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OIL SHALE PROGRAMS
FOURTH QUARTERLY REPORT
OCTOBER 1976 THROUGH DECEMBER 1976

MASTER

A. L. STEVENS, EDITOR

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Edited by A. L. Stevens, Supervisor
Oil Shale Programs Division 5734
Sandia Laboratories
Albuquerque, New Mexico

5669000

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INTRODUCTION

This is the fourth Quarterly Report for the Sandia Laboratories Oil Shale Programs and summarizes activities between October 1, 1976 and December 30, 1976, on three programs funded by the Oil, Gas and Shale Technology Division of the U.S. Energy Research and Development Administration. These programs are:

1. Diagnostics and Rock Mechanics Support for the Laramie Energy Research Center (LERC) In Situ Oil Shale Program,
2. Advanced Instrumentation and Field Projects for In Situ Oil Shale Processing, and
3. In Situ Oil Shale Bed Preparation Study.

In the following three sections each of these respective programs will be treated individually. The scope and plans necessary to meet the objectives listed in the respective statements of work will be briefly stated. In this context, then, the activities on each program over the past quarter will be discussed.

I. DIAGNOSTICS AND ROCK MECHANICS SUPPORT FOR THE LERC IN SITU OIL SHALE PROGRAM

The objective is to provide technical assistance and field support to LERC to assist in the development of in situ oil shale processing technology and in the demonstration of its technical feasibility and assessment of its economic viability. The tasks required to accomplish this objective are identified with experiments at specific sites in accordance with the test program and experiment schedule mutually agreed upon between LERC and Sandia Laboratories. In each case, the specific tasks fall into five main categories:

- (1) Site specific formation property measurements, as required,
- (2) Numerical studies to assist in the design of rubblizing and retorting experiments,
- (3) Instrumentation to monitor and diagnose the bed preparation experiments,
- (4) Instrumentation to monitor and diagnose the subsequent retorting experiment, and
- (5) Instrumentation, integrated with (3) and (4) above, to monitor the characteristics, quantity and migration of effluents resulting from the in situ oil shale processing.

A. General Design of Field Experiments (A. L. Stevens, 5734)

LERC and Sandia are in the process of revising their joint, long-range program aimed at developing a true in situ commercial capability of recovering oil from oil shale. Included in the program will be the evaluation of the limiting characteristics required of a rubble bed to permit reliable in situ retorting, and the design and evaluation of methods required to produce the desired rubble beds for overburden depths of interest in the western United States. Planning for LERC and Sandia participation in PON2 and other ERDA funded oil shale process research programs was undertaken. The revised long-range program is expected to be completed early in 1977.

B. Site 6A Fielding and Data Evaluation
(B. L. Sewell, 5734; V. E. Kerr, 1133)

Explosive fracturing of the Site 6A formation was completed in July 1976. Air flow and acoustic survey testing was performed to evaluate the communication established between Holes A and B (see Figure IB-1). Core drilling operations to recover samples of the fractured shale were conducted in an effort to develop a means for clearly distinguishing explosive induced fracture from those caused by the coring operation, and to determine the crack widths and hence induced void volume.

1. Post Shot Core Drilling -- The first attempts to mark or tag the in situ fractures involved injecting grout into Holes A and B (the two explosive holes) and attempting to force the grout into the fracture matrix. Prior to grout injection, a dye solution was placed in the explosively produced cavity. Both of these methods of marking

ROCK SPRINGS SITE 64 HOLE LAYOUT

Figure IB-1

existing fractures were completely unsuccessful. Eight conventional cores (see Figure IB-2) were removed from the formation and compared to a core which was obtained from Hole F prior to the explosive fracturing. If it can be proven that the conventional coring operation does not contribute significantly to the fracturing of the cores, these cores indicate considerable fracturing between Holes A and B.

The most promising technique developed to date to acquire the needed proof involves drilling a small diameter hole into the fractured shale bed. This hole is then filled with grout (cement) or another bonding agent and subsequently overcored with a large diameter core bit. In cementing these small holes, which were partially filled with ground water, it was determined that in order to obtain structurally competent grout for the overcoring operation the grout must be pumped into the bottom of the hole until all water and dilute grout is expelled from the top of the hole. This is also a rather slow process since a curing time of about seven days is desirable to obtain the strongest overcored column possible. Two such overcored specimens have been removed from Site 6A. Some core loss was experienced with each core especially in areas where dense fracturing was encountered. Although close correlation of the overcored and conventional cores has not been accomplished yet, it is fairly evident from the preliminary examinations of the overcored specimens that most of the fracture widths are very small.

Figure IB-3 shows the fracture density with depth of core holes 01 and 03 as determined from the conventional cores.

Figure IB-2

Figure IB-3

2. Acoustic Survey (B. L. Sewell) -- Prior to explosive fracturing the acoustic wave attenuation characteristics of the shale formation between Hole A and two of the instrumentation holes was determined. A acoustic pulse generator placed in Hole A was used to supply the input acoustic wave. A hydrophone placed first in Hole C and then Hole E sensed the attenuated acoustic wave transmitted through the shale. The vertical positions of the acoustic wave generator and hydrophone were changed in 2 1/2' increments to acquire a complete mapping of the acoustic properties of the shale regions between Holes A and C and Holes A and E. (See Figure IB-4).

After the second explosive springing of Hole A, an acoustic survey similar to the pre-test survey described above was performed. Comparison of pre- and post-test signal attenuation along individual ray paths results in an outline of a fractured volume as described in Section IIB.

3. Stress Wave Measurements (R. P. Reed) -- Stress measurements were made at various depths in four instrumentation wells. Figure IB-5 shows where the different stress gages were located. The gage nomenclature (Example: 1 LF + 60) is explained as follows.

- 1 indicates the gage was used in the first springing of Hole A.
- L indicates a lithium niobate gage.
- F indicates the gage was located in Hole F.
- +60 indicates that the gage was located 60 decameters (6 meters) above the detonation point, i.e., above the bottom of the explosive charge.

SITE 6A
TEST SCENARIO
ACOUSTIC SCANNING EXPERIMENT

Figure IB-4

STRESS INSTRUMENTATION LOCATIONS - ROCK SPRINGS SITE 64

Figure IB-5

Stress measurements were made during each of the three springing (Hole A - 1 and 2; and Hole B - 1) operations. Figure IB-8 shows the variation in total pressure calculated for radii up to 10 meters from the explosive column for the horizontal planes in which the stress gages were located. For example, $Z = 0$ identifies the horizontal plane at the base of the explosive column, $Z = -2\text{m}$ the plane 2m lower, etc. Stress values recorded by those gages which are known to have functioned satisfactorily are superimposed for comparison with the calculated stresses.

It should be noted that it was impossible to orient the stress gages in the instrumentation holes. Most of the gages were encapsulated in grout and, therefore, experienced a non-spherical stress field, i.e., a field in which principal stress components are not all of equal magnitude. An effort is currently underway to determine the extent to which gage response depends on orientation relative to principal stress components. Some additional comments regarding stress measurements and calculations will be made later in this report.

4. Air Flow and Tracer Tests (R. D. Klett) -- In past tests at Rock Springs and at other sites, the Laramie Energy Research Center has deduced conductivity through a fractured bed by observing total flow and arrival time at observation wells of air and tracer pulses of radioactive Krypton (^{85}K) introduced in another nearby well. The LERC air flow technique with some modification was used to evaluate the conductivity between Holes A and B and between Holes A and H after the second springing of Hole A and after the detonation in Hole B.

Figure IB-6

Prior to the detonation in Hole A the shale formation was so tight that flow from Hole A could not be detected at any of the instrumentation holes. After the first detonation in Hole A, considerable communication between the two holes was indicated by the water flow between them.

After the second springing in Hole A, Hole B was drilled and air flow between these two holes was determined. (Holes C and E had been grouted full just before the second spring of Hole A). Although airflow between Holes A and B was clearly evident at this time, the quality of the data was not adequate to derive consistent values for conductivity parameters.

After the detonation in Hole B, the ^{85}K concentrations in Hole B and Hole H from injection in Hole A were observed as a function of time. The results are shown in Figure IB-7. Total flow rates obtained in both high and low pressure tests were found to be in good correlation with the tracer pulse data. The flow and tracer tests were run using LERC hardware and procedures while formation flow properties were computed using techniques developed by Sandia. Effective flow conditions between the two holes were determined rather than local permeability. The formation properties deduced were hydraulic transmittance defined as the total flow rate divided by the pressure head (in feet of water), hydraulic conductivity, thicknesses of permeable regions, effective thicknesses of fractures, and equivalent diameters of pipes that would transmit the same amount of gas as the flow paths in the formation. The tracer output of the high pressure test between Holes A and B is shown in Figure IB-9.

Figure IB-7

The first pulse can be identified with a fracture that passed about 27% of the flow and the second pulse can be identified with a permeable region that passed the remainder of the flow. There were no other significant flow paths. Table IB-1 summarizes the formation flow properties between the two sets of holes. Additional flow tests are planned between core holes to obtain more localized formation flow properties.

C. Site 12 Experiment Activities
(B. L. Sewell, 5734; V. E. Kerr, 1133)

Late in the third quarter of CY 1976, the Site 12 Experiment at Rock Springs was begun. The objectives of the experiment are,

1. to characterize the fracturing of the Tipton shale member which will be produced by detonating an explosive slurry emplaced in two previously formed horizontal hydraulic fractures,
2. to compare the pressures measured and the fracturing produced to those predicted by the analytical model of the system,
3. to develop and evaluate site preparation techniques for use in future production efforts, and
4. to develop and evaluate physical property measurements systems for use in future experimental and production efforts.

Table IB-1

Site 6A Flow Tests

	A - B Wells		A - H Wells	
	Pulse 1	Pulse 2	Pulse 1	Pulse 2
Hydraulic Transmittance $\left(\frac{\text{ft}^2/\text{sec}}{\text{ft (H}_2\text{O)}}\right)$	8.1×10^{-7}	2.2×10^{-6}	1.3×10^{-6}	1.3×10^{-6}
Hydraulic Conductivity $\left(\frac{\text{ft}}{\text{sec}}\right)$	--	1.8×10^{-6}	--	9.1×10^{-6}
Thickness of Permeable Layer (in)	--	36.4	--	7.3
Fracture Thickness (in)	.002	--	.002	--
Equivalent Pipe Diameter (in)	.30	.58	.65	.65

1. Experiment Description -- The general approach to the Site 12 experiment is to separately form three horizontal hydraulic fractures with a minimum of vertical separation. The lower two fractures will be filled with a slurry explosive and the explosive will then be detonated. The upper fracture will serve as a void from which the stress wave from the middle fracture will reflect. The void formed by the expansion of the middle fracture will serve the same purpose for the stress pulse from the lower fracture. By keeping the formed hydraulic fractures on the order of five feet apart, considerable horizontal fracturing should result. Vertical fracturing by flexural or shear stresses should "block" the shale.

Observation wells at 25 and 50 foot radii will be drilled to permit the evaluation of the hydraulic fracture and explosive insertion operations. Pressure and time-of-arrival gages will be placed at or near fracture depth in instrumentation wells. Figure IC-1 shows a plan view of the hole pattern anticipated for Site 12. Figure IC-2 is a simplified cross section showing the relationship of the boreholes to the hydraulic fractures

2. Site Characterization and Fracture Instrumentation -- Of equal importance to the conduct of the overall experiment are the characterization of each hydraulic fracture during their formation, controlling the placement of the explosive and characterization of the explosively induced fractures. Table IC-1 lists the instrumentation techniques planned and the purpose of each measurement.

A. Hydraulic Fracture Formation and Characterization -- Because of the need to understand the configuration of each of the

Figure IC-1

Figure IC-2
 Proposed Site "12" Layout

Table IC-1
Site 12 Instrumentation

<u>Technique</u>	<u>Purpose</u>
<u>HYDRAULIC FRACTURE</u>	
Hydrophones in observation wells	Passive acoustic mapping (2D) of the fracture front.
Current probes in observation wells (O.W.)	Time of arrival of the fracture fluid at the O.W.
Current probes on the surface	Resistivity mapping of the fracture front.
Pressure measurements at the observation wellhead	Time of arrival of the fracture fluid at the O.W.
Pressure measurement at the frac. well	Independent pressure profile of the fracture.
Recording of the Halliburton pressure and flow	Correlation with Sandia data.
<u>HYDRAULIC FRACTURE CHARACTERIZATION</u>	
Air flow and tracer injection	Permeability of the fracture.
Flow Logging	Fracture location and permeability.
T.V. Logging	Fracture location.
Temperature Logging	Fracture location.
Gamma Log	Fracture location if proppant sand is radioactively loaded.
Impression Packer	Fracture location.

Technique
EXPLOSIVE CHARACTERIZATION

Pressure measurement at
Holes B and C

Time of Arrival

Stress Measurements

Round Robin

RUBBLE IDENTIFICATION

Core-back, log and photograph

Air flow and tracer
injection

Air flow logging

T.V. Log

Temperature Log

Purpose

Pressure profile on explosive emplacement
holes.

Verification of detonation quality
and symmetry.

Correlation with the theoretical
model.

Verification of the slurry emplacement.

Ground Motion.

Rubble I.D.

Permeability of the rubble area.

Location of the rubble area.

Location.

Location.

three hydraulic fractures after they are formed, considerable effort will be expended observing various dynamic parameters during the hydraulic fracture operation which will be descriptive of the fracture rate, fracture thickness, fracture radius and proppant placement. Subsequent to each fracture, the desired communication between appropriate holes and the desired isolation of other holes will be verified. Acoustic, resistivity, and pressure measurements will aid in observing hydraulic fracture formation while flow, TV, temperature and gamma logs combined with mechanical impression packer will help characterize the formed hydraulic fracture.

- B. Explosive Placement and Performance -- Approximately 7500 pounds of explosive slurry will be placed in each of the two lower hydraulic fractures. Special attention will be given to instrumentation which will sense the arrival of the explosive at the detonator/booster assembly and at each of the observation wells. Pressure and time-of-arrival measurements of the stress waves will be made at the locations shown schematically in Figures IC-3 and IC-4.
- C. Rubble Characterization -- Post-shot coring, air flow logging, tracer gas flow logging, and TV logging techniques will be used to evaluate the fracturing produced by the explosion.

Figure IC-3
Preliminary Stress Gage Array
Rock Springs Site 12 Experiment

Figure IC-4
Preliminary Stress Gage Array
Rock Springs Site 12, Experiment

3. Current Status and Future Schedule -- The hydraulic fracture and explosive installation holes have been drilled to total depth. The observation wells have been drilled to the top of the Tipton shale member. Drilling of the instrumentation holes to the top of the Tipton is underway. The balance of the experiment is expected to follow the schedule shown in Figure IC-5.

Figure IC-5
Site 12 Experiment Schedule

II. ADVANCED INSTRUMENTATION AND SUPPORT PROJECTS FOR IN SITU OIL SHALE PROCESSING

The objective is to develop instrumentation techniques and equipment to monitor and control in situ fracturing and retorting experiments. Specifically, the activities include (1) the development of a solid state specific oxygen probe to permit continuous monitoring of oxygen fugacities during retorting, (2) the examination of the applicability of an acoustic technique to determine the extent and degree of fracturing within an oil shale bed that has been rubblized preparatory to retorting, and (3) the development of a data acquisition system including both equipment and software which is to be used to acquire and process data during in situ oil shale retorting experiments.

A. Oxygen Probe Development (D. M. Haaland, J. M. Freese, 5825)

During this reporting period, much of the emphasis was placed on approaches to eliminate the catalytic interference of platinum electrodes on the operation of the oxygen probe in non-equilibrium environments. Because non-equilibrium gas mixtures are often encountered in oil shale retort operations, this problem must be solved in order to extend the useful range of the probe. For this purpose, the catalytic behavior of gold electrodes in the presence of non-equilibrium mixtures of CH_4 , O_2 , and N_2 was determined.

Gold electrodes prepared by vapor deposition, sputtering, and thick-film painting techniques were compared. Vapor-deposited gold exhibited poor current-voltage characteristics while sputtered gold proved to be comparable to painted platinum in current-voltage response. The thick-film painted gold electrodes showed much greater current (i.e., greater oxygen pumping rates) for a given applied potential but exhibited large hysteresis comparable to that found previously with silver electrodes. However, it was discovered that after a current treatment of approximately 1 amp on the painted electrode, the response of these electrodes improved significantly. Not only did the current levels increase by more than a factor of 2, but hysteresis was also eliminated. Figure II A-1 shows the current voltage characteristics of painted gold electrodes on a disc of 16.9 wt. % yttria stabilized zirconia. Electrode area is 2.0 cm^2 and disc thickness is 1.5 mm. Curve A is the original response of the gold electrode. Curve B was obtained after a current treatment at an applied voltage of 6 V for approximately 5 minutes. Note the lack of hysteresis and the introduction of ohmic behavior after current treatment. SEM photographs of the electrode surfaces showed that the current treated surfaces were both more porous and more structured than the untreated electrodes. Thus the improved electrode response observed with current conditioning is probably a result of more rapid transport of oxygen through the porous electrode as well as the introduction of additional active sites for oxygen absorption.

A gold-electrode probe was constructed using current treated electrodes. Because cracks were present in the solid electrolyte,

Figure IIA-1
Current voltage response of probe with painted gold electrodes

the accuracy of this probe was limited. Unfortunately these gold electrodes were catalytic well below 800°C in contrast to our previous results with air-reference oxygen probes. The current treatment necessary for efficient oxygen pumping must be considered responsible for the increased catalytic behavior of these treated electrodes. At any given temperature, these gold electrodes are, however, less catalytic than platinum since a larger stoichiometric excess of methane was required before the probe operation failed. At temperatures less than 700°C, this probe responded significantly slower than the platinum electrode probes, this limiting its use at low temperatures. Because only limited success was achieved with this probe, other methods described later in this report were investigated to eliminate the catalytic interference of the platinum.

In another phase of this work, two solid-electrolyte oxygen sensors described in the previous quarterly report were tested in the 10-ton-shale vessel at the Laramie Energy Research Center in Laramie, Wyoming. Two problems were encountered which limited the amount of data obtained. First, an unusual burn front advanced more rapidly down the sides of the retort than at the center where the sensors were located. This caused the non-equilibrium mixture of gases (O_2 , CH_4 , H_2 , etc.) withdrawn from the cooler center section through a stainless-steel sampling tube to be driven to equilibrium during passage through the hotter burn zone near the walls. Thus gas samples withdrawn through these tubes always yielded O_2 concentrations of $< 0.1\%$ as determined with a paramagnetic oxygen analyzer. This eliminated an independent O_2 analysis at the location of our solid-

electrolyte oxygen sensors. The second problem involved an unexpected and early shorting of the stainless-steel sheathed electrical lead wires. This resulted from the higher temperatures and a more corrosive atmosphere developed during the retorting of Michigan shale than had been previously encountered with Colorado oil shales. After reaching an operating temperature of 450°C, one O₂ sensor did function normally for a short period of time yielding a free oxygen content of 0.7%. From previous experiments with other analysis techniques, this is a reasonable O₂ concentration for the area in advance of the burn front. One of the oxygen sensors was recovered from the spent shale and was returned intact to Sandia for analysis. Future field experiments will be designed to eliminate the problems encountered in this test.

These field experiments were conducted with oxygen sensors using platinum electrodes. However, as described earlier, platinum has been shown to catalyze the reaction of methane and oxygen, and, therefore, sensors constructed with platinum electrodes will not normally measure non-equilibrium oxygen values when a large excess of methane is present. However, one sensor did appear to operate normally in the 10-ton retort even though an excess of methane was probably present. A possible explanation for this result is that the catalytic electrode surfaces were poisoned by one or more components of the retort gas which allowed O₂ values to be measured accurately. This hypothesis was recently tested in a controlled environment.

Because sulfur is known to be an effective poison for platinum, the response of a platinum electrode probe was tested after treatment with H_2S . The electrodes were effectively poisoned after the addition of approximately 0.5% H_2S to the flowing gas mixture for several minutes at 600°C. The probe was then capable of accurately monitoring oxygen content in a non-equilibrium mixture of oxygen and methane. In addition, oxygen pumping rates were increased by a factor of 5 to 10 with this treatment. Studies of these rapid pump rates will be continued since they may lead to lower temperature operation of the probe. The poisoning effects of the sulfur were found to continue at 700°C. However at 800°C, the sulfur rapidly desorbed and the catalytic properties of the platinum returned at this and lower temperatures. This is consistent with results on the sulfur poisoning of platinum catalytic converters used for auto emission control. Unfortunately, later H_2S treatments with the above probe did not reproduce the earlier poisoning effects. Therefore, further poisoning studies with a new platinum electrode probes will be initiated. In light of the partial success of the oxygen sensor in the 10-ton retort and its survival even after the harsh treatment in the Michigan shale retorting, it is concluded that only the successful and persistent poisoning of the catalytic platinum electrodes is required to provide a sensor that will yield accurate oxygen concentration data in an oil shale retort environment.

B. Particle Size Distribution - Acoustic Technique
(L. W. Beckham, 5732)

The acoustic data collected at Rock Springs has been analyzed and interpreted to evaluate the spatial extent of the test effects in the planes between the source well (well A) and the receiver

wells (wells C and E). These results are presented in Figures II B-1 and II B-2 for planes A to C and A to E respectively. These figures were drawn by assuming that an observed change in the time of arrival along a particular ray path was significant enough to delete the direct path ray. A consistent change observed over an area gives the spatial extent of the test effects. The criterion chosen for deleting a ray path was a change in the time of arrival of approximately 0.25 - 0.30 milliseconds since this threshold seemed to give the most consistent results and was not too sensitive to timing errors in the method of data collection.

Figures II B-1 and II B-2 suggest that the effects of the test for the well A-C plane are more severe than the well A-E plane. A nonsymmetric distribution of the fracture pattern around well A is also evidenced in the cores taken to date.

The above results indicate the feasibility of the acoustic technique for determining the spatial extent of fracturing. However, it is not clear at the present time that this technique can be used to achieve the ultimate goal of mapping the rubble size distribution of an underground shale bed as a function of position.

The mathematical techniques which have been developed for reconstruction of images from x-ray data have been proposed as being possibly useful in this program. However, there are a number of experimental and theoretical problems associated with the extension of the x-ray techniques to this acoustic problem. Among them are:

- diffraction effects
- longitudinal shear mode conversion

POST-TEST RESULTS: WELL A-C PLANE

Figure IIB-1

POST-TEST RESULTS: WELL A-E PLANE

Figure IIB-2

- large acoustic attenuation
- inhomogeneity of velocity even in original oil shale
- anisotropic acoustic behavior in layered medium
- multiple propagation paths from source to detector

These effects have no x-ray counterpart and greatly complicate the experimental and theoretical problems.

Considering the problems outlined above and with a realization of the high risk nature of the program, a small scale laboratory program is being considered prior to collecting further field data. The laboratory program would attempt to demonstrate the feasibility of the technique and address those problems which would arise in the implementation and analysis of a field program.

C. Field Data Acquisition System (K. M. Kimball, 1123)

A data acquisition system including both equipment and software is being developed to acquire and process data during in situ oil shale retorting experiments. The system is designed to perform the following functions:

- (1) Acquire data from a wide variety of sensors located throughout the test bed.
- (2) Preprocess (e.g., thermocouple linearization, conversion to engineering units, data compression, formatting, etc.) and store the data on disc or magnetic tape.
- (3) Process data as required for immediate on-site analysis and interpretation of the experiment status.
- (4) Display and produce hard copy of data in graphic and tabular forms.

(5) Perform a limited number of control functions.

A block diagram of the system is shown in Figure II C-1.

An order has been placed for the electronic equipment; delivery is presently scheduled for mid-March. The system is planned for installation in a currently available Sandia trailer.

Figure II C-1

III. IN SITU OIL SHALE BED PREPARATION

The objective of the current program is to provide realistic computational tools to aid in the design and understanding of in situ field experiments. To accomplish this goal, extensive characterization of the mechanical properties of oil shale is necessary so that constitutive models suitable for use in numerical computational schemes can be developed. The thrust of the early stages of the program has been toward the experimental characterization of mechanical properties; this characterization has now developed to the point that serious modeling efforts can begin.

The triaxial tests, which provide a significant portion of the input to constitutive models, are essentially completed. These tests, involving work at Sandia and Terra Tek, have explored the stress-strain response of two types of shale (with different kerogen contents) as functions of confining pressure and bedding plane orientation. In those areas where test conditions were duplicated, the results from the two laboratories are in agreement, regardless of the fact that the specimen sizes were significantly different. As the first step in model characterization, the shear stress invariant versus mean pressure response is being extracted from the data. This can be done unequivocally at 0° and 90° bedding plane orientations, and at other orientations once a correction for induced bending stress is made. Results acquired to date are quite consistent.

The fracture toughness testing has been devoted in large part to the performance of peripheral, but highly essential, tests to establish criteria for sizing specimens, to verify material variability effects, and to acquire tensile strength data. It has been found that the larger of the two specimen sizes considered does yield larger values for fracture toughness; however, the trend observed is not uniform and may display some effects of material variations. Also, during this quarter, the preliminary tests on "stress corrosion" were performed. The results were positive in that an effect was observed.

Significant progress in our ability to make the computer computations was made this quarter. The computational effort has until now been directed primarily at the Rock Springs Site 6A experiment and has involved the use of the two-dimensional wavecodes, TOODY and CSQ. Calculations were performed using simple elastic-plastic constitutive models both with fracture suppressed and with fracture permitted. In the "fracture permitted" calculations the BFRAC model was used with tentative parameter values. We are just beginning to evaluate the implications of the calculated fracture patterns. The layering of the shale bed has also been introduced in the calculations with the result being that very definite effects are evident; one example is higher peak pressures in regions of high densities.

A. Triaxial Testing of Competent Oil Shale
(K. W. Schuler, A. K. Jones, T. J. Lutz, 5163)

The triaxial and unconfined testing of competent 20 and 40 gal/ton oil shale is now complete. In this phase of the program, over 130

tests were performed. The effort was approximately equally divided between Sandia and Terra Tek. Both laboratories tested specimens which were obtained from carefully selected regions of two large blocks. The blocks had been selected on the basis of density and NMR analysis to yield reasonably uniform specimens with nominal kerogen concentrations of 20 and 40 gal/ton. The primary difference between the specimens tested at the two laboratories was their size -- at Sandia, specimens were 4.5 cm in diameter by 9.0 cm long while at Terra Tek they were 1.9 cm in diameter by 3.8 cm long. The testing matrix provided some duplication; thus, a comparison of the data from the two laboratories provided an indication of sample size effects.

Figure III A-1 shows a comparison of the axial stress-axial strain response obtained on the two different specimen sizes. In both tests, the specimen was a 20 gal/ton shale with bedding planes oriented at 90° to the specimen axis. The data for the two specimen sizes lie within ~ 15 percent of each other. Since this is about the same reproducibility which we have observed in specimens of a given size, we can conclude that there is little, if any, size effect in the range of sizes tested to date. More detailed comparisons of the data from the two laboratories will be made using our PDP-11 computer facility to produce merged plots of the test data for similarly loaded and oriented samples.

A comparison of the dependence of the strength of the shale on confining pressure can readily be made for the 0° and 90° orientations. For other orientations, a correction must be applied to the strength

AXIAL STRESS-STRAIN CURVES OF TWO
DIFFERENT SIZES OF 20 GAL/TON SPECIMENS

Figure III A-1

data using finite element calculations to account for the stress nonuniformity effect which was outlined in prior quarterly reports. The calculations require a consistent set of elastic moduli which should be readily determined from the computer generated merged plots.

For the 20 gal/ton material, the maximum stress difference-pressure data for both the 0° and 90° orientations are shown in Figure III A-2. It is clear from the plot that there is good agreement between the data obtained by Sandia and Terra Tek on the two different size samples. More importantly, there does not appear to be any significant differences between the two orientations. This observation should greatly simplify the modeling of the strength data. We cannot conclude from this observation that there is no strength anisotropy since in both the 0° and 90° orientations the maximum shear stresses develop at 45° with respect to the bedding planes. Thus, if the bedding planes are planes of weakness, we would not expect to see slippage in these tests. The two data points obtained at 800 MPa appear to reverse the trend of the lower pressure data and have lower than expected strengths. In that we have no reason to suspect the accuracy of these data, these results suggest that a few tests at higher confining pressure should be performed.

Figure III A-3 shows a similar plot for the two orientations of the 40 gal/ton material. Once again there is good agreement between data obtained at the two laboratories and there is little difference between the two orientations. Comparing the results of 20 and 40 gal/ton material, we note that the richer shale is considerably weaker. The dashed line shown in Figure III A-3 is the strength-pressure

Figure III A-2

Figure III A-3

data obtained on Polyvinyl chloride (P.C.V.) by Pae and Sauer.¹ This polymer has an unconfined strength which is quite comparable to the 40 gal/ton shale. We see that the shale strengthens more with pressure; however, above about 400 MPa the rate of increase is approximately the same.

The dashed line on Figure III A-2 is the strength-pressure data obtained on Hasmark dolomite by Handin and Hager.² We chose this material to compare with the 20 gal/ton results because the major mineral constituent of the shale is dolomite and this particular dolomite has about the same unconfined strength as the oil shale. The shale data lies below that of the dolomite; however, at lower pressures the rate of rise appears to parallel that of the dolomite. These comparisons suggest the possibility of developing a mixture theory for oil shale which would use dolomite and a typical polymer as the two constituents.

B. Fracture Mechanics of Oil Shale
(R. A. Schmidt and C. W. Huddle, 5163)

In situ bed preparation of oil shale by rubblization involves rock fracture regardless of the actual technique employed, be it hydraulic fracturing or detonation of explosive-filled fractures or boreholes. Most failure theories that exist for rock apply only to competent rock and do not deal directly with the fracture process.

¹Pae and Sauer, in Eng. Solids Under Pressure, Institution of Mech. Engrs., 1 Birdcage Walk, Westminster, London SW1H 9JJ, p. 69, 1971.

²Handin and Hager, Bull. Am. Assoc. Pet. Geol. 41, 1, 1957.

As a result, they cannot be expected to deal with questions of crack propagation such as (1) the length of major crack extension in processes such as hydraulic fracturing, (2) failure loads necessary to extend pre-existing fractures as might be encountered in repeated borehole explosive tests and (3) conditions for crack bifurcation or branching necessary to create rubble in a rock fragmentation process. Many similar questions have been answered for fracture problems in metal alloys, glasses, and polymers using the well-founded discipline of linear elastic fracture mechanics. Encouraging results of recent investigations at Sandia have indicated that much of the theory, principles, and testing techniques of fracture mechanics are also applicable to the study of fracture behavior of rock. The value of the stress intensity factor, K , at which a crack will propagate in a material under the limiting conditions of plane strain is called the plane strain fracture toughness, K_{Ic} , or simply fracture toughness. Fracture toughness has been measured and shown to be a material constant for two rock types, Indiana limestone and charcoal granite, using ASTM-recommended techniques.

Results of a series of comprehensive, unconfined fracture toughness tests on oil shale were reported in the Third Quarterly Report. These tests provided preliminary baseline values of fracture toughness for two grades of oil shale (20 and 40 gal/ton) and for the three principal crack orientations with respect to bedding planes (divider, arrester, and short transverse). Duplicate tests were also performed which indicated excellent reproducibility of results.

During this past quarter, effects of two additional parameters -- specimen size and crack tip environment -- were addressed.

Toughness data on larger specimens were necessary to determine the extent to which the baseline data are affected by the smaller "subsize" specimens. The previous specimens measured 2.5 x 5 x 20 cm and are considered subsize according to ASTM testing standards for metallic materials. The baseline toughness values obtained should, therefore, be considered preliminary. Five large specimens (5 x 10 x 40 cm) were machined from the two main blocks of oil shale (blocks B and D). Two initial tests were unsuccessful since the technique of specimen immersion in dye penetrant used to mark the position of the fatigue crack left residual penetrant in the crack. This excess penetrant ran during final crack advance, obliterating the mark for the original crack front. This problem was alleviated by placing the samples in a vacuum after immersion to extract the excess penetrant. Results from the three remaining tests are summarized in Table III B-1.

Table III B-1

Nominal Assay (gal/ton)	Crack Orientation	Preliminary Values for Fracture Toughness, K_Q	
		(MN m ^{-3/2})	
		2.5 x 5 x 20 cm specimens	5 x 10 x 40 cm specimens
40	Divider	660	800
20	Arrester	940	2070
20	Divider	1070	1560

Note that the fracture toughness values are greater for the larger specimens, a trend that has been observed for other rock types. The trend evident from our three experiments, however, is by no means uniform. This nonuniformity in the increase is most likely caused by material differences among specimens; the large specimens could not be obtained from the same beds of material in the two large blocks as the smaller specimens. To provide for a more direct comparison of results, several small specimens are presently being machined from pieces of the large specimens.

Some initial results on the effect of a fluid environment on fracture toughness were obtained this quarter. The phenomenon known as stress corrosion cracking occurs when a crack, which is subjected to a particular environment, propagates in a stable manner while the stress intensity is below the critical level. Many metallic materials are affected in this way, with some titanium alloys displaying crack propagation in the presence of distilled water at levels of stress intensity as low as 25 percent of the critical level. In addition, several investigators have reported degradations in material properties of some rocks when exposed to various fluid environments.

Three stress corrosion tests were performed on oil shale this quarter with test durations as long as 122 hours. The details of these tests and results are summarized in Table III B-2. Note that while subcritical crack growth did occur in test #3 (lab air), the time-to-failure was more than ten times longer than for test #2 (distilled water) which was performed at the same level of stress

intensity. This behavior is similar to that of many metallic materials and implies that water is an agent of stress corrosion cracking in oil shale. For additional verification, a test is being readied in which the sample will be subjected to an inert environment of dry argon.

TABLE III B-2

Test No.	Nom. Assay (gal/ton)	Crack Orientation	Environment	Maximum Stress Intensity (% of K_Q)	Failure (hours)
1	40	Divider	Distilled Water	78	No failure after 122 hr.
2	20	Divider	Distilled Water	90	.71
3	20	Divider	Lab Air (7% R.H.)	90	8.87

Table III B-2 also indicates that while oil shale does appear to exhibit the phenomenon of stress corrosion in distilled water, the level of stress intensity necessary to produce significant crack growth is nearly 90 percent of the critical value, K_Q . This would indicate that little benefit can be obtained for enhancing crack growth with the presence of water. Tests with other fluids should be performed however, before this approach is abandoned.

Plans are also being made to perform a comprehensive series of "direct pull" tensile tests to measure tensile strengths on samples with orientations both parallel and perpendicular to the bedding planes and under both confined and unconfined test conditions. The results of these tests are important for a proper understanding of the fracture toughness measurements because sample size effects in fracture

toughness tests are directly related to tensile strength magnitudes. The tensile strength experiments will also provide additional information about the fracture response of oil shale under various triaxial stress conditions. Samples for the tensile strength experiments are currently in the final machining phase.

c. Simulation of Explosive Detonations in Oil Shale with the CSQ Wavecode
(R. R. Boade, 5734)

The ability to predict with confidence the degree of rubble-ization that will result from the detonation of an explosive charge in an oil shale bed is regarded as an important objective of Sandia's oil shale program. For many practical explosive configurations, this objective can, in principle, be realized with the use of two-dimensional wavecodes, provided the physical characteristics of the explosive and oil shale are adequately known and properly utilized in the code. To develop the desired computational ability, two separate, but related, efforts are currently underway. One of these efforts involves the use of the Eulerian-Lagrangian code, CSQ; this work will be discussed in this section of the quarterly report. The other effort involves the use of the Lagrangian wavecode, TOODY, and will be considered later in the report.

The bulk of the work to date with the CSQ code has been directed at the Rock Springs Site 6A experimental activity, although some preliminary calculations pertinent to the upcoming Site 12 experiment have been made. The calculations for the Site 12 experiment were made primarily to determine appropriate separation distances for the three horizontal hydraulic fractures which will

be produced in the oil shale bed. A slurry explosive will be introduced into the lower two fractures while the upper fracture will contain only propping sand. The CSQ calculations for the Site 12 experiment were augmented by additional calculations made with the one-dimensional code, CHART D. By considering compressive stress pulse amplitudes predicted to exist in the oil shale surrounding the explosive-filled cracks, and also by considering tensile stress amplitudes generated after interaction of the compressive waves with the cracks, it was concluded that separation distances should be about 1.5 m (or less). With these separation distances, tensile stress amplitudes should be greater than 1 kbar, which is felt to be large enough to produce interlaminar spallation in the oil shale. The computer examination of the Site 12 experiment will proceed with increased intensity during the early part of 1977, as the time for execution of the various aspects of the experiment becomes appropriately imminent.

The calculations for the Site 6A experiments have been made in part (i) to evaluate methods for deducing equation-of-state and constitutive-relation parameters for the explosive and oil shale, (ii) to determine the effect of shale bed stratigraphy on stress histories at various locations around the explosive charge, and (iii) to examine the nature of the transient stress field in the oil shale with special attention given to departures from spherical conditions. The configuration assumed for all calculations performed to date has been that of the first detonation in Hole A and the only detonation in Hole B; for each of these cases, a vertical column

(3.7 m high) of explosive was emplaced at the bottom (~ 195 ft. deep) of a 20 cm diameter well. Experimental measurements obtained with lithium niobate and ytterbium gages located in grout-filled wells 5 and 10 m distant from the explosive wells have been used for comparison with the calculations.

While several sets of calculations have been performed in the process of selecting proper methods for addressing problems similar to those of the Site 6A experiments, two sets are of particular interest at this time. In one of these, the shale bed was assumed to be homogeneous with a density of 2.30 g/cm^3 and in the other a layered configuration was assumed which approximated the stratigraphy deduced from logs acquired in the vicinity of Site 6A. The upper graph in Figure III C-1 shows a comparison between density log data (dashed curve) and the approximation (solid line segments) assumed for the calculation. Abscissas for all the graphs in Figure III C-1 correspond to vertical position, or elevation; the zero position was taken to be the bottom of the explosive column which is also the point at which the detonation was initiated.

The oil shale for both the homogeneous and layered configurations was treated as a non-porous elastic-plastic material. Constitutive relation parameters were deduced from properties of the two constituents of oil shale (namely kerogen and marlstone) using a standard mixture theory. The explosive (an IRECO slurry) that was used at Site 6A has not been characterized in sufficient detail to make meaningful calculations with CSQ; because of this, the properties of another commercial explosive (Unigel Dynamite) were assumed. Based on comparisons of those properties which are

Figure III C-1

Upper Graph: Density variations deduced from logs (dashed curve) and assumed for CSQ calculations (solid line segments) for the oil shale bed near the Rock Springs Site 6A explosive rubblization experiments.

Lower Graphs: Plots of calculated peak pressure levels and measurements as a function of vertical position at radial distances of 5 and 10 meters from the 3.7m high explosive columns. The bottoms of the explosive columns are at zero vertical position.

known for both explosives, it appears that Unigel Dynamite is a reasonable substitute. This was partially substantiated by the fact that calculated post-detonation cavity diameters were essentially equal to measured diameters (~ 33 cm).

The curves on the lower two graphs in Figure III C-1 represent calculated peak pressure levels plotted as a function of vertical position at radial distances of 5 and 10 m from the axis of the explosive column. The calculations for both shale configurations indicate a general increase in peak pressure with vertical position up to the point of maximum pressure, which occurs at an elevation above the top of the explosive column, i.e., above 3.7 m. As the radial distance is increased, the vertical position for the maximum peak pressure becomes greater. At a radial distance of 10 m (lower graph in Figure III C-1), the maxima occurs slightly beyond the right hand extreme of the plot. The calculations for the layered shale configuration (dashed curves), when compared with the calculations for the homogeneous shale, show a tendency for enhanced peak pressure level regions of high density. Comparisons between calculations and measurements however does not suggest an obvious preference for either set of calculations. With two exceptions, the measurements fall below the calculations. Several speculative reasons for this latter observation can be proposed. For example, dispersive effects which are known to be characteristic of stress wave propagation in oil shale are not properly taken into account in the elastic-plastic constitutive formulation. Additional dispersive and attenuative effects would also be introduced with a

more detailed treatment of the layering and by accounting for the porosity which undoubtedly is present to at least some degree in the oil shale.

The CSQ calculations also showed a large departure from a spherical stress field at the 5 and 10 m radial distances. This is evident in Figure III C-2 where calculated peak values for the maximum and minimum principal stress amplitudes are plotted for the homogeneous shale configuration. Also plotted are the corresponding curves for pressure as well as the measurements. In general, the maximum principal stress component lies along the wave front propagation direction; the minimum stress component then lies in a plane tangent to the wave front. Results similar to those plotted in Figure III C-2 were also obtained for the layered shale configuration. Some effort is currently being expended to determine whether or not gage orientation relative to the direction of the transient stress can influence gage response. Prior use of the gages has been in weak or water saturated earth media where departures from spherical stress field are insignificant.

D. Computer Simulation of Borehole Explosive Detonations (M. E. Kipp, 5162, and E. G. Young, 5163)

The computer simulations of borehole explosive detonations performed this quarter with the TOODY Lagrangian wavecode are of two major kinds. The first kind has a restriction placed upon the oil shale that the fracture be suppressed. That is, although regions exist where the critical tensile stress associated with fracture has been exceeded, no material degradation occurs to

Figure III C-2

Graphs illustrating departures from a spherical stress field for the homogeneous oil shale calculation of the Rock Springs Site 6A experiment.

influence the wave propagation characteristics of the material. The second kind of calculation has employed the crack nucleation and growth model, BFRACT, to determine the fractured regions. In this model, the fractures degrade material strength, and thus alters material response. Comparisons of the results of the two type of calculations show significant differences in stress histories for material in proximity of the explosive column.

The initial explosive detonation in the fracture suppressed calculation increased the nominal hole diameter from 0.2 m to 0.44 m. Peak pressures from the calculations can be compared with field measurement obtained at selected points around the explosive. For example, at a radial distance of 5 m from the axis of the explosive, the calculated peak pressure is 51 MPa as compared to a measurement of 39 MPa; again at 5 m, but at a different vertical position, the calculated peak is 10 MPa and the measurement is 5 MPa; at 10 m, the calculated peak is 11 MPa while the measurement is 4 MPa. The success in being able to calculate the propagation of a wave to a radial distance of 10 m with reasonable resolution is encouraging. Calculated fracture-producing stresses, however, occur within a 5 m radius, where numerical resolution is well known. In the initial fracture suppressed calculation, locations of tensile stresses that exceeded the critical level for failure were marked; these are plotted in Figure III D-1. The stress component primarily responsible for the failure is the circumferential (or hoop stress) stress. It is clear from the figure that there are two distinct, unconnected regions of concentrated failure.

SPRINGS SITE 04 - FRACTURE SUPPRESSED
TIME 0701776-02 SEC. CYCLE 1230 E VS. X

Figure III D-1

The results of the initial fracture suppressed calculation was used as an initial configuration, with new explosive emplaced in the expanded hole, to simulate a second detonation. The previously noted failures do not disperse the new pressure wave because no material degradation was permitted in the initial calculation. The larger quantity of explosive in the second calculation resulted in new failure regions. These are denoted by the s's in Figure III D-2; the failures from the first calculation are denoted by the p's. The new stress waves form tensile regions that are further away from the borehole than before, but still with two distinct regions of concentrated failure. The "fractured" regions now extend past 4 m. The nominal hole diameter has been increased to 0.94 m.

Fracture parameters became available for BFRACT this quarter, so a calculation of the same event just discussed could be made with a model in which the fractures affect wave propagation. The information of interest was obtained from Stanford Research Institute and modified to fit Sandia spallation data. The modifications were necessary to increase the range of validity of the as-received parameters. The initial detonation produced the fracture region seen in Figure III D-3. When compared to Figure III D-1, one notes that now the fractured regions have joined, and some fractures extend to 4 m from the hole. We observed that the fractures initially formed in similar positions in both types of calculations; however, as the material lost strength in the BFRACT calculation, the energy was dissipated locally, so some fractures seen in Figure III D-1 are not evident in Figure III D-3. The additional fractures in the BFRACT calculation occur because tensions develop

Figure III D-2

Figure III D-3

in regions which have been weakened.

As an example of an effect of fracture, Figure III D-4 depicts stress histories at a monitoring point near the borehole. One profile was obtained in the calculation with fracture suppressed and the other in the BFRACT calculations; in the latter case the stress is reduced as the material fractures and loses its strength. The stress plotted is the circumferential stress, the main driving component for the failure model in this geometry.

Figure III D-4

Distribution:

Mr. L. M. Burman
Oil, Gas and Shale Technology
U. S. ERDA Fossil Energy
20 Massachusetts Ave. NW
Washington, DC 20545

Harry C. Carpenter
H. B. Jensen
Robert Kerr
Andrew Long
R. E. Paulson
R. L. Wise
Laramie Energy Research Center
P. O. Box 3395
University Station
Laramie, Wyoming 82071

W. J. Carter, M-6
Los Alamos Scientific Laboratory
Los Alamos, New Mexico 87544

Mr. Hugh Guthrie, Director
Oil, Gas and Shale Technology
U. S. ERDA Fossil Energy
20 Massachusetts Ave. NW
Washington, DC 20545

Ronald Hofmann
Science Applications Inc.
8201 Capwell Drive
Oakland, CA 94621

A. E. Lewis
Lawrence Livermore Laboratory
University of California
P. O. Box 808
Livermore, CA 94550

Mr. Jerry Ramsey
Oil, Gas and Shale Technology
U. S. ERDA Fossil Energy
20 Massachusetts Ave. NW
Washington, DC 20545

Mr. H. E. Thomas
U. S. ERDA Fossil Energy
20 Massachusetts Ave. NW
Washington, DC 20545

Dr. Phillip C. White
Assistant Administrator
U. S. ERDA Fossil Energy
20 Massachusetts Ave. NW
Washington, DC 20545

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for ERDA/TIC (25)
Attn: R. P. Campbell, 3171-1