FINAL REPORT
CAPABILITIES TO MEASURE GEOTHERMAL MATERIAL PROPERTIES
AT SIMULATED *IN SITU* CONDITIONS

by

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ABSTRACT

The lack of reservoir material property data has been identified as a major obstacle to geothermal development and commercialization. To meet this need a high pressure/high temperature test facility was designed and constructed at Terra Tek under Department of Energy funding. Testing capabilities include confining and pore fluid pressures to 200 MPa and temperatures to 535°C. Rock samples to 10.2 cm diameter can be accommodated.

The program summarized herein is the second year (October 1977-September 1978) effort (DOE Contract DE-AC07-77ET28301). Test techniques were developed to meet the geothermal community needs and tests were conducted to determine: mechanical properties, thermal conductivity, thermal diffusivity, thermal expansion, permeability, electrical resistivity and ultrasonic velocities. Sandstones, graywackes, granites, and basalts, from KGRA's in Baca, New Mexico; Milford, Utah; Geysers, California and Northern Nevada, respectively were characterized.

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INTRODUCTION

In recent years it has become evident that geothermal energy has the potential of becoming an important energy resource. Both high temperature ($>150^{\circ}\text{C}$) and moderate temperature ($<150^{\circ}\text{C}$) resources could conceivably be exploited to support a significant portion of the energy demand now shouldered by diminishing fossil fuels. However, certain technological limitations exist, many of which are geoscience related (rock mechanics, geochemistry, etc.), that have inhibited the rapid, economic and efficient development of this potential resource. The work described herein is the second year (October 1977-September 1978) of a DOE/DGE funded program to provide rock mechanics support to geothermal development. This second year included primarily materials testing and continued test technique development. As of this writing, several specific programs in stimulation, subsidence, reservoir engineering and formation damage are utilizing the geothermal rock mechanics facility.

The first year of this DOE/DGE program is described in Terra Tek report TR 78-11¹ and provides a detailed description of the test facility. Briefly the testing capabilities include confining and pore fluid pressures to 200 MPa and temperatures to 535°C . Sample sizes to 10.2 cm can be accommodated.

This report contains two sections: the first is a general facilities description and the second is a detailed description of each of the individual test techniques, summarized in the following table.

TABLE 1
Geothermal Core Testing Capabilities

Test Parameter	Maximum Confining Pressure MPa (ksi)	Maximum Pore Pressure MPa (ksi)	Maximum Temperature °C	Pore Fluid	Overall Accuracy	Application**
Mechanical Response	200 (30)	200 (30)	535	Gas Water Brine	±1%	Drilling Stimulation Subsidence
Thermal Conductivity	200 (30)	200 (30)	535	Gas Water Brine	±5%	Surface Exploration Subsurface Evaluation Reservoir Engineering
Thermal Diffusivity	200 (30)	200 (30)	535	Gas Water Brine	±5%	Surface Exploration Subsurface Evaluation Reservoir Engineering
Thermal Expansion	200 (30)	---*	535	Gas Water Brine	±5%	Reservoir Engineering Subsidence
Permeability	200 (30)	69 (10)	535	Gas Water Brine Acid Caustic	±10%	Reservoir Engineering Stimulation
Electrical Resistivity	200 (30)	200 (30)	300	Gas Water Brine	±2.5%	Surface Exploration Subsurface Evaluation
Ultrasonic Velocity	200 (30)	200 (30)	300	Gas Water Brine	±1%	Surface Exploration Subsurface Evaluation

* Not available at present.

** References 2-7

Contained within the appropriate individual description sections are data on sandstones, granites, graywackes, and basalts. The tests conducted include materials from KGRA's at Baca, New Mexico; Milford, Utah; Geysers, California and Northern Nevada. The data have been generated in support of the DOE/DGE supported programs in surface exploration, subsurface evaluation (logging), drilling, stimulation, reservoir engineering and subsidence, and in support of resource development activities by several industrial groups.

FACILITY DESCRIPTION

Capabilities

The geothermal material property test facility is shown in Figure 1. Data acquisition and associated control equipment are shown in the foreground separated from the load frame and pressure vessel (background) by a clear protective barrier. A schematic detailing major components of the load frame is contained in Figure 2. Capabilities of the high pressure-high temperature test facility are summarized:

- Confining pressure to 200 MPa (30,000 psi)
- Temperature to 535°C (1000°F)
- Axial load to $4.5 \times 10^6 \text{ N}$ (10^6 lbs)
- Sample size: 5.1 cm (2") diameter (to 535°C)
10.2 cm (4") diameter (to 200°C)

While maintaining these previously described environments the following geothermal material properties can be determined:

- Mechanical properties: complete stress-strain response including longitudinal and lateral strains (volume response) and pore fluid pressure.
- Thermal properties: thermal conductivity, thermal diffusivity, thermal expansion coefficient
- Matrix and fracture permeability/conductivity to gases and liquids
- Electrical resistivity
- Ultrasonic velocities

Figure 1. Geothermal material test system.

Figure 2. Geothermal material test system schematic.

Figure 3 contains a photograph of complementary equipment at Terra Tek including an identical vessel in a configuration designed specifically for long term consolidation (creep) tests, and a high temperature, high flow circulation system. This flow system is capable of pressures to 69 MPa (10,000 psi), temperatures to 312°C (600°F), and flow rates to 37 liters per minute (10 gal/min). As well as circulating fresh waters, it can transport mixed brines, light acids and light caustics.

Figure 3. Complementary test equipment.

Pressure Vessel

The pressure vessel, in which the material property testing is conducted, is a 15.25 cm (6 inch) inside diameter hardened steel cylinder capable of internal pressures to 200 MPa (30,000 psi). A mild steel

cooling jacket/safety liner surrounds the vessel. Cooling water circulating in the jacket maintains a constant vessel wall temperature during extended duration testing. Elevated sample temperatures are achieved by internally mounted heaters (up to 9 kilowatts) which promote fast test times due to the avoidance of long heat up and cool down times associated with externally heated vessels.

Dowtherm A (liquid) was selected as a confined medium after testing determined that it remained electrically non-conductive at 200 MPa (30,000 psi) and 535°C (1000°F). Degradation begins in the fluid at approximately 400°C (750°F) but this does not prove to be a serious limitation since very little of the total fluid is exposed to the high temperature. Dowtherm A will not vaporize at atmospheric pressure until approximately 290°C (550°F). A pressure of 7 MPa (1000 psi) is sufficient to maintain the fluid phase to 535°C (1000°F)

All load, strain, temperature, and pore pressure measurements are made inside the pressure vessel. Pressure effect on each of these transducers is carefully documented. Samples ranging from 2.5 cm (1 inch) diameter x 5.1 cm (2 inch) length to 10.2 cm (4 inch) diameter x 20.3 cm (8 inch) length can be accommodated.

Load Frame and Control

The load frame is capable of applying an axial load of 4.5×10^6 newtons (1 million pounds) via the servo-controlled upper actuator. Column and rod design are used in the load frame. Prestressed rods and columns maintain a rigid frame. Pressure vessel to lower platen clearance allows for assembly of the test stack with heaters and multiple ceramic

shrouding prior to the insertion of the lower closure into the vessel. A reaction column is used to support the lower closure. This type of design allows for quick stack and vessel assembly thereby reducing test times and costs. The capacity is sufficient to provide loading to failure of all except the strongest rocks.

Load feedback and displacement feedback are available to provide constant load or actuator position operational modes. An electronic ramp generator is used to establish a constant displacement rate of the upper actuator and therefore allows accurate control of strain rate during triaxial compression testing.

TEST TECHNIQUES AND TEST DATA

Sample Preparation

When applying a confining stress to a rock sample it is necessary to isolate the internal pore fluid from the confining fluid. In low temperature rock mechanics tests, thin urethane jackets are used to seal the sample. At temperatures above 175°C (350°F) urethane degrades. For temperatures to 300°C (~600°F) a pre-molded silicone rubber (RTV) jacket is used. End sealing is accomplished by wiring the jacket to stainless steel endcaps. Testing at temperatures above 300°C (~600°F) requires the use of 0.025 cm (0.010 inch) thick annealed copper jackets. Sealing is accomplished by tapered endcaps; press-fit sealing rings allow quick assembly and reuse of the endcaps. Jacketing of this type has been used frequently in high temperature research and has been shown to have negligible influence on the measured strength of competent rock (granite, basalt)⁸. Jacketing effects of weaker, less competent materials (sandstone, shale) at large strains require documentation on a case by case basis.

For tests requiring no pore fluid between the sample and the inside of the jacket (electrical resistivity and permeability), RTV is applied directly to the sample surface. This provides a good adhesion plus an ideal jacket fit. The extent of silicone rubber infiltration is not significant.

The complete range of sample sizes, 2.54 cm (1.0 inch) diameter to 10.2 cm (4.0 inch) diameter can be accommodated by both sample preparation techniques.

Mechanical Response

The quasi-static stress-strain response of a material under simulated geothermal conditions is determined in the testing configuration of Figure 4. Both lateral and axial strains can be measured allowing the evaluation of failure characteristics, elastic properties and volumetric response characteristics. Pore pressure, confining pressure, and axial load are controlled by separate closed loop servocontrol systems allowing simulation of various pressure-load application "paths".

The test sample, prepared as discussed in a previous section, is located in the upper portion of the test vessel. Ceramic insulation is used on each end of the sample to minimize the longitudinal temperature

Figure 4. Mechanical response test configuration.

gradients. Heating elements are coiled about the lower aluminum insulator and the lower portion of the sample. A cylindrical ceramic shroud is placed over both the sample and the coiled heater to localize the fluid convection currents. Convection within the vessel is further minimized by a second all-encompassing ceramic shroud. These ceramic convection baffles enable temperatures of 535°C (1000°F) to be reached within the inner shroud while maintaining the fluid surrounding the exterior shroud to remain at less than ~100°C (200°F), well below the maximum allowable for the internal instrumentation and pressure seal.

Supporting this sample stack is a strain-gaged load cell selected to give optimum output for the anticipated load levels. Measurement of axial load in this manner eliminates the possibility of seal friction errors. Axial strain is measured by two LVDT's (linearly variable differential transformers) contained in the lower vessel closure. These transducers are attached to the top of the test sample by quartz rods. Quartz was selected because of its very low thermal expansion. Lateral strain measurement is made by a cantilever arrangement using long, stiff cantilever arms with strain-gaged sections near the bottom of the pressure vessel outside of the high temperature environment.

Axial strain measurements with appropriate calibration for temperature and pressure effects can be made over a range of ± 1.27 cm (0.5 inches) and are accurate to 0.0013 cm (0.0005 inches). Lateral strains of ± 0.64 cm (0.75 inches) can be measured and are accurate to 0.003 cm (0.001 inches). Confining pressure, pore pressure and axial load are accurate to within ± 1 percent of the measured value.

During debugging of the mechanical response system, various unconfined and triaxial compression tests were conducted. Figures 5, 6, 7 and 8 contain typical mechanical response data. Figure 5 shows the stress-strain response of a granite material undergoing triaxial loading to failure. This sample was tested dry, without any pore fluid, at a temperature of 200°C (~400°F) and a confining pressure (σ_3) of 13.8 MPa (2000 psi). Both axial and lateral strains are shown versus axial stress difference ($\sigma_1 - \sigma_3$). Figure 6 shows the volume response for the same test. Note the difference in behavior of the saturated and dry samples.

Figures 7 and 8 contain a failure envelope and failure stress versus temperature behavior respectively, of Westerly granite.

Figure 5. Mechanical response of a dry granite material at temperature.

Figure 6. Volumetric response of a granite material at temperature.

Figure 7. Failure envelope for Westerly granite.

Figure 8. Failure stress as a function of temperature for Westerly granite confined at 34.5 MPa (5000 psi).

Thermal Conductivity

Thermal conductivity can be measured in the simulated temperature-pressure environment by the transient line heat source ("needle-probe") method as used by Woodside and Messmer⁹. A detailed discussion of the theory applicable to this testing is contained in Appendix A.

Basic sample heating and insulation are accomplished in the same manner as previously discussed in the mechanical response testing. A schematic of the thermal conductivity test set-up is contained in Figure 9. A 0.32 cm (0.125 inch) diameter heater approximating the behavior of an ideal line heat source is placed along the longitudinal axis of

Figure 9. Thermal response test configuration.

a 5.1 cm (2.0 inch) diameter by 10.2 cm (4.0 inch) long test sample. A thermocouple is attached to the heater at the mid-sample location. The entire probe assembly is then "potted" in place with a mixture of ceramic cement and powdered copper to minimize the contact resistance between the probe and the sample. Ceramic insulators are used to minimize heat loss from the ends of the test sample. The overall internal heater length, 13.4 cm (5.25 inches), gives a heater length to heater diameter ratio of 42. Blackwell¹⁰ has shown that a L/D ratio of 30 is minimum for an acceptable approximation of an infinite line source in this type of test.

Power, in the form of carefully monitored voltage and current, is applied to the probe heater (for periods less than one minute) while the test sample exterior is maintained at the desired temperature and pressure. Sample conductivity determines the probe heater temperature rise. The exterior temperature of the sample does not change; a sample of finite dimension can behave (as the theory requires) as though it were infinite in size. Thermal conductivity for the material is calculated from the internal heater temperature history and the power input. Due to the absence of thermal conductivity standards in this range, accuracy definition is uncertain. It is estimated that thermal conductivity values are accurate to within ± 5 percent.

Thermal conductivity testing was performed following calibration with fused quartz ($K @ 20^\circ\text{C} = 1.36 \text{ W/m}\cdot\text{K}$) at temperature and pressure. Major emphasis during debugging was placed on developing a standardized probe cementing procedure. Once this was established, consistent results were achieved.

An example of thermal conductivity testing performed on Westerly granite is contained in Figure 10. Comparison is made with published data contained in Clark's Handbook of Physical Constants¹¹. The only significant deviation from the published values occur at 200°C . Also contained in this figure are thermal conductivity values for fused quartz. Confining pressure results in increased thermal conductivity as can also be seen from Figure 11. Even for a very low porosity rock the variation of thermal conductivity with pressure is significant.

Figure 10. Thermal conductivity variation with temperature.

Figure 11. Thermal conductivity variation with confining pressure.

Thermal Diffusivity

Thermal diffusivity is the ratio of the system's ability to conduct heat to the system's ability to store heat. Thermal diffusivity (κ), thermal conductivity (K), density (ρ) and specific heat (c_p) are related by the following:

$$\kappa = \frac{K}{\rho c_p}$$

Thermal diffusivity measurement of core material under simulated *in situ* conditions is important: 1) for geothermal reservoir modeling and 2) as an alternate method of determining thermal conductivity (assuming the density and specific heat are adequately known).

Density can be measured and corrections for overburden pressures and temperature can be obtained from hydrostatic compression tests and thermal expansion tests, respectively. Because specific heat is a mass related phenomenon it can be accurately estimated for rock from mineralogical composition using Kopps Law (Figure 12) which averages the mass weighted specific heats of the individual mineralogical components. Somerton¹² has shown the changes in heat capacity with temperature for rock are similar for rocks of different mineralogical composition (Figure 13). Corrections must be included to account for the influence of pore fluid.

Several methods have been developed to experimentally determine thermal diffusivity of thermal systems¹³. Of these various approaches, the one best suited for determining thermal diffusivity in geologic materials under *in situ* conditions is based upon a classical heat transfer

Figure 12. Comparison of calculated and measured heat contents.

Figure 13. Experimental heat capacities of different rock materials.

model presented by Carslaw and Jaeger¹⁴. This model requires that a sinusoidal temperature oscillation be induced at the surface of a finite cylindrical sample while the internal temperature is monitored. Under such conditions the center temperature will lag the surface temperature by some time, t_1 . This measured time lag, period of oscillation, and sample radius are then used to analytically determine diffusivity. A detailed presentation of the theory is contained in Appendix B.

The testing configuration is very similar to that of the thermal conductivity test set-up (see Figure 9). A thermocouple is plotted along the center axis of the sample cylinder using a potting mixture of ceramic cement and powdered copper. A second thermocouple is placed upon the outer surface of the sample and cemented to the rock surface. The sample is jacketed with material suitable for the anticipated temperature and pressure.

The sample is convectively heated with the temperature oscillation controlled by a programmable temperature controller. The insulating shroud minimizes the quantity of heated fluid and confines it to the region near the sample surface. Because of the simplicity of the measuring devices and the accuracy with which the radius of the sample, period of oscillation and time lag can be determined, diffusivity can be determined to within 5 percent.

Thermal diffusivity changes significantly with temperature, see Figure 14. This is to be expected due to the temperature dependence of both heat capacity and thermal conductivity (and to a much lesser extent, density). Figure 15 shows the comparison of thermal conductivity

Figure 14. Thermal diffusivity response to temperature of Westerly granite.

Figure 15. Calculated versus experimental thermal conductivities.

measured directly (needle-probe method) and values calculated from thermal diffusivity measurements with the appropriate density and heat capacity. Estimates are based upon published mineralogical compositions and density values.

Thermal Expansion

Thermal expansion characteristics of geologic materials have not been studied extensively at *in situ* conditions. Studies performed at elevated temperatures have shown the expansion coefficient to have considerable temperature dependence and significant nonrecoverable thermal strain, generally attributed to microcracking. During heating and cooling, fragmentation may occur within a rock sample as a result of anisotropy and thermoelastic property differences between adjacent grains. The magnitude of internal stresses will depend on the distribution and orientation of individual grains. The thermal fragmentation process depends, among other factors, on thermal gradients during heating and the reaction of rock material to induced thermal stresses¹⁵. Heating rates less than 2°C/min. have been found to cause significantly less fragmentation in small rock samples than higher heating rates¹⁶.

Thermal expansion of geologic materials can be evaluated at *in situ* conditions in the configuration shown in Figure 16. Although the basic components, such as the ceramic shrouding and heater, are the same as previously presented, several unique aspects of this set-up are worth noting. All non-sample stack components exposed to the high temperature environment are fused quartz, with a thermal expansion

Figure 16. Thermal expansion test configuration.

coefficient of $0.5 \times 10^{-6} \text{ cm/cm}^\circ\text{C}$. Fused quartz endcaps and a fused quartz stack spacer support the sample. Quartz rods attached to the top of the test sample suspend the core elements of the LVDT transformers at the base. The expansion of the rods and stack components tend to offset one another except for the expansion of the sample. System expansion consists of the expansion of the fused quartz rods over a distance equal to the sample length. Compared to the anticipated range of rock expansion ($\sim 8\text{-}10 \times 10^{-6} \text{ cm/cm}^\circ\text{C}$), the expansion of the quartz is small.

High resolution LVDT's located near the base of the stack measure the axial strain induced by sample temperature change. Thermal strains of up to $\pm 2.5 \times 10^{-2}$ cm (0.010 inch) can be measured with an accuracy of $\pm 6.2 \times 10^{-5}$ cm (2.5×10^{-5} inch). Averaging the outputs of the two LVDT's removes any false strain due to tilting. Calibration was accomplished with three materials; fused quartz, steel, and aluminum. Using accepted expansion coefficients for these materials, the system expansion was determined as that amount of expansion in excess of published values for the calibration samples. The system expansion is typically 0.5×10^{-6} cm/cm°C, and is repeatable regardless of the sample material.

Sample temperature is monitored by a thermocouple placed at mid-point on the sample, continually in contact with the sample jacket. Sample temperature is increased at a predetermined rate depending on the test material. It is desirable that the sample temperature increase occur uniformly. Rates can be controlled from zero to 5°C/min., with rates of 0.5°C/min. generally being the most desirable. System accuracy can be strongly affected by the heating rate. Heating too rapidly produces increased hysteresis. With heating rates of less than 1°C/min., hysteresis is minimized and overall accuracy of ± 5 percent is achieved.

Sample dimensions are; 2.54 cm (1.00 inch) diameter, 5.1 cm (2.00 inches) length, with the ends ground flat and parallel to within 2.5×10^{-3} cm (0.001 inch). Before jacketing, the sample is bonded to

the endcaps, with a minimum sized peripheral ring of adhesive along the interface line. A prestretched silicone jacket is sealed to the fused quartz endcaps by lock wire. Silicone jackets are preferred to copper in this type of testing because of the low Young's modulus of the silicone.

A typical test procedure includes stabilization at room temperature, followed by a programmed heating and cooling cycle. Stabilization at particular temperatures for up to 10 minutes assure uniform temperature distribution and increase the accuracy of strain measurements over a certain temperature range. However, best results over a broad temperature range are achieved by imposing a quasi-static temperature increase upon the sample and surrounding system. Results of such a test with a steel sample is shown in Figure 17. When scaled from the graph, the coefficient of expansion for the steel sample is $12.2 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$; this includes system expansion. This compares well with published values for the thermal expansion coefficient of steel, which is approximately $11.6 \times 10^{-6} \text{ }^{\circ}\text{C}^{-1}$. An example of thermal strain versus temperature for a geologic material, welded ash fall tuff, is shown in Figure 18. Note the non-linear thermal strain.

Permeabilities

Permeability measurements at temperature, confining pressure and pore fluid pressure are accomplished in a test set-up similar to those previously described. Although both gas and liquid can be used as flow media, this discussion will focus on the liquid (principally water and brine) flow medium. Figure 19 shows a typical test configuration.

Figure 17. Thermal expansion of steel calibration sample.

Figure 18. Thermal expansion of a welded ash fall tuff.

Figure 19. Permeability test sample schematic.

The test sample has two stainless steel diffusion discs placed at each end. These highly permeable, isotropic discs distribute the pore fluid over the sample end faces. Behind each disc is a ceramic insulator and stainless steel endcap. A coating of two component catalyzed silicone rubber (RTV) is applied to the test sample, adhering to the circumference of the sample and filling any irregularities. Flow is therefore prevented from occurring down the sides of the sample. Sealing is further enhanced by the application of confining pressure. An additional silicone rubber tube is placed over the sample and sealed by lock wire to the endcaps.

Flow lines are attached to the base discs. Inlet flow is introduced at the top of the test sample to allow preheating of the permeating fluid prior to entry into the top endcap. Confining pressure and pore fluid pressure are established and the sample is allowed to stabilize. Stabilization times vary based on porosity and estimated permeability. For the tests, flow across the sample can be controlled by two different means, constant flow rate or constant head pressure.

In the constant flow rate system, Figure 20, a constant displacement pump is used to establish flow. Differential pressure is measured between the upstream and downstream faces of the sample. The flow is several hundred times the total sample pore volume in order to achieve steady-state.

The other steady-state method currently being used is the constant head pressure technique. Schematically the system is shown in Figure 21. Gas pressure, held constant by precision regulators, is used to pressurize one side of a separation accumulator. This in turn pressurizes the fluid at the upstream end of the sample. After flowing through the test sample, the permeating fluid is collected in a downstream separation accumulator maintained at a lower constant pressure. Piston location in each accumulator is monitored electronically and is used to determine flow rate. Differential pressure across the sample is monitored by a transducer and recorded continually. Pressure and flow rate accuracy is better than ± 1 percent. Provisions have been made to allow sampling or precision weighing of the circulated fluid.

Figure 20. Constant flow permeability schematic.

* DISPLACEMENT OF ACCUMULATOR PISTON IS MEASURED ELECTRONICALLY

Figure 21. Constant head pressure permeability system schematic.

Permeability is calculated according to the Darcy law:

$$K = \frac{v_t}{v_r} \frac{Q_r \mu L}{A \Delta P}$$

where:

K = permeability, darcies

v_t, v_r = specific volume of fluid at temperature and at reference point, respectively, m^3/kg

Q_r = flow rate at reference point, ml/sec (measured at $T = 23^\circ\text{C}$, constant)

μ = viscosity at flow temperature, c_p (temperature dependent)

L = sample length, cm

A = cross-sectional flow area, cm^2

ΔP = pressure differential, bars (absolute)

The specific volume ratio at the front of this equation constitutes a volumetric correction for fluid volume expansion at the flow temperature since flow rate measurements are made at a reference point with constant 23°C temperature. Pressure differential is maintained constant during the course of the test. All other parameters on the right side of this equation can be physically measured from the sample or determined from published data on the properties of water at temperature and pressure appropriately corrected for salinity¹⁷.

The above techniques allow fluid permeability (brines, water, oils, or light acids) to be determined under simulated *in situ* conditions for rocks ranging in permeability from $50 \mu\text{darcies}$ to 1 darcy.

Special systems allowing permeating fluid preheat are available for high flow-high temperature circulations. Transient techniques can be employed for materials below this permeability range.

Permeability responses for two sandstones are shown in Figures 22 and 23. Test temperatures were limited to 200°C because pore fluid pressure was less than 2.07 MPa (300 psi) and flashing of the water to steam was not desired. In both cases, permeability declined significantly with temperature. This effect is discussed in detail by Casse and Ramey¹⁸ and Danesh, *et. al.*¹⁹ and is generally attributed to water-pore surface interactions.

Figure 22. Castlegate sandstone permeability to water.

Figure 23. Mixed Company sandstone permeability to water.

Electrical Resistivity

Parameters relevant to the resistivity of geothermal rock are the temperature, pressure, pore fluid pressure, initial saturation, pore fluid chemistry and frequency. This particular area of study is somewhat complex and only a brief discussion will be given here. An expanded presentation is contained in Appendix C.

A detailed schematic of the test sample is shown in Figure 24. A four pole electrode arrangement is used; two current, two potential. A porous, 0.03 cm (0.010 inch) thick diameter ceramic disc is placed at each sample face, followed by a platinum mesh potential electrode. A porous ceramic spacer connects the titanium endcaps to the

Figure 24. Electrical resistivity sample configuration and equivalent circuit.

sample. The entire assembly is then coated with high temperature silicone rubber for isolation from the confining medium. Samples of porous material are saturated for approximately 24 hours under vacuum with jacket material in place to prevent leakage paths due to jacketing wrinkles. Since the response of the test sample is electrically complex, impedance and phase angle measurements are made to determine the electrical response. Measurements are made using a Hewlett-Packard model 4274A Multi-Frequency LCR meter at approximately 2 volt alternating current (to prevent electrode polarization) and at frequencies ranging from 100 Hz to 10 KHz. The potential electrodes are isolated by

approximately 10^{12} ohms impedance improving the instrument's accuracy at high impedance values ($10\text{ K}\Omega$ and up). A schematic for the instrument and sample arrangement is shown in Figure 25.

Data obtained during the debugging of this test set-up is contained in Figure 26. As expected, the resistivity of the sandstone material declines with temperature, an effect that is primarily due to the increasing charge mobility caused by decreasing pore fluid viscosity. Pressure and temperature effects on the pore fluid also contribute to this effect*. In a material of this type, electrolyte effects tend to dominate, especially with heavy concentrations of ions.

Present capabilities allow measurement of electrical resistivity to 300°C and 207 MPa ($30,000\text{ psi}$) confining pressure. Accuracy of resistivity measurements are ± 2.5 percent.

Figure 25. Electrical resistivity circuit configuration.

* See appendix for more complete discussion.

Figure 26. Electrical resistivity of partially and fully saturated Colton sandstone at temperature.

Ultrasonic Velocity Measurements

The longitudinal (p-wave) and shear (s-wave) velocities can be measured at elevated temperature, confining pressure and pore fluid pressure. These data, together with the bulk density of the material, are used to calculate dynamic elastic moduli.

The velocities are obtained using a "through transmission system" adapted from a technique introduced by Mattaboni²⁰. The equipment configuration is shown in Figure 27. This method of measuring velocities in solids has been employed for some time at Terra Tek^{21,22}, and is not part of the development in this program.

Figure 28 shows the sample configuration that is presently being used. A ferroelectric ceramic material (lead titanate zirconate, PZT5)

THROUGH TRANSMISSION SYSTEM

Figure 27. Ultrasonic measurement system schematic.

Figure 28. Ultrasonic velocity sample configuration.

was selected for use as the transducer elements. The desirable features of this material is its high Curie point (360°C) and piezoelectric coupling coefficient^{23*}. A single transducer, cut to produce a shear wave is used as the emitter. It has been found from experience that this transducer produces sufficient p-wave energy to allow both shear and longitudinal velocity measurements if separate receivers are used to detect the respective signals. The electrical leads are ceramic insulated and all connections are either brazed or mechanically made with a positive contact force.

The temperature effect on the endcaps and ceramic spacers are determined by conducting a test without a sample. To date, successful measurements have been made to 250°C. No problems are foreseen in extending to 300°C in the near future.

Figure 29 shows the velocity data obtained on an aluminum reference sample and Westerly granite. The least squares fit of the aluminum data is well within 1 percent agreement with published data²⁴ as indicated in Table 2. The velocity data for aluminum and Westerly granite were obtained with the hydrostatic confining pressure held constant at 13.8 MPa (2000 psi) for all measurements. The slope of the least squares fit of the granite data is -4.65×10^{-3} per °C for the p-wave and -2.46×10^{-3} per °C for the s-wave. The RMS error is 3.75×10^{-2} (km/sec) for the p-wave and 1.88×10^{-2} (km/sec) for the s-wave. The measured velocities, calculated Young's moduli and Poisson's ratios are tabulated in Table 3.

* Approximately a factor of seven (7) larger than that of standard quartz transducers.

Figure 29. Ultrasonic velocity response of Westerly granite and aluminum.

TABLE 2

Comparison of Terra Tek and Published Data on 6061-T6 Aluminum

	ASAY ⁽¹⁷⁾	TTI
Density (g/cc)	2.704	2.702
Longitudinal velocity (km/sec)	$6.428 - 9.1 \times 10^{-4}T^*$	$6.459 - 9.4 \times 10^{-4}T^*$
Shear velocity (km/sec)	$3.188 - 8.6 \times 10^{-4}T$	$3.217 - 1.16 \times 10^{-3}T$
Poisson's ratio	$0.336 + 1.1 \times 10^{-4}T$	$0.337 + 0.82 \times 10^{-4}T$
Young's modulus	$7.35 \times 10^4 - 41.6T$	$7.38 \times 10^4 - 43.49T$

*T = Temperature in °C

TABLE 3

Ultrasonic Velocities and Elastic Moduli as a Function of Temperature with the Confining Pressure at 13.8 MPa

Material	Density (gm/cc)	Sample Length (mm)	Temp. (°C)	Velocities		Young's Modulus (GPa)	Poisson's Ratio
				P-Wave (km/sec)	S-Wave (km/sec)		
A1 (6061-T6)	2.702	50.80	76	6.384	3.115	70.46	0.3438
A1 (6061-T6)	2.702	50.80	99	6.358	3.094	69.57	0.3449
A1 (6061-T6)	2.702	50.80	152	6.313	3.035	67.18	0.3497
A1 (6061-T6)	2.702	50.80	173	6.295	3.021	66.00	0.3504
A1 (6061-T6)	2.702	50.80	202	6.278	2.983	65.12	0.3542
A1 (6061-T6)	2.702	50.80	225	6.252	2.954	63.96	0.3563
A1 (6061-T6)	2.702	50.80	250	6.216	2.930	62.96	0.3572
Granite	2.631	48.97	75	5.624	3.297	70.82	0.2382
Granite	2.631	48.97	101	5.478	3.245	68.14	0.2297
Granite	2.631	48.97	151	5.184	3.154	63.14	0.2061
Granite	2.631	48.97	200	4.978	3.020	58.01	0.2088
Granite	2.631	48.97	250	4.822	2.860	52.88	0.2287

SUMMARY

The following geothermal material properties can be evaluated at simulated *in situ* conditions:

- Mechanical response
- Thermal conductivity
- Thermal diffusivity
- Thermal expansion
- Permeability
- Electrical resistivity
- Ultrasonic velocity

Major developments are now complete in each of these areas allowing basic testing capability in each. Sample configurations and testing procedures have been defined and verified through both "debugging" and actual testing experience.

Following is a general discussion of the data obtained in each testing area with references where appropriate to similar work in the literature. The data contained in the text of this report is representative of the data generated during testing development. Proprietary data is not included.

Mechanical Response

Data obtained on various materials have shown ultimate failure stress and failure mode (ductile or brittle) to be strongly pressure dependent. The nature of the pore fluid medium (liquid, gas, or two-phase

fluid) and its pressure state can also have considerable influence on the elastic modulus of the material. Temperature demonstrates a significant effect upon ultimate failure strength and may play an important role in determining the material compaction characteristics²⁵.

Thermal Conductivity

Thermal conductivities generally decline with increasing temperature in the granite and sandstone materials tested. This is consistent with published data²⁶. The application of confining pressure can alter this decline and in some cases reverse it. Materials in which fractures and micro-cracks control the porosity seem more susceptible to the influence of pressure. The nature of the pore fluid can also significantly affect the thermal conductivity with water saturated rocks being more thermally conductive than dry or partially saturated rock²⁷.

Thermal Diffusivity

Thermal diffusivity of geologic materials tested decrease with increasing temperature. This effect is interrelated to the dependence of diffusivity upon thermal conductivity and specific heat. Specific heat has been shown to be strongly temperature dependent¹² and therefore has a significant influence on thermal diffusivity. Thermal conductivity is also temperature dependent and the two parameters act in such a way (see text for complete discussion) as to cause a decreasing thermal diffusivity with temperature.

Thermal Expansion

Data obtained under *in situ* pressure and temperature states have indicated considerable variation in expansion coefficients over the temperature ranges 23°C to 300°C. Due to the nature of rock (brittle, polycrystalline, heterogeneous and anisotropic mineral aggregates), microcracking can occur during temperature cycling as a result of dissimilar expansion and contraction¹⁵.

Permeability

In general, permeabilities in porous materials (e.g. sandstone) demonstrate significant declines with increasing temperature. This phenomenon is believed to be due to rock-water interaction at the higher temperatures. Clay swelling and adsorption characteristics are believed to play a big role. Some very low permeability materials (granites in particular) have shown a somewhat different trend. Data obtained on a few such materials demonstrated permeabilities that increase with temperature^{28,29,30}.

Electrical Resistivity

Data generated in this test system have demonstrated trends and phenomenon that agree with published values. High porosity materials such as sandstones show dramatic resistivity decreases with temperature, an effect that is primarily due to increasing mobility caused by decreasing pore fluid viscosity^{31,32}.

Ultrasonic Velocity

To date successful testing has been conducted to 250°C and it is anticipated that testing capability can be extended to 300°C+ in the

near future. In general, velocities in very competent crystalline materials, such as granite, tend to decline with increasing temperature. Different tendencies have been observed in recent testing on saturated porous materials at elevated temperatures³³.

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APPENDIX A

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Capabilities to Determine Rock Properties at Simulated Geothermal Conditions

To date, a very limited amount of rock property research has been conducted under conditions simulating actual geothermal resource conditions. Consequently, limitations exist in the definition and modeling of potential geothermal resources. A test machine was, therefore, constructed specifically to meet the geothermal community data requirements in the areas of surface exploration and subsurface evaluation, drilling, reservoir engineering, stimulation, and subsidence prediction. The machine is capable of testing up to 10.2-cm-(4-in-) dia rock cores at confining pressures up to 200 MPa and 538 C (1000 F) temperature. Mechanical and thermal response tests at conditions simulating downhole pressures and temperatures have been conducted thus far.

Introduction

One of the major technological factors inhibiting the development of geothermal resources is the lack of definition of the resource potential, from the initial surface evaluation through the reservoir assessment. All phases of recovery, from exploration and evaluation through production and environmental impact, require a suitable conceptual and theoretical model for the reservoir. A few of the factors contributing to this lack of resource understanding are: (a) the newness of interest in this energy source, (b) the variety and complexity of the resources encountered, (c) the infancy of geothermal reservoir engineering, and (d) the lack of material properties data in the temperature range of 205 C to 538 C (400 F to 1000 F). Rock mechanics research for the defense, mining and oil communities has contributed a good deal toward the development of rock material properties testing capability. Rock mechanics research has continued to demonstrate the necessity of simulating the in-situ sample environment [1, 2]. Most of this work, however, has focused on relatively low temperature studies (<150 C or <300 F) while basic earthquake research has focused on the ultrahigh temperature-pressure response of rock material (>815 C or >1500 F). Data between 200 C and 538 C (400 F to 1000 F) are lacking. It is in this temperature region that there exist a substantial number of promising geothermal resources.

Recognizing this limitation, a test machine was, therefore, constructed specifically to meet the geothermal community data requirements in the areas of surface exploration and subsurface evaluation, drilling, reservoir engineering, stimulation, and subsidence prediction. The machine is capable of testing up to 10.2-cm-(4-in-) dia rock cores at confining pressures to 200 MPa and 538 C (1000 F) temperature. Mechanical and thermal response tests at conditions simulating downhole pressures and temperatures have been conducted thus far.

Design Criteria

It has been demonstrated many times that simulation of in-situ sample conditions is critical to providing applicable material properties data [2]. In a typical geothermal reservoir, the principal environmental conditions to which the rock material is exposed are the overburden stress, tectonic stress, pore pressure, pore fluid chemistry, and formation temperature. Overburden stress levels to 35–105 MPa (5000–15,000 psi) are common for most current resources. Stress levels up to 207 MPa (30,000 psi) might well be possible for deep hot dry-rock systems that might be considered in the future. Tectonic stresses (horizontal stresses) are anticipated to be as high as 70 to 80 percent of the overburden. Pore pressures of up to 11 KPa/m (0.5 psi/ft) are estimated (excluding geopressured reservoirs). Formation temperatures range up to approximately 430 C (800 F) for current resources.

Sample material obtained from a resource varies from extremely hard rock materials, e.g., granites, basalts, to essentially unconsolidated materials (East Mesa). Failure stress for granites

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confined at 207 MPa (30,000 psi) can often exceed 1.38 GPa (200,000 psi). Sample availability as well as grain size dictate the size of the sample to be tested. It is necessary, therefore, that the test system handle a variety of samples ranging from 2.5-cm (1-in.) diameter to 10.2-cm (4-in.) diameter with L/D ratios of 2.

Design criteria established for the test apparatus were selected to allow for maximum flexibility. Environmental extremes such as 207 MPa (30,000 psi) confining pressure and 538 C (1000 F) sample temperatures were provided to maximize the usefulness of the test equipment. Emphasis was placed on extending current low temperature rock mechanics capabilities to high temperature.

System Development

The geothermal material property test system is shown in Fig. 1. A schematic detailing the significant components is shown in Fig. 2.

Pressure Vessel. The pressure vessel in which the material property testing is conducted is a 15.25-cm (6-in.) I. D. hardened steel cylinder capable of internal pressures to 207 MPa (30,000 psi). A mild steel cooling jacket/safety liner surrounds the vessel. Cooling water circulating in the jacket maintains a constant vessel wall temperature during extended duration testing. Elevated sample temperatures are achieved by internally mounted heaters (up to 9 kW) which promote fast test times due to the avoidance of long heat up and cool down times associated with externally heated vessels.

Various confining fluids were tested in an effort to select a fluid that would remain stable and nonconductive while under the designed temperature and pressure conditions. Liquids were preferred to gases primarily for safety considerations. Dowtherm A was selected after testing determined that it remained electrically non-conductive while at 207 MPa (30,000 psi) and 430 C (800 F). Degradation begins in the fluid at approximately 399 C (750 F) but this does not prove to be a serious limitation since very little of the total fluid is exposed to the high temperature. Dowtherm A will not vaporize at atmospheric pressure until approximately 290 C (550 F). A pressure of 7 MPa (1000 psi) is sufficient to maintain the fluid phase to 430 C (800 F).

All load, strain, temperature, and pore pressure measurements are made inside the pressure vessel. Pressure effect on each of these transducers is carefully documented. Samples ranging from 2.5-cm (1-in.) diameter $\times$ 5.1-cm (2 in.) length to 10.2-cm (4-in.) diameter $\times$ 20.3-cm (8-in.) length can be accommodated.

Load Frame and Control. The load frame is capable of applying an axial load of 960,000 lb (4.3×10^6 N) via the servo-controlled upper actuator. Column and rod design is used in the load frame. Prestressing of the rods and columns maintains a rigid frame. Pressure vessel to lower platen clearance allows for assembly of the test stack with heaters and multiple ceramic shrouding prior to the insertion of the lower closure into the vessel. A reaction column is used to support the lower closure. This type of design allows for quick stack and vessel assembly thereby reducing test times and costs. The capacity is sufficient to provide shear stress to failure on most materials.

Load feedback and displacement feedback are available to provide constant load or actuator position operational modes. An electronic ramp generator is used to establish a constant dis-

Fig. 1 Geothermal material test system

Fig. 2 Geothermal material test system schematic

placement rate of the upper actuator and therefore allows accurate control of strain rate during triaxial testing.

Sample Preparation. When applying a confining stress to a rock sample it is necessary to isolate the internal pore volume of that sample from the confining fluid. In low temperature rock mechanics tests, thin urethane jackets are used to seal the sample. At temperatures above 230 C (450 F), however, the urethane cannot be used because of its degradation and loss of strength. Polymeric type materials were subsequently ruled out as possible jacket materials at elevated temperatures because of this loss of strength.

High temperature research has frequently used a thin [<0.025 -cm (0.010-in.) wall thickness] copper tubing as a jacket material. This technique employs special tapered endcaps or welded end-

Nomenclature

C = Euler's const (dimensionless)
 C_p = specific heat (cal/g K)
 $J_0(x)$, $J_1(x)$ = Bessel functions
 K = thermal conductivity (W/m·K)
 P = power (w/m)

r = radius (cm)
 r_o = outer radius (cm)
 t = time (s)
 V = temperature at sample surface (C)
 θ = temperature (C)
 $\Delta\theta$ = temperature rise (C)

ρ = rock density (gm/cc)
 α = thermal diffusivity (m^2/s)
 δ_n = positive roots of $J_0(\alpha x_n)$
 $\sigma_1 - \sigma_3$ = stress difference
 σ_3 = confining pressure
 ϵ_a = axial strain

Fig. 3 Comparison of copper tubing before and after etching process

Fig. 4 Assembled copper jacketed sample sealed with tapered end-caps and lock rings

caps for sealing. Since thin-walled (0.025-cm) copper tubing was not commercially available, a technique was developed to reduce the wall thickness by chemically etching the available 0.066-cm (0.026-in.) wall to less than 0.025 cm (0.010 in.). Hot, circulating ferric chloride solution removes the unwanted wall material. A comparison of tubing etched in this manner is shown in Fig. 3. Wall thickness variation can be controlled within 0.005 cm (0.002-in.).

Final copper jackets are less than 0.025-cm (0.010-in.) wall thickness and are fully annealed. Jacketing of this type has been shown to have negligible influence on the strength of competent rock (granite, basalt) [3]. Jacketing effects on weaker, less competent materials (sandstone, shale) will require documentation as these materials are encountered. Sealing is accomplished by tapered endcaps as shown in Fig. 4. Sealing rings of this type allow quick assembly and reuse of the endcap. The complete range of sample sizes from 2.54-cm (1.0-in.) diameter to 10.2-cm (4.0-in.) diameter can be accommodated in a similar manner.

Safety Considerations. From a safety standpoint, considerable caution must be exercised when working with high pressures and high temperatures. As was mentioned previously, a liquid confining medium was selected primarily for safety reasons. Large volumes of high-pressure gas contain tremendous amounts of stored energy and can result in catastrophic failures. A compressed liquid, on the other hand, stores much less energy and loses pressure very quickly upon even a small amount of leakage. Very small amounts of the Dowtherm confining fluid are exposed to temperatures exceeding the vaporization point and, therefore, this does not pose a hazard upon sudden depressurization.

For protection, the vessel is completely enclosed by four 0.635-cm- (0.25-in.-) thick lexan sheets. An additional lexan safety barrier exists between the operator and the pressure vessel. Total lexan thickness between the operator and the test apparatus is 1.27 cm (0.5 in.) (equivalent to 2.54 cm (1.0 in.) of bullet-proof glass). All control and testing functions are conducted from be-

hind the safety wall. Control of ventilation, confining fluids, high pressure valving, electronics, and heaters can be made from this remote location. This limits the operator exposure to the hazards of the high pressure and the possibility of being exposed to high temperature fluid in the event of a seal failure.

The lexan shields surrounding the pressure vessel also serve as a fume hood where Dowtherm A and rinsing solvent vapors are collected and removed by a ventilating system. Dowtherm A has been shown to be nontoxic at low vapor concentration levels [4]. However, even at extremely low levels the odors are unpleasant and special precautions were undertaken to minimize operator contact with the fluid. A special rinsing cycle in the pressure vessel is employed to remove residual Dowtherm fluid from the test stack prior to sample removal following a test.

Testing Configurations

Mechanical Response. A schematic of the vessel interior components for the mechanical response setup is shown in Fig. 5. Thermal expansion coefficients are also determined in this configuration.

The test sample, sleeved and sealed within a copper jacket, is located in the upper portion of the vessel. Ceramic insulation is used on each end of the sample to help minimize the longitudinal temperature gradients. Heating elements are coiled around the lower alumina insulator and the lower portion of the sample. A cylindrical ceramic shroud is placed over the sample and the coiled heater to localize the fluid convection currents. Convection within the vessel is further minimized by a second ceramic shroud which contains the first. Alumina (~2.4 GPa (350,000 psi) compressive strength) is used as a cap for this shroud, through which axial loading is applied. These ceramic convection baffles enable temperatures of 532 C (1000 F) to be reached within the inner shroud while maintaining the exterior of the outer shroud at less than ~100 C (200 F), which is well below the maximum allowable for the internal instrumentation and pressure seals.

The lower endcap of this configuration contains the pore pressure transducer. Measurement is made at the very base of the sample thereby minimizing the amount of fluid volume between the transducer and the sample. Pore pressure measurement can be made to 312 C (600 F) in this configuration. Supporting the stack is a strain-gaged load cell. Measurement of axial force in this manner eliminates the possibility of seal friction errors.

Fig. 5 Mechanical response test configuration

Axial strain is measured by two quartz rods connecting the top of the sample to LVDT's (linearly variable differential transformers) contained in the lower vessel closure. Axial measurements with appropriate calibrations for temperature and pressure effects can be made over a range of ± 1.27 cm (0.5 in.) and are accurate to 0.0013 cm (0.0005 in.).

Thermal Response. Thermal conductivity and specific heat testing can be performed in the test configuration of Fig. 6. Basic sample heating and insulation are accomplished in the same manner as previously discussed in the mechanical response set-up (Fig. 5).

Thermal Conductivity. Thermal conductivity at temperature and pressure is determined by the transient line heat source ("needle-probe") method as used by Woodside and Messmer [5]. A detailed discussion of the theory applicable to this testing is contained in Appendix A. In this type of testing a 0.32-cm- (0.125-in-) dia heater approximating the behavior of an ideal line heat source is placed along the longitudinal axis of a 5.1-cm- (2.0-in-) dia by 10.2-cm- (4.0-in-) long test sample. A thermocouple is attached to the heater at the midsample location. The entire probe assembly is then "potted" in place with a mixture of sauerisen cement and powdered copper to minimize contact resistance between the probe and the sample. Ceramic insulators are used to minimize heat loss from the ends of the test sample. Overall internal heater length is 13.4 cm (5.25 in.) giving a heater L/D ratio of 42. Blackwell [6] has shown a L/D ratio of 30 to be minimum for an acceptable approximation of an infinite line source in this type of testing.

Power, in the form of carefully monitored voltage and current, is applied to the probe heater while the test sample is maintained at the desired temperature and pressure. The rate of temperature rise of the probe heater is determined by sample conductivity. Power is supplied to the sample for a short duration only (< 1 min). The exterior temperature of the sample does not change and, therefore, a sample of finite dimension can behave (as the theory requires) as though it were infinite in size. Thermal conductivity for the material is calculated from the temperature history of the internal heater and the power input.

Specific Heat. The thermal conductivity (K), specific heat (C_p), and thermal diffusivity (α) are interrelated as shown by the expression

$$C_p = \frac{K}{\rho\alpha} \quad (1)$$

Fig. 6 Thermal response test configuration

Fig. 7 Axial temperature profiles for a triaxial test sample

where ρ is the rock density (known). If any two of these thermal properties are known, the third can be determined.

Procedures were outlined in the previous section for experimental determination of thermal conductivity. Using the same configuration, a relatively straightforward determination of thermal diffusivity can be made. Classical heat transfer methods allow evaluation of thermal diffusivity in the case of an infinite cylinder of uniform initial temperature responding to an altered surface temperature [7]. In theory, the surface temperature must be uniformly changed. This is difficult to accomplish but is of less importance for poor conductors of heat such as rocks [8]. Also the assumption that the sample will behave as an infinite cylinder must be justified. The theoretical response of a finite cylinder (length equal to diameter) differs from that of an infinite cylinder by less than 10 percent [8]. Since both cases are quite easily determined, the thermal diffusivity values can be bracketed. A more detailed discussion of this and the theoretical basis for this technique is contained in Appendix B.

In the experimental case, thermocouples are brazed to the copper jacket containing the sample. Interior temperature is monitored as the external surface temperature is incremented by 10 to 15 C and maintained. Time-temperature history for the interior thermocouple is recorded and used in the theoretical calculations to determine thermal diffusivity. Using this thermal diffusivity value, and the previously determined thermal conductivity value, the specific heat of the sample can be determined via equation (1).

Data

Mechanical and thermal data have been generated for calibration purposes. Testing was performed on Westerly granite, a very hard, low porosity granite from Westerly, Rhode Island.

Temperature Profile. In any type of temperature testing it is important to provide a uniform temperature distribution throughout the sample. Nonuniform temperature distribution can result in thermal stress, thermal cracking, nonuniform saturation and other deleterious effects. To verify the effectiveness of the heater design used in the various test configurations, temperature profiling tests along the center axis of the sample were conducted.

The results of one such test on a 2.54-cm (1.0-in.) diameter by 5.1-cm (2.0-in.) length of Westerly granite sample are shown in Fig. 7. Confining pressure of 1.72 MPa (2500 psi) was applied during the test. Maximum axial temperature variation of 16 C occurs at 300 C (5 percent temperature variation). Measured radial temperatures indicated gradients of typically 6 C or less at steady state conditions.

Mechanical Response. During the debugging of the mechanical response setup, unconfined and triaxial strength tests were performed. An example of the type of data generated during this testing is contained in Fig. 8. Results of two triaxial tests are shown. Samples were tested dry and pore pressure was not established in an effort to limit variables. In the figure, axial stress difference, $\sigma_1 - \sigma_3$, is plotted versus axial strain, ϵ_a . Con-

Fig. 8 Mechanical response testing

Fig. 9 Failure envelope for Westerly granite

fining pressure, σ_3 , is designated for each test. Sample temperature in both cases was 200 C.

A failure envelope determined from such testing is shown in Fig. 9. Failure stresses to 69 MPa (10,000 psi) confining stress were determined for sample temperatures of 200 C.

Failure stress variation with temperature is contained in Fig. 10. Samples in this testing were confined at 34.5 MPa (5000 psi). Failure stress shows a general decline with increasing sample temperature.

Thermal Conductivity. Thermal conductivity testing was performed following calibration with fused quartz ($K @ 20^\circ \text{C} = 1.36 \text{ W/m} \cdot \text{K}$) at temperature and pressure. Major emphasis during this debugging was placed on developing a standardized probe cementing procedure. Once this was established, consistent results were achieved.

An example of thermal conductivity testing performed on Westerly granite is contained in Fig. 11. Comparison is made with published data contained in Clark's Handbook of Physical Constants [9]. The only significant deviation from the published

Fig. 10 Failure stress as a function of temperature for Westerly granite confined at 34.5 MPa (5000 psi)

values occur at 200 C. Deviation is approximately 5 percent. Also contained in this figure are thermal conductivity values for fused quartz. Confining pressure results in increased thermal conductivity as can also be seen from Fig. 12. Even for a very low porosity rock such as this the variation of thermal conductivity with pressure is significant. A more porous rock would show even greater variation.

Summary

Environmental conditions simulating those of geothermal resources have been routinely achieved with the new test system demonstrating the effectiveness of the machine design. Initial tests have been completed on two major test configurations; mechanical response and thermal conductivity. Detailed test programs can now be conducted in these two areas while development continues on the permeability, ultrasonic velocity, and elec-

Fig. 11 Thermal conductivity variation with temperature

Fig. 12 Thermal conductivity variation with confining pressure

trical resistivity measurements.

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Acknowledgment

The theoretical and, to a limited extent, the technical approaches applied in the thermal conductivity and specific heat testing are based upon published literature. The intent of this paper is to present the modifications and new developments required to advance these measurement techniques and theories to the temperature and pressure conditions of in-situ geothermal environments. We, the authors, gratefully acknowledge the work of numerous individuals responsible for advancing the technology in these areas.

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The following appendices are included as a convenience to the reader and contain well established theories readily available in the literature.

APPENDIX A

Discussion of Transient Thermal Conductivity Testing

Theory. The equation governing the temperature, θ , at a point in an infinite mass containing a uniform linear heat source can be determined as [7]:

$$\theta(r, t) = \frac{P}{2\pi K} I \left[\frac{r}{2(\alpha t)^{1/2}} \right] \quad (2)$$

where

P = power input per unit length of heat source (W/m)

K = thermal conductivity of the mass (W/m-K)

α = thermal diffusivity of the mass (m-m/s)

r = radial distance from source (m)

t = time from start of energy input (s)

and I is the function

$$I(x) = C - \ln x + \frac{x^2}{2} - \frac{x^4}{8} + \dots \quad (3)$$

where C is Euler's const, 0.5772.

Considering the case where the radial distance becomes very small ($r \rightarrow 0$) the term, $x = [r/2(\alpha t)^{1/2}]$, also becomes very small. Assuming that this situation occurs in the experimental setup, the terms in the expansion of $I(x)$ of order x^2 and higher can be regarded as negligible. This assumption appears to be good for the present experimental system as the thermocouple is attached directly to the heater.

Therefore,

$$\theta(r, t) = \frac{P}{2\pi K} \left[C - \ln \left(\frac{r}{2(\alpha t)^{1/2}} \right) \right] \quad (4)$$

Temperature rise, $\Delta\theta$, between times t_1 and t_2 can then be described:

$$\Delta\theta = \theta(r, t_2) - \theta(r, t_1) \quad (5)$$

This expression with some algebraic manipulation gives:

$$\Delta\theta = \frac{P}{4\pi K} \ln \frac{t_2}{t_1} \quad (6)$$

From equation (6) it can be seen that the term $(P/4\pi K)$ represents the slope of θ versus $\ln(t)$ curve. With carefully monitored voltages and current applied to the linear heat source, the power dissipated per unit length, P , can be determined allowing the calculation of the thermal conductivity.

Several major sources of error are possible in this method. The first is due to the elimination of the higher order terms of the $I(x)$ function. Direct attachment of the thermocouple eliminates this error. Secondly, the contact resistance of the probe to the sample can influence the time-temperature curve. This error is eliminated by the use of a standardized, highly conductive potting compound and the disregarding of the initial portion of the curve. Finally the power input to the probe can vary due to the resistance change of the probe with temperature. This effect can be minimized by the use of a constant current power supply and careful monitoring of the probe voltage. Probes showing voltage variation greater than 1 percent are not used.

APPENDIX B

Discussion of Transient Thermal Diffusivity Testing Theory

Thermal diffusivity can be evaluated from classical theory treating an infinite cylinder with zero initial temperature and a constant surface temperature, V . For times $t > 0$ the temperature at any point in that cylinder can be expressed [7]:

$$\theta(r, t) = V - \frac{2V}{r_0} \sum_{n=1}^{\infty} e^{-\alpha \delta_n^2 t} \frac{J_0(r \delta_n)}{\delta_n J_1(r_0 \delta_n)} \quad (7)$$

Fig. 13 Values of $\theta(2t)/\theta(t)$ as a function of $T = \alpha t/r_o^2$

where

θ = temperature at arbitrary point in sample (C)

V = temperature at sample surface (maintained) (C)

r_o = outer radius (m)

r = radius (m)

t = time (s)

$J_o(x)$, $J_1(x)$ = Bessel functions

δ_n = positive roots of $J_o(ax_n) = 0$, $N = 1, 2, \dots$, where a is a const (dimensionless)

α = thermal diffusivity (m-m/s)

Substituting $T = \frac{\alpha t}{r_o^2}$

$$\theta(r, t) = V - \frac{2V}{r_o} \sum_{N=1}^{\infty} e^{-\delta_n^2 r_o^2 T} \frac{J_o(r \delta_n)}{\delta_n J_1(r_o \delta_n)} \quad (8)$$

Substituting $\beta_n = r_o \delta_n$ the foregoing expression can be simplified to the form

$$\theta(r, t) = V - 2V \sum_{N=1}^{\infty} e^{-\beta_n^2 T} \frac{J_o\left(\frac{r \beta_n}{r_o}\right)}{\beta_n J_1(\beta_n)} \quad (9)$$

where β_n , $N = 1, 2, 3, \dots$ are the roots of $J_o(\beta) = 0$. The first few roots of this expression along with corresponding values of $J_1(\beta_n)$ can be found in The Chemical Rubber Company Standard Mathematical Tables, 18th Edition, p. 519.

Since the temperature profile of interest is the center of the cylinder, $r = 0$, therefore

$$\theta = V - 2V \sum_{N=1}^{\infty} \frac{e^{-\beta_n^2 T}}{\beta_n J_1(\beta_n)} \quad (10)$$

In this situation, temperature, $\theta(t)$, measured at some point in the cylinder at time, t , after the beginning of the experiment has the form

$$\theta(t) = A(K, \alpha) f(\alpha t/r_o^2) \quad (11)$$

where A is some constant dependent on the thermal conductivity and thermal diffusivity of the material. It follows therefore that [8]

$$\frac{\theta(2t)}{\theta(t)} = \frac{f\left(\frac{2\alpha t}{r_o^2}\right)}{f\left(\frac{\alpha t}{r_o^2}\right)} \quad (12)$$

$\alpha t/r_o^2$ can then be found from an experimental value of $\theta(2t)/\theta(t)$ and the theoretical curve of the function $f(2T)/f(T)$ where $T = \alpha t/r_o^2$. Such a theoretical curve is contained in Fig. 13 [8]. Also included is the theoretical case for a finite cylinder of length equal to diameter. The similarity of these curves especially at early times verifies the assumption of treating the test sample as an infinite cylinder.

APPENDIX B

THERMAL DIFFUSIVITY

Thermal diffusivity can be evaluated from classical theory of radial heat flow in a cylinder with zero initial temperature and surface temperature $\phi(t)$. For times $t > 0$ the temperature at any point in that cylinder can be expressed

$$\theta(r,t) = \frac{2\alpha}{r_0} \sum_{n=1}^{\infty} e^{-\kappa \delta_n^2 t} \frac{\delta_n J_0(r \delta_n)}{J_1(r_0 \delta_n)} \int_0^t e^{\kappa \delta_n^2 \lambda} \phi(\lambda) d\lambda \quad (1)$$

where

θ = temperature at an arbitrary point in the sample ($^{\circ}\text{C}$)

$\phi(t)$ = temperature at the sample surface

r_0 = outer radius (meters)

r = radius (meters)

t = time (seconds)

$J_0(x), J_1(x)$ = Bessel functions

δ_n = positive roots of $J(ax_n) = 0, n=1,2,\dots$, where a is a constant

κ = thermal diffusivity (meter-meter/second)

If the surface temperature is oscillated, i.e. $\phi(t) = V \sin(\omega t)$, then the integral in Equation (1) can be computed to yield the temperature at any point in the cylinder as a function of r and t .

$$\begin{aligned} \theta(r,t) = & \frac{V M_0(\omega'r)}{M_0(\omega'r_0)} \sin [\omega t + \theta_0(\omega'r) - \theta_0(\omega'r_0)] \\ & + 2 \frac{\kappa V}{r_0} \sum_{n=1}^{\infty} e^{-\kappa \delta_n^2 t} \frac{\delta_n \omega J_0(r \delta_n)}{(\kappa^2 \delta_n^4 + \omega^2) J_1(r_0 \delta_n)} \end{aligned} \quad (2)$$

where

V = amplitude of the sinusoidal oscillation ($^{\circ}\text{C}$)

ω = angular frequency

and $\omega' = (\omega/\kappa)^{1/2}$

$$M_0(z)e^{i\theta_0(z)} = \text{ber } z + i \text{bei}(z) = J(ze^{3/4 \pi i})$$

In sufficiently long time, $e^{-\kappa\delta_n t}$ approaches zero and the transient portion of Equation 2 vanishes. Mathematically:

$$\left. \frac{2\kappa V}{r_0} \sum_{n=1}^{\infty} e^{-\kappa\delta_n t} \frac{\delta_n \omega J_0(r\delta_n)}{(\kappa^2 \delta_n^4 + \omega^2) J(r_0 \delta_n)} \right|_{t \rightarrow \infty} \rightarrow 0$$

$$\theta(r,t) = \frac{V M_0(\omega'r)}{M_0(\omega'r_0)} \sin [\omega t + \theta_0(\omega'r) - \theta_0(\omega'r_0)] \quad (3)$$

If the temperature is measured at $r = 0$ then

$$\theta(0,t) = \frac{V}{M_0(\omega'r_0)} \sin [\omega t - \theta_0(\omega'r_0)]$$

Since the steady-state solution is a function of the same angular frequency as the surface temperature and differs only in amplitude and phase (Figure B1), then the oscillating portion of the solution can be expressed as:

$$\sin [\omega t - \theta_0(\omega'r_0)] = \sin [\omega(t - \Delta t_c)] \quad (4)$$

Figure B1. Temperature versus time for the surface and center of a cylinder.

where

t_c = time for one period of oscillation

Δt_c = time lag between a temperature at the surface of the cylinder and the temperature at the center of the cylinder

thus

$$\omega t - \theta_0(\omega' r_0) = \omega(t - \Delta t_c)$$

or

$$\theta_0(\omega' r_0) = \omega \Delta t_c \quad (5)$$

where $\omega = \frac{2\pi}{t_c}$ and $\omega' = \left(\frac{\omega}{\kappa}\right)^{\frac{1}{2}}$

Solving Equation 5 for thermal diffusivity, κ , we obtain

$$\kappa = \left(\frac{r_0}{\theta_0^{-1}\left(\frac{2\pi}{t_c} \Delta t_c\right)} \right)^2 \frac{2\pi}{t_c} \quad (6)$$

where

$$\theta_0(z) = \tan^{-1} \frac{\text{bei}(z)}{\text{ber}(z)} .$$

Thus by knowing the time lag Δt_c , the period of oscillation $\frac{2\pi}{t_c}$, and the sample radius r_0 , the thermal diffusivity κ can be computed.

APPENDIX C

TECHNICAL DISCUSSION OF RESISTIVITY MEASUREMENT

Background

Electrical conduction in rock materials is achieved principally by electrolytic means in the pore spaces and not significantly by or through the mineral grains (matrix). Most rock forming minerals are insulators at the temperatures which they are typically encountered in the upper crust, with resistivities above 10^8 ohm-meters. Notable exceptions are the native metals and various sulfosalts which, when interspersed, will contribute to the overall conduction of the rock material³¹.

Although essentially non-conductive, the rock matrix dictates the pore-distribution and porosity and therefore controls the electrolytic paths through the material. Various approaches have been made to modeling the conducting pore space geometry. A parameter defined as a "formation factor" was defined by Archie³⁴ and has been used extensively in oil field electric log interpretation. This parameter is expressed as:

$$F = \frac{\rho_r}{\rho_1} \quad (1)$$

where ρ_r is the resistivity of rock material (with electrolyte in pore space) and ρ_1 is the electrolytic resistivity of the saturating fluid. The formation factor is therefore a unique quantity dependent solely upon the rock pore space geometry. A restrictive assumption that the rock is entirely devoid of conducting solids is tied to this particular approach. A more generalized relation, the apparent formation factor,

has been defined to represent the more common case of a conducting material (clay or mineral) being present and is as follows:

$$F_a = \frac{\rho_r}{\rho_l} = \frac{\text{rock resistivity (both electrolytic and by conductive solids)}}{\text{electrolyte resistivity}} \quad (2)$$

Surface conduction via clays is due to the nature of the cation adsorption and exchange process that occurs at the clay surface. A "double layer" of cations are held in two zones near clay surface, an inner or "fixed zone" and an outer or "diffuse" zone. The cations of the diffuse layer are quite mobile and are free to move under the influence of an applied electric field. Cations from this "layer" can add to the normal charge carrier density and result in an increased "surface conduction".

The effect of disseminated clays or shales on rock resistivities becomes more important as the conduction by electrolytic pore paths decreases. Most connate waters possess resistivities in the range of 10 to 100 ohm-meters, indicating salinities of the order of 0.001N to 0.01N. In geothermal situations, the equilibrium concentrations can range much higher due to the higher formation temperatures (to 6N, Niland, Calif.).

By definition, resistivity, $\rho = \frac{1}{\sigma_r}$ where σ_r is conductivity. Therefore:

$$F_a = \frac{\rho_r}{\rho_l} = \frac{\sigma_l}{\sigma_r} \quad (3)$$

As electrolyte ion density increases the electrolyte conductivity, σ_l also increases. Thus the relative contribution of the fixed non-electrolytic conduction components will decrease. It has been shown by Wyllie³⁵

that a "true" formation factor could be defined for a porous rock material by the inverse slope of a linear plot of rock conductivity versus saturating electrolyte conductivity. An example of this is shown in Figure C1.

As can be seen the nonlinear effects take place at the very low ionic concentrations and represent the relatively more significant contribution of the non-electrolyte conducting elements.

It has been shown by the well known "Archies Law" that the porosity of the matrix material can be related to the formation factor of a material,

$$F = \phi^{-m} \quad (4)$$

Figure C1. Rock conductivity versus electrolyte conductivity for Berea and Boise sandstones.

where ϕ represents the fractional porosity and m is a "empirically defined cementation" factor ranging between 1.3 and 2.3. Generally the following holds true:

$$\text{Non-consolidated Sands} \quad F = \phi^{-1.4}$$

$$\text{Consolidated Sandstones} \quad F = \phi^{-1.7}$$

$$\text{Limestones and Dolomites} \quad F = \phi^{-2.0}$$

Winsauer³⁶ generalized the Archies Law equation by the addition of a resistivity index constant, c :

$$F = c\phi^{-m} \quad (5)$$

The constant c in most cases is very close to unity and is generally excluded.

Archies Law has had very successful application in oil field use in sedimentary formations but recent work has suggested that it may not hold in fracture dominated systems, particularly volcanic ones³⁷.

Porosity has been demonstrated to be both pressure and temperature dependent. Because the formation factor and rock resistivity are dependent on porosity (via Archies Law) they will also demonstrate pressure and temperature effects. Pore fluids, as pointed out earlier, play the dominant role in the conduction process. Studies have demonstrated the pressure and temperature dependence of resistivity of aqueous solutions³² with varying ionic concentrations. One such study is shown in Figure C2.

Solution conductivity increases with temperature to a pronounced maximum. Below 300°C pressure effects are of second order. The maximum is shifted to higher temperatures with increasing pressure. Following

Figure C2. Isobaric conductivity of NaCl solution (0.01 m) versus temperature.

this peak, conductivity demonstrates considerable pressure dependence and, in general, decreases with temperature. Viscosity of the fluid decreases with temperature as does the dielectric constant (amount of polarization that takes place when an electrical field is applied) and to a much smaller extent the density. Initially this viscosity decrease dominates; ion mobility increases and the carrier charge density increases. However, at the higher temperatures, the decrease in dielectric constant begins to dominate the process resulting in decreasing conductivities.

Measurement Technique

Conductive processes in a rock material are complex and considerable reliance has been placed upon the use of empirical relations such

as Archies Law. Studies have been undertaken to generate simplified models of these processes such as the following equivalent circuit for a mineralized rock, Figure C3. In this figure;

Figure C3. "Equivalent" circuit for "simplified" mineralized rock.

w = Warburg impedance (discussed in more detail later in appendix)

$= K(1 + i)/\sqrt{f}$, K being a constant (i = current, f = frequency)

C_f = double layer capacitance (clays)

C_{ch} = chemical capacitance

R = reaction resistance

R' = second order and higher reaction resistances

R_i = resistance of the ionic path

R_m = resistance to metallic or conducting mineral elements

Noting that the complexity of this "simplified" representation is beyond the intended scope of this discussion, future schematics of equivalent rock circuits will be shown:

where the function $Z(f)$ will represent the electrically complex* elements.

A four pole electrode arrangement used in the sample configuration of Figure 25, described earlier in the text, is desirable for the laboratory measurement of complex electrical resistivity. As can be seen from this figure, the electrodes are actively involved in the equivalent sample circuit.

Two paths, faradaic and non-faradaic, are available by which current can be carried across an interface between an electrolyte and a metal surface, Figure C4. Current flow by faradaic means is a result of an electro-chemical reaction such as an oxidation or reduction of some ion and involves the migration (diffusion) of ions toward or away from the interface. In this manner there is an actual physical transport charge across the interface. In the non-faradaic process, current is carried by the capacitive effect charging and discharging of a double layer of ions on the interface surface. This is generally treated as a

* Electrically complex components are defined here as circuit elements that demonstrate non-resistive responses to electric current and are frequency dependent.

Figure C4. Circuit analog of interfacial impedance.

capacitance in series with the electrolyte resistance. Circuit representation is not as straightforward as in the case of the faradaic path. It is generally represented by a impedance element customarily referred to as a Warburg impedance with is frequency and current dependent:

$$W = K(1 + i)/\sqrt{f} \quad (6)$$

where K is a constant, i is current and f is the frequency at which current is flowing. Since this type effect will occur at all metallic/electrolyte interfaces, potential drop is measured at a location different from current application. As can be seen from Figure C4, the potential is measured across the sample by two platinum mesh electrodes located 0.03 cm (0.010 inch) from the sample faces. This prevents the measurement of system induced interface impedances and therefore erroneous resistivity values. For voltage measurement a finite current must

Figure C5. Electrical resistivity sample configuration and equivalent circuit.

flow in the potential measuring circuit inducing additional electrolyte metallic impedances and induces a non-sample voltage drop. The magnitude of this drop will depend on the effective impedance of the measuring instrument. For this reason isolation amplifiers, effective input of 10^{12} ohms, are used. The current in the potential measurement circuit will see a minimum impedance 10^8 times larger than the circuit path through the sample.

The Hewlett-Packard model 4274A Multi-Frequency LCR meter is a fully automated test instrument designed to measure various component parameters over frequency range 100 Hz to 10 KHz. Sample impedances are measured using a driving voltage of approximately 2 vrms. The absolute

value of the vector impedance ($|Z|$) and the phase angle (θ) are measured during a resistivity test. The test instrument will allow considerable flexibility in the selection of these test parameters. Measurement is made via a four pole configuration with lead length effects deleted. Isolation amplifiers are used in the potential circuit to maximize system accuracy at the higher sample impedances.

Overall impedance and phase angle values are accurate to ± 2.5 percent.

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