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Yucca Mountain Site Characterization Project

Procedure Development Study: Low Strain Rate and Creep Experiments

R. J. Martin III, P. J. Boyd, J. S. Noel, R. H. Price

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PROCEDURE DEVELOPMENT STUDY: LOW STRAIN RATE AND CREEP EXPERIMENTS

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ABSTRACT

Licensing of the potential nuclear-waste repository at Yucca Mountain by the Nuclear Regulatory Commission would require, among other things, demonstration of the long term usability of the underground facilities. Such a demonstration involves analysis of the mechanical response of the rock to the presence of underground openings and heat-producing waste, which in turn requires data on the mechanical properties of the rock. This document describes the experimental results from a scoping study which led to the development of procedures for performing quality-affecting rock-mechanics experiments on intact rock. The future experiments performed with these procedures will produce information on the time-dependent deformation of welded tuff and represent one aspect of the overall effort to characterize the rheology of the rock mass.

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PROCEDURE DEVELOPMENT STUDY: LOW STRAIN RATE AND CREEP EXPERIMENTS

1.0 Introduction

Yucca Mountain, Nevada, has been selected as a potential repository of nuclear wastes. The Yucca Mountain Site Characterization Project (YMP) of the Civilian Radioactive Waste Management Program has been assigned the task of determining the suitability of the Yucca Mountain site. Among the concerns being investigated, the characterization of the mechanical properties of the host rock has direct relevance to design activities, as well as to pre- and post-closure performance assessment.

This report presents the results from a scoping study designed to develop procedures for performing rock mechanics experiments on intact rock. The experiments to be run with these procedures will yield information on the time-dependent deformation of the potential repository-horizon welded tuff (TSw2 in the thermal/mechanical stratigraphy defined by Ortiz et al., 1985), and represent one aspect of the overall effort to characterize the rheology of the rock mass.

2.0 Summary of Experiments

Experiments were performed at three sets of conditions on samples of the Topopah Spring Member of the Paintbrush Tuff. These initial experiments were conducted to develop procedures necessary to perform quality-affecting experiments and calibrations. Appropriate samples of rock from the potential repository horizon are presently scarce; therefore, because of the scoping nature of these tests, all of the samples used in this study had characteristics which would invalidate their use in quality-affecting experiments (i.e., the data presented in this report should not be construed to be indicative of the actual intact rock properties of this tuff unit). The samples either contained large lithophysal voids (too large to consider the results valid for this sample size) or contained non- or partially-healed fractures (a fracture in the sample violates the assumption that the resultant properties are for intact rock). The samples were gathered from an outcrop of the Topopah Spring tuff on the southeast flank of Busted Butte, southeast of Yucca Mountain in southern Nevada. For a mineralogic and petrologic description of adjacent samples from this outcrop, see Price et al. (1987).

The water-saturated samples were tested in compression under the following three sets of experimental conditions.

- Unconfined Compression at Ambient Temperature

Strain Rate: 10^{-9} s^{-1}

Temperature: Ambient

Pressure: Ambient

- Confined Compression at Elevated Temperature

Strain Rate: 10^{-8} s^{-1}

Temperature: 250°C

Confining Pressure: 5.0 MPa

Pore Pressure: 4.5 MPa

- Constant Stress Tests

Creep Stresses: 50 and 70 MPa

Duration/Temperature: 168 hours at Ambient Temperature and 1,246 hours at 250°C

Confining Pressure: 5.0 MPa

Pore Pressure: 4.5 MPa

All experiments were conducted on ground right-circular cylinders approximately 50.8 mm in diameter and 101.6 mm in length. Following machining, the samples were placed in an oven and slowly heated to 110°C, held at temperature for at least 120 hours, cooled in the oven, removed, and weighed. They were subjected to as many additional drying cycles, including heating to 110°C for 24-36 hours followed by cooling and weighing, as necessary to meet a constant-weight criterion for a dry sample. The dry weight and sample dimensions were used to calculate a dry bulk density for each sample. Subsequently, the samples were submerged in distilled water and subjected to 3 or more saturation cycles, each of which included at least 18 hours under an active vacuum followed by submersion for at least 6 hours at ambient pressure. After each saturation cycle the samples were weighed, and the sequence of cycles were continued until a constant-weight criterion for saturated samples was met. For each sample, a saturated bulk density was calculated. The bulk density data are presented in Table 1. (Note: The formal identification of the samples used in this study are of the form: BB-10AE-23W-SNL, where BB

refers to the sample location - Busted Butte, 10AE is the large rock the sample was taken from, 23W is the position within block 10AE, and SNL is the sample user. For brevity, the majority of the sample references in this document will delete the BB and SNL modifiers.)

TABLE 1.
BULK DENSITY PROPERTIES

Sample	Dry Bulk Density	Saturated Bulk Density
—	<u>g/cc</u>	<u>g/cc</u>
10AE-23W	2.16	2.30
10AE-50X	2.28	2.38
10AE-19Z	2.24	2.34
10AE-48X	2.24	2.33
10AE-37Z	2.29	2.37

The results of the mechanical experiments are summarized in Table 2. The following data are presented for each sample: sample identification, test temperature (T), strain rate ($\dot{\epsilon}$) or creep stress (σ_c), confining pressure (P_c), pore pressure (P_p), Young's modulus (E), Poisson's ratio (ν), and fracture strength (σ_f) where appropriate. As mentioned above, the quality of the samples used in these tests varied greatly. Due to the large lithophysal cavities, fractures, and other large inhomogeneities in these samples, the compressive fracture strengths determined in this study cannot be considered representative of the tuff as a whole. On the other hand, the resultant elastic properties (Young's modulus and Poisson's ratio) are in good agreement with previously reported values on the same lithology. Furthermore, for several samples on which compressional and shear wave velocities were measured prior to testing and the dynamic Young's modulus and Poisson's ratio were computed.

As an integral part of this study, system calibration-checks were performed on aluminum standards. Prior to each experiment at ambient temperature, a specimen of 2024-T351 aluminum was uniaxially loaded to half its yield strength at a strain rate of 10^{-5} s^{-1} . The moduli computed from these tests agree to within 5% of the values determined from the open literature

for this material. Based on the ability to measure these constants to within an accuracy of 5%, the performance of the system was judged acceptable to perform the experiments.

TABLE 2.
STATIC MECHANICAL PROPERTIES

Sample	T	$\dot{\epsilon}$	σ_c	P_c	P_p	E	ν	σ_f
_____	°C	s ⁻¹	MPa	MPa	MPa	GPa	_____	MPa
10AE-23W	25	10 ⁻⁹	-	0	0	23.9	0.17	36
10AE-50X	25	10 ⁻⁹	-	0	0	30.7	0.12	75
10AE-19Z	250	10 ⁻⁸	-	5	4.5	41.6	0.09	71
10AE-48X	250	10 ⁻⁸	-	5	4.5	36.4	-	58
10AE-37Z	25	-	49	5	4.5	38.7	0.13	-
	250	-	51	5	4.5	39.4	0.17	-
	250	-	51	5	4.5	40.9	0.18	-
	25	-	50	5	4.5	35.9	0.22	-
	250	-	70	5	4.5	39.0	0.11	-

Prior to both the constant strain rate and creep experiments at 250°, a single high temperature calibration was performed on a specimen of 6061-T6511 aluminum. The calibration-check was performed using the same sample geometry employed during the tuff measurements; that is, the sample was jacketed with 0.13 mm thick copper and subjected to a confining pressure of 5 MPa and a temperature of 250°. The sample was loaded in compression to half of its reported yield strength at a strain rate of 10⁻⁶s⁻¹. Young's modulus and Poisson's ratio were determined for this data set. The only high temperature modulus reported in available references is Young's modulus. The value from the calibration experiment differed from this value by 10%.

Based on the results of these preliminary tests, the final procedures have been developed. This report will discuss the procedures; more detailed descriptions of the step-by-step process are provided in YMP procedures.

3.0 Constant Strain Rate Experiments at 10^{-9} s^{-1}

Two specimens of Topopah Spring Member tuff were tested in uniaxial compression at a strain rate of 10^{-9} s^{-1} . The experiments were carried out under ambient temperature conditions on saturated rock cores.

3.1 Sample Preparation and Experiment Apparatus

The saturated samples were jacketed with polyolefin heat shrink tubing and positioned between two hardened steel end caps. The jacket was sealed to the end caps with silicone adhesive to ensure that the samples remained saturated during the preparation stages and testing.

Each sample was instrumented with three LVDTs (linear variable differential transformers). Two LVDTs were used to measure the axial displacement on the specimen during deformation; the third LVDT monitored the change in one diameter of the specimen. The axial LVDTs were mounted in an aluminum ring which was positioned concentrically about the sample. The cores for the LVDTs were attached to a second ring separated from the first ring by a fixed distance.

For specimen 10AE-23W the rings were mounted directly on the sample, and the axial strain was computed by averaging the two axial displacements and dividing by the separation between the center of the rings (approximately 50.8 mm). A schematic of this arrangement is shown in Figure 1.

For specimen 10AE-50X, the axial LVDT support rings were mounted on the steel end caps. This geometry was employed to alleviate potential problems that might be encountered in securing the LVDT support rings to the specimen. During rock failure, shifts in the support rings will result in anomalous displacements. A calibration sequence using aluminum specimens of different lengths was performed to calculate the contributions of the interfaces and the end caps to the total displacement. The elastic displacement of the interfaces and the portion of the steel end caps between the support rings was measured as a function of axial force. The axial strain was computed by averaging the two axial displacements, subtracting the interface and end cap correction, and dividing by the sample length (approximately 101.6 mm).

The radial displacement on each sample was measured with a radial displacement gauge similar to that described by Holcomb and McNamee (1984). Lateral displacement was divided by the original sample diameter in order to calculate lateral strain.

The instrumented sample was placed onto the base plug of the pressure vessel. Water, at ambient pressure, was allowed to flow into the specimen through a pore pressure inlet. A water filled reservoir was attached to the pore pressure inlet. The head of water was adjusted so that it was level with the top of the specimen. This technique was used to maintain complete saturation of the specimen throughout the experiment. After the saturation system was in place, the LVDTs were electrically connected and adjusted so that their output was slightly positive. Once all the initial checks on the instrumentation were completed, the sample assembly was inserted into a pressure vessel. Although the experiments were run at room pressure, a pressure vessel was used to prevent thermal fluctuations that might adversely influence the test data.

The uniaxial compression tests were carried out in a servo-controlled hydraulic loading frame with an axial load capacity of 6.7×10^5 N. The system was operated in displacement feedback; the sample strain rate was nominally 10^{-9} s⁻¹. The outputs from seven transducers (three LVDTs, two load cells, a ram displacement gage, and a thermocouple) were amplified and recorded in a computer-based data-acquisition system. A schematic diagram of the data-acquisition system is shown in Figure 2.

3.2 Sample Description

BB-10AE-23W-SNL: Major, sharp, compositional contact that is nearly perpendicular to core axis, approximately dividing the sample in half. One half is fine grained, and has consistent pink coloration. The other is coarser grained with variegated coloration. Very minor lithophysae are present in the coarser end only, and no obvious fractures exist in the sample.

BB-10AE-50X-SNL: Open lithophysal slits near each end face, nearly parallel with the faces. One minor partially calcite-filled lithophysis in middle of sample. No obvious fracturing.

3.3 Experimental Results

Two Topopah Spring Member tuff specimens were loaded to failure in uniaxial compression at a strain rate of about 2.5×10^{-9} s⁻¹. These results are presented in Figures 3 through 8.

The first experiment was conducted on specimen 10AE-23W. A Young's modulus of 23.9 GPa and a Poisson's ratio of 0.17 were computed from the strain data collected between 10 and 50% of the ultimate axial stress. The observed fracture strength was approximately 36 MPa.

Several difficulties were encountered in this experiment. Approximately 2.5×10^5 seconds into

the experiment, a power failure occurred. The duration of the power loss was only momentary. Since the data-acquisition system runs on an uninterruptable power supply, data collection was continuous throughout this event. However, when the power came back on an electrical surge caused some disruption in the performance of the servo-system. This can be seen in Figure 3, which shows the axial strain as a function of time for the experiment. Fortunately the disruption of the test occurred at a stress less than one-half the fracture strength, when the specimen deformation was essentially elastic. No effects are apparent on the elastic properties and the ultimate strength as a result of the non-uniform loading.

The axial strain is shown as a function of axial stress in Figure 4. When compared to previous data, the trend of the axial stress-axial strain data appears fairly representative for tuff specimens (Price et al., 1987). The radial strain/axial strain relation is linear at axial strains less than 2×10^{-3} (Figure 5). As the sample approached failure, the strain relationship became non-linear. The offset in the radial strain occurred during pervasive fracturing.

The Young's modulus computed from the stress-strain data for specimen 10AE-50X (Figure 7) was 30.7 GPa. A Poisson's ratio of 0.12 was computed from the strain data shown in Figure 8. The sample failed at a stress of about 73 MPa.

While the axial strain behaved as expected during this experiment, the initial radial strain measurements proved to be anomalous. In fact, the initial loading sequence of the specimen was terminated after two days because no change in radial strain was detected. The sample was removed from the pressure vessel and all the instrumentation was thoroughly examined to ensure that it was operating correctly. The radial displacement gage was placed in a different orientation through the midplane of the specimen and the assembly was reinserted into the pressure vessel. The experiment was restarted. Once again, no change in radial displacement was observed after several days; however, the experiment was continued to failure. It appears that the lack of radial strain is most likely related to the actual rock behavior rather than any problem with the experimental setup or test procedure.

Ultrasonic velocities were measured on specimen 10AE-50X before initiating compressional loading. The compressional and shear wave velocities were observed for both the room dry and saturated states; the dynamic elastic constants were computed from this data set. The results of these measurements are given in Table 3.

TABLE 3.
VELOCITY AND DYNAMIC MODULI DATA FOR SAMPLE 10AE-50X

<u>Property</u>	<u>Units</u>	<u>Room Dry</u>	<u>Saturated</u>
Compressional Velocity	km/s	4.59	4.62
Shear Velocity	km/s	2.78	2.70
Young's Modulus	GPa	42.6	43.1
Poisson's Ratio		0.21	0.24

3.4 Discussion

The elastic constants observed on specimens 10AE-23W and 10AE-50X are somewhat lower than those reported for samples from the same Busted Butte outcrop by Price et al. (1987). For example, Price et al. (1987) reported that the Young's moduli were 40.1 ± 3.8 GPa and the Poisson's ratios were 0.20 ± 0.02 for saturated samples tested at ambient pressure and temperature conditions and a strain rate of 10^{-7} s^{-1} . Because the numbers of samples being reported are relatively small (two in this study and four in the Price et al. study), the difference could very well be a product of material variability rather than a rate effect.

The dynamic elastic moduli computed from the velocity data collected on specimen 10AE-50X provide an upper bound to the quasi-static moduli measured during compressional loading. The dynamic Young's modulus is 41% greater than that determined from the stress vs strain data. Similarly, the dynamic Poisson's ratio is 50% greater than the static value. These differences are large; however, the dynamic elastic constants are well within the range of static values reported by Price et al. (1987). It must be pointed out that this is one set of measurements on a single specimen, and many more comparative measurements must be made before a meaningful comparison can be established.

As expected (see earlier discussion on sample characteristics), the fracture strengths were significantly less than those reported by Price et al. (1987). For example, for the same samples referred to above (that were tested at ambient pressure and temperature conditions and a strain rate of 10^{-7} s^{-1}), the fracture strengths were 131.0 ± 25.2 MPa. It is highly unlikely that merely reducing the loading rate two orders of magnitude will explain the observed strength differences. Most probably, sample quality is the reason.

4.0 Constant Strain Rate Experiments at 10^{-8} s^{-1}

Two specimens of Topopah Spring Member tuff were tested to failure at a strain rate of 10^{-8} s^{-1} , a confining pressure of 5.0 MPa, a pore pressure of 4.5 MPa, and a temperature of 250°C. Young's modulus, Poisson's ratio, and fracture strength were computed from the data set.

4.1 Sample Preparation and Experimental Apparatus

Prior to static mechanical testing, the compressional and shear wave velocities were measured on the saturated samples.

Because the experiments were to be conducted at high temperature, a simple jacketing arrangement was not suitable. Consequently, the sample was positioned between low thermal conductivity Macor ceramic spacers. The entire sample assembly (i.e., tuff specimen and Macor spacers) was jacketed with 0.13 mm thick copper. The jacketed sample was then swaged onto a stainless steel plug to seal the jacket and isolate the sample from the confining medium. Figure 9 shows a schematic diagram of the sample assembly, furnace, and pressure vessel used in these measurements. The loading frame and the pressure vessel were the same ones used in the experiments described in Section 3. Once the sample was positioned on the base plug of the pressure vessel, the furnace was lowered into place and the LVDTs connected to the electrical feed-throughs and checked for continuity.

The furnace used in these experiments has two zones with independent controls and feedback thermocouples for each zone. The thermocouple for the lower zone is located in the pore pressure inlet tube and senses the temperature at the bottom of the tuff specimen. The upper zone of the furnace is monitored with a thermocouple which enters the pressure vessel through the loading piston and impinges on the top of the sample assembly. A calibration sequence was carried out prior to initiating the high temperature experiments to determine the power required by each zone to develop a temperature of 250°C with a gradient along the axis of the sample of less than $\pm 4^\circ\text{C}$. The furnace profile obtained during this calibration sequence is shown in Figure 10. The position of the tuff specimen with respect to the profile is shown in the diagram. Note that the temperature over the length of the specimen is very uniform, and that the temperature drops off rapidly away from the sample.

The jacketed sample was instrumented to measure the axial and radial displacement during loading. The technique for measuring the displacement is identical to that described for the

10^{-9} s^{-1} , room temperature experiments. LVDTs were used to monitor the change in sample geometry. Due to the high temperature it was necessary to locate the axial LVDTs near the base plug of the system to remove them from the temperatures encountered within the furnace assembly. To accomplish this, the LVDT barrels were supported in extended tubes attached to the lower end of the sample (See Figure 9). The cores were secured on extended stainless steel rods mounted in a second ring located near the top of the sample. The extension rods for the cores passed through the tubes supporting the LVDT barrels, and into the transducer.

The radial strain was measured with a radial displacement gage very similar to that described in the previous section; however, the support ring for the LVDT was positioned outside the furnace assembly. Radial gage access to the sample was achieved by passing the extension rods through holes in the wall of the furnace. The LVDT core was coupled to the rock with an extended rod that passed through the furnace wall. The most important consideration in this arrangement is the isolation of the LVDT barrels from the high temperatures inside the furnace. Every effort has been made to ensure that the temperatures experienced by the LVDTs do not exceed 105°C , because the output of the LVDTs become distinctly nonlinear at higher temperatures. Thermocouples, which are not shown in Figure 9, were positioned next to the LVDTs so their temperature could be monitored.

Following an initial check of all the electrical connections, the confining pressure was increased to 5.0 MPa. The confining medium was argon. After the confining pressure stabilized and no leaks in the jacket were detected, the pore pressure was increased to 4.5 MPa. Once the pore pressure was in equilibrium, the sample was heated to a temperature of 250°C at a rate not exceeding 2.0°C per minute. During the heating and differential-stress loading phases of the experiment, the confining pressure and pore pressure were held constant (with the ratio of P_c/P_p remaining at a constant 1.1) by a servo-controlled intensifier. The sample was then loaded monotonically to failure at a constant, nominal strain rate of 10^{-8} s^{-1} . The data were collected using the data-acquisition system described previously and schematically shown in Figure 2.

4.2 Sample Description

BB-10AE-19Z-SNL: Homogeneous, fine-grained composition, except for a compositional contact at the corner of the sample at one end between a dark reddish brown matrix and a lighter red matrix. Small, partially-filled lithophysae are elongated perpendicular to the core axis throughout the sample. No fracturing is evident.

BB-10AE-48X-SNL: Homogeneous, fine-grained composition. A large lithophysal cavity is nearly perpendicular to the core axis, and is within one inch of one end of the core. No fractures are evident.

4.3 Experimental Results

Two experiments were conducted to test the apparatus and to establish the required procedures. Specimen 10AE-19Z was tested first. Figure 11 shows the axial strain plotted as a function of time. Note that for the first 2×10^4 seconds or so, no strain was measured. The axial strain then increased linearly with time until the sample failed. In Figure 12, stress difference is plotted as a function of axial strain. At strains less than 1.5 millistrain, the sample deformed more or less linearly. The Young's modulus was computed using the data between 10 and 50% of the ultimate stress. Young's modulus was 41.6 GPa. At higher stresses, the stress-strain curve became non-linear. At a stress of approximately 55 MPa, an experimental difficulty was encountered. The pore pressure intensifier ran out of stroke and the pore pressure began to decrease. This increased the effective confining pressure (i.e., confining pressure minus pore pressure) and strengthened the rock. The increase in effective confining pressure can be seen in Figure 12. The pore pressure did not drop to zero, but gradually decreased to approximately 3 MPa. Figure 13 shows the correlation between stress difference and pore pressure during the experiment. From this plot it is very clear to see that the strengthening of the rock occurred as a direct consequence of the decrease in pore pressure. Furthermore, the rock ultimately failed when the drop in pore pressure was noted and corrected; the correction, in this case, was increasing the pore pressure to its original value of 4.5 MPa. The rock failed at a fracture strength near 71 MPa.

Radial strain is plotted as a function of axial strain in Figure 14. The change in radial strain was extremely small during this experiment. In fact, Poisson's ratio computed from the data set was only 0.09. This is consistent with the Poisson's ratios computed for the low strain rate ambient temperature experiments described in Section 3.

A second experiment was performed under the same conditions on specimen 10AE-48X. Figure 15 shows the change in axial strain as a function of time. The sample deformed linearly up to failure. After 1.25×10^5 seconds, the sample relaxed in response to increasing stress. That is, following the stress difference being increased to 56 MPa, the stress would relax to 52 MPa. This process continued up to failure. The stress relaxation is most likely due to crack growth

and the development of the fracture zone which eventually produced the macroscopic failure of the sample. Consequently, the data are relatively noisy in that region of the plot. Figure 16 presents the stress difference as a function of axial strain. The sample deformed more or less linearly for the first half of the experiment and then began to exhibit a nonlinear behavior up to failure at a stress of about 58 MPa. The Young's modulus was computed to be 36.4 GPa at stress levels between 10 and 50% of the fracture strength. Figure 17 shows the radial strain as a function of axial strain. Poisson's ratio was very low. In fact, no meaningful value could be computed from this data set.

A very interesting point is emerging from these low strain rate experiments. Both at room temperature and 250°C, the radial strain observed is extremely small. As yet, there are insufficient data to make any generalizations regarding this observation. As further experiments are conducted, specific attention will be paid to the radial strain in hopes of understanding the effect.

Ultrasonic velocities were measured before jacketing the specimens. The compressional and shear wave velocities were observed for the saturated state; the dynamic elastic constants were computed from this data set. The results of these measurements are given in Table 4.

TABLE 4.
VELOCITY AND DYNAMIC MODULI DATA FOR
SATURATED SAMPLES 10AE-19Z AND 10AE-48X

<u>Property</u>	<u>Units</u>	<u>10AE-19Z</u>	<u>10AE-48X</u>
Compressional Velocity	km/s	4.51	5.23
Shear Velocity	km/s	2.67	2.98
Young's Modulus	GPa	39.2	52.2
Poisson's Ratio		0.23	0.26

4.4 Discussion

Results of the two experiments described above are internally very consistent. The data sets show that the Young's moduli do not differ greatly between the two samples. Static Poisson's ratios for both rocks were extremely low. The dynamic moduli measured at room pressure and temperature were significantly greater than the static measurements at 250°C. Obviously, it is difficult to compare these two types of measurements at different temperatures. However, the

capability is being developed to make ultrasonic measurements at high temperatures, so that the effect of temperature on both static and elastic constants can be meaningfully evaluated. As discussed before, the sample quality for these scoping experiments was poor; consequently, the fracture strengths are not representative of the actual intact tuff strength.

5.0 Constant Stress (Creep) Experiments

Ideally, each specimen designated for creep testing is to be loaded to a differential stress of 80 MPa at a confining pressure of 5.0 MPa and a pore water pressure of 4.5 MPa. The specimen is to be tested under ambient laboratory temperature conditions for 168 ± 4 hours; then the temperature on the specimen is to be increased to 250°C and the test continued for $1,246 \pm 4$ hours. A single experiment was performed on a specimen of Topopah Spring Member tuff to develop the test procedures and experimental conditions for these measurements.

5.1 Sample Preparation and Experimental Apparatus

A creep experiment was carried out on specimen 10AE-37Z. The experiment was run in the apparatus described in Sections 3 and 4. The servo-controlled hydraulic press, the pressure vessel, and the data-acquisition system were identical for all three types of experiments. The experimental set-up, sample assembly, furnace, and LVDT arrangement are shown in Figure 9 and described in Section 4.1.

Specimen 10AE-37Z was saturated, jacketed, instrumented, and pressurized to a confining pressure of 5.0 MPa with an internal pore water pressure of 4.5 MPa.

The constant stress experiment was carried out in two stages. First, the saturated specimen was monotonically loaded at a constant strain rate of 10^{-5} s^{-1} to a differential stress of about 50 MPa at a confining pressure of 5.0 MPa, a pore pressure of 4.5 MPa, and ambient temperature. Differential stresses lower than specified were used due to poor sample quality and the likelihood that the sample would fail before a differential stress of 80 MPa could be reached. In light of the fact that this experiment was designed to establish test procedures and develop experimental technique, the lower axial stress was deemed necessary and acceptable. The stress was held constant, while temperature, axial force, axial and radial displacement, confining pressure, and pore pressure were recorded. After the initial loading phase, the differential stress on the specimen was removed, but the confining and pore pressures were maintained. The temperature of the specimen was then increased to 250°C . Once the sample and test apparatus were in

temperature equilibrium, a differential stress of 50 MPa was again applied to the specimen. The differential stress was held constant, reduced to hydrostatic, and raised to 50 MPa. After fixing a furnace problem, the sample was reloaded, first at ambient temperature and then at elevated temperature. During the second loading sequence, stress differences of 50 and 70 MPa were applied to the sample at elevated temperature.

5.2 Sample Description

BB-10AE-37Z-SNL: Homogeneous fine-grained composition. No compositional contacts or fractures evident. Lithophysae are small, partially filled, and elongated perpendicular to the core axis.

5.3 Experimental Results

Following application of the confining and pore pressures, specimen 10AE-37Z was loaded to a differential stress of 49 MPa at a constant strain rate. The results of the initial loading are shown in Figure 18 (stress difference plotted as a function of axial strain) and Figure 19 (radial strain plotted as a function of axial strain for the same loading sequence). A Young's modulus and Poisson's ratio were calculated using the data between 10 and 50% of the applied differential stress. The values were approximately 39 GPa and 0.13, respectively. The stress was held constant for 6.5×10^5 s (181 hours). Figure 20 shows the axial strain during the constant stress segment of the test. No change in axial strain was observed during this time period.

The differential stress was removed and the temperature was increased to 250°C. The specimen was then reloaded at a constant strain rate to a stress difference of 51 MPa. The results of this loading sequence are presented in Figures 21 and 22. These data at high temperature are somewhat noisier than those observed during the room temperature loading sequence. In spite of this, a Young's modulus of about 39 GPa and Poisson's ratio of 0.17 were calculated for this tuff specimen at 250°C. The creep data at 250°C are shown in Figures 23, 24, and 25. There appears to be a very small increase in axial strain as a function of time (Figure 23). Superimposed on this trend are several large positive and negative short term deviations. The radial strain as a function of time data (Figure 24) show a very slight decrease in radial strain as the experiment proceeded. Once again, there are several significant deviations from the long term trend of the data. These spikes correspond in time with the spikes seen in the axial strain.

Figure 25 shows the temperature of the specimen as a function of time. Several large devia-

tions in temperature with amplitudes up to 15°C are evident. These deviations coincide in time with the irregularities in the axial and radial strain data; the axial stress, confining pressure, and pore pressure remained constant.

It was apparent from the data presented in Figure 25 that the furnace was not performing satisfactorily so the creep experiment was terminated. The pressure vessel was separated from the sample assembly and the furnace was visually examined. No apparent reason for the temperature fluctuation was detected. Rather than completely disassembling the experimental set-up, the sample was reinserted into the pressure vessel. The sample was reloaded to 50 MPa differential stress, with a temperature of 250°C at a confining pressure of 5.0 MPa and a pore pressure of 4.5 MPa. Young's modulus and Poisson's ratio were computed from the data collected during the loading cycle and are presented in Table 2. Several minutes into the constant stress phase of the experiment, it was apparent that the problem with the furnace persisted so that the experiment was terminated, once again.

The sample was removed from the pressure vessel, the furnace was inspected, and a loose connection was found. This connection was repaired and the sample was again inserted into the test apparatus. The entire experimental sequence was repeated; that is, the specimen was loaded to a differential stress of 50 MPa and held constant for 168 hours at ambient temperature conditions. The ambient temperature data for this loading are not presented. No axial strain was observed during the constant stress phase of the test; Young's modulus and Poisson's ratio computed during the constant strain rate loading phase are presented in Table 2.

The sample was unloaded, the temperature was increased, and the specimen loaded to a differential stress of 70 MPa. The differential stress-axial strain curve for the reloading segment at 250°C is shown in Figure 26. The radial strain is shown as a function of axial strain for the same loading sequence in Figure 27. The Young's modulus and Poisson's ratio computed from these data are 39 GPa and 0.11, respectively. The data from the creep experiment are shown in Figures 28 through 33. The specimen failed 2.25×10^5 s (approximately 62 hours) after initial loading, apparently the result of premature failure of the jacket. Considering the number of thermal and stress cycles that the specimen and jacket were subjected to, this result is not surprising. All other aspects of the experiment were satisfactory.

The axial strain showed a slight increase with time (Figure 28) prior to failure. The data do not show any of the irregularities that characterize the data in Figure 23. The strain began

to decrease as the jacket failed 1.75×10^5 s after loading. The radial strain appeared to increase at a relatively constant rate (Figure 29) throughout the experiment. When the jacket failed, the radial strain increased precipitously and went off scale. In general, the strain data are of very high quality with very little noise. Stress difference, confining pressure, pore pressure, and temperature were held constant during the experiment (Figures 30, 31, 32, and 33, respectively). The stress difference, confining pressure, and temperature remained stable throughout the experiment. Small deviations were observed in the pore pressure prior to the leakage of the jacket; however, the deviations were less than 0.1 MPa. When the jacket failed, the pore pressure increased to 5.0 MPa, the value of the confining pressure.

5.4 Discussion

The required parameters for the creep experiments on Topopah Spring Member tuff easily can be obtained using the procedures developed from this activity. The experiments on specimen 10AE-37Z show that the creep rates will undoubtedly be extremely small at 250°C and virtually nonexistent at lower temperatures. It would be premature, however, to make any further quantitative statements on the rheological behavior of tuff based on this single experiment on an inhomogeneous sample.

Young's modulus and Poisson's ratio were determined from the data collected from five loading sequences on specimen 10AE-37Z; the elastic constants computed for these data are presented in Table 2. It is interesting to note that Young's modulus for this specimen was relatively insensitive to temperature (i.e., there was no evidence of lower values being associated with higher temperatures). For these five cycles, Young's modulus varied between 35 and 41 GPa. Similar results were noted for Poisson's ratio values, which ranged from 0.11 to 0.22.

6.0 Aluminum Calibration-Check Experiments

Calibration-check experiments were carried out at room temperature and at 250°C on aluminum specimens with the same nominal dimensions as the tuff samples (i.e., 50.8 mm in diameter and 101.6 mm in length). The purpose of these measurements is to verify the accuracy and reproducibility of the entire testing system (i.e., test frame, calibrated instrumentation, and data-acquisition system). If the elastic constants determined by the calibration-check at ambient temperature agree to within 5% of the published values for the aluminum specimen, the system is deemed ready to begin the experiments. Because values of the elastic constants at elevated temperatures are not well documented, a deviation of $\pm 10\%$ of the published value(s)

will be acceptable. Failure to meet these specifications prior to or subsequent to an experiment performed on a tuff sample requires an evaluation and corrective action before additional experiments can be conducted.

6.1 Aluminum Calibration-Check at Ambient Temperature

Calibration-check experiments were conducted before and after each experiment on a tuff specimen. Specimens of 2024-T351 aluminum were tested in uniaxial compression at a strain rate of 10^{-5} s^{-1} . Calibration-checks AL9 and AL16 were carried out before and after, respectively, the test on tuff specimen 10AE-23W. Calibration-checks AL17 and AL50X were carried out before and after, respectively, the test on tuff specimen 10AE-50X. For tests AL9 and AL16 the axial LVDT support rings were mounted directly on the sample, whereas for tests AL17 and AL50X the LVDT support rings were mounted on the end caps at each end of the specimen. The results of the calibration-check tests are presented in Figures 34 through 39 and in Table 5.

TABLE 5.
AMBIENT TEMPERATURE CALIBRATION-CHECK RESULTS

<u>Test</u>	<u>E, GPa</u>	<u>Deviation, %</u>	<u>ν</u>	<u>Deviation, %</u>
AL9	74.94	+2.5	0.324	+1.3
AL16	74.57	+2.0	0.323	+0.9
AL17	73.11	+0.0	0.317	-0.9
AL50X	72.58	-0.7	0.317	-0.9

It is obvious that these results are well within the stated requirements (i.e., that the elastic constants computed for the calibration-checks be within 5% of the published values for the standard specimen). Furthermore, there appears to be no significant difference in the values determined when the axial displacement rings are mounted directly on the sample compared to those observed when the rings are mounted on the end caps.

6.2 Aluminum Calibration-Check at High Temperature

An aluminum calibration-check was performed on a specimen of 6061-T6511 aluminum at a temperature of 250°C, prior to performing the experiment on tuff sample 10AE-37Z. The aluminum was jacketed in copper with the same Macor spacer geometry utilized for the tuff specimen. The sample was instrumented and positioned in the furnace and pressure vessel according to the same procedure described in Section 4. The sample assembly was then pressurized

to 5.0 MPa and the temperature increased to 250°C. Once thermal equilibrium was achieved, the specimen was loaded to half its published yield strength at a strain rate of 10^{-6} s^{-1} .

The axial strain as a function of time for the loading cycle is presented in Figure 40. Figure 41 shows the stress difference as a function of axial strain. Young's modulus computed from these data was 62.91 GPa. The published value for 6061-T6511 aluminum at 250°C is 56.96 GPa. Our value is somewhat higher and differs by 10.5%. However, it is not clear that the standard value published by Alcoa Aluminum is a reliable value for compression. Their data were collected in tension and a correction factor must be uniformly applied to convert it to compression. In addition, the modulus begins to change very rapidly at temperatures above 200°C. In light of these uncertainties, it is not readily apparent that our data are significantly in error. We are currently seeking more detailed information from aluminum manufacturers.

Radial strain is plotted as a function of axial strain in Figure 42. A Poisson's ratio of 0.31 was computed from this curve. No values of Poisson's ratio are available from published sources for aluminum at high temperature.

For quality-affecting high-temperature activities, the aluminum calibration-checks at high temperature will be performed at a loading rate not greater than 0.3 MPa/s, but at a confining pressure of 0.5 MPa. Although aluminum is generally insensitive to small changes in confining pressure, this value more appropriately represents the effective pressure conditions under which the tuff specimens were tested.

7.0 References

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8.0 Figures

Figure 1. Schematic diagram of the sample geometry, instrumentation, and loading apparatus used for the experiments on tuff samples at an axial strain rate of 10^{-9} s^{-1} and ambient temperature.

**TUFF
SPECIMEN**

Figure 2. Block diagram of data acquisition, storage, and reduction during an experiment.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE-23W

Figure 3. Axial strain is plotted as a function of time for the experiment on tuff specimen 10AE-23W. The mean axial strain rate computed from these data is $2.6 \times 10^{-9} \text{ s}^{-1}$.

**TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE-23W**

Figure 4. Axial stress is plotted as a function of axial strain for the experiment on tuff specimen 10AE-23W. The test was run at an axial strain rate of 10^{-9} s^{-1} , atmospheric pressure, and ambient temperature.

**TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE-23W**

Figure 5. Radial strain is plotted as a function of axial strain for the experiment on tuff specimen 10AE-23W. The test was run at an axial strain rate of 10^{-9} s^{-1} , atmospheric pressure, and ambient temperature.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE-50X

Figure 6. Axial strain is plotted as a function of time for the experiment on tuff specimen 10AE-50X. The mean axial strain rate computed from these data is $2.5 \times 10^{-9} \text{ s}^{-1}$.

**TOPPAH SPRING MEMBER TUFF
SPECIMEN: 10AE-50X**

Figure 7. Axial stress is plotted as a function of axial strain for the experiment on tuff specimen 10AE-50X. The test was run at an axial strain rate of 10^{-9} s^{-1} , atmospheric pressure, and ambient temperature.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE-50X

Figure 8. Radial strain is plotted as a function of axial strain for the experiment on tuff specimen 10AE-50X. The test was run at an axial strain rate of 10^{-9} s^{-1} , atmospheric pressure, and ambient temperature.

Figure 9. Schematic diagram of the pressure vessel, furnace, sample assembly, and instrumentation used for the confined compression experiments tests at a strain rate of 10^{-8} s^{-1} and a temperature of 250°C , and for the constant stress tests at 250°C .

Figure 10. Temperature is plotted as a function of position along the axis of the sample column. The position of the tuff specimen in a sample column is shown. The zero position is the end of specimen toward the base plug of the pressure vessel (See Figure 9). The measurements were carried out at a confining pressure of 5.0 MPa. The sample assembly was identical to that shown in Figure 9. An axial hole was drilled through the tuff specimen to allow a thermocouple to move freely through the sample column.

TOPOPAH SPRING MEMBER TUFF
BB-10AE-19Z

Figure 11. Axial strain is plotted as a function of time for the experiment on tuff specimen 10AE-19Z. The test was run at an axial strain rate of 10^{-8} s^{-1} , a confining pressure of 5.0 MPa, a pore water pressure of 4.5 MPa, and a temperature of 250°C.

**TOPOPAH SPRING MEMBER TUFF
BB-10AE-19Z**

Figure 12. Stress difference is plotted as a function of axial strain for the experiment on tuff specimen 10AE-19Z. The test was run at an axial strain rate of 10^{-8} s^{-1} , a confining pressure of 5.0 MPa, a pore water pressure of 4.5 MPa, and a temperature of 250°C.

Figure 13. Stress difference and pore pressure are plotted as a function of time for the experiment on tuff specimen 10AE-19Z. The test was run at an axial strain rate of 10^{-9} s^{-1} , a confining pressure of 5.0 MPa, and a temperature of 250°C.

TOPOPAH SPRING MEMBER TUFF
BB-10AE-19Z

Figure 14. Radial strain is plotted as a function of axial strain for the experiment on tuff specimen 10AE-19Z. The test was run at an axial strain rate of 10^{-8} s^{-1} , a confining pressure of 5.0 MPa, and a temperature of 250°C.

TOPOPAH SPRING MEMBER TUFF
10AE-48X

Figure 15. Axial strain is plotted as a function of time for the experiment on tuff specimen 10AE-48X. The test was run at an axial strain rate of 10^{-8} s^{-1} , a confining pressure of 5.0 MPa, a pore water pressure of 4.5 MPa, and a temperature of 250°C.

Figure 16. Stress difference is plotted as a function of axial strain for the experiment on tuff specimen 10AE-48X. The test was run at an axial strain rate of 10^{-8} s^{-1} , a confining pressure of 5.0 MPa, a pore water pressure of 4.5 MPa, and a temperature of 250°C.

TOPOPAH SPRING MEMBER TUFF
10AE-48X

Figure 17. Radial strain is plotted as a function of axial strain for the experiment on tuff specimen 10AE-48X. The test was run at an axial strain rate of 10^{-8} s^{-1} , a confining pressure of 5.0 MPa, a pore water pressure of 4.5 MPa, and a temperature of 250°C.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37Z
CREEP @ 25 C

Figure 18. Stress difference is plotted as a function of axial strain for the loading segment of the ambient-temperature creep experiment on tuff specimen 10AE-37Z.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37Z
CREEP @ 25 C

Figure 19. Radial strain is plotted as a function of axial strain for the loading segment of the ambient-temperature creep experiment on tuff specimen 10AE-37Z.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37Z
CREEP @ 25 C

Figure 20. Axial strain is plotted as a function of time for the creep experiment on tuff specimen 10AE-37Z. The test was run at a differential stress of 50 MPa, a confining pressure of 5.0 MPa, a pore pressure of 4.5 MPa, and ambient temperature.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37ZB
CREEP @ 250 C

Figure 21. Stress difference is plotted as a function of axial strain for the loading segment of the first high-temperature creep experiment on tuff specimen 10AE-37Z.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37ZB
CREEP @ 250 C

Figure 22. Radial strain is plotted as a function of axial strain for the loading segment of the first high-temperature creep experiment on tuff specimen 10AE-37Z.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37ZB
CREEP @ 250 C

Figure 23. Axial strain is plotted as a function of time for the creep experiment on tuff specimen 10AE-37Z. The test was run at a differential stress of 50 MPa, a confining pressure of 5.0 MPa, a pore pressure of 4.5 MPa, and temperature of 250°C.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37ZB
CREEP @ 250 C

Figure 24. Radial strain is plotted as a function of time for the creep experiment on tuff specimen 10AE-37Z. The test was run at a differential stress of 50 MPa, a confining pressure of 5.0 MPa, a pore pressure of 4.5 MPa, and temperature of 250°C.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37ZB
CREEP @ 250 C

Figure 25. The temperature of a tuff specimen is plotted as a function of time for the creep experiment on tuff specimen 10AE-37Z. The test was run at a differential stress of 50 MPa, a confining pressure of 5.0 MPa, and a pore pressure of 4.5 MPa.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37Z
CREEP @ 250 C

Figure 26. Stress difference is plotted as a function of axial strain for the loading segment of the second high-temperature creep experiment on tuff specimen 10AE-37Z.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37ZG
CREEP @ 250 C

Figure 27. Radial strain is plotted as a function of axial strain for the loading segment of the second high-temperature creep experiment on tuff specimen 10AE-37Z.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37ZG
CREEP @ 250 C

Figure 28. Axial strain is plotted as a function of time for the creep experiment on tuff specimen 10AE-37Z. The test was run at a differential stress of 70 MPa, a confining pressure of 5.0 MPa, a pore pressure of 4.5 MPa, and temperature of 250°C.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37ZG
CREEP @ 250 C

Figure 29. Radial strain is plotted as a function of time for the creep experiment on tuff specimen 10AE-37Z. The test was run at a differential stress of 70 MPa, a confining pressure of 5.0 MPa, a pore pressure of 4.5 MPa, and temperature of 250°C.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37ZG
CREEP @ 250 C

Figure 30. Stress difference is plotted as a function of time for the creep experiment on tuff specimen 10AE-37Z. The test was run at a confining pressure of 5.0 MPa, a pore pressure of 4.5 MPa, and temperature of 250°C.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37ZG
CREEP @ 250 C

Figure 31. Confining pressure is plotted as a function of time for the creep experiment on tuff specimen 10AE-37Z. The test was run at a differential stress of 70 MPa, a pore pressure of 4.5 MPa, and temperature of 250°C.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37ZG
CREEP @ 250 C

Figure 32. Pore pressure is plotted as a function of time for a the creep experiment on tuff specimen 10AE-37Z. The test was run at a differential stress of 70 MPa, a confining pressure of 5.0 MPa, and temperature of 250°C.

TOPOPAH SPRING MEMBER TUFF
SPECIMEN: 10AE 37ZG
CREEP @ 250 C

Figure 33. Temperature of the tuff specimen is plotted as a function of time for the creep experiment on tuff specimen 10AE-37Z. The test was run at a differential stress of 70 MPa, a confining pressure of 5.0 MPa, and a pore pressure of 4.5 MPa.

ALUMINUM CALIBRATION
AL 9
SPECIMEN: 2024-T351

Figure 34. Axial strain is plotted as a function of time for the aluminum calibration-check prior to loading tuff specimen 10AE-23W. The mean axial strain rate was $1.4 \times 10^{-5} \text{ s}^{-1}$.

ALUMINUM CALIBRATION
AL9
SPECIMEN: 2024-T351

Figure 35. Axial stress is plotted as a function of axial strain for the aluminum calibration-check prior to loading tuff specimen 10AE-23W.

ALUMINUM CALIBRATION
AL9
SPECIMEN: 2024-T351

Figure 36. Radial strain is plotted as a function of axial strain for the aluminum calibration-check prior to loading tuff specimen 10AE-23W.

ALUMINUM CALIBRATION
AL 16
SPECIMEN: 2024-T351

Figure 37. Axial strain is plotted as a function of time for the aluminum calibration-check subsequent to loading tuff specimen 10AE-23W. The mean axial strain rate was $1.6 \times 10^{-5} \text{ s}^{-1}$.

ALUMINUM CALIBRATION
AL16
SPECIMEN: 2024-T351

Figure 38. Axial stress is plotted as a function of axial strain for the aluminum calibration-check subsequent to loading tuff specimen 10AE-23W.

ALUMINUM CALIBRATION
AL16
SPECIMEN: 2024-T351

Figure 39. Radial strain is plotted as a function of axial strain for the aluminum calibration-check subsequent to loading tuff specimen 10AE-23W.

**ALUMINUM CALIBRATION
SPECIMEN: 6061-NER-88**

Figure 40. Axial strain is plotted as a function of time for the calibration-check on 6061-T6511 aluminum. The test was conducted at a confining pressure of 5.0 MPa and a temperature of 250°C. The mean axial strain rate was $1.3 \times 10^{-6} \text{ s}^{-1}$.

**ALUMINUM CALIBRATION
SPECIMEN: 6061-NER-88**

Figure 41. Stress difference is plotted as a function of axial strain for the calibration-check on 6061-T6511 aluminum. The test was conducted at a temperature of 250°C and a confining pressure of 5.0 MPa.

**ALUMINUM CALIBRATION
SPECIMEN: 6061-NER-88**

Figure 42. Radial strain is plotted as a function of axial strain for the calibration-check on 6061-T6511 aluminum. The test was conducted at a temperature of 250°C and a confining pressure of 5.0 MPa.

Appendix A

This report contains no information from the Reference Information Base (RIB).

This report contains no candidate information for the RIB or the Site & Engineering Properties Data Base (SEPDB).

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