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Sandia National Laboratories
Waste Isolation Pilot Plant

Test Plan TP 17-04

Microstructural Investigations on Natural and Laboratory Tested Salt

Task 4.4.2.4.1

Revision 0

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APPROVALS

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1 ABBREVIATIONS, ACRONYMS AND INITIALISMS

CBFO	Carlsbad Field Office
EDX	Energy Dispersive X-ray
EBSD	Electron Back Scatter Detector
M&TE	Measure and Test Equipment
NIST	National Institute of Standards and Technology
NP	Nuclear Waste Management Procedure
SEM	Scanning Electron Microscope
WIPP	Waste Isolation Pilot Plant

2 REVISION HISTORY

This is the initial issuance of this Test Plan.

3 PURPOSE AND SCOPE

This Test Plan describes observational petrofabrics and physical measurements of natural and experimentally tested intact salt core samples from domal and/or bedded formations as well as crushed salt reconsolidated experimentally and in-situ. Details of the forensic examinations are explained here in sufficient detail that the research could be repeated by independent peers with equal expertise. Several of the techniques applied and measurements described in this Test Plan were reported previously (Bechthold et al. 2004, Hansen et al. 2012).

Understanding salt rock mechanics related to closure and seal systems is important for predicting repository performance. This includes predictions of mined salt cavern creep rates along with the performance of crushed salt backfill. Room closure rates can be slow and difficult to measure, which makes predictability challenging. Therefore, laboratory creep tests on intact salt cores are advantageous to determine responses of salt behavior for long-term performance assessment modeling of salt repository systems over long time scales. Understanding and gaining insight into the micromechanisms occurring during slow creep provides characteristics significant to constitutive model development for the thermal-mechanical behavior of salt. Additionally, understanding creep mechanisms in intact salt cores establishes assessment criteria of reconsolidated granular salt, which is expected to consolidate to a porosity and permeability comparable to the intact host rock.

Crushed salt reconsolidation is an important consideration for the Waste Isolation Pilot Plant (WIPP) and any other salt repository in which crushed or run-of-mine salt plays a role in isolation systems. Salt as a geologic medium has several attributes favorable to long-term isolation of radioactive waste. Salt formations are largely impermeable where induced fractures heal. Permanent isolation also depends on our ability to construct geotechnical barriers that achieve high-performance characteristics attributed to the native salt formation. Crushed salt is commonly used as backfill in operating mines for structural and sealing purposes. Salt repository seal concepts have often included granular salt. There is a lingering uncertainty about the timing of salt reconsolidation to native salt porosity and permeability in geotechnical barriers constructed of granular salt. The uncertainty in the timing pertains mostly to the limits involved with laboratory testing over extended time periods and at an appropriately large scale. To strengthen the technical basis for salt seal systems, natural and anthropogenic analogues can be used to illustrate cases where granular salt reconstitutes itself into a low-porosity, impermeable medium. Invoking analogue examples provides an independent line of reasoning to safety case performance arguments. Therefore, these forensics add to the analogue database as well as provide pertinent information for technical assessments being made by other colleagues.

In previous cooperation with international colleagues, the Carlsbad Field Office (CBFO) advanced scientific knowledge of salt properties pertinent to WIPP through multiple Work Packages (i.e. Geochemistry, Geohydrology, Rock Mechanics, Process Model, and International). Overall the consolidation or creep experiment itself and the post-test forensics comprise many elements of direct relevance to WIPP operations and its performance assessment as well as salt repository future activities. Studies for this Test Plan characterize basic properties (i.e. porosity, pore size, grain contact angles, etc.)

and observable characteristics (i.e. deformation mechanisms, subgrain size/shape, etc.) of experimentally tested cores and reconsolidated crushed salt backfill from various entities, as noted by the principal investigator.

4 EXPERIMENTAL PROCESS DESCRIPTION

4.1 Overall Strategy and Process

The author of this Test Plan is collaborating with various entities to further the technical basis of salt micromechanics. This is through an integrated program of laboratory measurements and microstructural and petrofabric observations of experimentally tested (e.g., creep, uniaxially, or triaxially loaded) cores along with consolidated granular salt samples against natural analogs. Characterization of the microstructure can further be utilized with and implemented in numerical models. The author has been taught specimen preparation techniques, measurement methods, and received hands-on observational instruction and interpretation.

4.2 Equipment

Microstructural observations are made with optical and electron beam microscopes. There are many optical scopes and scanning electron microscopes (SEM) that would provide equally sufficient images. Interpretation of microstructures, porosity, and other characteristics will enhance the database and provide high-quality results for national and international collaborators. The equipment and techniques described in this Test Plan are being employed in their respective research. Below are descriptions of equipment and techniques applied, such that any other equally trained person can replicate this work. Other types of investigative equipment may be used at the principle investigators discretion with the possibility of improving experimental outcomes. Emerging techniques and equipment may also be implemented for certain research initiatives to possibly improve the understanding and outcomes pertinent to petrofabric properties and microstructure characterization. All instruments and equipment used will be maintained according to manufacturer specifications.

> ***Note: Reference to specific commercial equipment does not constitute or imply endorsement. There are many types of instruments with similar capabilities that might be used depending on availability to the investigator.***

Optical Microscope: An optical microscope (see Leitz Ortholux II in Figure 1 as an example) uses combined transmitted and incident polarized light to examine samples. The microscope is equipped with a camera to capture images. The optical scope will be used for point-counting porosity and petrophysical descriptions. Much of the microscopic documentation is captured by photography.

Figure 1. Leitz Ortholux II optical microscope

Scanning Electron Microscope: An SEM (see Tescan Vega3 LM SEM in Figure 2 as an example) is equipped with a conventional tungsten heated cathode that can be used in high vacuum or low vacuum. The SEM should have 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 to produces images for observational work. The analytical chamber should have a 5-axis motorized stage to permits easy placement of multiple sample types. The SEM should have energy dispersive x-ray spectroscopy (EDX) and an electron back scatter detector (EBSD) capabilities to allow for elemental analysis spectrums and back scattering detection.

Figure 2: Tescan Vega3 LM SEM.

Sputter Coater: To prepare non-conducting or poorly conducting samples for observation in an SEM, a sputter coater must be used. Figure 3 shows a standard sputter coater (Denton Vacuum Desk IV) with optional rotating stage to distribute a conductive layer of Au/Pd on samples for use in the SEM to obtain high resolution images. The sputter coater should have a two-stage, direct-drive mechanical pump to evacuate the chamber, and an automatic vent valve that opens when the mechanical pump is turned off to vent to atmosphere.

Figure 3: Denton Vacuum Desk IV sputter coater.

Diamond Wire Saw: Some samples may need to be cut and prepared to make thin-sections, obtain aggregated samples, and extract single crystals. A diamond wire saw (example shown in Figure 4) can be used to prepare subsamples. Samples of interest are clamped in place on the platform to ensure they do not move while cutting. The saw mechanism changes rotating directions using magnetic sensors, which allows the wire to be pulled back and forth. A small amount of weight (about 5 grams) on the wire arm slowly lowers the wire through the sample. Sensors located on the sides of the arm control the stroke and shut-off.

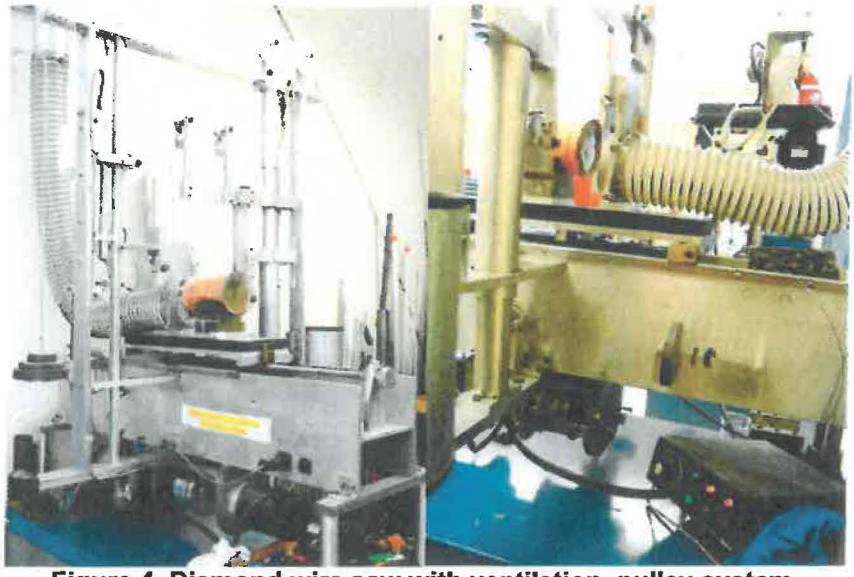

Figure 4. Diamond wire saw with ventilation, pulley system, adjustable table, wheel, pump and pump controller

Low Speed Saw: A low speed saw (example Buehler IsoMet shown in Figure 5) is used for making precision cut thin sections with a diamond tipped saw. 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. A holder is then screwed onto the arm above the blade and its position adjusted by a micrometer. A micrometer allows for manual determination of thickness for each cut. Special care must be used to maintain the speed of the cut. For salt specimens, isopropanol is used as the cutting liquid; the saw's reservoir must be refilled regularly due to evaporation.

Figure 5. Buehler IsoMet low speed saw

Polisher and Grinder: A polisher and grinder (example Buehler Ecomet III shown in Figure 6) is used to polish thin sections after cutting and also to create smooth, even surfaces for porosity samples. Abrasive, circular paper, ranging from 400 to 1200 grit, 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 attained.

Figure 6. Buehler Ecomet III polisher and grinder

4.3 Sample Types

Below we describe several types of sub-samples that could be used in these observational studies. We may not create all these types of sub-samples for each type of test or analysis, because they may not provide distinguishing features or information.

Cleavage Chips: Cleavage chips produced from individual crystals can be used to evaluate deformational processes in that grain. A single grain is isolated by disaggregation. A small, sharp chisel is used to cleave along the Miller Indices {100} plane. With practice, flat chips 1 to 3-mm thick can be produced. Typically, the chips are etched. Observations can be made using the optical microscopy or SEM.

Thick-Thin Sections and Thin Sections: Sample material is impregnated with epoxy as described in the next section on sample preparation. Excess epoxy can be cut away with a knife, band saw, or other convenient method. Impregnated samples are then cut to size for a petrographic slide. Thin sections for optical petrography are challenging to make because they are extremely thin; having an optical path difference between 530 and 560 nanometers. Fortunately, much of the information of interest can be obtained from thick-thin sections (1 to 3 mm thick). Optical microscopy is used for observations of deformation processes, grain boundary conditions, void spaces, fabric, mineralogy and any other notable characteristics. Petrographic sections can also be etched and examined optically or by SEM.

Fragments: Fragments can be obtained several ways. Part of observational studies of experimentally tested or reconsolidated granular salt involves use of a "fresh face" that has not been cut or polished. Usually fragments from cut ends can simply be broken by flexure. This exposes a clean surface, which exhibits diagnostics of sample cohesiveness, grain boundary characteristics, and other evidence of microprocesses. The SEM is better for observation because of the uneven surfaces. Fragments provide a sense of 3-D imaging, particularly useful for examining grain boundaries.

4.4 Sample Preparation

Epoxy Impregnation: Typically, the solid state of the sample is locked in place by impregnation with low-viscosity epoxy. Samples are shaped to a convenient size for thin- or thick-section preparation using the slow-speed, low damage wire saw described in the equipment section. There are many types of epoxy that can be used for impregnation, the choice of which is at the discretion of the investigator. RF 1366 is a two-component epoxy that is stained blue and has performed well in similar sample preparation (RF 1366 is a commercially available epoxy from Resin Formulators). The sub-sample is placed in a convenient disposable container (a paper cup or aluminum foil, for example). The sample is completely immersed in epoxy and placed in a bell jar, which is put under approximately 10 psi vacuum (approximately 3 psia inside the bell jar). After the system has been evacuated, the vacuum is removed slowly by venting and atmospheric pressure helps force epoxy into the evacuated voids of the specimen. This process is repeated 3-4 times to ensure that the specimen has been fully impregnated. The epoxy is then allowed to cure.

Polishing: Cutting with the diamond-wire saw creates a relatively smooth surface with small ridges. To improve imaging on the optical microscopes, uneven surfaces need to be smoothed by polishing, which is done using a clean, flat surface, a sheet of sandpaper, and isopropanol. For salt, 400 to 600 grit silicon

carbide sand paper has been shown to produce an acceptable surface. Isopropanol is used as the wetting agent and a figure-eight motion seems to create a nice finish.

Etching: Individual cleavage chips or thin sections can be etched by multiple methods. One that leads satisfactory results is to submerge the sample in a solution of methanol saturated with $PbCl_2$. The etching process is stopped with butanol (Hansen et al. 2012). Etching requires practice. Typically, the single chip is held with a tweezers and agitated in the $PbCl_2$ solution for 3 to 4 seconds and stopped immediately by transfer to butanol. Excess liquid can be drawn off by dropping the chip on a dry Kimwipe®. Etching highlights the substructure in the crystal lattice, which can be immediately examined on an optical scope. This quick examination also allows evaluation of how well etching was done. Crisp etches with sharp contrast and resolution allow for better evaluation of the substructure. After cleavage chips are successfully etched, they are mounted with carbon tape and coated in gold-palladium for observation of the etched surface under the SEM at a higher magnification and resolution.

Another technique, described by Spiers et al. (1986) and Urai et al. (1987), involves a slightly undersaturated solution of NaCl (~5.5 M) with 0.8 wt% $FeCl_3*6H_2O$. The cleavage chip is immersed in the solution while agitating for 10 seconds, followed by blasting with a jet of n-hexane. Methods are at the discretion of the principal investigator depending on sample type and condition.

4.5 Observation Techniques

Point counting: Point counting is a common technique for manually determining the porosity of a sample by counting the number of void spaces and solid spaces. A thick-thin or thin section is placed on a mechanical sliding stage (example Leitz stage shown in Figure 7), on an optical microscope. We have found that for salt thin or thick-thin sections, racking the stage horizontally 40 mm and 20 mm vertically at nominally 1-mm intervals provides accurate coverage. The petrographic section is moved within the stage in a grid-like pattern. At each grid point a determination (one count) of solid or void under the cross hairs is made by the observer. Typically, 300 to 500 counts are made to obtain a statistical estimate considered representative for the entire sample. A single sample can be re-measured by changing the direction of movement.

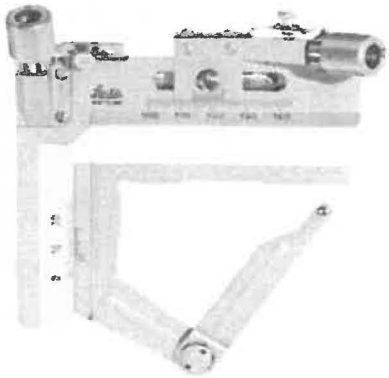

Figure 7. Leitz mechanical slide stage for optical microscope

An example from previous work is shown in Table 1. The ratio of void to solid space was calculated and multiplied by 100 to determine the percent porosity. There are other means to estimate porosity. In this

particular example, laboratory measurements of geometric change roughly approximated the porosity to be 14%. The average obtained by point counting is 13.8%. On a section by section basis, point counting porosity is quite precise, depending on the skill of the microscopist. The accuracy shown here is characteristic of the data quality objective—justifiably only one significant figure after the decimal point.

Table 1. Results of the point counting technique on a thin section sample

Trial #	Void #	Solid #	Ratio	Porosity
1	44	257	0.146	14.6%
2	40	260	0.133	13.3%
3	41	259	0.136	13.6%
			Average	13.8%

4.6 Sample Control

Sample control for the experiments carried out under the TP will follow NP 13-1 "Control of Samples and Standards". For samples created during collaborative endeavors, samples will either be given directly to the Principal Investigator and hand-carried to the Geomechanics Laboratory or Building 823 at Sandia Laboratories, or shipped to the Principal Investigator. Other samples may be created in the SNL Geomechanics Laboratory. The Principal Investigator will retain possession during sample preparation and observational work. If necessary, a Chain of Custody according to procedure SP 13-1 will be created by the Principal Investigator. Prior to any work, sample names and information will be documented in a Scientific Notebook designated with appropriate title and number.

4.7 Data Quality Control

4.7.1 Measuring and Test Equipment

In accordance with NP 12-1, Control of Measuring and Test Equipment (M&TE), measuring devices will be identified in the Scientific Notebook in a manner that allows for easy retrieval and reproduction.

4.7.2 Data Acquisition

Data collection will be recorded in the Scientific Notebook. Most information comes by qualitative observations made on the optical or SEM microscope, which is recorded in the Scientific Notebook. Accompanying photographs will be accumulated in a Scientific Notebook Supplement. There are no plans to acquire data by typical electronic data acquisition systems. Procedure NP 20-2 Scientific Notebooks will be followed. As described in the text above, much of the scientific inquiry here is observational in nature.

5 TRAINING

Any personnel involved in sample preparation, microscopy, and measurements described in this Test Plan must have the requisite training and experience by virtue of hands-on experience within their respective research projects. Personnel must adhere to WIPP requirements identified in NP 2-1, Qualification and Training, and fulfill all necessary procedures and documentation.

6 HEALTH AND SAFETY

Research associated with this Test Plan will be undertaken in two locations: 1) Building 849 Sandia Geomechanics Laboratory and 2) Building 823 of Sandia Tech Area 1. Scanning electron and optical microscopy along with sample sputtering will take place in Building 823 and involve operator M. Mills.

Sample sawing, impregnation, and polishing will occur as necessary within the Geomechanics Laboratory at Sandia Building 849 and are governed by SNL NEPA ID: NM16-0004; Bldg 849 Geomechanics Laboratory Materials Characterization Testing and PHS #: SNL07A00110-010, entitled: Bldg 849 Geomechanics Laboratory. Laboratory operations describe the non-radiological hazards associated with these activities and procedures to deal with those hazards, including training requirements for personnel active in the laboratory.

7 PERMITTING/LICENSING

There is no special license or permit requirement for the activities described in this Test Plan.

8 REFERENCES

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