SANDIA NATIONAL LABORATORIES
CIVILIAN RADIOACTIVE WASTE MANAGEMENT
TECHNICAL PROCEDURE (TP)

TP-092

CONSTANT STRESS (CREEP) EXPERIMENTS AT ELEVATED TEMPERATURE

Revision 02

Effective Date: December 11, 2003

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(Reviewer signatures above document the review and resolution of comments.)

REVISION HISTORY

Revision	Description
0	Initial issue
1	TP-092 was deactivated during Audit BSC-ARC-01-010. It is now reactivated for additional work to be performed. No major technical revisions were required from the previous revision, only references to current procedures and other minor editorial revisions.
2	A slight modification to the loading path is implemented in this revision so that elastic moduli data can be collected and other minor editorial revisions.

1.0 Scope and Objective

The objective of this Technical Procedure (TP) is to define the process for New England Research, Inc. (NER) to perform constant stress (creep) experiments at elevated pressure and temperature. This procedure is intended for implementation in a laboratory environment, in conjunction with work for the Yucca Mountain Project (YMP).

2.0 Prerequisites

Before performing work under this technical procedure, personnel must be trained by the author and/or the Principal Investigator (PI), and demonstrate their proficiency in performing the work in this procedure. The trainer has the responsibility for generating a record of the personnel proficiency training, as well as the responsibility that work is performed and documented in accordance with this procedure.

The personnel using this procedure are responsible for ensuring that a controlled copy of this procedure is available and used for performing the work in this procedure.

3.0 Description of Activity

This TP details procedures for laboratory mechanical experiments on rock specimens. The experiments will be performed on right-circular cylinders of tuff with a nominal length to diameter ratio of 2:1. The specimens will be water saturated prior to testing. The mechanical experiments will be run by maintaining a constant differential stress (the magnitudes of the stress will be determined based on prior baseline testing), a confining pressure of 5.0 ± 0.1 MPa, a pore water pressure of 4.5 ± 0.1 MPa, and a temperature of 125 ± 5^{o} C.

For this experimental series, outputs from a variety of transducers (load, pressure, temperature, and strain) may be monitored. The output voltage from each device is conditioned, amplified, converted to digital format, and recorded as a function of time. The outputs from the devices are recorded with a PC-based data acquisition system (DAS). All devices (with the exception of strain gages, which are calibrated after the specimen is set up using a shunt resistor in parallel with one leg of the Wheatstone bridge) will be calibrated, in accordance to AP 12.1Q, *Control of Measuring and Test Equipment and Calibration Standards*.

A description of the equipment and an overview of the test procedures will place the step-by-step procedures in the proper context. All of the creep tests and system checks will be carried out in servo-controlled hydraulic test systems with a minimum axial force capacity of 5.1×10^5 N. The servo-controller is a self-contained digital unit, which operates in either force or displacement feedback. Mineral oil is used as the confining pressure fluid and distilled water as the pore fluid. Both the confining pressure and the pore fluid pressure will be held constant using servo-controlled intensifiers. The axial and radial strains of the specimen will be measured with strain gages and/or LVDTs. The force on the test column is measured with an internal load cell. The position of the loading piston is observed with a displacement transducer. This transducer provides feedback control in constant strain rate tests and is continuously monitored along with all the other transducer outputs during all tests, regardless of the loading method.

Confining stress and axial load are both generated within a single pressure vessel. A moveable platen divides the pressure vessel into two chambers. The confining pressure on the rock is developed in the lower chamber. When the pressure in the top chamber is greater, a differential stress is exerted on the specimen by the moveable platen that separates the two chambers. The axial deformation is controlled by prescribing either a force or displacement on the specimen. The sample assembly consists of the rock core (nominally 101.6 mm in length and 50.8 mm in diameter) with titanium spacers at each end of the specimen. The bottom titanium spacer has an axial hole to permit pore fluid to access the specimen. The specimen and titanium pieces will be jacketed with 0.13mm thick copper sheeting, or rubber tubing depending on the deformation instrumentation employed. The ends of the jacket are swaged onto the endcaps.

The temperature of the specimens is controlled by adjusting the temperature of the confining fluid (mineral oil). Heating bands mounted on the outside diameter of the pressure vessel heat the vessel, which in turn transfers the heat to the mineral oil. A thermocouple, secured between the heating bands and the vessel, serves as the feedback for the temperature controller. The heating bands are positioned so as to minimize the temperature gradient along the specimen; the gradient will not exceed 4^{o} C over the specimen length during a test. The variation in temperature during the experiment will be less than $\pm 5\ ^{o}$C. A second thermocouple inside the vessel senses the temperature at the midpoint of the sample assembly.

4.0 Operations

4.1 System Checks

Prior to any series of compression tests, when test results are suspect, or at other times specified by the PI, an evaluation of the operation of the entire test procedure will be performed. The evaluation consists of performing a constant strain rate compression test on a specimen material having known elastic properties (Young's modulus and Poisson's ratio). An acceptable check will result in a determination of the elastic properties to within ±5% of the published values. Otherwise, the overall procedure must be checked before tests on rock specimens can be performed, or continued. The proper operation of the test apparatus, measurement devices, and data acquisition system are verified by a successful check. The check shall be documented on the Test Data Report (TDR) in Appendix A, noting that the test is a system check.

4.1.1 System Check Procedure

1. Apply strain gages either directly onto the standard material specimen, or onto a copper jacket on the specimen. If a jacket is used, it must be seated to the specimen with a pressure equal to that to be used during the system check. Position the three gages in appropriate locations so as to sense radial strain, and axial strain at two diametrically opposed positions.

2. Place the specimen on the base plug of the test apparatus, and swage the ends of the specimen to the end pieces.

3. Where LVDTs are to be used, place the gages in the appropriate set-up on the sample assembly. The axial gage(s) is (are) positioned in such a way to ensure that the line of the displacement measurement(s) is (are) parallel to the axis of the specimen. The radial displacement gage (as described in Holcomb and McNamee, 1984) is positioned in such a way to ensure that the line of the displacement measurement: (1) passes through, and (2) is perpendicular to the axis of the specimen.

4. Attach the strain measurement devices to their appropriate feedthru electrical connectors.

5. Make the final mechanical adjustments on the LVDTs, if in use.

6. Position the sample assembly to ensure that all loading column components are coaxial.

7. Load the sample assembly into the pressure vessel.

8. Increase the confining pressure to 5 MPa.

9. Advance the loading platen and apply a differential stress of approximately 0.5 MPa on the specimen. This seats the specimen and all the elements in the loading column. This procedure is accomplished by manually operating the servo-controller in load feedback.

10. Change the servo-controller feedback to displacement control. Retract the loading platen until there is no force on the loading column.

11. Once again, advance the loading platen and apply a differential stress of approximately 0.5 MPa on the specimen. Back the platen off until the load is off, but the platen continues to just touch the specimen.

12. Initiate data acquisition. The amplified outputs from all transducers are monitored and recorded using a microprocessor based DAS. The conditioned output signals from the strain devices, pressure transducers, thermocouples and the force cell are presented to an A/D converter, and the voltage data are stored on the data acquisition computer.

13. Adjust the setting on the displacement rate controller to the displacement rate that corresponds to a nominal axial strain rate of 10^{-5} s^{-1}.

14. After a final check of all the transducer values, load the standard specimen to a differential stress of nominally one half of its yield strength.

15. Unload the specimen, at the same axial displacement rate, until the loading piston is out of contact with the specimen.

16. Terminate data acquisition.

17. Decrease the confining pressure to ambient conditions.

18. Remove the specimen from the test apparatus.

4.1.2 Data Processing

Reduce the data. Commercial software with a built-in linear fitting function can be used. The voltages recorded by the DAS will be converted to engineering units by employing simple mathematical formulae that incorporate the scale factors determined for the individual measurement devices during calibration. The following elastic constants will be computed:

Young's modulus, E (GPa), where E= Δaxial stress/Δaxial strain and

Poisson's ratio, ν, where ν = Δradial strain/Δaxial strain.

The elastic constants will be calculated by least-squares linear fits to the uploading data collected between approximately 10 and 50% of the yield stress for the specimen. Axial stress is computed by dividing the axial force by the initial cross-sectional area of the specimen. Stress will be reported in MPa. When using LVDTs, axial strain is obtained by dividing the axial displacement by the gage separation. The measured displacement is corrected for any endcap contribution, if the support rings for the axial LVDTs are attached to the endcaps. The average axial strain recorded by the two axial devices is used in the moduli calculations. Radial strain is computed by dividing the change in specimen diameter observed by the radial displacement gage by the initial specimen diameter. Where strain gages are used, their outputs are multiplied by their respective scale factors, as determined by their response to the shunt resistor. All strains will be reported as strain, percent strain, millistrain, or microstrain.

Compare the elastic constants determined above with those documented for the standard material. If either, or both of the values deviate from those expected by more than 5% the system check indicates unacceptable operation of the entire test system. No further rock properties testing will ensue until a resolution of the problem(s) is determined. If the results meet the 5% tolerance, the test system is deemed to be operating satisfactorily, and tests on rock specimens may continue.

4.2 Constant Stress Tests

Specimens of tuff will be tested at a constant differential stress, a confining pressure of 5.0 MPa, a pore pressure of 4.5 MPa, and a temperature of 125^{o} C. In order to determine the elastic moduli, a constant strain rate loading cycle, to approximately one half of the expected ultimate strength, will precede the constant stress portion of the test. The differential stress to be applied to the specimen will be a percentage of the expected ultimate strength, as predicted from previous confined compression tests results and correlations of strength with porosity and moduli. The following sections include the step-by-step procedures for the mechanical experiments.

4.2.1 Experimental Procedures

1. The specimen designated for testing will be prepared per TP-51, entitled “Preparing Cylindrical Samples, Including Measurement of Dimensional and Shape Tolerances.” If drying and/or saturation are required, use SNL TP-65, entitled “Drying Geological Samples to Constant Weight” and/or SNL TP-64, entitled “Vacuum Saturation of Geologic Core to Constant Weight.” All initial conditions, work activities and a list of all measurement devices (with relevant information) will be documented on the TDR.

2. Visually inspect the rock core. Any major surface irregularities/imperfections should be noted on the TDR, along with a sketch and/or photograph of the specimen.

3. Jacket the specimen in an appropriate material.

4. If strain gages are to be used, seat the jacket, and apply gages in appropriate locations.

5. Place the specimen on the base plug of the test apparatus, and swage the ends of the specimen to the end pieces.

6. Where LVDTs are to be used, place the gages in the appropriate set-up on the sample assembly. The axial gage(s) is (are) positioned in such a way to ensure that the line of the displacement measurement(s) is (are) parallel to the axis of the specimen. The radial displacement gage is positioned in such a way to ensure that the line of the displacement measurement: (1) passes through, and (2) is perpendicular to the axis of the specimen.

7. Attach the strain measurement devices to their appropriate feedthru electrical connectors.

8. Make the final mechanical adjustments on the LVDTs, if in use.

9. Position the sample assembly to ensure that all loading column components are coaxial.

10. Load the sample assembly into the pressure vessel.

11. Apply the defined confining and pore pressures.

12. Advance the loading platen and apply a differential stress of approximately 0.5 MPa on the specimen. This seats the specimen and all the elements in the loading column. This procedure is accomplished by manually operating the servo-controller in load feedback.

13. Change the servo-controller feedback to displacement control. Retract the loading platen until there is no force on the loading column, leave space to allow for the unloaded expansion of the sample assembly.

14. Increase the temperature to the defined value at a rate of $\leq 2^{o}$C/min. Allow thermal equilibration for a minimum of two hours.

15. Advance the loading platen and apply a differential stress of approximately 0.5 MPa on the specimen. Back the platen off until the load is off, but the platen continues to just touch the specimen.

16. Initiate data acquisition. The amplified outputs from all transducers are monitored and recorded using a microprocessor based DAS. The conditioned output signals from the strain devices, pressure transducers, thermocouples and the force cell are presented to an A/D converter, and the voltage data are stored on the data acquisition computer.

17. Adjust the setting on the displacement rate controller to the displacement rate that corresponds to the nominal strain rate to be used in testing.

18. After a final check of all the transducer values, cycle the axial load on the specimen to one half of the expected ultimate strength and back to zero.

19. With the DAS data collection off, return the servo-controller to load feedback and advance the loading platen to apply a differential stress of approximately 0.5 MPa on the specimen. Back the platen off until the load is off, but the platen continues to just touch the specimen.

20. Reactivate the DAS data collection.

21. Quickly (in approximately 10 to 15 seconds) load the specimen to the differential stress that will be held constant.

22. Maintain the temperature, differential stress, confining pressure, and pore pressure constant until the specimen fails, or the test is terminated. A maximum four week test duration limit is allowed. If the specimen has not failed after four weeks at constant stress, the differential stress will be increased at a constant axial strain rate of nominally 10^{-5} s^{-1} until a failure stress is reached.

23. Terminate the data acquisition.

24. Unload the specimen until the loading piston is out of contact with the specimen.

25. Allow the specimen to cool to ambient conditions.

26. Reduce the confining and pore pressures to ambient conditions.

27. Remove the specimen from the test apparatus and examine the manner in which the specimen failed. Record the observations on the TDR.

4.2.2 Data Processing

Reduce the data. Commercial software with a built-in linear fitting function can be used. The voltages recorded by the DAS will be converted to engineering units by employing simple mathematical formulae that incorporate the scale factors determined for the individual measurement devices during calibration efforts. Ultimate strength, and the following elastic constants will be computed:

(a) Young's modulus, E (GPa), where E= Δaxial stress/Δaxial strain and

(b) Poisson's ratio, ν, where ν = Δradial strain/Δaxial strain.

The elastic constants will be computed from the constant strain rate loading cycle data collected between approximately 50 and 100% of the maximum stress applied during the constant strain rate loading cycle. Axial stress is computed by dividing the axial force by the initial cross-sectional area of the specimen. Stress will be reported in MPa. When using LVDTs, axial strain is obtained by dividing the axial displacement by the gage separation. The measured displacement is corrected for any endcap contribution, if the support rings for the axial LVDTs are attached to the endcaps. The average axial strain recorded by the two axial devices is used in the moduli calculations. Radial strain is computed by dividing the change in specimen diameter observed by the radial displacement gage by the initial specimen diameter. Where strain gages are used, their outputs are multiplied by their respective scale factors, as determined by their response to the shunt resistor. All strains will be reported as strain, percent strain, millistrain, or microstrain.

4.2.3 Electronic Media and Hard Copy Document Handling

Data in digital format, and hard copy documents are handled so as to maintain their integrity, and protect information from damage, or destruction. All digital and hardcopy documentation of the testing will be retained at NER for a minimum of three years following the end of the testing sequence.

Data collected during the testing is stored directly on to the hard disk of the data acquisition computer in the laboratory. At least once each day, the data files are accessed, over the NER internal network, by the operator using the non-laboratory computer on which data processing will occur. The original, unmodified content of the accessed raw file is also maintained on the data processing computer. The raw data will be manipulated in order to convert the voltage information to engineering units from which to determine the mechanical properties. System checks serve to verify proper input, transfer and manipulation of the data, so as to insure completeness and accuracy of the information, including changes thereto. The file generated by the data processing software program will include the unmodified raw data and the processed data, and it will also be retained on the hard disk of the operator's computer.

All computers are password protected, and behind a firewall. Access to the computers is limited to the employees qualified for YMP activities. Backups of non-laboratory computers are automatically performed nightly, with a redundant technique. All backups contain the actual date and time stamps of the original files, so no time stamp information is lost. The date and time that the backup was performed is also stored. Integrity verification is done by using checksums on source and backup files.

Multiple labeled, and redundant copies of all backups are kept offsite on removable hard disks in order to maintain retrievability. One copy, on disk, is rotated into onsite use on a weekly basis, while rotating another copy offsite. Additional integrity checks are performed when an offsite copy is rotated into redundant onsite service. In addition, the operator will backup all files on the data processing computer, weekly, by copying them to a compact disk that will be stored offsite along with the hard copy documents.

Hard copy documentation shall be maintained by ensuring that there is a copy of completed originals available offsite at all times, and until the documents have been formally submitted to the YMP. All completed documents and copies shall be stored in an environment suitable for their maintenance.

5.0 Safety

Operations shall be in accordance with safety requirements of the facility where the work is being performed and those of the employer of person(s) performing the work. No safety hazards other than normal hazards of the equipment.

6.0 Nonconformance, Deviations, and Corrective Actions

Any nonconformances or deviations must be reported to the PI as soon as possible. Deviations, deficiencies and corrective actions must be determined and documented in accordance with AP-16.1Q, *Condition Reporting and Resolution*

7.0 QA Records

QA records, and any corrections or changes thereto, generated as a result of implementing this procedure will be prepared and submitted as inclusionary QA records (QA:QA) by the PI in accordance with AP-17.1Q, *Records Management*. These records include:

Proficiency training records (Section 2.0)

Test Data Reports (TDR) (Section 4.0)

Calibration records (if applicable)

8.0 References

Holcomb, D. J. and M. J. McNamee, 1984. *Displacement Gage for the Rock Mechanics Laboratory*. SAND 84-0651. Albuquerque, New Mexico: Sandia National Laboratories.

TP-51, *Preparing Cylindrical Samples, Including Measurement of Dimensional and Shape Tolerances*

TP-64, *Vacuum Saturation of Geologic Core to Constant Weight*

TP-65, *Drying Geological Samples to Constant Weight*

AP-12.1Q, *Control of Measuring and Test Equipment and Calibration Standards*

AP-16.1Q, *Condition Reporting and Resolution*

AP-17.1Q, *Records Management*

TEST DATA REPORT (TDR)

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Test Plan Number / Rev.:________________________________Specimen ID:__________________________

Moisture Condition:_______________Length: ____________Diameter:____________Weight:_____________

Dry Bulk Density:_________Saturated Bulk Density: _________Grain Density:_________Porosity: _________

Required Test Conditions (e.g., differential stress, pressures, temperatures):___________________________

____Test

Apparatus Used:_________________________________Latest System Check Date:_________________

Measurement Devices (Some categories not relevant to all devices) / Label unused channels “Inactive”:

Raw DAS Channel (Column)	Device/Model/Serial#	Latest Calibration Date	Gain Setting	Gage Factor	“Zero” Voltage	Shunt Voltage Difference	Scale Factor: Units/Volt	Notes

DAS raw voltage data file: ____

Processed data file:____

Operator(s): Name:________________________Sign/Date:___________________________________

Name:________________________Sign/Date: ___________________________________

Name:________________________Sign/Date: ___________________________________

Name:________________________Sign/Date: ___________________________________

Organization / Location of Testing:____

TEST DATA REPORT (TDR)

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Continuation Page

Specimen ID: ______________________________

Operator(s): Name:________________________Sign/Date:____________________________________

Name:________________________Sign/Date: ____________________________________

Name:________________________Sign/Date: ____________________________________

Name:________________________Sign/Date: ____________________________________