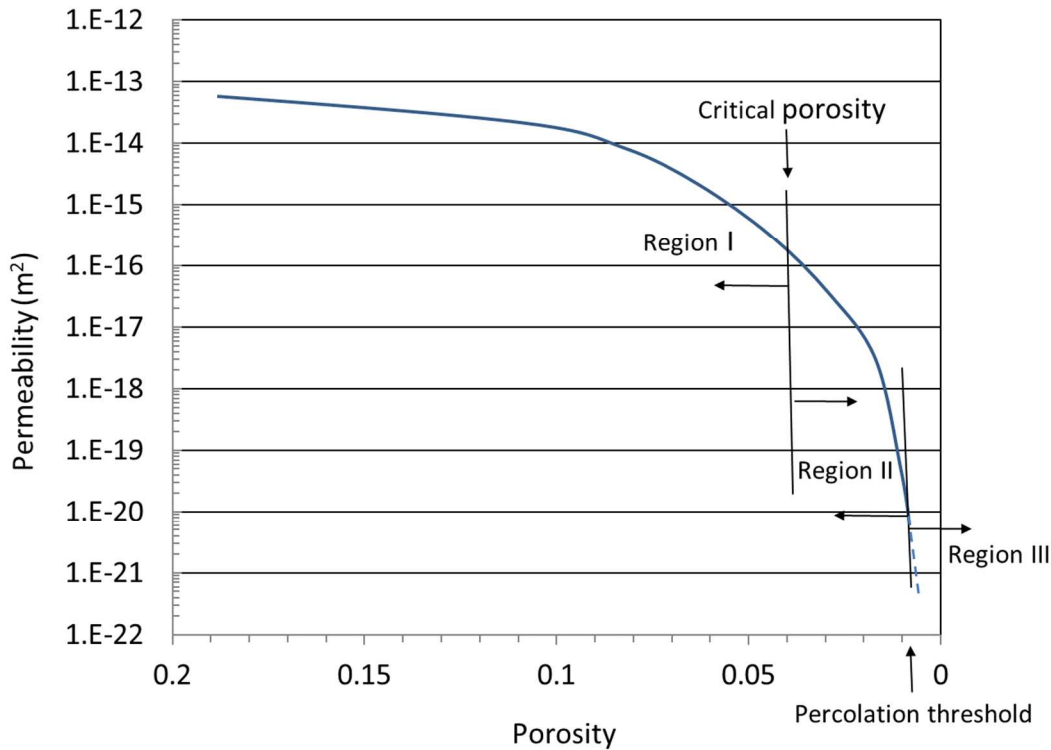


Fig. 3.34 - Proposed generalized permeability – porosity response of granular salt in response to consolidation.

Others have estimated the pore network becomes largely disconnected at a porosity of around 0.05 (Keller et al., 2012; Spiers and Brzesowsky, 1993; Holcomb and Shields, 1987). Our results suggest that, for these test conditions, the pore network remains sufficiently connected so that granular salt is much more permeable than intact salt until porosities of 0.01 or smaller.

The permeabilities given in Fig. 3.33 do not account for two important effects; gas viscosity changes with temperature and gas slip effects. During these measurements, the gas temperature may increase from contact with the heated sample, and consequently increase the gas viscosity. Attempts to measure the temperature of the gas immediately leaving the sample were unsatisfactory as the thermocouple could not be located sufficiently close to the exit from the sample. If the flowing gas is assumed to equilibrate with the temperature of the sample, the

viscosity change would increase the calculated permeability by 15% at 90 °C, 35% at 175 °C, and 50% at 250 °C.

Gas flow in low-permeability media can be affected by interaction between gas molecules and the flow path surface referred to as gas slip. This phenomenon results in a measured gas permeability that is greater than the intrinsic or liquid permeability. The measured single-phase gas permeability k can be expressed as (Klinkenberg 1941)

$$k = k_i \left(1 + \frac{b}{P_m}\right) \quad (3.5)$$

where k_i is the intrinsic permeability, P_m is the mean flowing gas pressure at which k is observed. The Klinkenberg coefficient, b , is a function of the pore dimensions and the properties of the gas. Jones and Owens (1980) developed an empirical expression for the Klinkenberg coefficient for air over a range of permeabilities from 10^{-14} to 10^{-19} m²

$$b = 0.98 k_i^{-0.33} \quad (3.6)$$

After correcting this expression for the test conditions here (Webb, 2006), the calculated Klinkenberg coefficient was used in Darcy's law to interpret the measured permeabilities as intrinsic permeabilities. Accounting for the slip effect by means of the assumed Klinkenberg coefficient in Equation 3.6 will lower the permeability compared to that reported in Fig. 3.33 by 20% to more than an order of magnitude depending on the test conditions.

In Fig. 3.35, the uncorrected permeability values (same data as Fig. 3.33) are given along with permeabilities corrected for both assumed viscosity changes and slip effects using the approach described above. These two corrections act in opposite sense, and so somewhat negate one another. The magnitude of the gas slip correction increases as permeability is reduced, so corrections at lower permeabilities are of greater magnitude. While the corrections mostly shift the permeabilities lower, the trends of the permeability with porosity do not change from those observed for the uncorrected permeabilities.

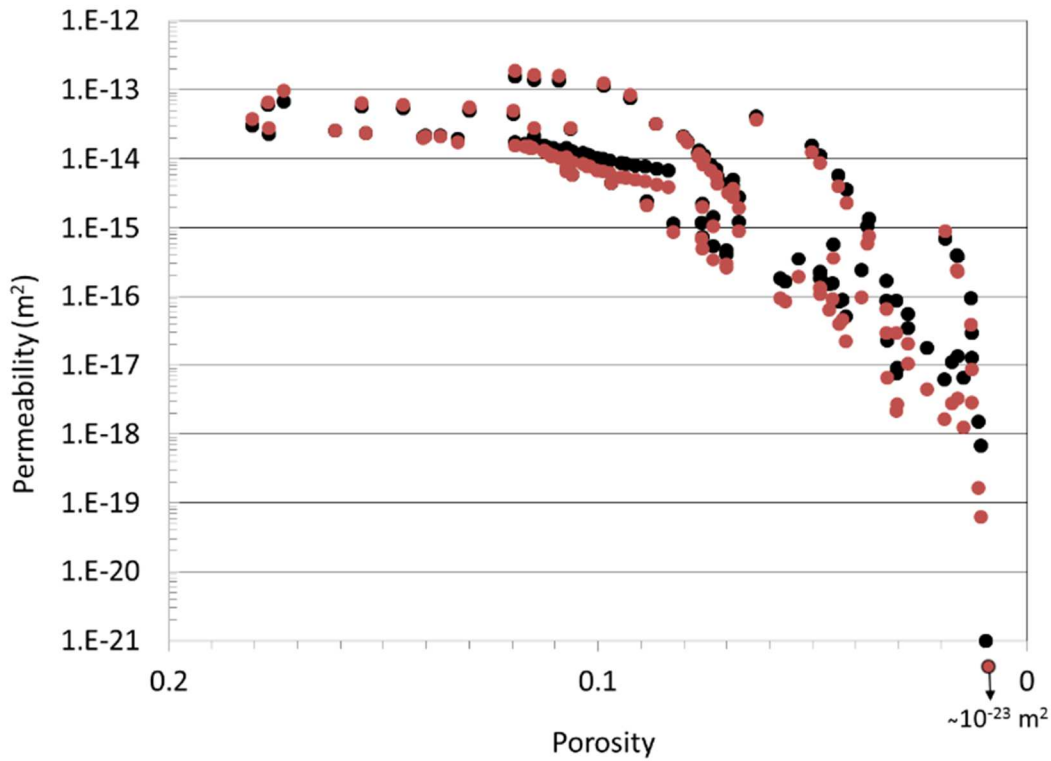


Fig. 3.35 – Permeabilities uncorrected (black) and corrected (red) for assumed viscosity and gas slip effects.

The aggregate data from all tests were fit to Equation 3.4. Using the corrected aggregate data, the best fit power law is given.

$$k = 2.55 \times 10^{-10} \varphi^{4.47} \quad (3.7)$$

It may be possible to fit the aggregate data with an exponent that increases as porosity decreases, but the scatter of data preclude generating a clear unique fit with this approach.

3.4 Conclusions

As the project progressed, we refined our capability to conduct consolidation tests under high pressures and temperatures with concurrent gas permeability measurements. This capability leveraged the unique facilities and experience of the SNL Geomechanics Lab. The consolidation tests go beyond previous test conditions, and are focused principally on achieving a high fractional density condition. Some of these tests included concurrent permeability measurements which provide important data to assess the thermal-mechanical-hydrological coupling of salt consolidation. Other tests included pore pressure during consolidation.

We summarize some conclusions from the consolidation testing below:

- Hydrostatic creep consolidation test response is strongly dependent on temperature. All consolidation tests conducted at 250 °C resulted in a final condition with a low porosity (<0.01) and high fractional density (>0.99). This final condition is independent of the type of salt, whether water added or not, and whether the sample was drained or undrained. Tests at 175 °C and 90 °C consolidate to low porosities, but at a slower rate.
- Water accelerates consolidation. This water can be “natural” or added. All samples – even those without added water – contain some water that may be released during heating and deformation during the consolidation testing. This water is available and likely involved with pressure solution mechanism. There appears to be more influence of added water on consolidation rate at lower temperature. The tests on the bedded salt at 90 °C clearly demonstrate the dramatic effect of added moisture on consolidation. In spite of a much shorter test duration, the sample with 1% additional water reached a significantly lower final porosity. The permeability of the sample without added water remained relatively great over the duration of the test, indicating this sample did not reach the critical porosity where the pores become disconnected from one another and the permeability decreases by orders of magnitude with small amounts of additional consolidation. Although the bedded salt contains water as evidenced by the water release of the sample tested at 250 °C, much of this water may not have been made available (released) at 90 °C and thus may not have enhanced the consolidation process.

- With the initial consolidation (preconsolidation) stage, samples can be prepared with a fractional density in the range of 0.8 to nearly 0.9 using hydrostatic stresses between 20 and 38 MPa. The initial consolidation is consistent with a range for compacted in place densities, depending on the particular placement method. Thus, the initial consolidation stage serves as a reasonable method for preparing samples for subsequent consolidation testing to evaluate as placed granular salt.
- There is evidence that pore pressure affects consolidation. An undrained test on the bedded salt generated a significant pore pressure and responded differently under low mean stresses compared to a comparable drained test. Subsequent multi-stage hydrostatic creep consolidation tests reveal that the volumetric strain rate (or rate of fractional density change) is responsive to effective stress changes and not to just confining pressure. These findings may be understood in terms of a local stress at the particle scale that varies from the macroscopic state of stress applied to the bulk sample. Based on a recognition of a local shear stress when interpreting these test results, P_p was observed to influence the shear stress within a bulk crushed-salt sample. This influence of P_p on $\dot{\epsilon}_v^p$ has largely been overlooked when interpreting the results from laboratory tests on ductile porous materials and does not appear to have been considered during the development of existing constitutive models for crushed salt.
- Although gas permeability measurements were conducted on samples during consolidation at different temperatures, pressures, durations and salt types, there is a similar general behavior between all samples. The gas permeabilities in consolidating rock salt decrease many orders of magnitude during consolidation. After the porosity reaches a critical value (in the 2 to 5% range), the permeability decreases at an increasing rate with continued consolidation. Consolidation to a low porosity (say <2%), the permeability may approach very small values. These results are consistent with the permeability evolving in three stages during consolidation. In the first stage, the porosity is decreasing but remains largely connected and involved in the flow. The second stage begins once a critical porosity is reached (perhaps on the order of 2 to 5%). In this stage, the permeability decrease with porosity is greatly accelerated. The decrease in permeability is due to the

loss of connectivity as some pores completely close. Isolated porosity may develop. Below a percolation threshold, there is no measurable permeability (the third stage). This percolation or porosity threshold corresponds to the porosity at which consolidation processes produce a complete loss of pore connectivity. The existence of a threshold porosity was not identified in this study. A very low permeability ($<10^{-20} \text{ m}^2$) was achieved from only one sample. These results suggest a very low porosity will have to be reached before the permeability of consolidating granular salt will be comparable to intact salt for conditions considered here.

4 Post-consolidation measurements

4.1 Sample preparation

After the consolidation test was completed, the consolidated salt was removed from the jacket assembly. Two discs, each approximately 25 mm thick, were cut from the top and bottom of the consolidated specimen using a diamond wire saw. The discs were used in the thermal properties testing and for microscopic observations. Subsequently, one of the discs was further cored to obtain 25.4 and 38.0 mm diameter sub-samples for porosity measurements.

The center portion of the cores was used for permeability and porosity testing. The center cores are variable and somewhat irregular in diameter (Figure 4.1). In order to give the sample a standard diameter to fit it in the permeameter, it is casted inside an epoxy cylinder of 10 cm (4 inch) diameter. After the dimensions and mass of the center core sample is measured, it is placed in a 10 cm diameter mold. Epoxy is poured in the mold so that the resulting sample is 10 cm diameter. The cylinder with the consolidated sample is placed in a vibrator and the epoxy mix is placed on the gap between the sample and the cylinder. After 18-24 hours, the sample is taken out of the cylinder and the excessive epoxy is removed. The sample is further cured at 65°C in an oven.



Figure 4.1 – Central core of post-consolidated sample prior to (left) and after being cast in epoxy (right).

In addition to laboratory consolidated samples, some of our testing included samples obtained from cores of partially consolidated granular salt backfill obtained from the Asse salt mine

located in Germany (referred to herein as BAMBUS samples). These samples were prepared in a manner similar to that for laboratory consolidated samples. We also performed thermal properties testing on two 100 mm diameter cylindrical cores of polycrystalline salt obtained from the WIPP facility. These cores had been previously subjected to uniaxial compression testing, one at 200°C and one at 250°C, which resulted in some dilation. Finally, we also tested the thermal properties of an intact salt crystal obtained from the Hockley Salt Dome in Texas.

4.2 Methods

4.2.1 Porosity

Porosity was measured in a number of different ways. On the discs sliced from the ends of the consolidated samples, porosity was measured with a gas porosimeter. Porosity was also estimated from the mass and volume of these sub-samples using an assumed grain density of 2.16 g/cc. These techniques are described in more detail in previous annual reports. Point counting on thin-sections produced from the discs also produced porosity estimates (described in Section 5).

Porosity measurements were made on the center core portion of the core using a differential pressure permeameter. Essentially, the permeameter is used as a gas porosimeter. Porosity estimates from fluid immersion (volume) measurements made on the prepared samples before and after consolidation testing were found to be unreliable. We have also used the vacuum sealing method for measuring the volume, specific gravity and porosity on post-consolidated samples. The system we have used is named *Corelok* supplied by *InstroTek Inc* (shown in Fig. 4.2). This system is predominantly used for sealing the specimen so that the bulk specific gravity of the sample can be measured using water displacement method (using Archimedes' Principle). As programmed, oven-dried salt samples are automatically sealed in a specially designed polymer membrane. A proprietary software named *CoreGravity* was used to compute the bulk specific gravity. Water temperature was also recorded during the test to calculate the total volume of the sample. For maximum specific gravity, the dry salt sample was placed inside the vacuum bags and sealed within the *CoreLok* Vacuum chamber. The sample sealed in polymer membrane are then cut open under fluid (Silicone Oil) of known density at laboratory

temperature and submerged weight was determined. The weight in air and submerged weight (in Silicone Oil) was used to assess the maximum specific gravity of the sample.

The total porosity of the sample was then calculated from the maximum specific gravity and bulk specific gravity data. The *GravitySuite* PC software package was used for calculating and managing data. Special care has been taken during each measurement to minimize the error.



Figure 4.2: CoreLok apparatus with sealed sample

4.2.2 Thermal properties

Thermal properties measurements were made using the transient plane source method with a Hot Disk® TPS 1500. In this method, a heat pulse is applied with a thin plane sensor that is sandwiched between two pieces of the material of interest. Thermal properties are interpreted numerically from the dissipation of the heat pulse with a time. The thermal properties that are obtained are thermal conductivity, diffusivity, and specific heat.

4.2.3 Permeability measurements

Gas permeability and porosity measurements were made on samples which had previously been consolidated under a wide range of stress, temperature, and moisture conditions. A description of the conduct of the consolidation tests is given elsewhere in this report and previous project reports. A summary of the samples and the test conditions are included in Table 4.1 below.

Table 4.1 – Summary of consolidation test condition for samples used for post-consolidation gas permeability tests.

Sample	Temperature (°C)	Maximum stress (MPa)	Added moisture?	Duration (days)
W250-03	250	38	n	9
W250-02	250	20	n	12
W250-01	250	20	n	12
W175-10	175	20	n	5
W175-03	175	20	n	3
W175-04	175	20	n	38
W175-01	175	20	n	2
W90-08	90	40	n	9
W90-07	90	20	1%	24
W90-04	90	20	1%	5
W90-03	90	20	1%	2
W90-01	90	20	n	112

The permeability and porosity measurements were made using the configuration shown in Figure 4.3. The test samples were placed in a triaxial cell capable of applying a confining pressure of 30 MPa and an independent axial load. All tests reported here were conducted under hydrostatic stresses, that is, the axial stress was equivalent to the lateral confining pressure. The triaxial cell includes ports on both end caps that allow fluids ingress or egress to the sample. One end of the ported end cap is attached to the “upstream” side of the measurement system, and the other is attached to the “downstream” side. The volumes of the components on the upstream and downstream sides (tubing, valves) were previously carefully measured. The gas used was nitrogen or helium. Upstream, downstream, and room temperatures were monitored with thermocouples. All measurements reported here were made at ambient temperature (nominally 23 C), and with a hydrostatic stress of 20 MPa.

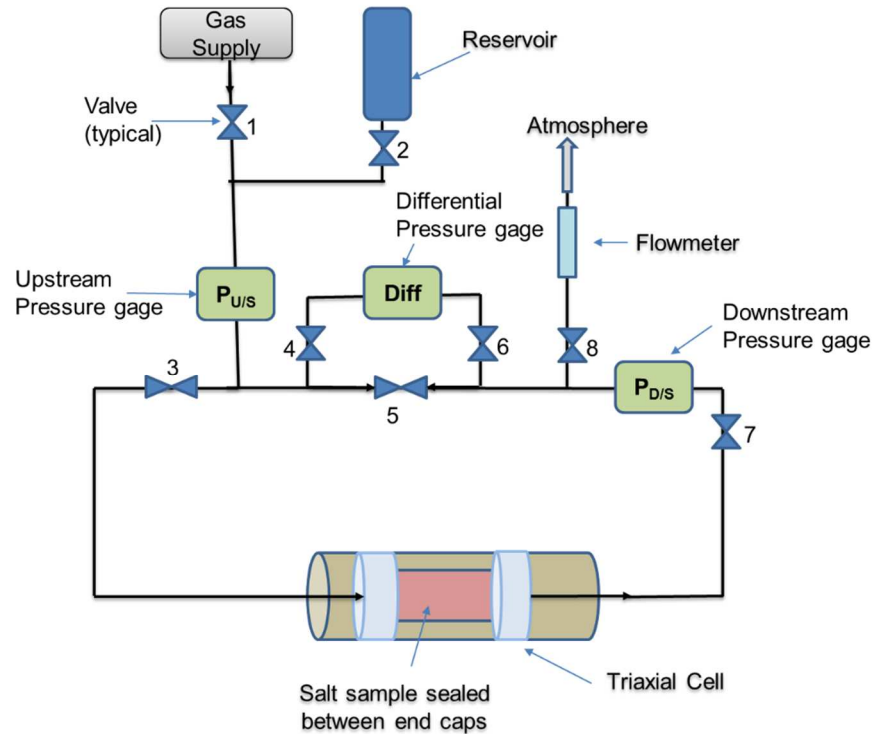


Fig. 4.3 – Schematic of system used to measure gas permeability and porosity on post-consolidated samples.

Porosity measurements were made using gas expansion methods. Gas under pressure in the upstream side is exposed to the sample and the downstream, and allowed to equilibrate. The pore volume can then be calculated from the equilibrium pressure and the known upstream and downstream volumes. This test can be conducted with the sample and the downstream volume at an initial pore pressure other than atmospheric.

Permeability was measured using three methods: steady-state (using a flowmeter); quasi-steady-state (using the so-called pressure fall-off method); and transient (using a pressure pulse method). The steady-state method uses a flowmeter on the downstream side of the sample. The flowrate is used in Darcy's law to interpret permeability. The quasi-steady-state method is similar to that described for the concurrent measurement system. From a measured pressure drop in the upstream volume, a flowrate can be interpreted (using ideal gas law). This flowrate is then used in Darcy's law to interpret permeability. In the transient test method (Brace et al., 1968), the upstream and downstream pressures are first equilibrated. The upstream pressure is then increased by nominally 10%, and the decay of the pressure difference is monitored with time as gas flows from the

upstream to the downstream. This technique can be used to interpret permeabilities as low as 10^{-21} m^2 .

All of the permeability measurements methods described above can be made with a backpressure; that is, with the downstream boundary at a condition other than atmospheric pressure. In this way, permeability at different pore pressures could be measured.

For each sample, tests were made under a range of pore pressures (back pressures) and differential pressures. These measurements provided the data to correct for non-linear flow and Klinkenberg effects. To account for possible non-linear flow, data were interpreted using the Forchheimer equation, which includes both viscous (Darcy) and inertial (non-linear) flow terms. The Forchheimer equation can be written as

$$-\frac{\nabla P}{Q} = \frac{\mu}{kA} + \frac{\beta\rho}{A^2} Q \quad (4.1)$$

where ∇P is the gradient, Q is the volumetric flowrate, k is the permeability, β is the inertial coefficient, A is the cross sectional area involved in the flow, μ is viscosity, and ρ is density. Plotting the left-hand side of Equation 4.1 vs. the flowrate yields a straight line with a slope that is a function of the inertial coefficient and an intercept proportional to permeability. In the absence of non-linear flow, the slope will be zero and Equation 4.1 reduces to Darcy's Law.

Gas slip effects were evaluated from data collected at different mean gas pressures (Klinkenberg, 1941). By fitting the gas permeability measured at different mean pressures to Equation 3.5, the intrinsic permeability (corrected for slip effects) and the Klinkenberg correction term are determined.

4.3 Results

4.3.1 Porosity

Porosity data are compiled in Figure 4.4 and Figure 4.5. The data in Figure 4.4 are from end pieces. In general, there is good agreement between the methods used to determine porosity on the discs cut from the sample ends. Point counting values tend to be somewhat lower than the other methods.

Data from central cores is given in Figure 4.5. Where there are data from both the vacuum sealing method (Corelok) and permeameter, the values are comparable. There is no discernable trend in the differences between the two methods. For W-175-13, the porosity was believed to be <0.01 , but the permeability was so low that a definitive measurement was not made.

The central core has a somewhat smaller porosity compared to the end pieces. This result is consistent with the observation that most samples appeared to have a slight hourglass shape after creep consolidation.

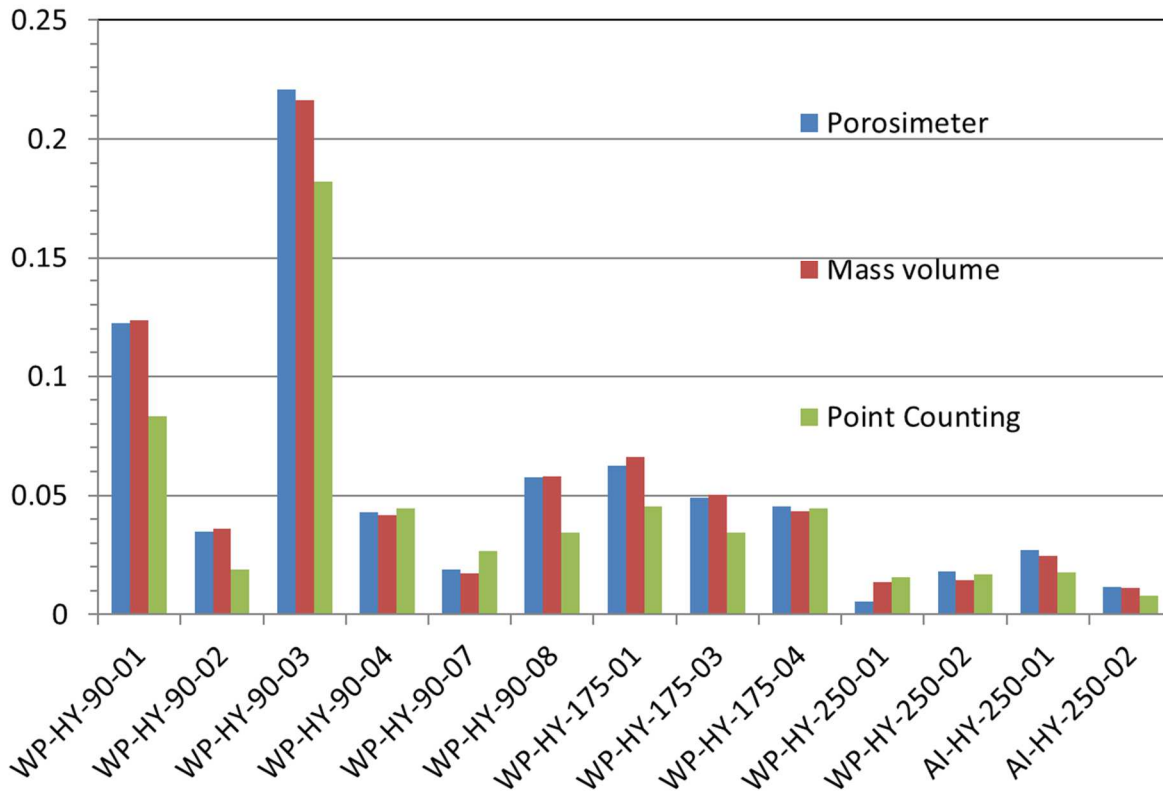


Figure 4.4 – Summary of porosity measurements on end pieces of post-consolidated samples.

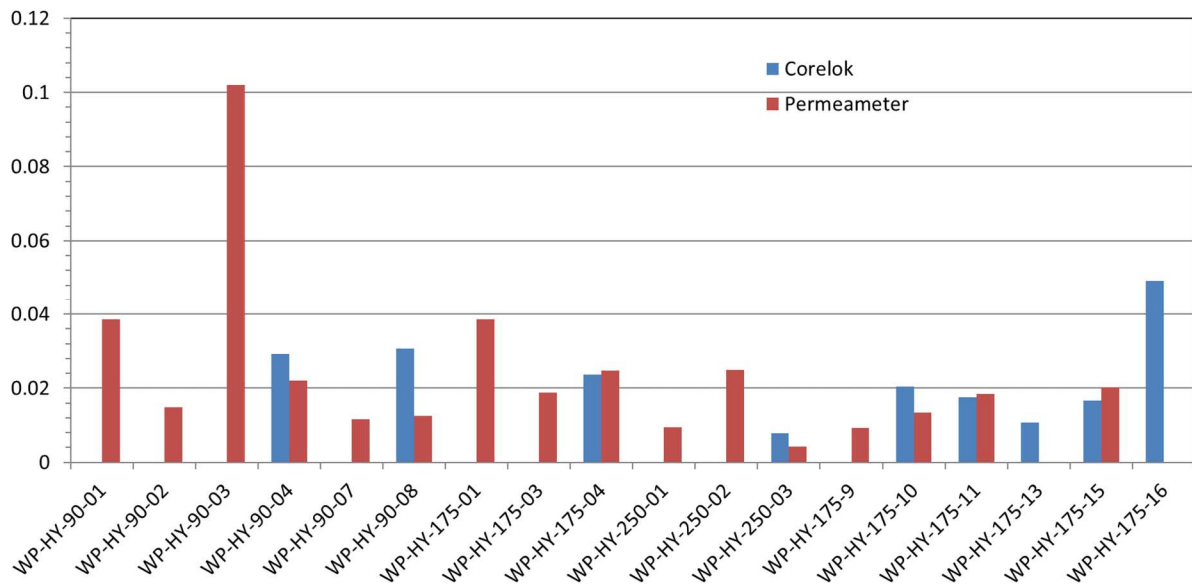
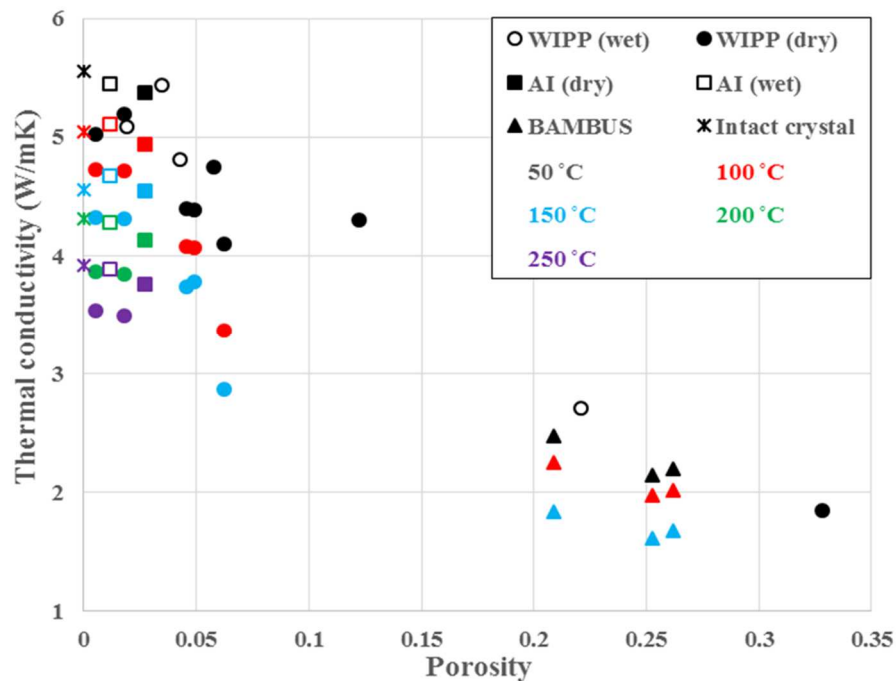


Figure 4.5 – Summary of porosity measurements on central core of post-consolidated samples.

4.3.2 Thermal properties

Thermal conductivity and specific heat of an intact salt crystal, laboratory consolidated salt and in situ consolidated salt at various temperatures are plotted against porosity in Figure 4.6 and 4.7, respectively. Results indicate that thermal conductivity of intact salt crystal decreases with increase in temperature, while specific heat appears to increase with an increase in temperature. For granular salt, thermal conductivity decreases with increase in temperature and porosity. Specific heat increases with increase in temperature. At lower temperatures, specific heat decreases with increase in porosity. At higher temperatures, porosity dependence is not apparent.



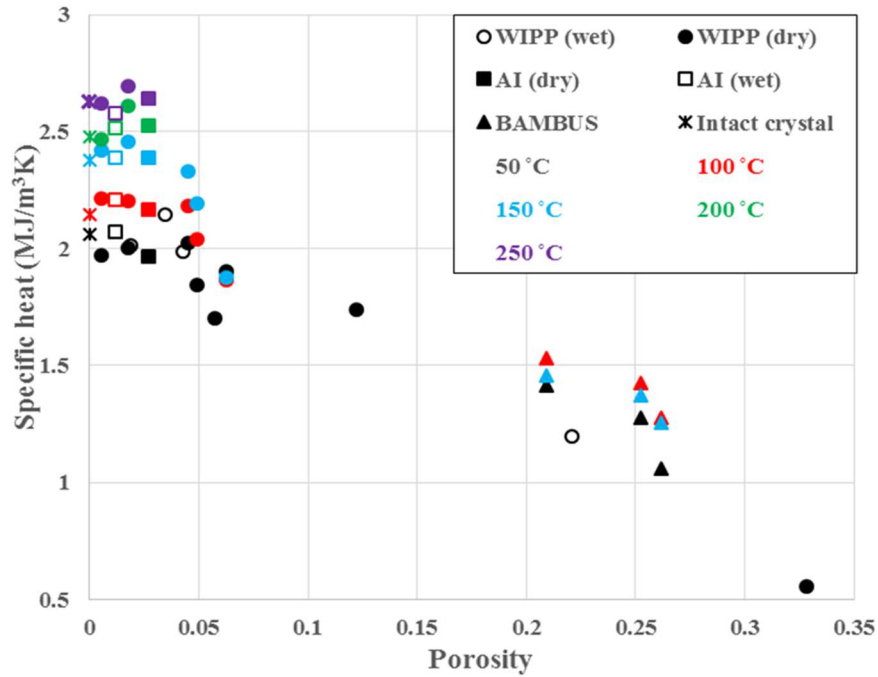


Figure 4.7 Specific heat of sub-samples vs porosity at various temperatures.

Measured thermal conductivity of dilated salt is given in Figure 4.8. For each sample, separate measurements were made in the vertical and horizontal plane. In one sample, the vertical and horizontal measurements nearly coincide, whereas in the other sample the values along the horizontal plane are larger. In all cases, the thermal conductivity decreases with temperature.

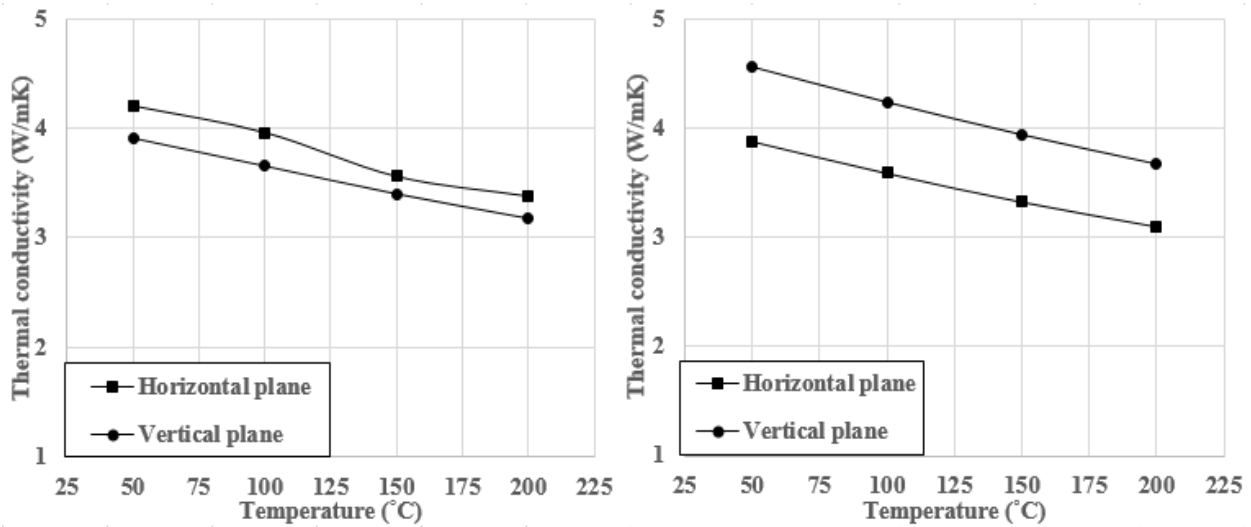


Figure 4.8 Thermal conductivity of dilated polycrystalline salt samples: WP-DL-200 (left) and WP-DL-250 (right).

The dilated salt has a lower thermal conductivity compared to granular salt at a comparable porosity (Figure 4.9).

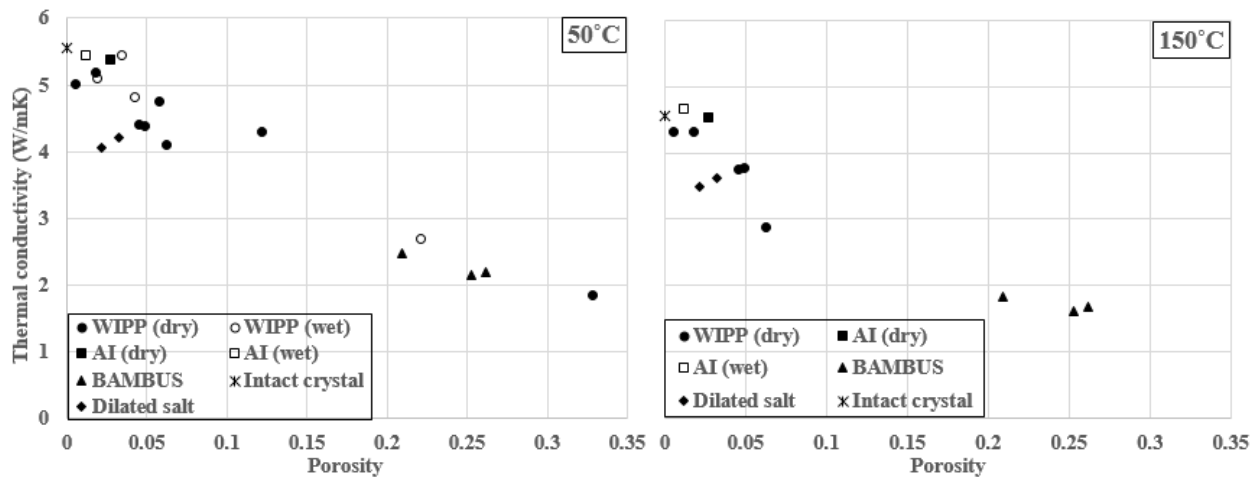


Figure 4.9 Thermal conductivities including dilated salt values.

4.3.3 Permeability

The data are summarized in Fig. 4.10. These data were conducted under a nominal confining pressure of 20 MPa. The data reveal the general trend of reduced permeability with decreasing porosity, although at porosities of 1% and lower the permeability appears to be more variable. The permeabilities from the samples that we consolidated with water do not appear to be systematically lower than those conducted without additional moisture.

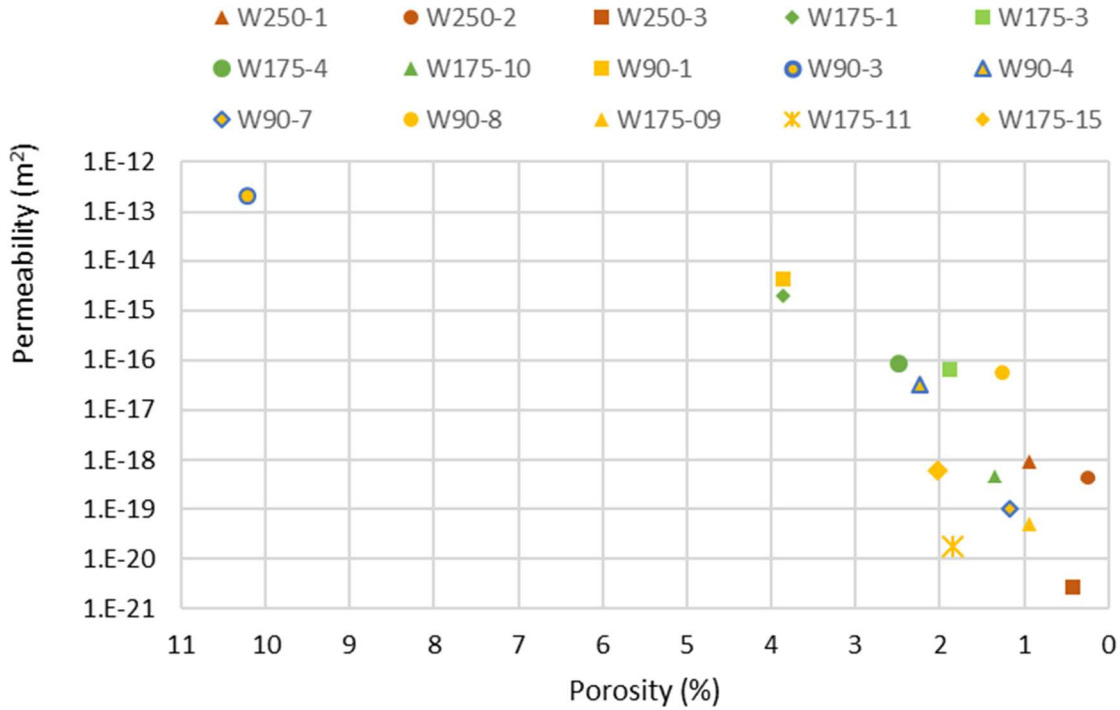


Fig. 4.10 – Summary of gas permeability vs. porosity data from tests on post-consolidated cores. The samples that had water added (1%) prior to consolidation are identified with a blue border around the symbol.

These data yield the following best fit to the power law expression of Equation 3.4

$$k = 6.64 \times 10^{-10} \phi^{4.47} \quad (4.2)$$

4.4 Discussion

4.4.1 Porosity

The similarity of porosities derived from the porosimeter, which determines connected porosity, and MV method, which estimate the total porosity, indicates the porosity of laboratory consolidated samples is largely connected. Below a total porosity of about 0.02, there is an indication that the connected porosity may be lower. Porosity of sub-samples taken from the sample ends is mostly greater than that from the central cores which suggests that the specimens may not have deformed uniformly. Typically the central portion of the core appeared to be smaller than near the edges, which would favor a lower porosity of the central cores compared to the ends where the sub-samples were obtained.

4.4.2 Thermal properties models

4.4.2.1 Intact salt crystal

Measured thermal conductivity and specific heat of an intact salt crystal were fit as a function of temperature to linear, quadratic, cubic, and exponential expressions (Table 4.2). The measured thermal properties of an intact salt crystal were also compared to other models. Sum of the squared errors (SSE) obtained from the measured and the predicted values are listed in Table 4.2.

The thermal conductivity of an intact salt crystal decreases with increasing temperature. The thermal conductivity of the intact salt crystal were reasonably well fit as a linear function of temperature. For a larger range of temperatures, a polynomial fit may be more appropriate as has been given by Urquhart and Bauer (2015) for a range from -75 °C to 300 °C. Specific heat of an intact salt crystal increases with increasing temperature. In contrast, Urquhart and Bauer (2015) found no clear temperature dependence of specific heat for intact salt crystal.

Table 4.2 Thermal properties of intact salt crystal fitted to empirical equations and compared to other models.

Thermal conductivity equations		SSE
Fitting to simple expressions	$K_0 = -7.98 \times 10^{-3}T + 5.876$	0.025
	$K_0 = 1.374 \times 10^{-5}T^2 - 1.21 \times 10^{-2}T + 6.116$	0.008
	$K_0 = -1.12 \times 10^{-7}T^3 + 6.414 \times 10^{-5}T^2 - 1.87 \times 10^{-2}T + 6.352$	0.005
	$K_0 = 5.996e^{-0.002T}$	0.218
Comparison to other models	$K_0 = 1.2 \times 10^{-1}T^{-2} - 6.11T + 7.01$ (Smith, 1976)	1.210
	$K_0 = -1.717 \times 10^{-8}T^3 + 3.12 \times 10^{-5}T^2 - 2.1 \times 10^{-2}T + 7.07$ (Yang, 1981)	0.646 ^a
	$K_0 = -1.51 \times 10^{-8}T^3 + 2.86 \times 10^{-5}T^2 - 1.838 \times 10^{-2}T + 5.734$ (Bechthold et al., 1999)	5.237
	$K_0 = -2 \times 10^{-7}T^3 + 1 \times 10^{-4}T^2 - 3.17 \times 10^{-2}T + 6.8203$ (Urquhart and Bauer, 2015)	6.887
Specific heat equations		SSE
Fitting to simple expressions	$C_0 = 2.92 \times 10^{-3}T + 1.9$	0.004
	$C_0 = -3.43 \times 10^{-7}T^2 + 3.02 \times 10^{-3}T + 1.894$	0.004
	$C_0 = -6 \times 10^{-8}T^3 + 2.665 \times 10^{-5}T^2 - 5.17 \times 10^{-4}T + 2.02$	0.005
	$C_0 = 1.929e^{0.0013T}$	0.006
Comparison to other models	$C_0 = -1.09 \times 10^{-3}T^{-2} + 2.83 \times 10^{-2}T + 2.06 \times 10^{-1}$ (Smith, 1976)	1.048
	$C_0 = 3.499 \times 10^{-10}T^3 - 8.453 \times 10^{-7}T^2 + 6.43 \times 10^{-4}T + 1.864$ (Yang, 1981)	0.210 ^a
	$C_0 = 0.177T + 855$ (Bechthold et al., 1999)	1.010

^aThermal conductivity and specific heat equations were obtained by fitting the recommended values. T is temperature in Celsius except for equations suggested by Smith, 1976 are in Kelvin.

4.4.2.2 Granular salt

Thermal properties of granular salt were fit to the following expressions

$$K_{gs}(T, \phi) = K_{int}(T)f(\phi) \quad (4.3)$$

$$C_{gs}(T, \phi) = C_{int}(T)g(\phi) \quad (4.4)$$

With these expressions, the dependence on temperature is accounted for by the term for the thermal property of intact salt. The second term in these expressions is a function of porosity only. Using the linear expressions for thermal conductivity and specific heat given in Table 4.3 for $K_{int}(T)$ and $C_{int}(T)$, various forms for the functional dependence on porosity ($f(\phi)$ and $g(\phi)$) were fit to data (Table 4.3). The data are also compared with other models in Table 4.3.

Thermal conductivity decreases with increase in temperature and porosity. We found that the data was well fit with the form of Equations 4.3 and 4.4 with various forms to capture the dependence

on porosity, with quadratic, cubic, and exponential forms performing nearly equally as well. Specific heat at lower temperatures is observed to decrease with increase in porosity. However, at higher temperatures porosity dependence is not apparent.

Table 4.3 Thermal properties of granular salt fitted to empirical equations and other models.

Thermal conductivity equations		SSE
Fitting to simple expressions	$K_{gs} = K_0 (1 - 2.46\phi)$	5.869
	$K_{gs} = K_0 (5.08\phi^2 - 3.696\phi + 1)$	4.140
	$K_{gs} = K_0 (-1.207\phi^3 + 5.556\phi^2 - 3.735\phi + 1)$	4.138
	$K_{gs} = K_0 (47.64\phi^4 - 29.44\phi^3 + 10.23\phi^2 - 3.89\phi + 1)$	4.111
	$K_{gs} = K e^{-3.73\phi}$	4.168
Comparison to other models	$K_{gs} = K_0 (1 - 2.7\phi)$ (Bechthold et al., 1999)	7.214
	$K_{gs} = K_0 (-54\phi^4 + 74\phi^3 - 27.2\phi^2 + 0.3\phi + 1)$ (Bechthold et al., 2004)	13.360
	$K_{gs} = K_0^{1-\phi} K_a^\phi$ (Macaulay et al., 2015)	10.464
Specific heat equations		SSE
Fitting to simple expressions	$C_{gs} = C_0 (1 - 1.7\phi)$	0.655
	$C_{gs} = C_0 (-2.525\phi^2 - 1.17\phi + 1)$	0.535
	$C_{gs} = C_0 (-8.305\phi^3 - 0.5\phi^2 - 1.04\phi + 1)$	0.477
	$C_{gs} = C_0 (-383.77\phi^4 + 221.75\phi^3 - 39.61\phi^2 + 0.43\phi + 1)$	0.372
	$C_{gs} = C_0 e^{-2.107\phi}$	0.850
Comparison to other models	$C_{gs} = C_0 (1 - \phi)$ (Bechthold et al., 1999)	2.257
	$C_{gs} = C_0 (1 - \phi) + C_a\phi$ (Jury and Horton, 2004)	2.259

T is temperature in Celsius and ϕ is porosity.

4.4.3 Permeability

All of the concurrent and post-consolidation data are plotted together in Fig. 4.11. The trend of permeability and porosity is consistent between the two data sets. The similarity of the response is expressed in the similar forms of the power law functions fit to the data sets. The testing of the post-consolidated samples was under more controlled conditions and fewer uncertainties; consequently, we believe this data set provides the best estimate of permeability as a function of porosity.

The permeabilities interpreted from the post-consolidated samples are somewhat greater than those measured concurrent with consolidation. For specific samples that were tested both concurrent

with consolidation and subsequently post-consolidation, the final permeability interpreted from testing during consolidation were always somewhat lower than that when tested on the post-consolidated sample. The cause of this response is not definitely known, but may be due to porosity changes upon cooling after the consolidation test. This response is difficult to measure, and the non-uniform porosity of consolidated samples makes this challenging to sort out.

Both data sets indicate that rock salt remains conductive until a very low porosity is achieved. Others have estimated the pore network in rock salt becomes largely disconnected at a porosity of around 0.05 (Keller et al., 2012; Spiers and Brzesowsky, 1993; Holcomb and Shields, 1987). Our results suggest that, for these test conditions, the pore network remains sufficiently connected so that granular salt is much more permeable than intact salt at least until very small porosities of 0.01 or smaller. Limited measurements by others on granular salt with porosities around 0.01 also indicate the material can have a permeability at least two orders of magnitude greater than that of intact salt (Krohn et al., 2015; Bechtold et al., 2004).

At the same porosity, there is no evidence that samples with 1% additional moisture have a systematically lower permeability than those without added water. This is not to say that water is not important to consolidation; indeed, it greatly affects consolidation rates. Also, more added water may have a greater effect on the resulting pore structure and permeability; however, the amount of added water will likely be limited to around 1% in order to prevent a significant brine-filled connected porosity as the final condition of consolidation. Finally, we recall that all samples contained some moisture, and that moisture may be released during consolidation.

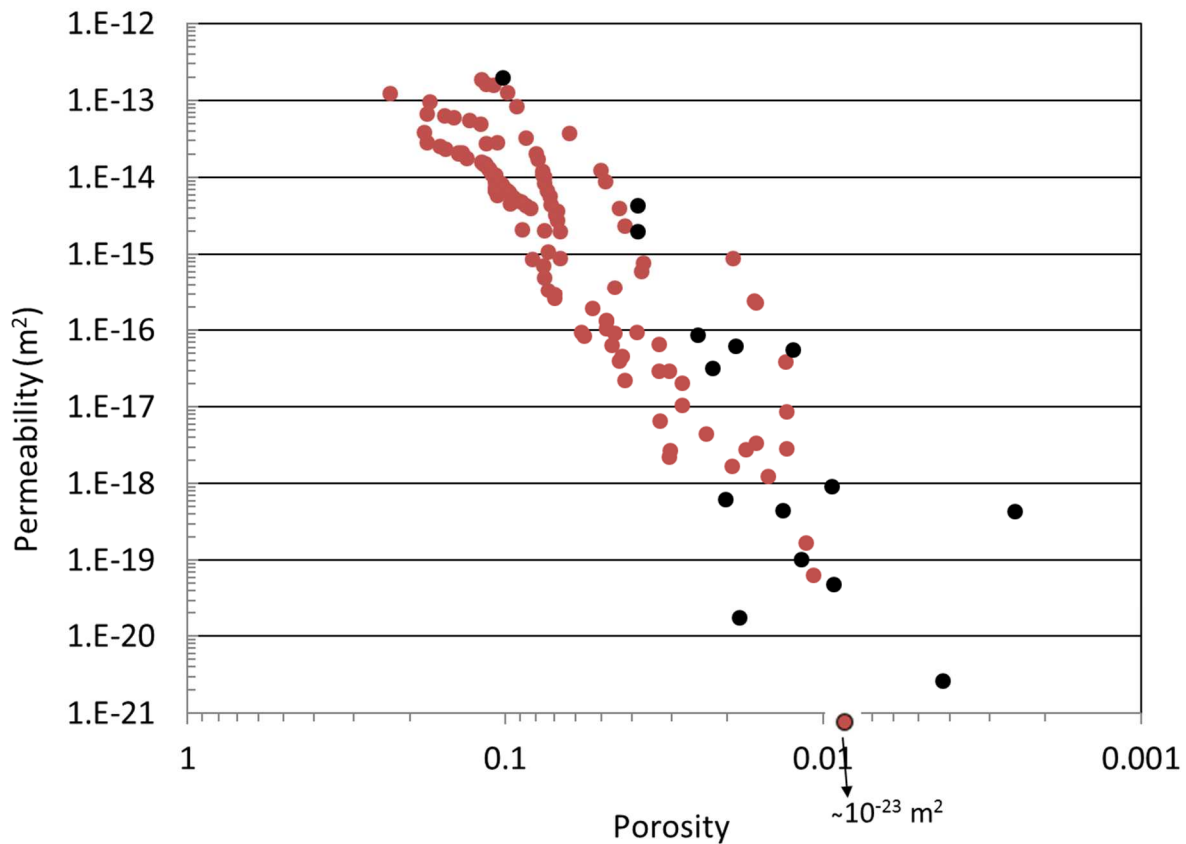


Fig 4.11 – Comparison of gas permeability and porosity data obtained concurrent with consolidation (corrected data, red) and on post-consolidated samples (blue).

In Figure 4.12, the power law relationships derived from the concurrent testing and the post-consolidation are plotted with expressions of others. We note that in spite of differences between the type and size of the salt used and the consolidation method, there is a remarkable similarity of the function derived by Cinar et al. and that derived from the measurements on the post-consolidated samples.

We conclude that intrinsic permeability can be reasonably expressed solely as a function of porosity to porosities of less than 2%, or fractional densities of greater than 0.98. Our results suggest the form of Equation 3.4 to describe this relationship. We note that permeability should

be adjusted for temperature to account for the temperature dependence of the fluid's viscosity. For gas flow, correction for the gas slip effect as a function of pore pressure will be required.

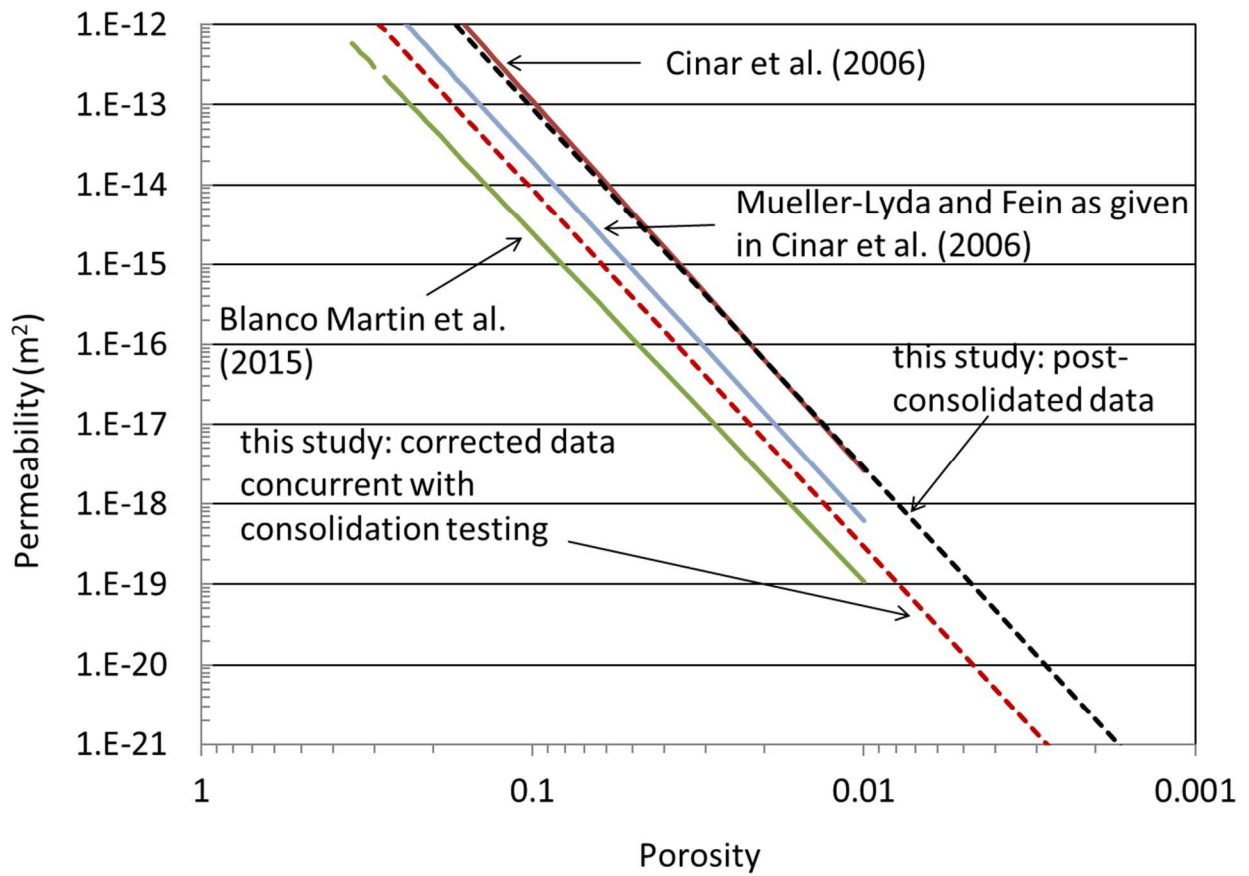


Fig. 4.12 – Power law relationships for gas permeability – porosity including those derived from this study.

5 Microstructural observations

Microstructural observations made on samples after they have been consolidated can be used to interpret deformation mechanisms and evaluate the ability of the constitutive model to predict operative mechanisms under different conditions. These observations require the use of optical microscopes and scanning electron microscopes. Considerable training in the use of these sophisticated devices is required. In addition, interpreting the images in terms of deformation mechanisms is somewhat subjective and requires experience and training. Consequently, much of this past year has involved training students on the obtaining and interpreting microstructural images using samples from previous investigations.

Because the permeability of granular salt at high fractional densities is of particular interest, there is a need to understand the pore structure that remains in the granular salt. In addition information gained from measuring transport properties (i.e., permeability, porosity) during the consolidation testing, we are investigating methods to interpret pore structure for subsequent microscopic observation.

5.1 Materials and Methods

5.1.1 Sample Preparation

In order to perform numerous types of observations, multiple samples were produced from both deformed and native salt. They comprise of polished thick petrographic sections and freshly broken surfaces. Ends of consolidated samples were cut with a diamond wire saw to minimize damage and create sub-samples. Six of these sub-samples were sent to Wagner Petrographic to be thin sectioned. Direction was given to have each sample be a standard 24 x 46 mm thin section, vacuum impregnated with blue epoxy, double polished, and be 2 mm thick. Due to long turn around times and uncertain methods performed commercially, thin sections for remaining samples were made in-house.

This involved sub-samples, typically a quarter of a cylinder end, to be placed in plastic bags where tape was applied around the outside to confine each side and then put into aluminum dishes to provide a flat bottom surface. Spurr Low-Viscosity resin doped with rhodamine-B was poured on the top, making sure an adequate amount puddled, followed by placement in a bell jar.

The bell jar was put under approximately 10 psi vacuum (approximately 3 psia inside the bell jar), then vented slowly to atmospheric pressure. This helps any evacuated voids be filled with epoxy and is repeated 2-3 more times, with the addition of more epoxy if needed, for assurance of a fully impregnated sample. Afterwards, the samples are placed in an oven at 50°C overnight to cure.

Once cured, samples were ground down on a Buehler Ecomet III polisher and grinder to expose a fresh, flat surface of the sample. Once reached, 5-minute Devcon epoxy was applied to the fresh surface and a glass slide, which was further placed on the sample with enough pressure to remove any air in between. Next, a Buehler IsoMet low speed saw with a vacuum thin section chuck was used to cut samples about a millimeter thick. Polishing was conducted on the same Buehler polisher, using a range of abrasive grit sizes from 400 to 1200 that is continuously wetted with isopropanol. A Leitz Ortholux II optical microscope equipped with a Leica camera and Leica Application Suite software was used to examine produced thin sections, point count, and capture images.

Etching, a technique that allows for heavily deformed grains under multiple mechanisms to be highlighted, was also conducted on thin sections. This was done by either submersion into a solution of methanol saturated with PbCl_2 as the etchant followed by transferring to butanol and rapidly dried, or by the swift swipe of a KimWipe dampened by DI water.

Fresh aggregate surfaces were broken off by hand from sub-samples to expose a clean surface, which can exhibit a diagnostics of sample cohesiveness, grain boundary characteristics, and other evidence of microprocesses. For examination, these aggregates were hot glued to a standard SEM stub containing a strip of carbon tape. Both thin-sectioned samples and aggregates were coated with AuPd and were investigated under two SEM's: a FEI Nova 200 Nanolab and a Tescan Vega3 LM.

5.1.2 Experimental Systems and Methods

A FEI Nova 200 Nanolab, Figure 5.1, is an instrument with a dual beam system of a focused ion beam column and SEM column. The SEM column is equipped with a tungsten filament for imaging in low or high vacuum levels. The detector has a resolution of 3.5 nm operating at 30 kV at high vacuum and less than 15 nm at 3 kV when operating in low vacuum. The instrument is also equipped with INCA Synergy 350 with HKL Premium EBSD System, which allows elemental analysis of samples as well as the capability of generating phase maps from elemental maps and Cameo data. Samples up to 12 inches in diameter can then be mapped. A HKL Premium Electron Backscatter Detector can be used within the machine for crystallographic determination to create orientation maps of microstructures, generate combined orientation and elemental maps, and generate pole figures.



Figure 5.1: FEI Nova 200 Nanolab SEM.

The Tescan Vega3 LM SEM, shown in Figure 5.2 and located at SNL, is equipped with a conventional tungsten heated cathode that can be used in high vacuum or low vacuum. Resolution down to at least 3 nm, an accelerating voltage range from 0.2 to 30 kV, and continuous magnification from 2.5x to 1,000,000x produces excellent images for observational work. The large analytical chamber with a 5-axis motorized stage permits easy placement of multiple sample types. The EDX and EBSD enabled ports allow for elemental analysis spectrums and back scattering detection.



Figure 5.2: Tescan Vega3 LM SEM

In order to prepare non-conducting or poorly conducting samples for observation in a SEM instrument, a sputter coater must be used. The EmiTech KX950 sputter coater, Figure 5.3, allows for samples to be thinly coated by carbon or gold-palladium to increase the electrical conduction. The palladium acts as a physical barrier to the gold, which attempts to conglomerate into large islands and restrict ultimate resolution performance. It is equipped with a turbo pump evaporator for complete automatic control during evaporation of the chamber to low pressures while having a dry gas inlet to improve deposition of the coating. This sputter coater is also used with a K150X film thickness monitor that measures the thickness of the coating that has been deposited on a crystal within the chamber. This monitor allows more control due to some samples having more delicate features than others, such as an etch, and too thick of a film will diminish these features.

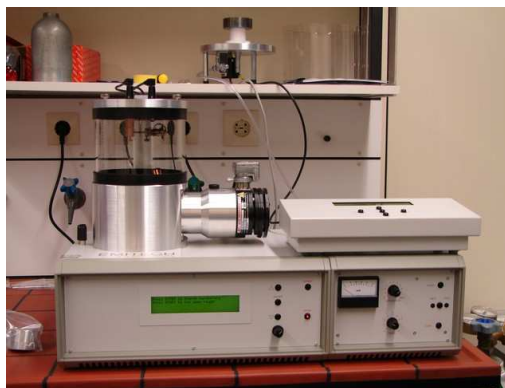


Figure 5.3: EmiTech KX950 sputter coater with K150X film thickness monitor.

Using the sputter coater, samples could be coated in carbon or gold-palladium to view within the SEM. A carbon rod with a 3mm sharpened spigot shaped head is ignited and evaporated producing a 25nm thick coat for a 5 second pulse. Carbon coating is generally used for the unetched, consolidated mass samples since greater detail is not as desirable. However, for samples that have been etched, gold-palladium is used to produce high-resolution coating. A consistent problem when coating has been figuring out the most favorable thickness. Too thick of a coating will cover the subgrain features produced by etching, while too thin will not highlight them enough to be observable. From a series of tests with samples each coated different thicknesses, 9.5nm produced the best results to view the etched subgrain features.

A Leitz Ortholux II optical microscope at SNL, Figure 5.4, uses combined transmitted and incident polarized light with a five objective nosepiece to examine samples. The microscope is equipped with a Leica camera and Lieca Application Suite software to capture images of any sample. It is also equipped with a Leitz mechanical slide stage used for point counting in a grid-like pattern. Point counting on thin sections was done to manually determine the porosity of a sample by counting the number of void spaces and solid spaces in a grid-like pattern for a minimal of 300 counts three times.



Figure 5.4: Leitz Ortholux II optical microscope

After salt is consolidated into lead jacketed cylinders, the samples need to be cut in order to make thin-sections, obtain aggregated samples, and perform thermal conductivity tests. A diamond wire saw in the Geomechanics Lab at SNL, shown in Figure 5.5, is the best type of saw to cut rock salt due to its semi-fragile nature. The sample is first placed on the platform, making

sure it is stable by using weights or clamps, so it does not move while cutting. The wire arm is then lowered just above the sample and then the pump is turned on for the rotating wheel. The wheel changes rotating directions due to magnetic sensors, which allows the wire to be pulled back and forth. By placing a small amount of weight of about 5 grams on the wire arm, the arm lowers slowly down through the sample. Sensors located on the sides of the arm control how low the wire goes and effectively shuts off the pump once the arm is close.



Figure 5.5: Diamond wire saw with ventilation, pulley system, adjustable table, wheel, pump and pump controller.

A Buehler IsoMet low speed saw, shown in Figure 5.6, is used for making precision cut thin sections with a diamond tipped saw and is also found at the Geomechanics Lab at SNL. Once the surface of an impregnated sample is glued to a glass slide, the slide is suctioned into a vacuum chuck holder with the help of a small amount of vacuum grease to create a tight seal. The holder is then screwed onto the arm above the blade and it's position adjusted by a micrometer. The micrometer allows for manual determination of thickness for each cut. Weights on the arm control the loading of the specimen, which governs the speed of the cut. For salt specimens, isopropanol is used as the cutting liquid and is poured into a reservoir below the blade, refilling regularly due to evaporation.



Figure 5.6: Buehler IsoMet low speed saw

Figure 5.7 shows a Buehler Ecomet III polisher and grinder, which is used to polish thin sections after cutting and also to create smooth, even surfaces for porosity samples. Abrasive, circular paper, ranging in grit size from 400 to 1200, is adhered to a rotating wheel. The paper is wetted with isopropanol and samples are held on the wheel with minimal pressure until a smooth surface is acquired.



Figure 5.7: Buehler Ecomet III polisher and grinder

Fiji is an open source image processing software, which includes ImageJ as well as multiple other plugins that provide a wide range of applications. For this project, Fiji is used to analyze images obtained by microscopes to determine pore size distributions that can be related to porosity and permeability of each sample, and to compare the influence of consolidation parameters. Primarily, after appropriate scales are established, images are thresholded to distinguish between pore and solid space; pore space being white and solid space being black.

Images are assessed for accuracy and then processed by the “Analyze Particles” function. Within this function, the size and circularity limits of the particle can be changed to filter out any background noise. Circularity measures how circular a particle is with 0 being a straight line and 1 being a perfect circle. The outlined image is normally selected as a type of output data. After analysis, a result window is displayed with the output data, which includes area, perimeter, and circularity, along with other set measurements chosen by the user. An outlined image of numbered particles can be shown as well in a separate window. This analysis tool will be useful when determining how pore size and geometry changes once samples have been consolidated and to further help determine the porosity of the sample.

5.2 Results

5.2.1 Point Counting

Thin sections of the following samples were point counted under the optical microscope to obtain porosity values shown in Table 5.1. One full count makes up 300 intersecting points on a grid.

Table 5.1: Point counting results for each thin sectioned sample with three separate counts averaged.

Sample	Count 1	Count 2	Count 3	Average
UNM-WP-HY-90-01	7.00%	6.67%	11.33%	8.33%
UNM-WP-HY-90-02	2.33%	1.67%	1.67%	1.89%
UNM-WP-HY-90-03	18.00%	18.60%	18.00%	18.20%
UNM-WP-HY-90-04	5.67%	2.67%	5.00%	4.45%
UNM-WP-HY-90-07	2.34%	2.50%	3.17%	2.67%
UNM-WP-HY-90-08	2.33%	4.67%	3.33%	3.44%
UNM-WP-HY-175-01	4.00%	5.33%	4.33%	4.55%
UNM-WP-HY-175-03	4.00%	2.67%	3.67%	3.45%
UNM-WP-HY-175-04	4.67%	5.00%	3.67%	4.45%
UNM-WP-HY-250-01	2.33%	1.00%	1.33%	1.55%
UNM-WP-HY-250-02	2.00%	1.33%	1.67%	1.67%
UNM-AI-HY-250-01	1.33%	2.33%	1.67%	1.78%
UNM-AI-HY-250-02	0.67%	0.67%	1.00%	0.78%

5.2.2 Microstructural Observations

5.2.2.1 *UNM-WP-HY-90-01*

Freshly broken aggregate pieces examined under the SEM show mainly mechanic and plastic processes of grain deformation and virtually no pressure solution processes. Figure 5.8a shows abraded grain surfaces with little cleavage. An SEM image of a thin section (Figure 5.8b) reveals a grain that has deformed plastically (center) deduced by its elongated shape and hook-like feature. Both images also show porous spaces where point counting analysis revealed a porosity of about 8.33%.

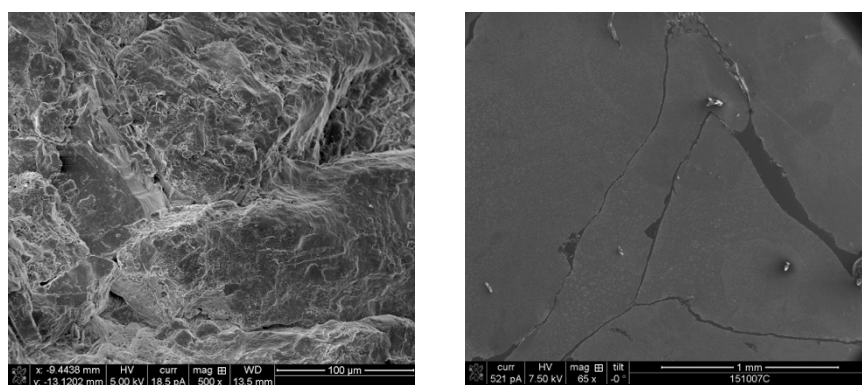


Figure 5.8: UNM-WP-HY-90-01 photomicrographs under the SEM. a) Broken aggregate piece with evidence of porous spaces, mainly abraded surfaces under SEM (500x magnification). b) Unetched slide showing deformed center grain (65x magnification).

Multiple images of a thin section were taken in wide field view on an SEM to process for pore size distributions (Figure 5.9). Five locations, ranging in view field size, were used to distinguish pore space using threshold techniques and analyze the size of each pore. During analysis, the particle size range was set from 0.0001 to infinity in order to filter out noise.

The results for the four locations were compiled and a histogram of the pore size areas were plotted using the StatPlus Toolbox in excel (Figure 5.10 and Table 5.2). This revealed a very right skewed distribution, showing 95% of particles are less than 0.073438 mm^2 with a bin size of 50. Average total porosity was calculated from the field view area of each image and the analyzed pore area, and was found to be 6.2%. Compared to point counting (8.33%), this lower porosity value could be a result from setting the smaller analyzing range too high, omitting lower

pore sizes. Further processing will evaluate the affect different ranges have on the calculated image porosity.

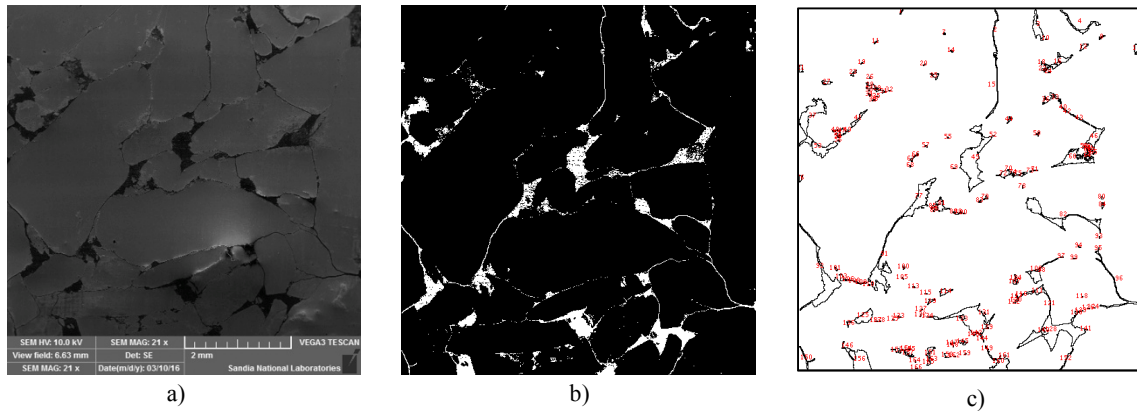


Figure 5.9: UNM-WP-HY-90-01 Fiji analysis stages. a) Raw SEM image with a view field of 6.63 mm. b) Thresholded SEM image with information bar cropped. c) Output image after “Analyze Particles” function outlining each particle counted with associated number.

Table 5.2: Output data from StatPlus Toolbox Histogram function.

<i>Frequency distribution of 0.0001164</i>				
	<i>Count</i>	<i>Cumulative Count</i>	<i>Percent</i>	<i>Cumulative Percent</i>
<i>0 To 0.073438</i>	751.	751.	0.95063	0.95063
<i>0.073438 To 0.14688</i>	18.	769.	0.02278	0.97342
<i>0.14688 To 0.22031</i>	10.	779.	0.01266	0.98608
<i>0.22031 To 0.29375</i>	6.	785.	0.00759	0.99367
<i>0.29375 To 0.36719</i>	1.	786.	0.00127	0.99494
<i>0.44063 To 0.51406</i>	1.	787.	0.00127	0.9962
<i>0.51406 To 0.5875</i>	1.	788.	0.00127	0.99747
<i>0.5875 To 0.66094</i>	1.	789.	0.00127	0.99873
<i>3.6719 To 3.7453</i>	1.	790.	0.00127	1.

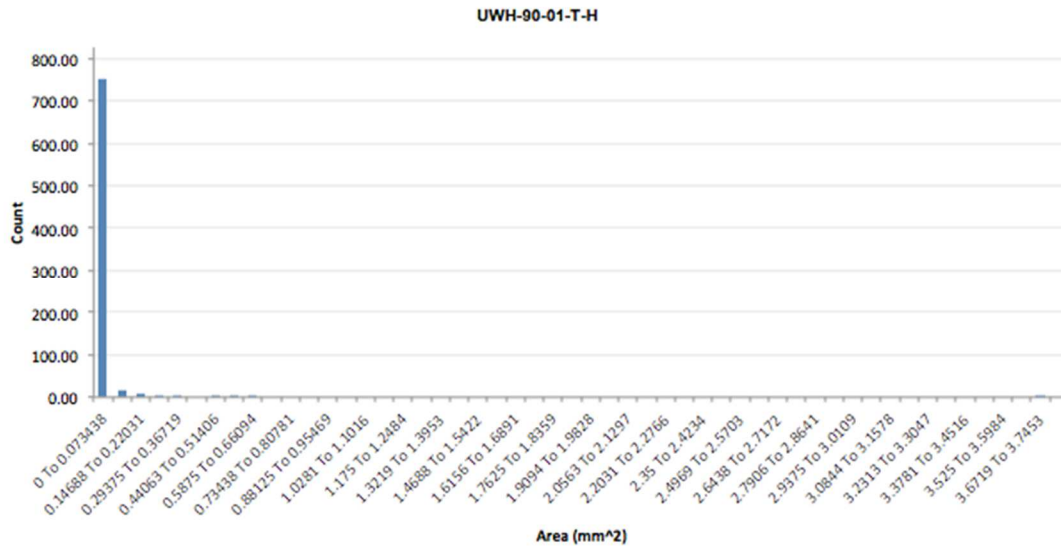


Figure 5.10: Histogram of pore area results for sample slide UWH-90-01-T-H.

5.2.2.2 UNM-WP-HY-90-02

SEM images of freshly broken aggregate surfaces reveal features of occluded fluid canals and pore spaces as well as tight cohesion and pressure solution processes, shown in Figure 5.11. When the sample was broken, grains fractured through the crystal structure instead of at grain boundaries, resulting in smooth, flat, cleaved surfaces. These indicate grain boundaries were more cohered and it required less energy to break through the crystal. While this sample had additional 1% moisture added, it is unclear why the fluid canal and pore features did not appear on further samples with added moisture with unvented conditions like sample UNM-WP-HY-250-02. An average porosity of 1.89% was found from point counting.

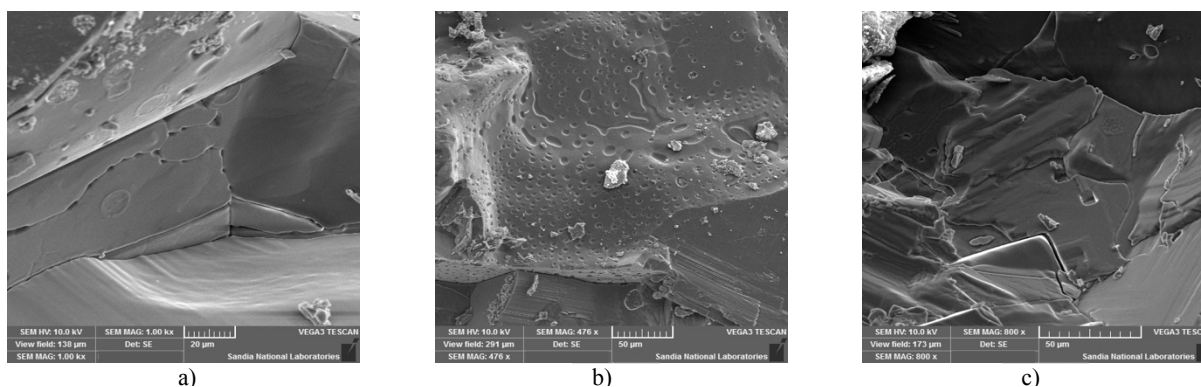


Figure 5.11: Photomicrographs taken on a SEM of a freshly broken aggregate piece of UNM-WP-HY-90-02. a) Occluded pores can be seen in the center along grain boundaries (1,000x magnification). b) Array of fluid canals and pore space imprinted on the grain boundary surface (476x magnification). c) Cleaved surfaces with evidence of pressure solution by cubic grain in center protruding onto adjacent grain and tight cohered grain boundaries (800x magnification).

5.2.2.3 UNM-WP-HY-90-04

Figure 5.12 shows a broken aggregate under the SEM and a thin section observed under the optical microscope. This sample also experienced cleaved surfaces. A grain experiencing deformation by pressure solution process can be seen in a), surrounded by cleaved surfaces. In Figure 5.12b), a healed grain boundary can be seen (center) with the assistance of fluid inclusions (black spots). A porosity of 4.45% was found from point counting.

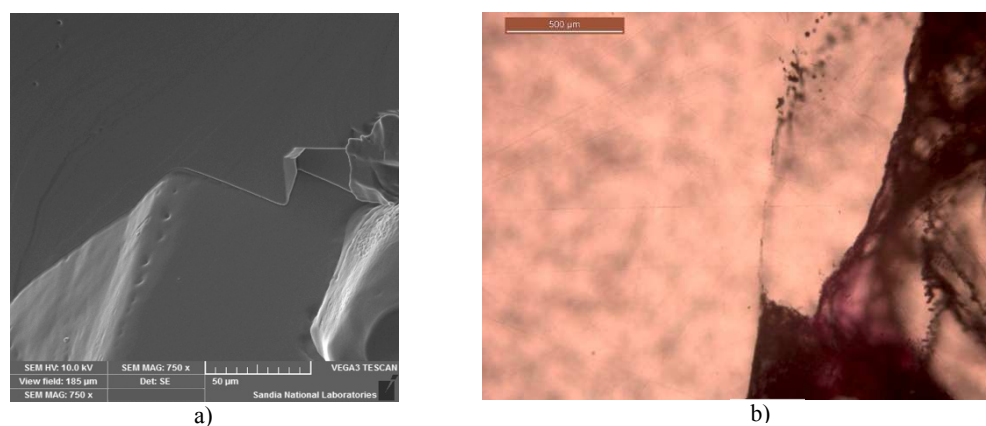


Figure 5.12: Sample UNM-WP-HY-90-04. a) Freshly broken aggregate piece under SEM showing evidence of pressure solution processes by upper deformed grain in center and traces of fluid inclusions on lower grain ridge (750x magnification). b) Photomicrograph of an impregnated thin section under the optical microscopes' reflected light showing healed grain boundary in the center aided by fluid inclusions (black spots) (5x magnification).

5.2.2.4 *UNM-WP-HY-90-07*

SEM photomicrographs can be seen on a broken aggregate piece in Figure 5.13. The surface of this sample was covered by a majority of smooth, cleaved planes, indicating again that grain boundaries experienced more cohesion during consolidation. There is also evidence of pressure solution processes indicated by large grain deformation, which is concurrent with the additional moisture added. Point counting revealed 2.67% porosity.

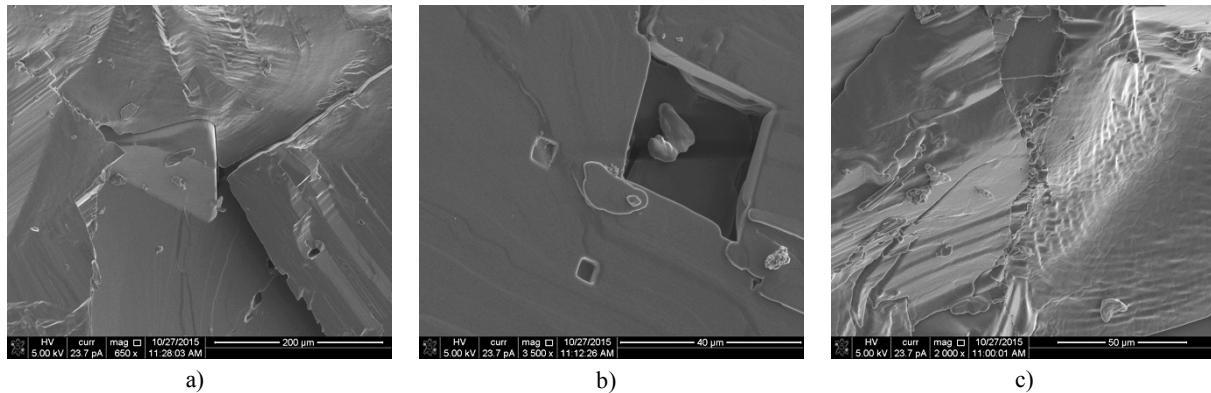


Figure 5.13: Freshly broken aggregate piece under SEM of UNM-WP-HY-90-07. a) Evidence of pressure solution in center by large grain deformation, surrounded by distorted grains from glide on $\langle 110 \rangle$ (650x magnification). b) Fluid inclusion showing intergrain crystal plasticity by slanted cubic structure and surrounding cleavage face pulled apart in tension (3,500x magnification). c) Grain boundary through the vertical center experiencing tight cohesion and glide in right grain indicated by wavy surface (2,000x magnification).

5.2.2.5 *UNM-WP-HY-90-08*

The SEM photomicrographs, Figure 5.14, show mainly abraded surfaces on an aggregate sample where breakage occurred on the grain boundaries. This sample also has no indication of pressure solution processes, which is consistent with the conditions of consolidation having no added moisture by comparison to UNM-WP-HY-90-02, UNM-WP-HY-90-04, and UNM-WP-HY-90-07. Porosity from point counting was 3.44%. When compared to UNM-WP-HY-90-01, which also had no added moisture, this lower porosity is more than likely experienced due to double the amount of stress at 40 MPa.

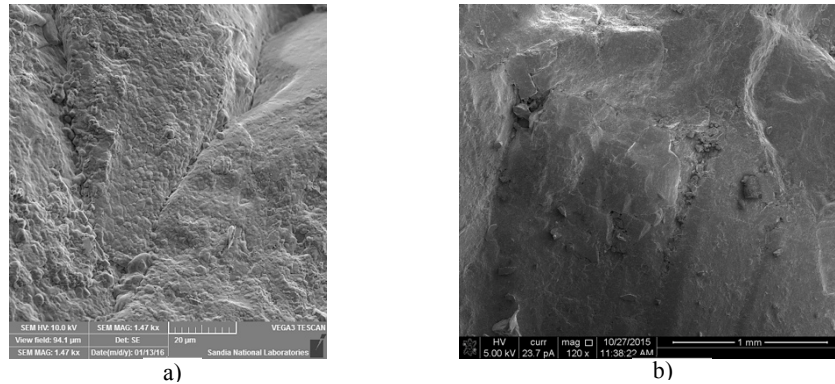


Figure 5.14: SEM photomicrographs of UNM-WP-HY-90-08 aggregate piece. a) Grain boundary with minute cohesion and rough, abraded surface (1,470x magnification). b) Low magnification (120x) showing the absence of cleaved surfaces.

5.2.2.6 UNM-WP-HY-175-01

In Figure 5.15, two SEM images are presented of an aggregate sample. Once again, ground surfaces are experienced through most of the sample. However, at higher magnification (Figure 5.15b), plastic deformation can be seen by the lower grain pushing on the upper grain creating warped 110 planes. The porosity of this sample was found to be 4.55% from point counting.

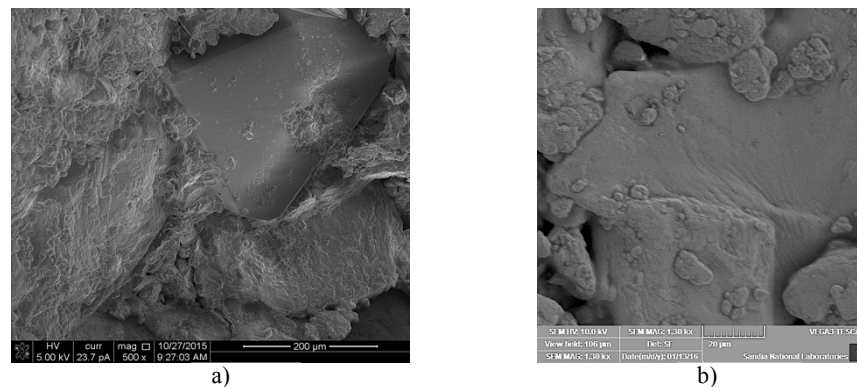


Figure 5.15: UNM-WP-HY-175-01 aggregate piece under SEM. a) Mechanically grinded surfaces with no evidence of pressure solution processes (500x magnification). b) Plastically deformed upper grain shown by curved 110 planes, (1,300x magnification).

5.2.2.7 UNM-WP-HY-175-03

UNM-WP-HY-175-03 was similar to UNM-WP-HY-175-01, by undergoing mechanically ground surfaces with scarce cleaved planes. A small example of a cleavage plane is

demonstrated in Figure 5.16b, where a remaining cubic grain is healed with larger grain. A porosity of 3.45% was obtained from point counting.

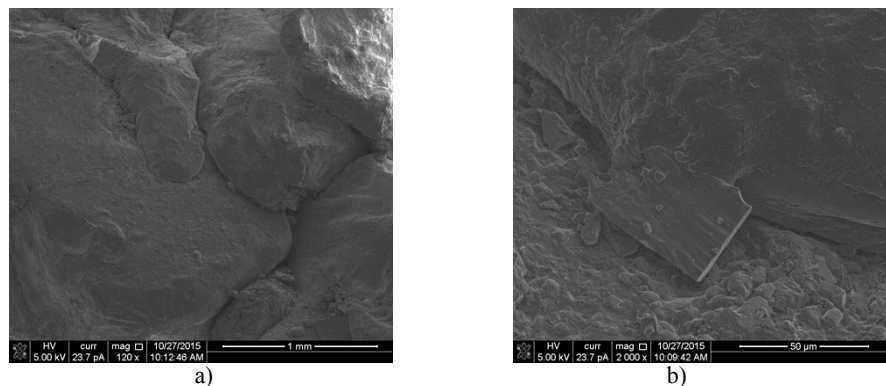


Figure 5.16: Broken aggregate piece of UNM-WP-HY-175-03 under SEM. a) Abrasive surfaces at low magnification (120x) with pore space. b) Higher magnification (2,000x) showing some plastic deformation with small cubic grain (pulled apart in tension due to cleaved face) healed with larger grain.

5.2.2.8 UNM-WP-HY-175-04

Similar to other samples consolidated at 175°C, UNM-WP-HY-175-04 experienced virtually no grains broken through the crystal structure, only at grain boundaries as seen in Figure 5.17. The grain boundaries are not tightly cohered, signifying pressure solution processes were not abundant. Point counting resulted in 4.45% porosity, similar to the previous 175°C samples.

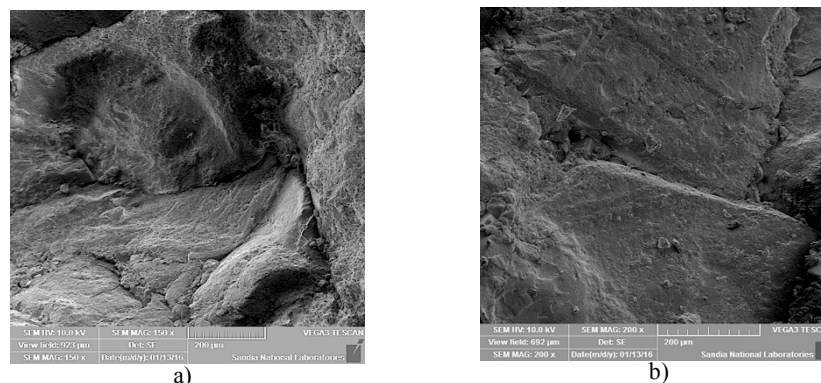


Figure 5.17: SEM images of UNM-WP-HY-175-04 aggregate piece. a) Abraded, ground surfaces with several grain boundaries (150x magnification). b) Ground surfaces with porous grain boundary not cohered tightly (200x magnification).

5.2.2.9 UNM-WP-HY-250-01

Samples from UNM-WP-HY-250-01 include a broken aggregate grain observed under the SEM and water-etched thin sections under the optical microscope. Figure 5.18 reveals cohered and tight grain boundaries created from plastic mechanisms, evident by cleaved surfaces. Etched thin sections in b) and c) are decorated with the subgrain structure, becoming denser near grain boundaries and showing areas of recrystallization where no subgrains exist. Recrystallization is the ultimate deformation mechanism, producing a form of the original, undamaged crystal structure. A porosity of 1.55% was found by point counting.

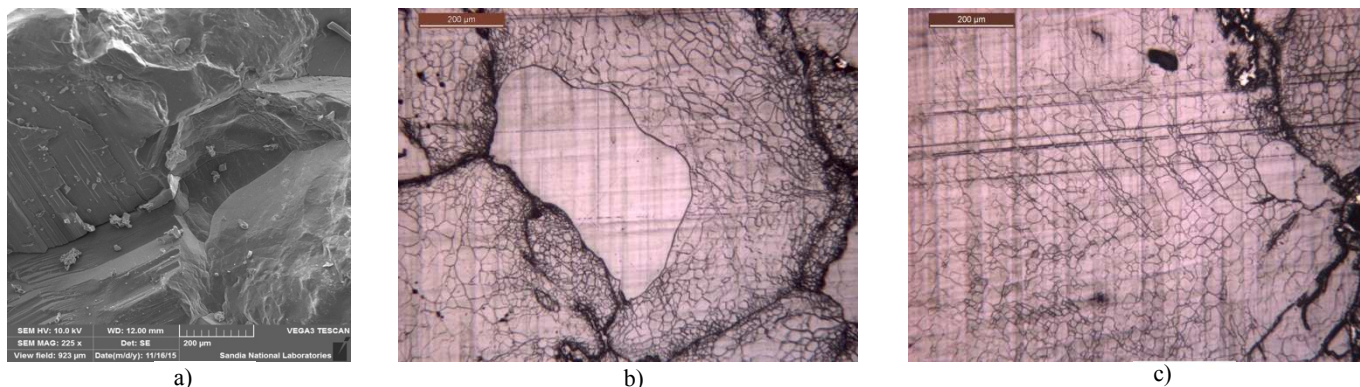


Figure 5.18: Microscope images of UNM-WP-HY-250-01. a) Intersection of grains at near orthogonal orientation with tight grain boundary achieved by crystal plasticity (225x magnification). b) Thin section with water-etched surface in reflected light displaying large recrystallized area as well as high-energy grain boundaries (10x magnification). c) Water-etched thin section with evidence of climb by elongated polygons in subgrain structure (10x magnification).

5.2.2.10 UNM-WP-HY-250-02

UNM-WP-HY-250-02 was observed on the SEM to have occluded fluid canal and pore space on broken aggregate surfaces shown in Figure 5.19a and b. It was believed these occluded areas were formed from the sample being unvented during consolidation, but similar occurrences found on UNM-WP-HY-90-02 that was vented suggest otherwise. An etched thin section under the optical microscope experiences areas of recrystallization and high-energy grain boundaries. Point counting analysis revealed a porosity of 1.67%.

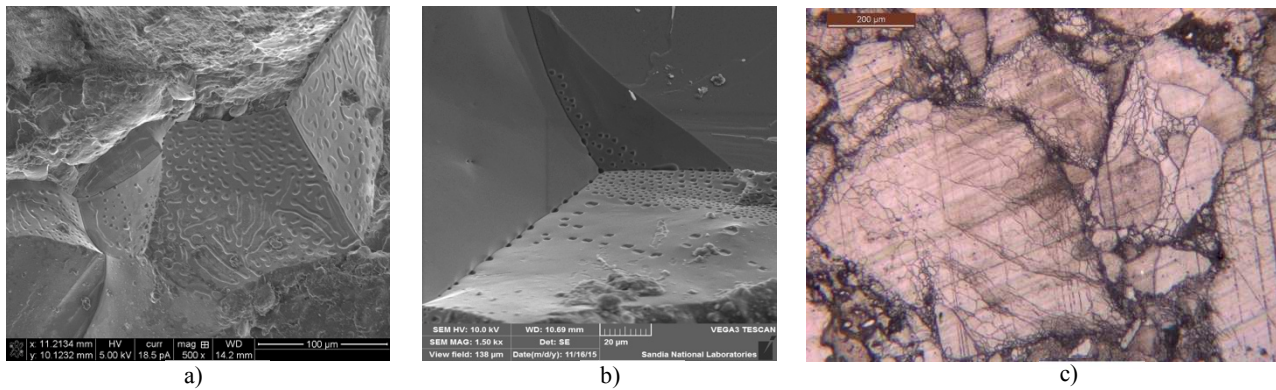


Figure 5.19: Microscope images of UNM-WP-HY-250-02 a) Occluded fluid droplets and canals on cubic grain boundary (500x magnification). b) Tight triple junction with canals of resided moisture displaying occluded pores (1,500x magnification). c) Thin section surface in reflected light etched with swipe of water revealing simultaneous recrystallization, internal grain recovery, and high energy grain boundaries (10x magnification).

5.2.2.11 UNM-AI-HY-250-01

Figure 5.20 shows SEM and optical microscope images of Avery Island consolidated salt.

Portions of the sample had cleaved surface and grain boundaries with some cohesion. The etched thin section (c) again has areas of dense subgrains near grain boundaries, with a recrystallized area in the center of a triple-junction. A similar porosity related to the other 250°C samples was 1.78% by point counting.

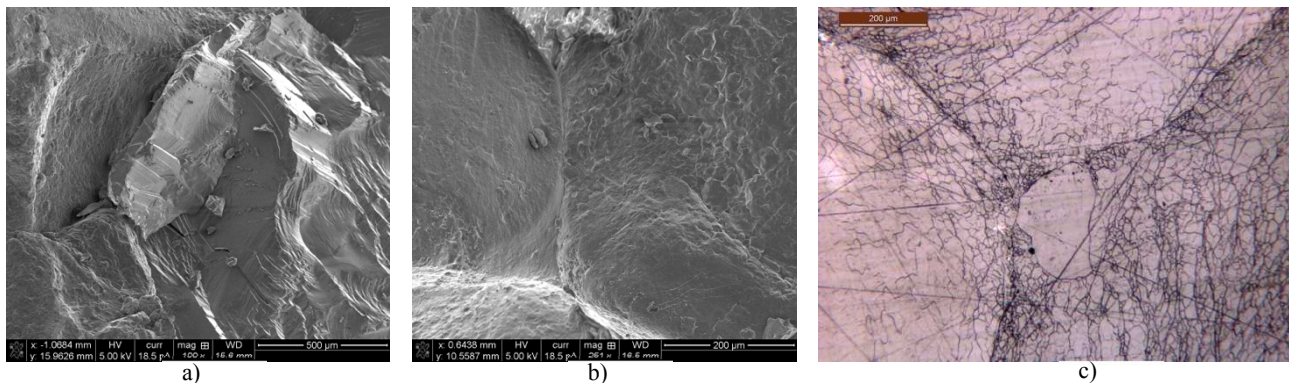


Figure 5.20: Photomicrographs of UNM-AI-HY-250-01. a) Grain boundary fracture on left and cleavage fracture on right (100x magnification). b) Evidently tight grain boundary, but not high cohesion (251x magnification). c) Water-etched thin section surface in reflected light showing triple-junction with recrystallized area in center and small subgrains decorating tight grain boundaries (10x magnification).

5.2.2.12 *UNM-AI-HY-250-02*

In Figure 5.21, photomicrographs analogous to UNM-AI-HY-250-01 are shown. However, possibly due to an additional 1% moisture during consolidate, it appears this sample experienced more cleaved surfaced and a higher level of cohesion at grain boundaries. An array of subgrains is displayed in c) along with clear, recrystallized areas. The porosity obtained from point counting was 0.78%.

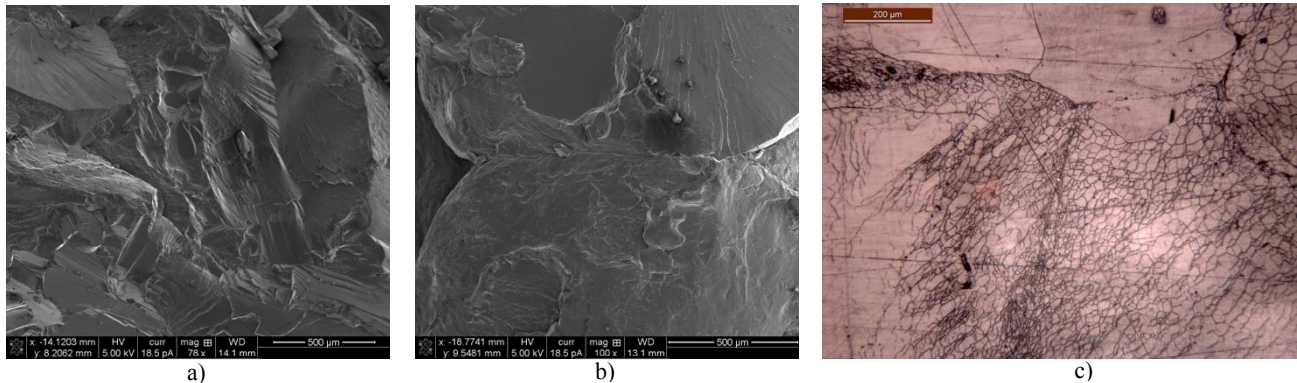


Figure 5.21: Microscope images of UNM-AI-HY-250-02. a) Cohered grains with fractured surfaces (78x magnification). b) Upper grain fractured through the crystal structure while lower grain fractured on boundary and minor residual porosity (100x magnification). c) Water-etched thin section surface in reflected light showing multiple grain boundaries and recrystallized area with smaller subgrains emanating through rest of crystal structure (10x magnification).

5.3 Summary

The techniques implemented here provide a suitable way to observe the microstructures of consolidated granular salt samples. Thin sections and broken aggregate samples expose deformation mechanisms taking place during consolidation experiments varying in pressure, temperature, and moisture conditions. From the photomicrographs displayed here, these conditions have a direct effect on the consolidation behavior and the types of mechanisms occurring. For instance, any sample with additional moisture appears to have a higher level of cohesion at grain boundaries and more instances of pressure solution processes when compared to no moisture samples having copious amounts of mechanically ground surfaces. At higher temperatures (250°C), samples experience recrystallization, apparent in etched thin sections under reflected light. Further investigations concerning the occluded pore spaces found in

UNM-WP-HY-90-02 and UNM-WP-HY-250-02 will be focused on determining their cause. The application of Fiji to process images and help create a pore area distribution is encouraging for use on additional thin-sectioned samples to see how the pore space evolves under different consolidating conditions. Investigation into additional measurements that can be made on processed images will also be conducted to provide adequate use in models.

6 Constitutive Model

A proper understanding of the phenomena causing the deformation and associated consolidation of granular salt is imperative for accurate long-term predictions of repository integrity. Many experimental works have been completed on the consolidation of granular salt over the past 40 years, and experimentalists and theoreticians in this field have widely accepted the notion that P_p only influences the mean stress (σ_m) within an porous sample (Spiers and Brzesowsky, 1993; Callahan et al., 1998; Olivella and Gens, 2002; Salzer et al., 2007; Czaikowski et al., 2012). Because of this, the influence of pore pressure (P_p) within the connected porosity (ϕ) on the deviatoric part of the stress tensor (σ^{dev}) and the associated deformations have largely been neglected. Understanding how P_p impacts the consolidation of granular salt is especially vital because gas pressure may developed within a sealed repository and influence its capacity to act as a seal. Therefore, the influence of P_p on the viscous deformation of granular salt has been experimentally evaluated through a series of hydrostatic compression and creep tests performed with independent control of the confining pressure (P_c) and P_p over a wide range of ϕ . Based on the results from these experiments, a poromechanical constitutive model has been developed and will be presented here.

6.1 Motivation

Development of this constitutive model was motivated by limitations of the existing constitutive models for granular salt. The principal shortcoming of existing constitutive models was that the influence of pore pressure on viscous deformation was not an explicit feature; although it has been experimentally shown and documented by Holcomb and Shields (1987) and Salzer et al. (2007), along with the experimental results provided herein. An additional shortcoming of existing models was the large number of material parameters included in the models (some have more than 20 material parameters). With this large number of material parameters, ascertaining the physical role a particular parameter has on the material deformation is very difficult. Additionally, when fitting parameters to existing models, we found equivalent model responses could be obtained with varying sets of parameter values, that is, the response of is non-unique. Because of this, relating an experimentally observed characteristic to a material parameter was impossible. Upon realizing these shortcomings of existing models, we did not attempt to modify an existing model; rather, we began anew with a fundamentally different approach that focused on making experimental observations and then relating these physical observations to a physically meaningful set of constitutive equations.

The constitutive model presented here was developed to achieve the following goals: (1) an accurate prediction of experimentally observed consolidation at conditions representative of a sub-surface repository, (2) utilization of basic poromechanic principals to predict material deformation, and (3) limiting the number of material-specific parameters that would need to be “fit” to experimental data. Unlike most existing constitutive models for intact and granular salt, the development of this model does not rely upon an accurate description of presumed micromechanisms; rather, the model described herein is based on a basic understanding of mechanics of porous media. Additionally, material parameters included in the model can be fit to experimentally measured bulk volumetric deformations of granular salt. The following subsections highlight the fundamental basis of this constitutive model.

6.1.1 Experimental Observations

In general, the deformation of granular salt may result from numerous factors, e.g., elastic volumetric compression, particle cataclasis, pressure solution along particle boundaries, and ductile isochoric plasticity. Observations and measurements made prior to and following consolidation experiments confirmed that granular salt consolidates in a predominately ductile manner under the application of hydrostatic (isotropic) P_c and P_p .

Ductile deformation is assumed to result from shear stresses within a material, as recognized by Huber (Huber, 1904) and later described mathematically by von Mises (Bohannon, 1965, pg. 189-199). To reconcile our experimental results with the well established theory on ductile deformation, the concept of local stresses was utilized. That is, the stress within a representative volume element (RVE) of a porous material is not constant, rather it varies spatially. By utilizing this paradigm, we could account for the development of local shear within a bulk porous sample that was under a macroscopically hydrostatic state of stress.

During our experiments, the deformation of granular salt was quantified under a macroscopically hydrostatic state of stress, where the magnitude of P_c and P_p were varied while samples were consolidated. Experiments were performed in an effort to describe how ϕ and P_d influence the potential for granular salt to deform and consolidate such that it may act as an effective sealing material. Experiments were completed on mine-run granular salt from the WIPP and performed over a range of stresses and temperatures consistent with expected repository conditions and other previous experimental works.

6.1.2 Poromechanics

Basic principles of poromechanics describe why local shear stresses exist within a porous sample whenever $P_p \neq P_c$; however, this phenomenon is not often recognized when evaluating experimental results from hydrostatic compression tests on porous materials. An illustration of this scenario is presented in Figure 6.1, where a porous material, under a macroscopically hydrostatic state of stress, is depicted as containing two discrete phases, fluid and solid. The static fluid phase is understood to only sustain a hydrostatic state of stress that is equal to P_p ; however, the solid phase can sustain a general state of stress that is dependent on both P_c and P_p . If $P_p \neq P_c$, then the stress distribution within the solid phase is not uniform and therefore by definition an isotropic state of stress does not exist over the bulk material. This implies the norm of the deviatoric stress tensor is nonzero and shear stresses exist within the bulk material. Because all practical compression tests on porous materials are in essence shear tests; albeit, the magnitude of shear during a common hydrostatic compression is typically less than the yield stress of most geologic materials. However, because salt yields under very small shear stresses (Bérest et al., 2015), the local stress must be considered for a proper understanding of mechanisms that cause granular salt to deform.

The magnitude of shear within the solid phase is understood to be proportional to the difference between the confining and pore pressures ($P_d = P_c - P_p$), and if $P_d = 0$ the solid phase would be under a hydrostatic state of stress and would experience no shear. That is, if $P_p = P_c$, then both phases are under the same hydrostatic state and no shear exists anywhere within the bulk material. Furthermore, the influence of local stresses diminishes as ϕ_p approaches zero or unity and only a single phase remains. No experiments to date on granular salt have been observed to utilize this conceptual understanding of a porous media. Additionally, because local shear stresses are

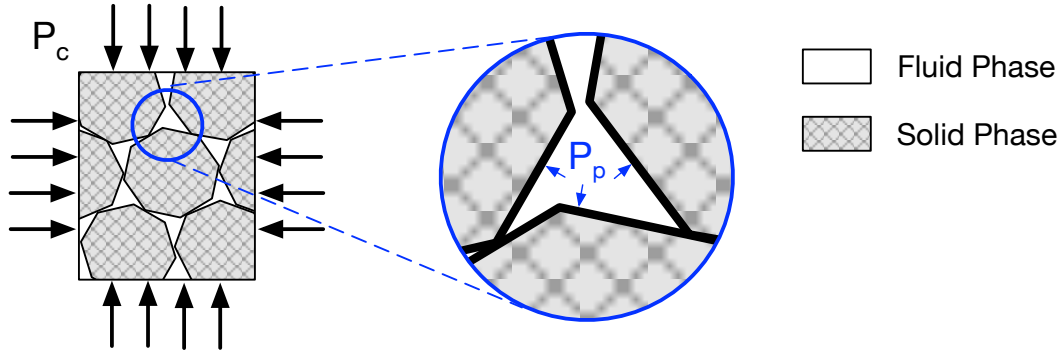


Figure 6.1 Graphical representation of a porous material under a P_c and P_p .

recognized to exist (except when $P_p = P_c$), all practical compression tests on porous materials impose shear stresses on the sample volume. Therefore, no fundamental difference is understood to exist between experiments performed on porous materials under a macroscopically hydrostatic or deviatoric state of stress other than the potential magnitude of shear.

6.1.3 Material Parameters

Material parameters were chosen to describe deformation of the solid phase under two distinct regimes, elastic and plastic. Under conditions of prescribed P_c and P_p , material parameters are only needed for the solid phase of granular salt, which is recognized to act as an isotropic elastoviscoplastic material. The elastic volumetric and shear deformations of the solid phase are described by the bulk (K) and shear (G) moduli, respectively, and the viscoelastic deformation is described with a single rate parameter (τ^{ve}). Plastic deformations are described by an initial yield stress (σ_0^y) along with nonlinear hardening parameters (H) and (n). The viscoplastic deformation is characterized by a rate-dependence parameter (τ^{vp}) and a temperature-dependence parameter (A) that are used to general-solid phase.

Values for K , G , and σ_0^y can be obtained from a uniaxial compression tests on intact rock salt, and values for such parameters on WIPP salt are widely available in existing literature (e.g., (Hansen et al., 1984; Senseny et al., 1992)). Therefore, experimental testing was needed to obtain the material parameters H , n , A , τ^{vp} and τ^{ve} , which were determined from experimental measurements acquired during hydrostatic consolidation tests on granular WIPP salt.

6.2 Constitutive Relations

The symmetric Cauchy stress tensor (σ) and the Lagrangian form of the logarithmic strain (e), which are energetically conjugate, will be used for all measures of stress and strain herein. Precise definitions of σ and e will be provided below; for the derivation of these terms see the report submitted previously in May of 2017 titled: *Constitutive model for granular salt consolidation* (Stormont et al., 2017). Components of σ and e will have a positive sign in compression and negative sign in tension, in accordance with traditional rock mechanics notation.

6.2.1 Stress

The development of σ in a material is assumed to result from elastic deformation; therefore, σ is a function of the elastic part of the total strain (e^e). Then, for an isotropic material:

$$\sigma = E_4 \cdot e^e \quad (6.1)$$

where E_4 is the fourth-order elasticity tensor. The modeled material is assumed to be isotropic at the scale of interest; therefore, E_4 is of the following form:

$$E_4 = 3K P_4^{sp} + 2G P_4^{dev} \quad (6.2)$$

where P_4^{sp} and P_4^{dev} are the fourth-order spherical and deviatoric projectors, respectively.

6.2.2 Strain

Principal components of the e are shown below in matrix notation:

$$[e] = \begin{bmatrix} \ln\left(1 + \frac{\partial u}{\partial r}\right) & 0 & 0 \\ 0 & \ln\left(1 + \frac{u}{r}\right) & 0 \\ 0 & 0 & \ln\left(1 + \frac{u}{r}\right) \end{bmatrix}$$

where u and r are the displacement and radial coordinate at a material point, respectively. For convenience, all calculations will be completed in a spherical basis in this report, let the following notation hold for the radial (rr) and circumferential ($\theta\theta$) strains.

$$e_{rr} = \ln\left(1 + \frac{\partial u}{\partial r}\right) \quad (6.3)$$

$$e_{\theta\theta} = \ln\left(1 + \frac{u}{r}\right) \quad (6.4)$$

This model has been developed to aid in the understanding of how granular salt consolidates, or stated in an alternative matter, this model has been developed to aid in the understanding of porosity decreases within a bulk volume of granular salt. Therefore, prediction of the volumetric strain is of great importance, and is shown below.

$$e^{vol} = tr(e) \quad (6.5)$$

6.3 Representative Volume Element

The constitutive model is based on a three-dimensional RVE as depicted in Figure 6.2 that is capable of capturing deformations resulting from P_c and P_p . The RVE takes the shape of a cube inscribed with a thick-walled hollow sphere, where each of the three distinct regions is capable of withstanding a prescribed state of stress. The outer region represents a solid phase with a uniform-hydrostatic state of stress that is equal to P_c . The inner-spherical region represents the pore-fluid phase with a uniform-hydrostatic state of stress equal to P_p .

The chosen geometry of the RVE allows for the development of local shear stresses in the intermediate region while the inner fluid phase and outer solid phase are under states of hydrostatic stress. Therefore, this geometry is capable of representing the state of stress understood to exist in a porous material under a macroscopically hydrostatic state of stress. However, the RVE is not intended to capture the actual geometry of a porous material.

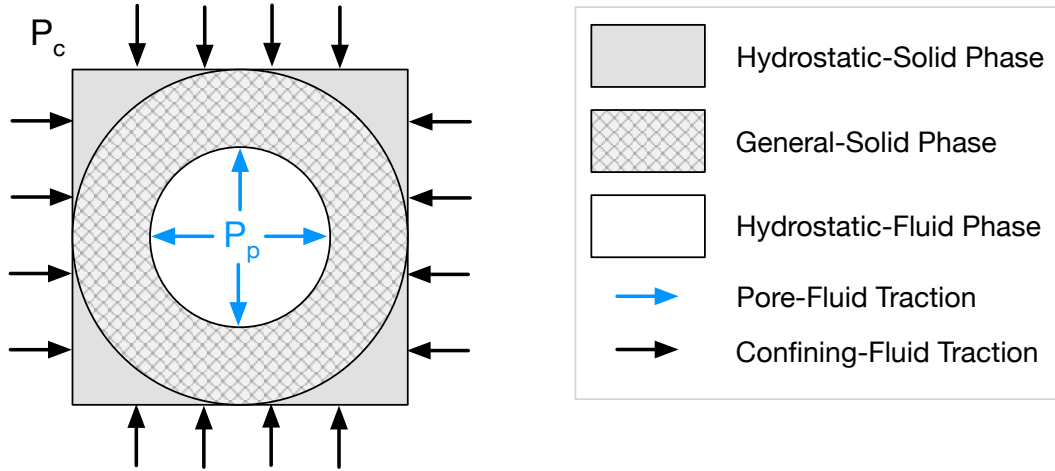


Figure 6.2 Two-dimensional slice through the spherical RVE utilized in the constitutive model, where the boundary regions can withstand only a hydrostatic state of stress while the intermediate region can withstand a general state of stress.

6.4 Constitutive Equations

The set of constitutive equations utilized to describe the viscoelastic-viscoplastic deformation of crushed salt are described here. Each of the three regions depicted in Figure 6.2 has a potentially unique state of stress; therefore, the states of stress must be represented by three distinct sets of constitutive equations, which are provided below. The bulk response of the constitutive model is simply the accumulation of the individual responses from each of the three regions.

6.4.1 Hydrostatic-Solid Phase and Hydrostatic-Fluid Phase

Regions described as the inner fluid-phase and outer solid-phase are assumed to have a linear-elastic response to a hydrostatic state of stress; therefore, the volumetric deformation of these regions may be described in terms of scalar quantities, such as:

$$P_p = K_{fluid} e_p^{vol} \quad (6.6)$$

$$P_c = K_{salt} e_c^{vol} \quad (6.7)$$

where K_{fluid} and K_{salt} represent the elastic bulk modulus of the inner pore fluid and outer solid region, respectively. While e_p^{vol} and e_c^{vol} represent the volumetric strains of the inner pore and outer solid regions, respectively. Because these regions are presumed to not undergo any shear, no further description of the constitutive response in these regions is needed.

6.4.2 General-Solid Phase

The intermediate region, with the geometry of a thick-walled hollow sphere, is described as the general-solid phase and is modeled as an elastic-viscoplastic material that may withstand a general state of stress. The general strain within this region is assumed to be composed of elastic (e^e), viscoelastic (e^{ve}), and viscoplastic (e^{vp}) components, as:

$$\mathbf{e} = \mathbf{e}^e + \mathbf{e}^{ve} + \mathbf{e}^{vp} \quad (6.8)$$

From this, $\boldsymbol{\sigma}$ may be written as:

$$\boldsymbol{\sigma} = E_4 \cdot (\mathbf{e} - \mathbf{e}^{vp} - \mathbf{e}^{ve}) \quad (6.9)$$

Viscoelastic Strain The viscoelastic component of deformation quantifies rate dependent deformation associated with the spherical part of $\boldsymbol{\sigma}$. Here, the viscoelastic strain rate ($\dot{\mathbf{e}}^{ve}$) is:

$$\dot{\mathbf{e}}^{ve} = \frac{1}{\tau^{ve}} (\mathbf{e} - \mathbf{e}^{vp}) - \frac{1}{\tau^{ve}} \mathbf{e}^{ve} \quad (6.10)$$

where τ^{ve} is a material parameter that governs the rate dependency. The viscoelastic component is essentially the standard linear solid model presented in the form of a backstrain.

Viscoplastic Strain The viscoplastic component of deformation quantifies the rate dependent deformation associated with the deviatoric part of $\boldsymbol{\sigma}$, and it relies on the concept of a yield surface and an associated effective stress. The following paragraphs will outline the viscoplastic component of the constitutive equation.

Effective Stress The von Mises yield criterion or the stress associated with the maximum distortional energy has been chosen to describe the viscoplastic deformation. Descriptions of deviatoric stress ($\boldsymbol{\sigma}^{dev}$) and the effective stress ($\bar{\sigma}$) associated with this criterion is given in a general form below.

$$\boldsymbol{\sigma}^{dev} = \boldsymbol{\sigma} - \frac{1}{3} tr(\boldsymbol{\sigma}) \mathbf{i} \quad (6.11)$$

$$\bar{\sigma} = \left[\alpha \left(\boldsymbol{\sigma}^{dev} \cdot \boldsymbol{\sigma}^{dev} \right) \right]^{1/2} \quad (6.12)$$

Here, $\mathbf{i}$ is the second order identity tensor and α is a scalar term that depends on the loading conditions. For comparison to results from uniaxial stress tests, let $\alpha = \frac{3}{2}$.

Yield Function The transition between elastic and viscoplastic domains is defined by means of a yield function, F , where if $F < 0$ then the deformation is viscoelastic and if $F \geq 0$ the deformation is viscoplastic. The yield function utilized here is given by Eqn. 6.13

$$F = \frac{\bar{\sigma}}{\sigma_y} - 1 \quad (6.13)$$

where σ_y is the yield stress..

Viscoplastic Flow Rule An associated viscoplastic flow rule is utilized here, and the rate-dependent evolution of the plastic strain is:

$$\dot{e}^{vp} = \dot{\lambda} \mathbf{N} \quad (6.14)$$

$$\dot{\lambda} = \begin{cases} \frac{1}{\tau^{vp}} \exp\left(-\frac{A}{T}\right) [(F+1)^{1+\phi_p} - (F+1)], & \text{if } F \geq 0 \\ 0, & \text{if } F < 0 \end{cases} \quad (6.15)$$

$$\mathbf{N} = \frac{\partial F}{\partial \boldsymbol{\sigma}} = \sqrt{\frac{3}{2}} \left[\frac{\boldsymbol{\sigma}^{dev}}{(\boldsymbol{\sigma}^{dev} \cdot \boldsymbol{\sigma}^{dev})^{1/2}} \right] \quad (6.16)$$

where $\dot{\lambda}$ and $\mathbf{N}$ are the plastic multiplier and unit-outward normal tensor relative to the yield surface, respectively.

Although the flow rule describes the deformation of the solid-intermediated region of the RVE, the flow rule is dependent on the bulk porosity of the RVE (ϕ_p). The dependence of $\dot{\lambda}$ on ϕ_p results in the ϕ_p having an influence on the stress distribution within the General-Solid phase of the RVE. This relationship has been observed experimentally. The flow rule given here is dependent upon the prescribed temperature (T), which has the consistent units of $^{\circ}C$.

Isotropic Hardening In general, a material may exhibit “strain hardening”, which results in σ_y not being constant but rather a function of other parameters. Laboratory tests have shown that crushed salt hardens under plastic deformation, and based on these observations a nonlinear hardening function has been employed as described in Eqn. 6.17.

$$\sigma_y = \sigma_y^0 + H \bar{e}^{[n(1-\phi_p)]} \quad (6.17)$$

$$\bar{e} = \int_0^t \|\dot{e}^{vp}\| dt \quad (6.18)$$

Here, $\bar{e}$ is the effective plastic strain, as given in Eqn 6.18, where $\bar{e}$ is a monotonically increasing function of the plastic strain increments ($\dot{e}^{vp}$) over the simulation time (t). The evolution of σ_y is nonlinear and defined by two material parameters (H and n). H is the hardening modulus with units of stress, and n is a dimensionless parameter that determines the influence of ϕ_p on hardening. Material hardening has been shown experimentally to be influenced by porosity; therefore, by including ϕ_p here the model is able to simulate this influence.

6.4.3 Summary of the Viscoelastic-Viscoplastic Constitutive Model

To solve for the complete deformation of the RVE, strains over each of the three subregions must be accumulated. This may be done by recognizing that when P_p and P_c are prescribed, strains over the inner and outer regions are as shown below.

$$e_p^{vol} = P_p / K_{fluid} \quad (6.19)$$

$$e_c^{vol} = P_c / K_{salt} \quad (6.20)$$

Volumetric strain of the RVE, e_{RVE}^{vol} , may then be determined by summing the product of the volume fraction of each subregion with the respective volume strain as:

$$e_{RVE}^{vol} = \phi_{int}e_{int}^{vol} + \phi_p e_p^{vol} + \phi_c e_c^{vol} \quad (6.21)$$

where ϕ_{int} , ϕ_p , and ϕ_c are the volume fractions of the intermediate, inner pore, and outer confining regions, respectively.

6.5 Validation of Constitutive Model

Samples of crushed salt were prepared and tested under hydrostatic loading and creep conditions where both a P_c and P_p were applied. Following these experiments, the initial and boundary conditions from these experiments were simulated to perform model predictions with the constitutive model described herein. The following subsections will identify how material parameters for the constitutive model were determined and the parameter values employed for the model predictions.

6.5.1 Determining material parameters

The constitutive model contains two different classes of material parameters, (1) rate independent and (2) rate dependent. The rate independent parameters describe the elastic deformation while the rate dependent parameters describe the viscoplastic material response. Elastic parameters utilized in the model were not fit here, rather they were based on values obtained from previous experimental works, as described by Senseny et al. (1992) and Bérest et al. (2015); these parameters are provided in Table 6.1. The value of σ_y^0 was chosen to be on the order of the accuracy of the pressure measurement devices utilized during these experiments.

Table 6.1 Summary of elastic and initial yielding parameter values

Paramter	Value	Units
K	$14.4(10^9)$	Pa
G	$12.6(10^9)$	Pa
σ_y^0	$1.0(10^3)$	Pa

Viscoelastic and viscoplastic parameters were determined by minimizing the scaled p-norm of an error vector between experimental measurements and model predictions. The chosen error vector is defined below:

$$\varepsilon = \phi^{meas} - \phi^{pred} \quad (6.22)$$

where ϕ^{meas} and ϕ^{pred} are the measured and model predicted porosities vectors. The error vector (ε) contains scalar error values for a particular test at discrete points in time. The $p - 2$ norm of ε was chosen for a scalar measure as shown below:

$$\|\epsilon\|_p = \left(\sum_i^n |\epsilon_i|^p \right)^{1/p} \quad (6.23)$$

$$p = 2 \quad (6.24)$$

where n is the number of discrete measurements in time. Because multiple experiments were performed, with test durations ranging from a single day to multiple weeks, a scaled norm was employed to normalize the error with respect to the number of measurements made during a test. To accomplish this a sum of scaled p-norms $\left(\|\epsilon\|_{sp} \right)$ was calculated as:

$$\|\epsilon\|_{sp} = \sum_i^m \frac{1}{n_i^{1/p}} \|\epsilon\|_{pi} \quad (6.25)$$

where n again is the number of discrete measurements in time, but here n acts as a scaling factor that allows for a normalized error value to be determined that is independent of the number of physical measurements made. The number of tests utilized in the fit is represented by m , where:

$$m = 10 \quad (6.26)$$

in this analysis. The viscoelastic and viscoplastic parameters were determined by approximating parameter values that provided a minimized value of $\|\epsilon\|_{sp}$.

6.5.2 Viscoelastic and viscoplastic parameters

Viscoplastic material parameters were fit to measured values of porosity obtained during consolidation tests on granular salt from the WIPP. Parameter values were fit by minimizing the cumulative sum of scaled error norms for five consolidation tests. Results from the parameter fits are provided in Table 6.2

Table 6.2 Summary of preliminary viscoelastic and viscoplastic material parameter values

Paramter	Value	Units
τ^{vp}	$3.78(10^4)$	<i>sec</i>
H	$8.59(10^6)$	Pa
n	3.25	-
A	$3.52(10^2)$	<i>°C</i>
τ^{ve}	$3.09(10^4)$	<i>sec</i>

This combination of parameters resulted in:

$$\|\epsilon\|_{sp} = 0.09 \quad (6.27)$$

which was the minimized using the Nelder-Mead minimization routine (Jones et al., 2017).

6.5.3 Numerical modeling results

Experimental results from ten consolidation tests on crushed salt along with predicted porosity and volumetric strain rate are illustrated in Figures 6.3 through 6.12. All values have been plotted with respect to time. Prescribed pressure and temperature as recorded in the laboratory and prescribed during modeling are presented in the upper two charts of each figure. Laboratory measured along with model predicted porosities and volumetric strain rates for each test are shown in the lower two charts of each figure, where laboratory measurements are labeled *Lab* and model predictions are labeled *Pred*.

6.5.4 Discussion of experimental and modeling results

The following paragraphs outline the experimental results and relate them to the proposed constitutive model.

Differential pressure A distinct advantage of proposed constitutive model over its predecessors is its ability to capture the influence of P_p on the viscous deformation of granular salt, where no other existing constitutive model has this ability. Experimentally, the influence of P_p on the deformation is especially evident in Figure 6.5, where dramatic increases in the measured and predicted volumetric strain rate correlate with increased P_d . Additionally, the influence of P_d is well illustrated at approximately day one in Figures 6.5 and 6.6, where the P_c and P_p are similarly altered and the volumetric strain rate remain approximately constant.

These experimental observation indicate the P_d may act as a measure of shear under these test conditions. Capturing this influence would not be possible with continuum theory without the use of a RVE. Because of the chosen RVE geometry, the constitutive model is able to predict shear stresses even when only hydrostatic stresses are applied at the boundaries.

Pore Pressure Increased P_p has been experimentally observed to cause tensile stresses, this phenomenon is evident in Figure 6.7 from day one to three, where increased P_p results in volume expansion of the sample (negative volumetric strain rate). Albeit not well, this characteristic is captured by the current constitutive model.

Temperature Experimentally, an increase of temperature results in an increase of the volumetric strain rate. This influence of temperature is especially evident in Figure 6.12, where at day two the volumetric strain rate abruptly increases with temperature while the pressure is held constant. The general influence of temperature is captured by the proposed constitutive model; although, the magnitude of the influence is substantially less than what has been experimentally observed. The proposed constitutive model incorporates this into the flow rate, as shown in Equation 6.15.

Hardening Strengthening as a result of plastic deformation is recognized here to occur as a result of a reduction in porosity. This was evident in all tests, where prior to testing the sample was loose unconsolidated particles of salt, and after undergoing substantial plastic deformation the sample was highly consolidated with a much greater yield strength. The proposed constitutive model incorporates this concept directly as shown in Equation 6.17.

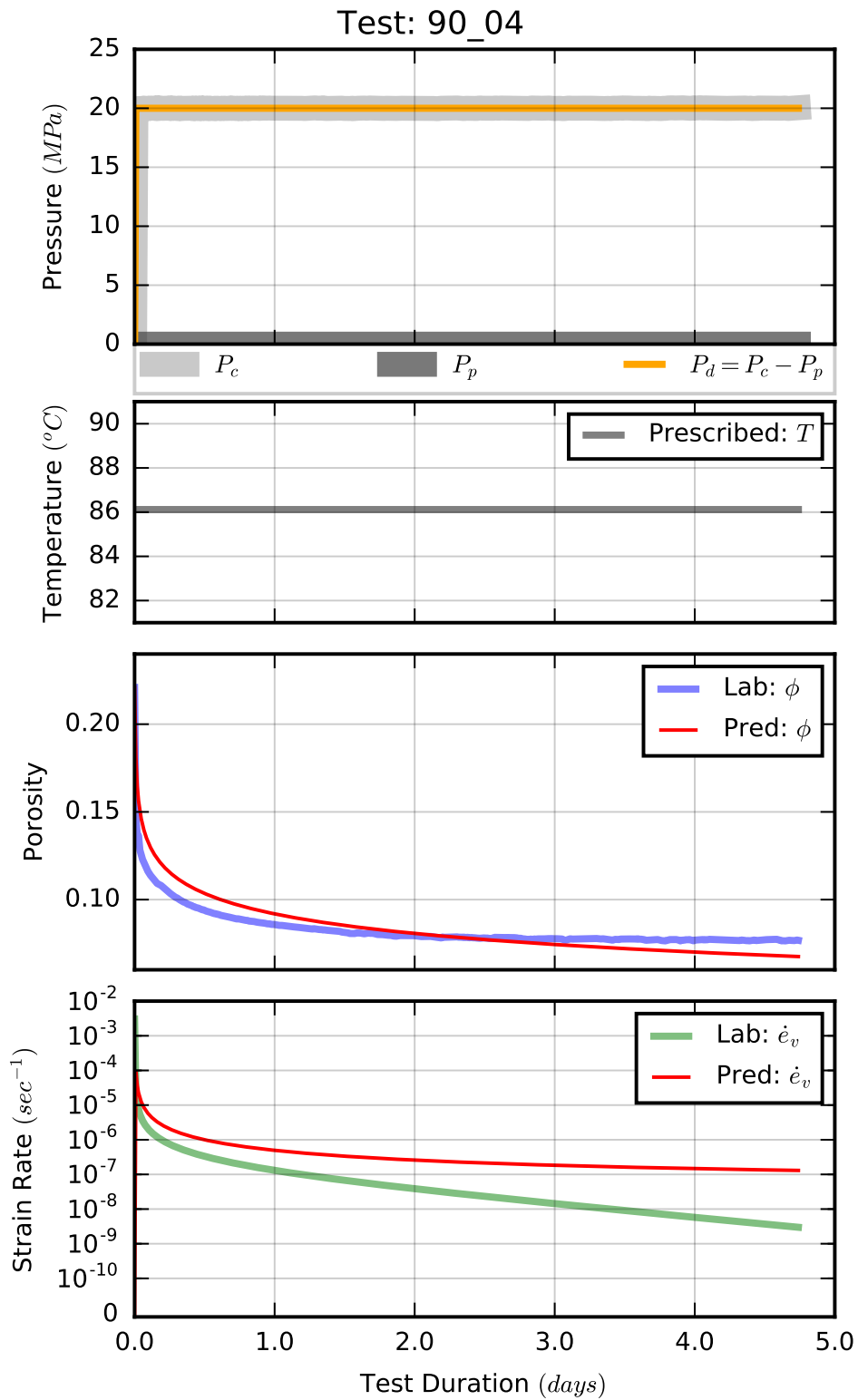


Figure 6.3 Experimentally measured pressure, temperature, porosity, and volumetric strain rate; model prescribed values of pressure and temperature; and model predicted values of porosity and volumetric strain rate for consolidation test 90_04.

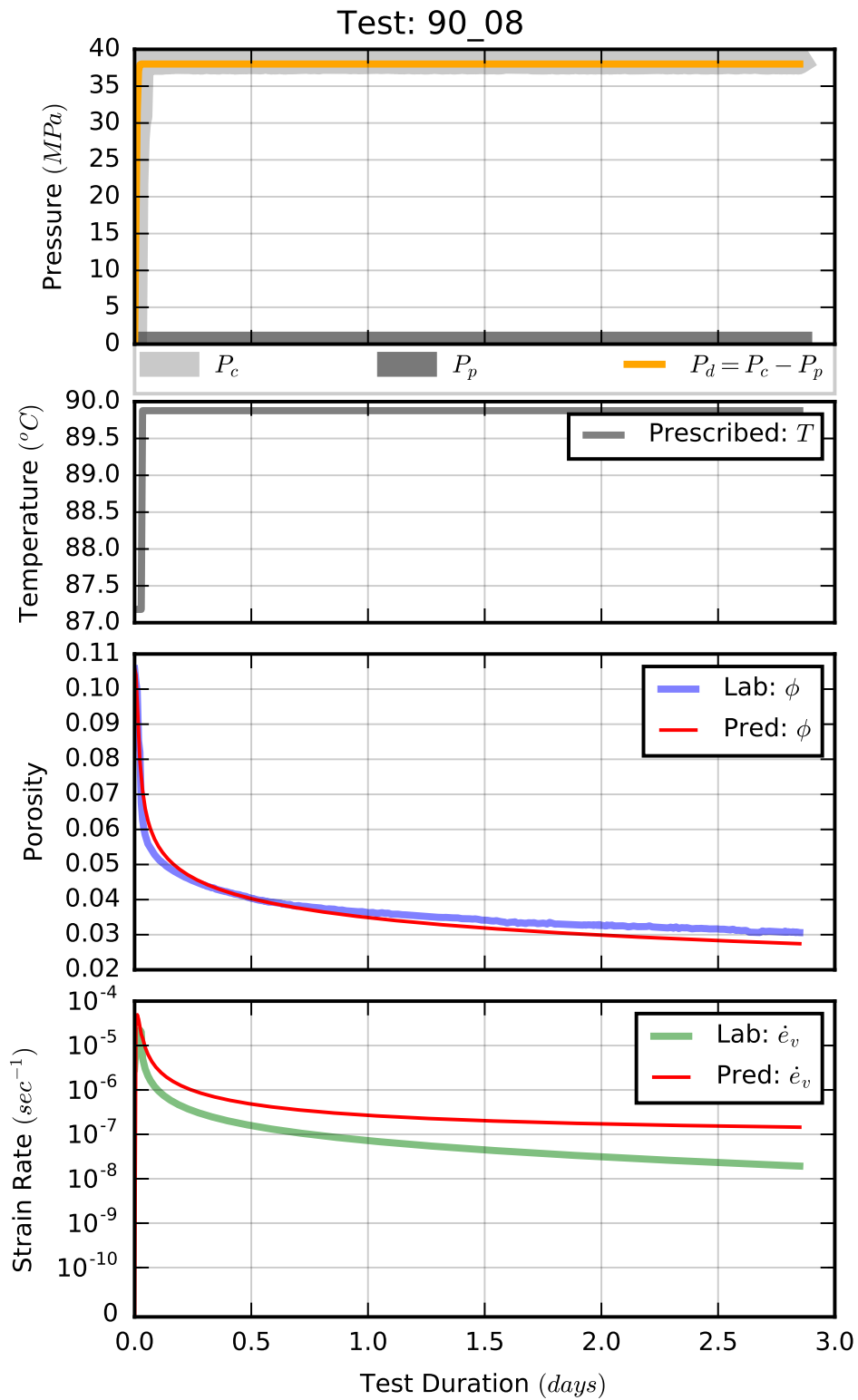


Figure 6.4 Experimentally measured pressure, temperature, porosity, and volumetric strain rate; model prescribed values of pressure and temperature; and model predicted values of porosity and volumetric strain rate for consolidation test 90_08.

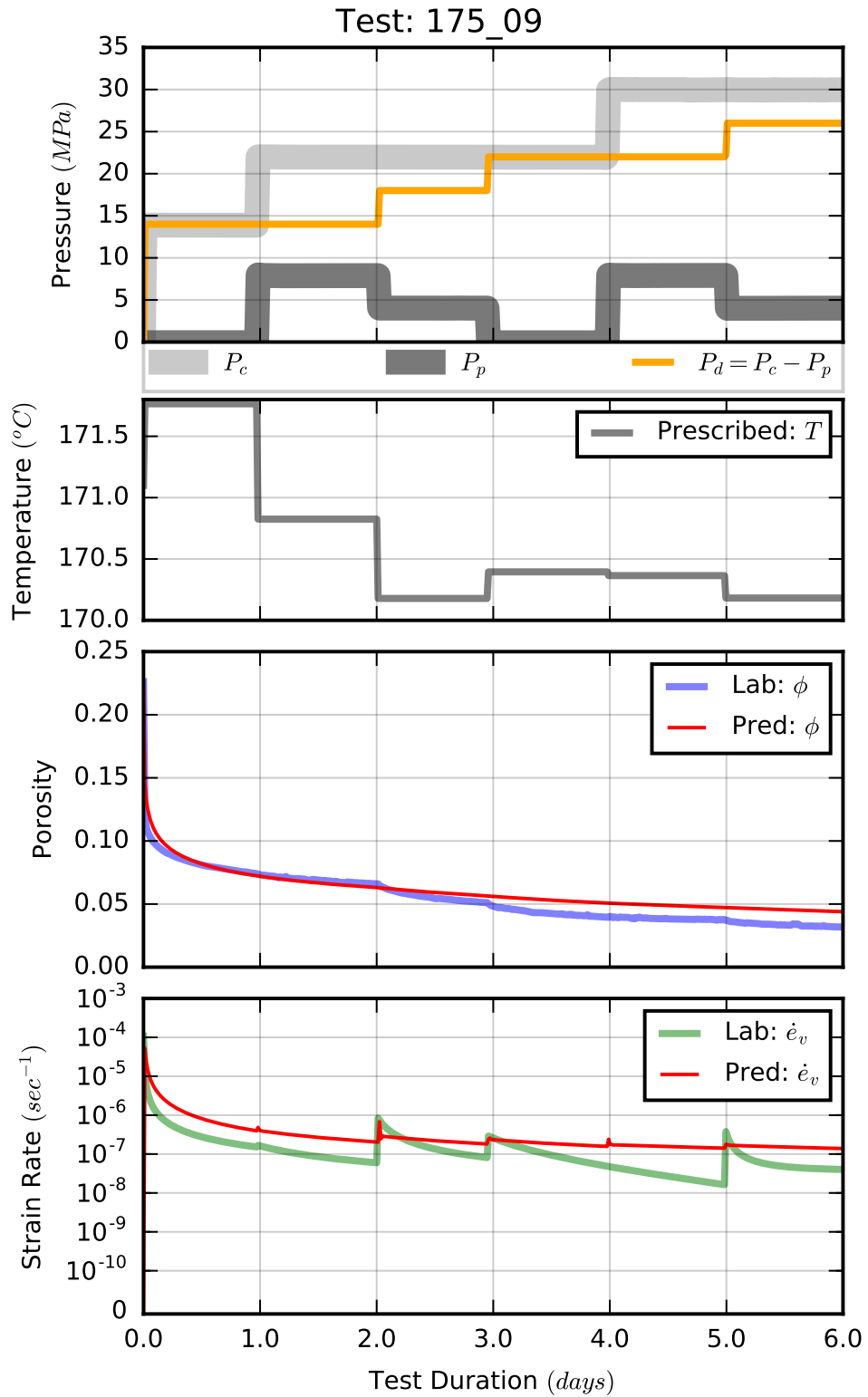


Figure 6.5 Experimentally measured pressure, temperature, porosity, and volumetric strain rate; model prescribed values of pressure and temperature; and model predicted values of porosity and volumetric strain rate for consolidation test 175.09.

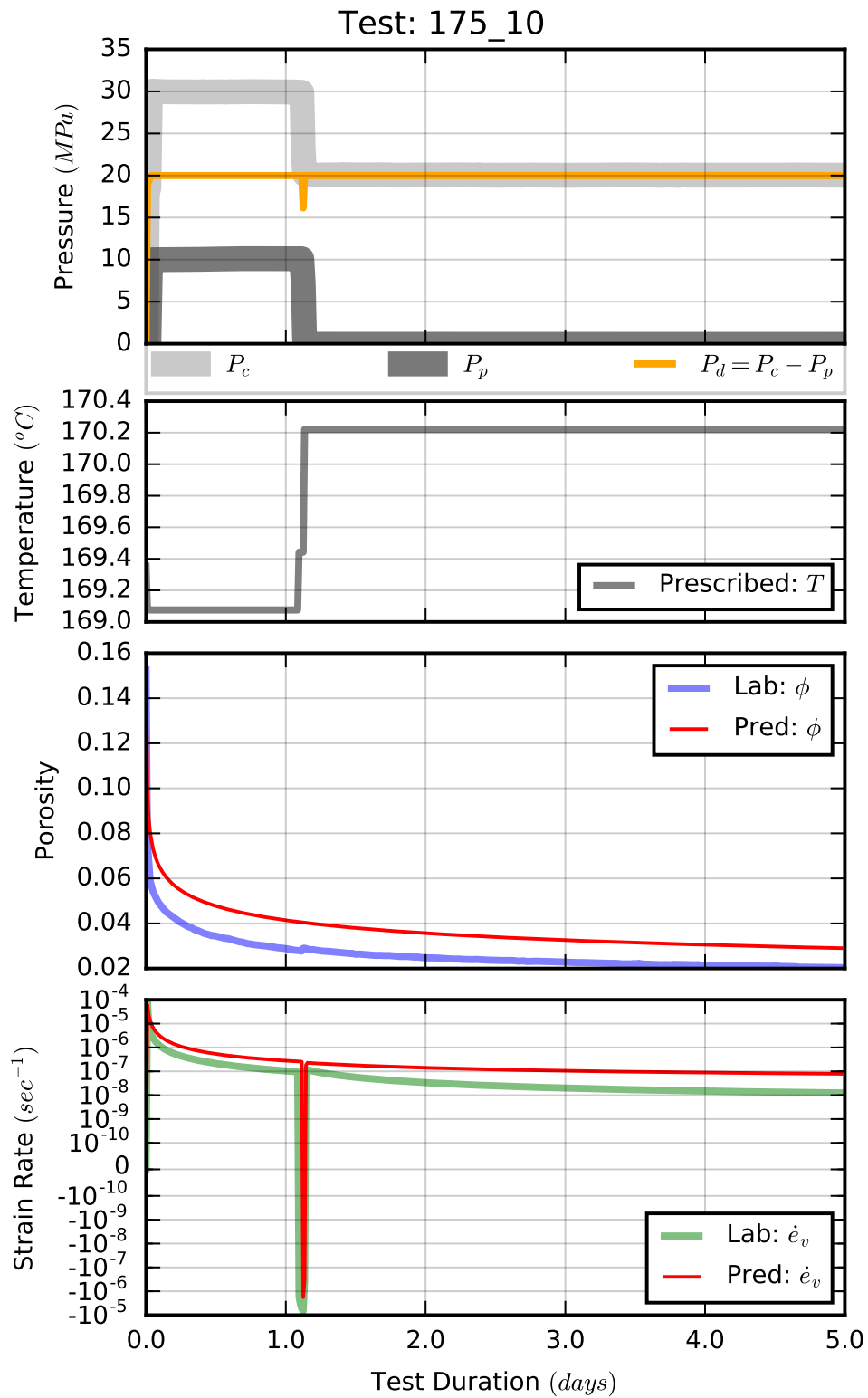


Figure 6.6 Experimentally measured pressure, temperature, porosity, and volumetric strain rate; model prescribed values of pressure and temperature; and model predicted values of porosity and volumetric strain rate for consolidation test 175_10.

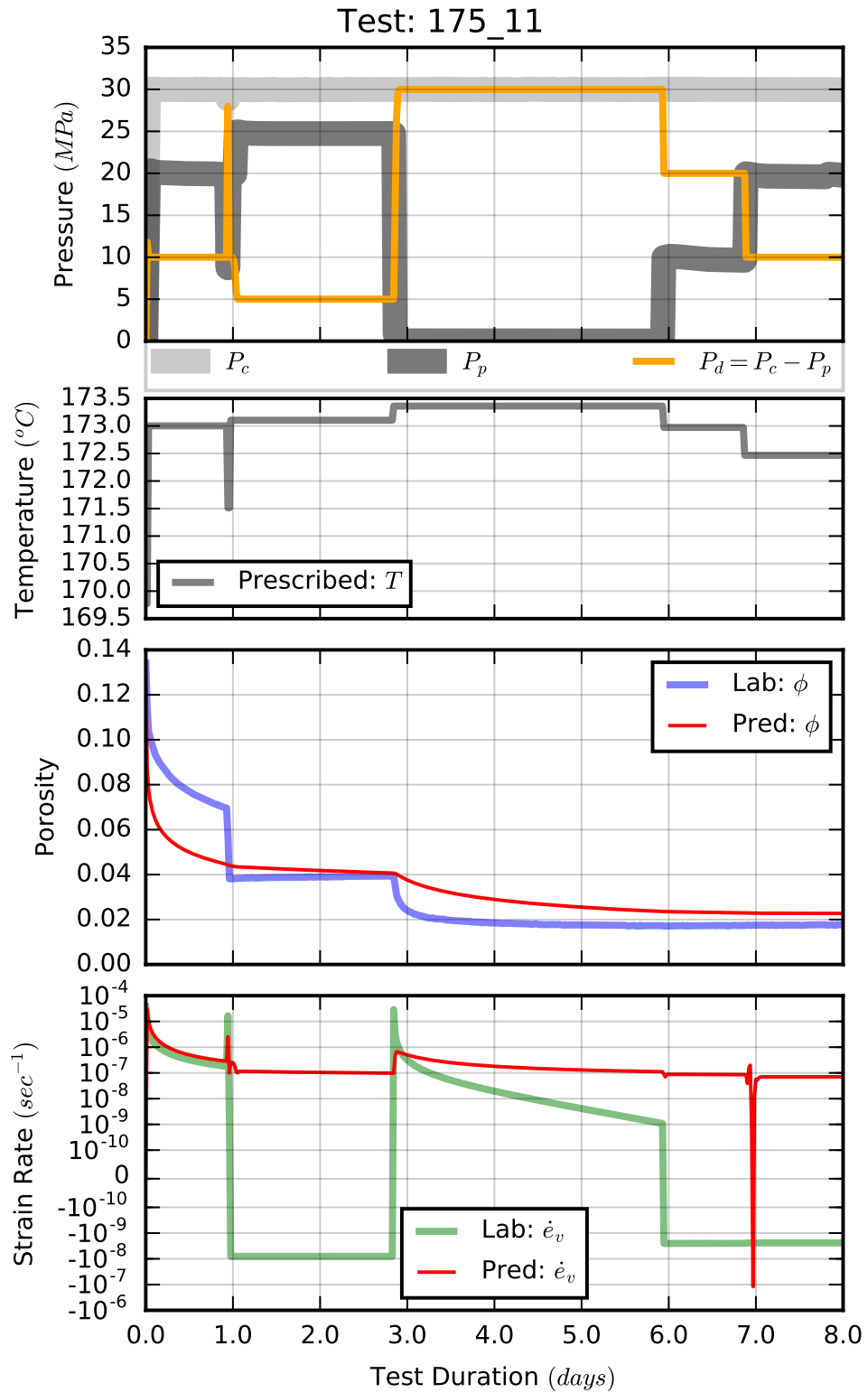


Figure 6.7 Experimentally measured pressure, temperature, porosity, and volumetric strain rate; model prescribed values of pressure and temperature; and model predicted values of porosity and volumetric strain rate for consolidation test 175_11.

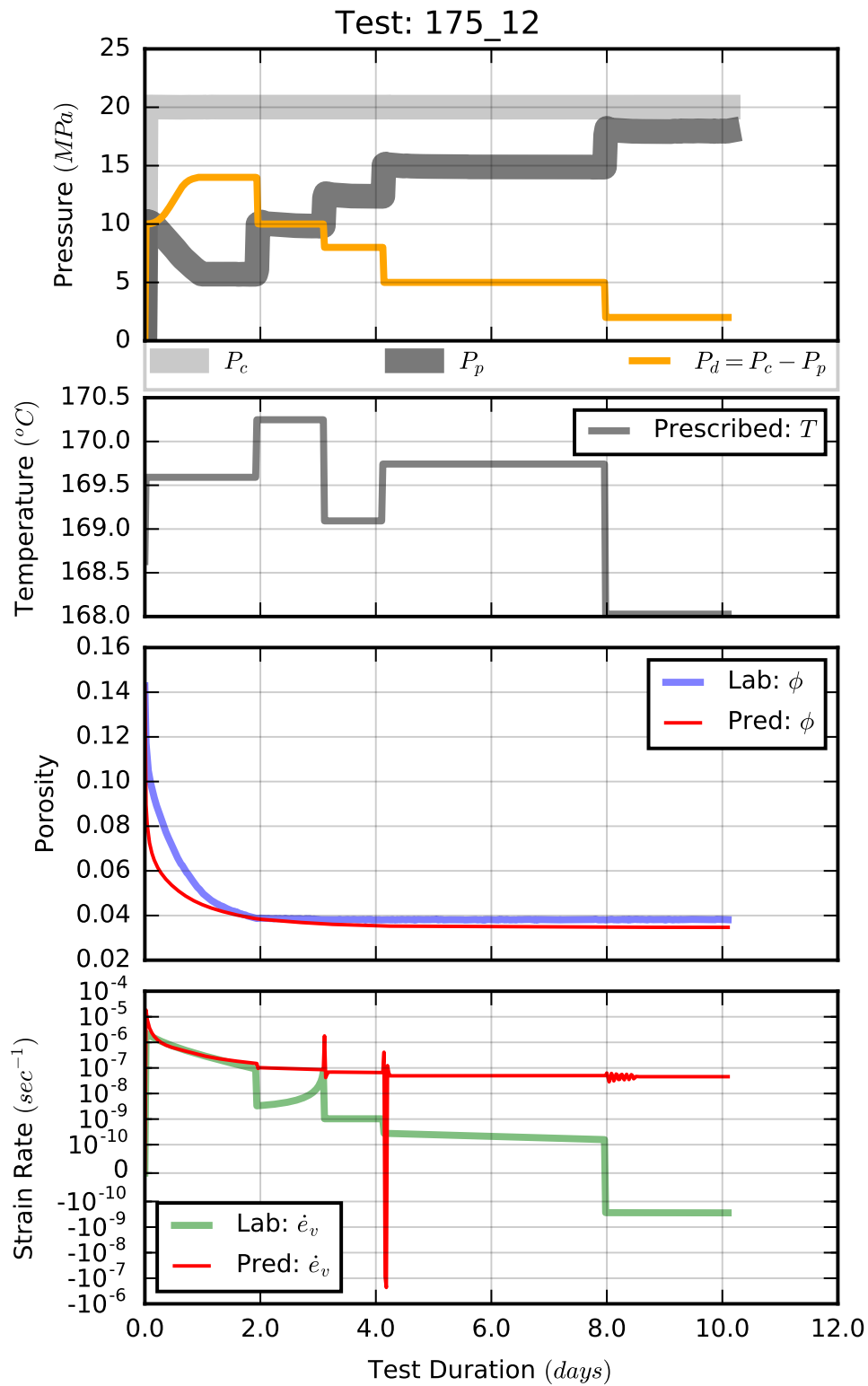


Figure 6.8 Experimentally measured pressure, temperature, porosity, and volumetric strain rate; model prescribed values of pressure and temperature; and model predicted values of porosity and volumetric strain rate for consolidation test 175.12.

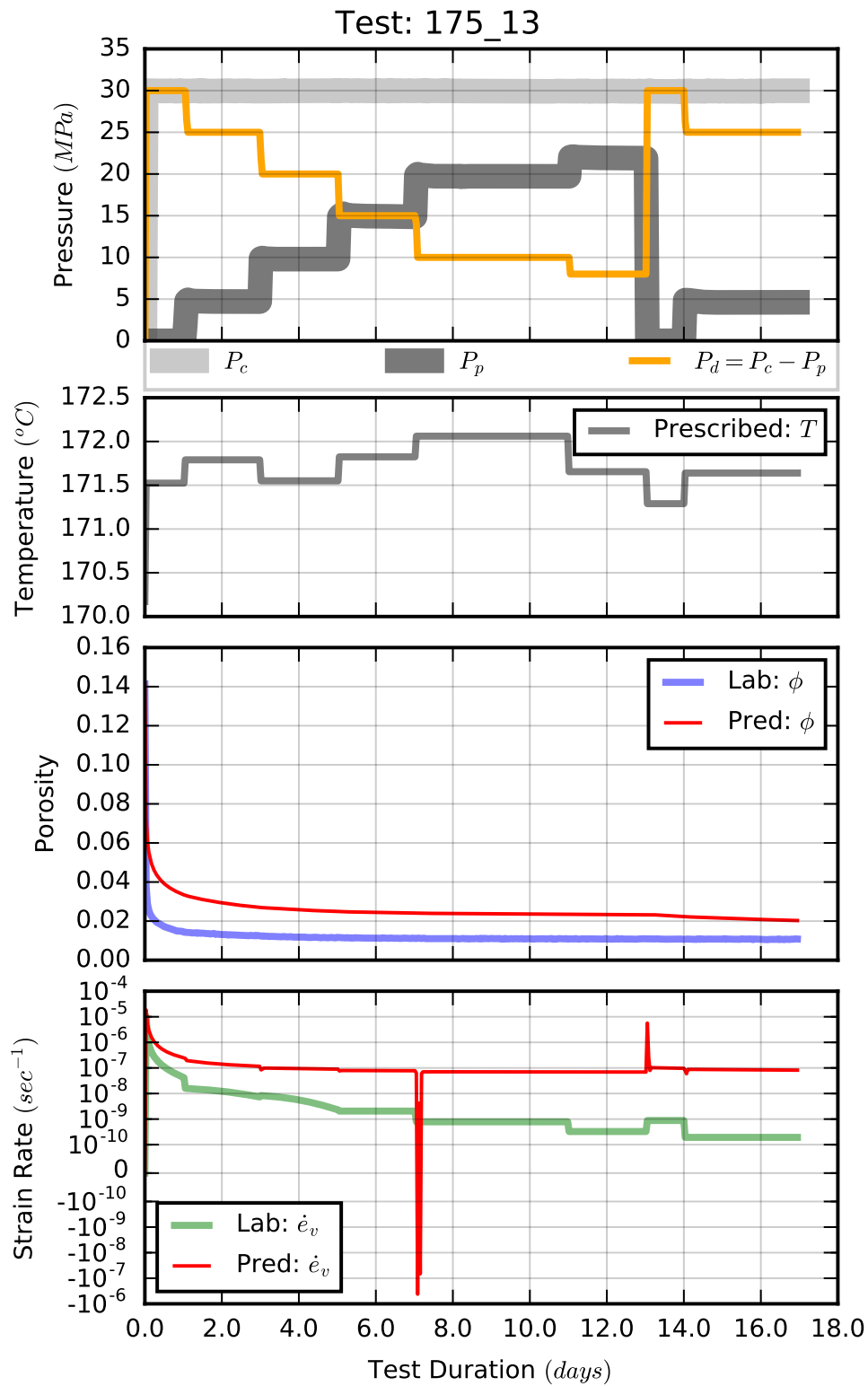


Figure 6.9 Experimentally measured pressure, temperature, porosity, and volumetric strain rate; model prescribed values of pressure and temperature; and model predicted values of porosity and volumetric strain rate for consolidation test 175_13.

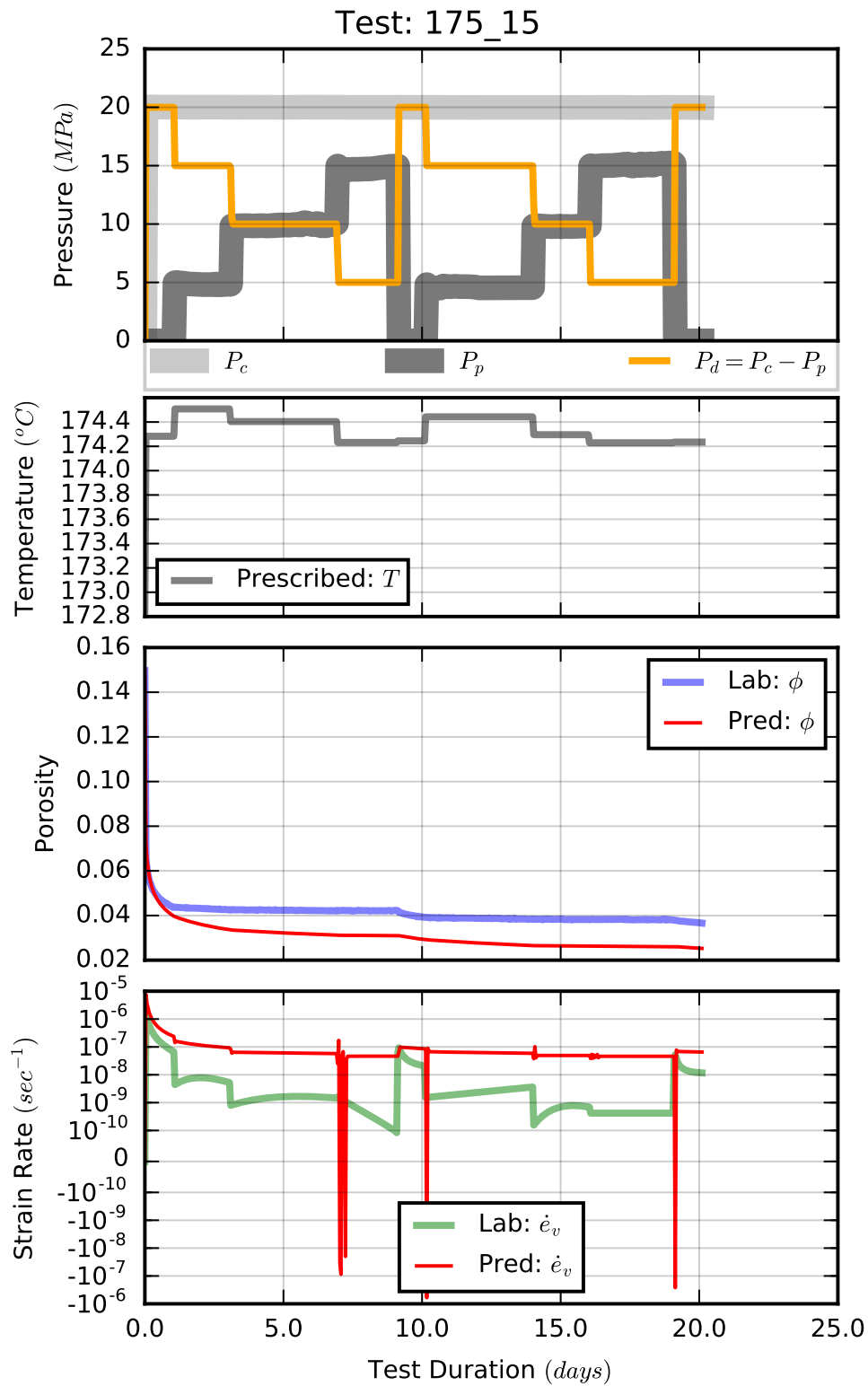


Figure 6.10 Experimentally measured pressure, temperature, porosity, and volumetric strain rate; model prescribed values of pressure and temperature; and model predicted values of porosity and volumetric strain rate for consolidation test 175.15.

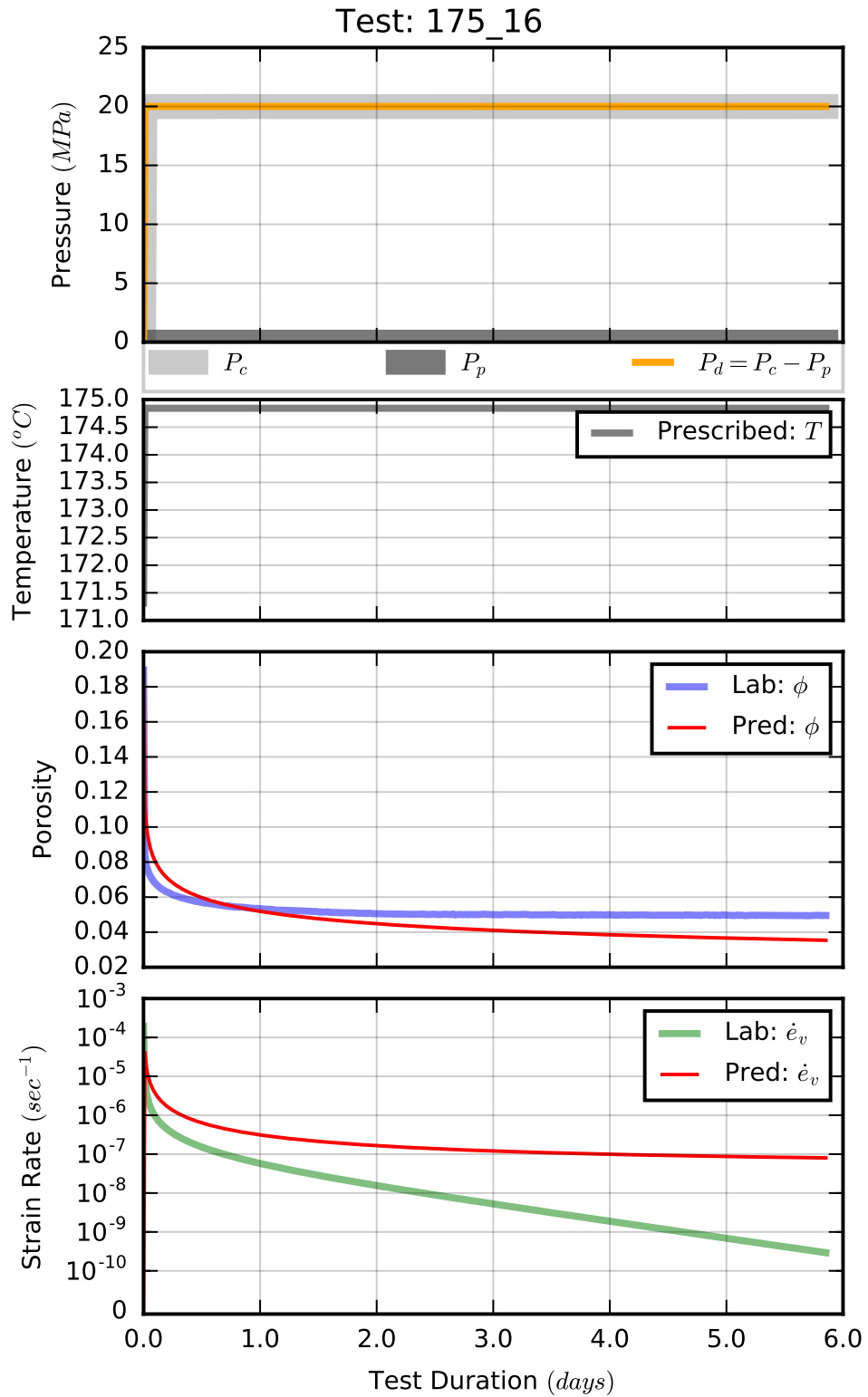


Figure 6.11 Experimentally measured pressure, temperature, porosity, and volumetric strain rate; model prescribed values of pressure and temperature; and model predicted values of porosity and volumetric strain rate for consolidation test 175.16.

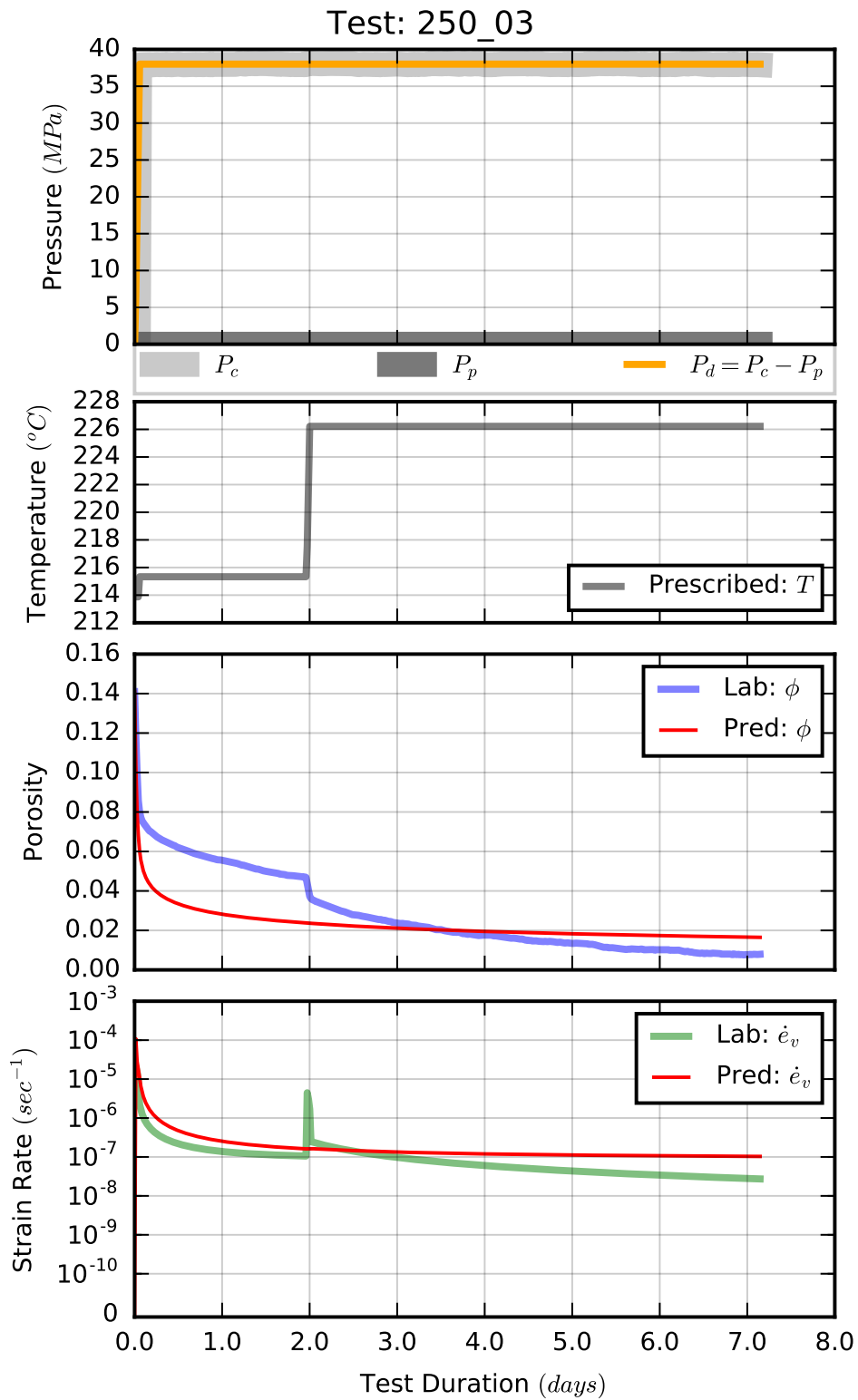


Figure 6.12 Experimentally measured pressure, temperature, porosity, and volumetric strain rate; model prescribed values of pressure and temperature; and model predicted values of porosity and volumetric strain rate for consolidation test 250_03.

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