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Structural Deformation of Green River Oil Shale as It Relates to In Situ Retorting

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STRUCTURAL DEFORMATION OF GREEN RIVER OIL SHALE AS IT RELATES TO IN SITU RETORTING

by

P. R. Tisot[1] and H. W. Sohns[2]

ABSTRACT

Structural response and/or deformation was determined for each of four different grade Green River oil shales (27.0-, 34.5-, 45.5-, and 63.5-gal/ton) as they were heated in an inert atmosphere, under constant compressive stress, from ambient to subretorting temperatures; that is, below those required for rapid pyrolysis of the organic matter. Small cores and small columns of confined fragments comprised the test specimens, which were heated, in a specially designed microunit, at a uniform rate either to 725° or to 825° F under three different stress levels, 80, 200, and 325 psi. Structural response of some cores was observed as they were heated in a stress-free environment. The obtained stress-strain-time-temperature relationships provide information regarding the oil shale's yield temperature, yield stress, rate of compressive strain, loss of mechanical strength, and nature of structural deformation. Effects of structural deformation on induced permeability in small columns of fragments are presented. Results indicate that underground retorting of Green River oil shale may be seriously impaired through loss of induced permeability brought about by structural deformation of rich oil shale under the influence of heat and compressive stress.

INTRODUCTION

Considerable worldwide research has been conducted on aboveground retorting of oil shale, and many retorts have been developed, some to commercial or semicommercial scale (5-6, 10-11, 20).[3] Underground processing, which in recent years has been given increasing consideration in the United States, is a second approach for shale oil recovery. The objectives of this approach are fourfold: (1) To utilize oil shale not readily processed by more conventional methods, (2) to eliminate mining, transporting, and crushing of the raw oil shale, (3) to eliminate disposing of the spent rock, and (4) to preserve the natural environment and surface ecology.

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[1] Research chemist.

[2] Research supervisor.

[3] Underlined numbers in parentheses refer to items in the list of references at the end of this report.

Green River oil shale is a highly consolidated rock composed of a complex mixture of organic and inorganic constituents in variable proportions. Because much of the rock formation is considered impervious for practical purposes, present oil-shale technology requires that permeability be induced in the process area prior to retorting. One possible method to induce permeability within the formation is to fracture oil shale between previously drilled wells. These fractures, in turn, provide flow paths for both the injected gases and the products (4, 13). The Bureau of Mines is presently conducting field tests at Rock Springs, Wyo., to evaluate the feasibility of this method. Thus far, field tests have included hydraulic fracturing, electrolinking, explosive fracturing using conventional explosives, and/or a combination of these techniques (1-3, 12-13).

Another way to induce permeability would be to use a nuclear explosion to create a large columnar mass of broken rock or rubble, commonly referred to as a nuclear chimney. The Atomic Energy Commission has conducted numerous contained nuclear explosions in tuff, alluvium, dolomite, granite, and salt. As yet no nuclear explosion has been conducted in oil shale. However, predictions have been made of the geometric configuration, size, and quantity of fragmented oil shale that may be expected from a nuclear explosion (7-9, 14). The rubble column's bulk porosity has been calculated to be 25 to 30 percent (7); therefore, its induced permeability should be high.

Fundamental engineering data need to be developed to understand and assess the feasibility of designs for underground retorting using either approach to induce permeability. Many problems, not critical to aboveground retorting, need to be considered for which few or no data based on previous experience exist. One such problem, the basis of this report, is the support capability of a column of rubble during retorting. One method considered for retorting oil shale in a nuclear chimney is downward flow; that is, advancement of a retorting zone from top to bottom of a rubble column (possibly several hundred to 1,000 feet tall). As the retorting zone advances, compressive stress at the interface between retorting zone and hot oil shale immediately ahead increases with depth as shown later in table 1. The question arises, will the hot oil shale immediately ahead of the retorting zone withstand the compressive stress incurred throughout the length of the rubble column, or will it undergo extensive structural deformation at certain depths and seriously impair induced permeability? If the latter should occur, it may impose a limit on chimney height.

Raw oil shale of all grades has high compressive strength (17), many times that required to withstand the compressive stresses in a nuclear chimney. This may not be the case with oil shales because they are progressively heated under compressive stresses incurred in a column of rubble. For intelligent engineering design it is important to know the structural behavior of oil shale, particularly in the retorting zone and in the hot unretorted zone immediately ahead.

A laboratory investigation was conducted to evaluate stress-strain-time-temperature relationships of small cores and of small columns of confined fragments prepared from four different grades of oil shales which varied from

27.0 to 63.5 gal/ton. The primary objectives of this research were the following: (1) To observe the sequence of events which occurred as the small specimens were progressively heated, under constant compressive stress, from ambient to subretorting temperatures (that is, below those required for rapid pyrolysis of the organic matter to produce oil); (2) to determine the extent of structural deformation in the specimens, both under different stress levels and in a stress-free environment; and (3) to determine if structural deformation seriously impaired induced permeability in small columns of confined fragments.

EXPERIMENTAL WORK

Specimen Preparation

A possible area for a nuclear explosion is the lower oil-shale zone in Colorado's Piceance Creek Basin. A histogram showing oil yield and weight-percent organic matter for two 100-ft intervals within this zone is presented in figure 1 (16).

Because appropriate size samples from this zone were not available, the samples used in this investigation were selected from the Mahogany Zone in the Bureau of Mines experimental mine near Rifle, Colo. The four oil shales selected assayed (to the nearest one-half gallon) 27.0, 34.5, 45.5, and 63.5 gal/ton. Assuming that the oil shales from this source are comparable, grade for grade, to those which occur in Colorado's Piceance Creek Basin, the experimental data should be applicable to oil shales from both areas. Approximately 90 percent of the oil shale shown in figure 1 has an oil content which varies from 27.0 to 63.5 gal/ton.

From each of the four oil shales a sample 2 inches thick and approximately 18 inches square was cut with the bedding plane parallel to the long axes. Each sample was carefully selected to insure that the organic matter and the mineral matter were fairly evenly distributed and that it contained no visible fractures. From each of the four samples, small specimens were prepared as cores, 3/4 in in diameter and 1-1/2 in long, cut perpendicular to the bedding plane. The ends of each core were ground smooth and normal to the core's long axis to insure that the ends evenly contacted the two platens in both the hydraulic tester and the stress-strain apparatus.

Specimens which consisted of small fragments, 3/16 to 3/8 in along their major axes, were also prepared from the four initial samples. These representative samples were cut into thin slices, approximately 1/4 inch thick, and in turn these slices were manually reduced to 3/16- to 3/8-in fragments with a shearing instrument. This technique minimized loss of fine particles which would have occurred had the samples been crushed to pass a 3/8-in screen and the 3/16- to 3/8-in fragments screened therefrom.

Compressive Strength Tests

Compressive strength of the four raw oil shales was determined with a hydraulic compression tester. Four cores, 3/4 in in diameter and 1-1/2 in

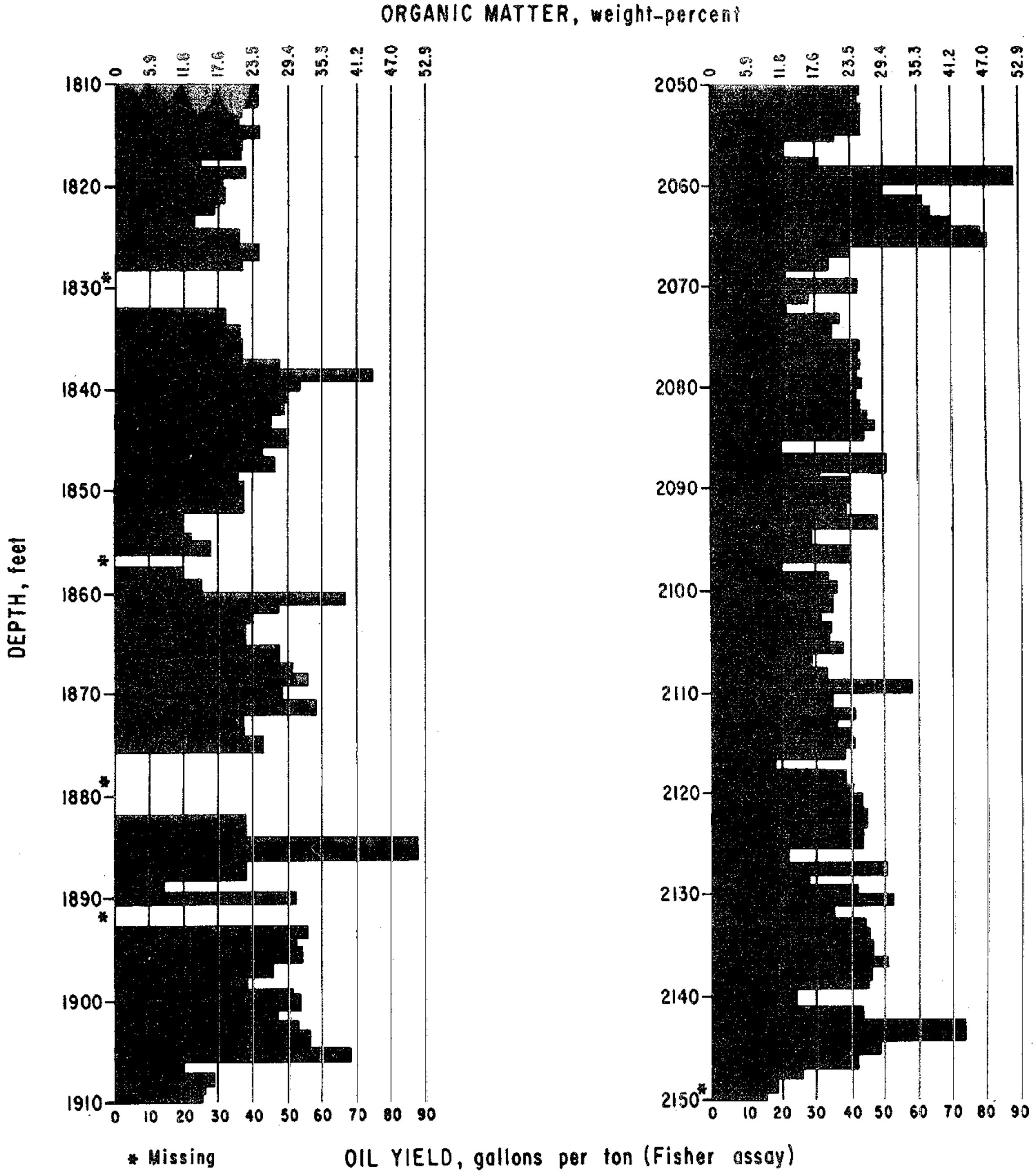

FIGURE 1. - Histogram of Organic Matter and Oil Yield of Typical Lower Oil-Shale Zone in Colorado's Piceance Creek Basin.

long, were individually tested for each oil shale. Each core was uniaxially stressed at a rate of 1,200 to 1,500 psi/min until structural failure occurred. The average compressive strength of the four cores is reported as that of the oil shale tested.

Stress-Strain Apparatus

A stress-strain microunit equipped with an electric heater was designed and fabricated in the laboratory such that compressive stress, temperature, rate of heating, and gaseous environment surrounding the specimen could be controlled. Besides glass wool, part of the insulation around the heating chamber included a double-walled stainless steel cylinder with its annular space highly evacuated. Compressive stress,[4] measured with a proving ring, was transmitted to the specimens through a gas-activated piston. Compressive strain[5] was measured with a dial gage sensitive to 0.0025 mm. The dial gage was attached to the piston resting on top of the specimens. The microunit shown in figure 2 served to study structural response under heat and stress for both the individual cores and the small columns of confined fragments.

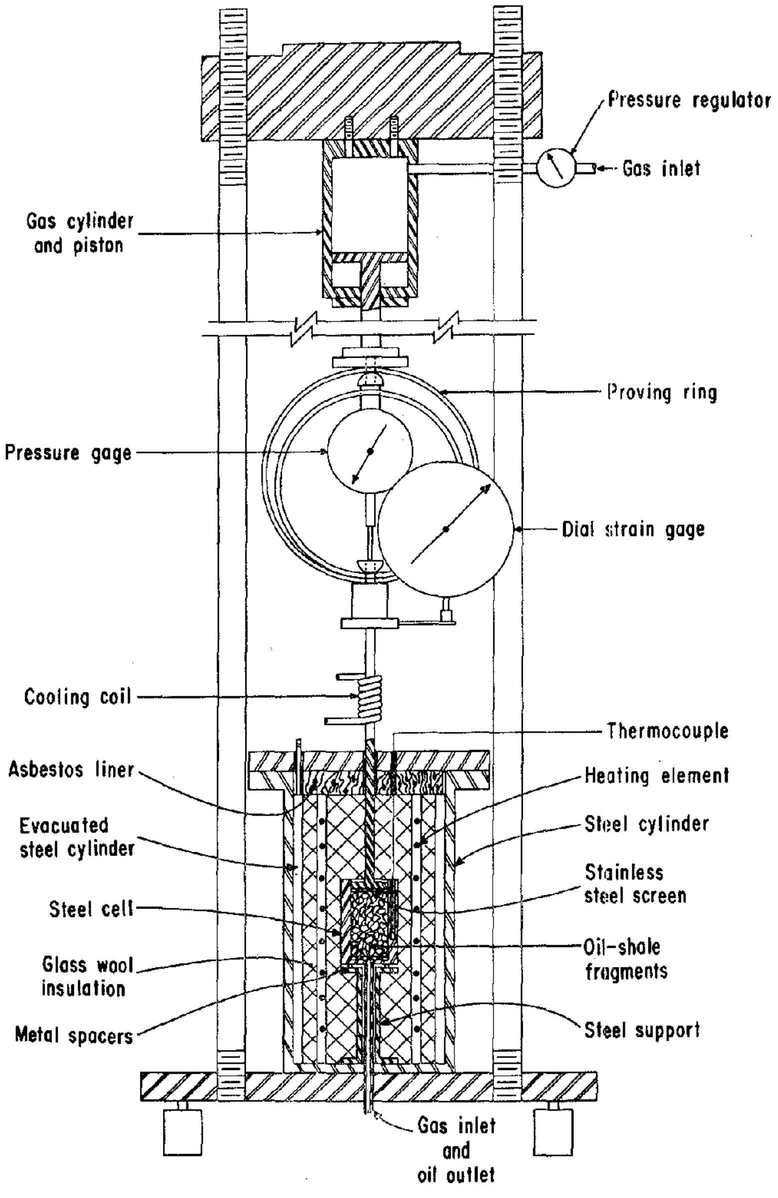

FIGURE 2. - Schematic Diagram of Apparatus for Testing Structural Response of Oil Shale Under Heat and Stress.

When cores were tested, they were placed upright in the apparatus between the piston and the steel support. Temperatures were measured by placing a small thermocouple next to the specimen, midway from top to bottom.

To study structural response of the fragments, the microunit was modified to include a small steel cell, 3/4 in in diameter and 2 in high, which served as a container to confine the column of fragments. The steel cell (fig. 2) had a hollow tube at the bottom which served as a gas inlet. Thin

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[4] Compressive stress is defined as pressure (psi) per unit cross-sectional area.

[5] Compressive strain is defined as percent decrease of initial height.

circular stainless steel screens placed at the bottom and at the top of the column of fragments separated the column from direct contact with the base of the steel cell and the face of the piston. Temperature was measured by inserting a thermocouple in the wall of the steel cell. A sensitive rotameter measured the incoming gas flow through the column of fragments. The gas escaped at the upper end of the column.

Operating Conditions

Many combinations of compressive stress, temperature, and heating rate may be selected to investigate structural response of oil shale to heat and stress. This study was primarily concerned with the structural response of the four different grade oil shales at temperatures below those required for rapid pyrolysis of the organic matter and under compressive stresses calculated to exist at three different depths within a nuclear chimney. Structural response of both the cores and the columns of confined fragments were determined at three stress levels (80, 200, and 325 psi).

Tests were also made to determine the extent of structural deformation as the cores were heated (1) in a stress-free environment, that is, with no restraint imposed on any of their surfaces, and (2) in an environment which restrained movement along the cores' major axes. All specimens were heated from ambient temperature to 725° or to 825° F at a heating rate of 2° F/min.

Treatment of Stress-Free and Restrained Cores

Stress-free and restrained cores were heated to determine structural deformation along their major and minor axes. Eight sets of cores were prepared. Each set contained one core from each of the four oil shales. Two sets of cores were used for each of four tests. No restraint was imposed on the first set, whereas the four cores from the second set were each placed upright in a viselike frame which restrained movement along their axes. These two sets of cores were placed in an electric muffle and then they were heated in an inert atmosphere from ambient temperature to 725° F. When the cores' temperature reached 725° F, they were removed from the electric muffle, cooled, and photographed. The test was repeated with two sets of fresh cores to determine whether structural deformation differed greatly between tests.

The above tests were repeated except that the cores were heated to 825° F to determine the extent of structural deformation which occurred between 725° and 825° F. The cores from these two tests were also photographed.

Treatment of Uniaxially Stressed Cores

Cores from each of the four oil shales were individually tested in the microunit (fig. 2) to observe their structural response to heat and compressive stress. Cores were uniaxially stressed under each of the three loads (80, 200, and 325 psi) before they were heated, and they remained under constant load throughout the tests. Three series of tests were made, each in triplicate. In one series of tests, the cores were heated to determine their yield temperature; that is, the minimum temperature at which the stressed specimens no longer could support the applied load. Once the specimen reached its yield temperature, the test was terminated and the loss of organic matter was determined. In a second series of tests each specimen was heated to 725° F. The temperature was maintained at this level and the time required for the core to undergo structural collapse together with the total loss of organic matter was determined. In a third series of tests the stressed specimens were heated to 725° F, and just prior to structural collapse, stress was progressively reduced to maintain structural configuration. Specimens were then cooled and photographed.

Treatment of Confined Oil-Shale Fragments

The primary purpose for determining structural response of small columns of confined fragments under heat and compressive stress was to determine the effect of structural deformation on induced permeability. Fragments, 3/16 to 3/8 in, were packed at random in a small steel cell, 3/4 in in internal diameter and 2-1/8 in deep, and the resulting column of fragments was then prestressed either to 80, 200, or 325 psi and allowed to equilibrate, which usually required less than 1 minute. If the stressed column was not 2 in deep, stress was relieved, additional fragments were added, and constant stress was reapplied. After a 2-in column (±1/32 in) was obtained, the charged cell was placed in the microunit and constant stress was applied before heating the fragments.

Initial porosity for each column of fragments was determined from the cell's volume (bulk volume) and the oil shale's weight-density ratio (material volume). Because the fine particles, minus 3/16 in, were eliminated, initial intraparticle permeability through each column was high.

Columns of confined fragments were heated either to 725° or to 825° F and then maintained at their respective temperatures for the remainder of the tests. Nitrogen was passed through the column at a slow rate, 0.04 to 0.05 cu in/sq in of cross-sectional area per minute. This flow rate was maintained during each test except when permeability measurements were made or when permeability through the column was lost owing to compressive strain. As permeability became impaired because of compressive strain, a gas pressure differential of either 3 or 14.7 psi, depending on the amount of permeability lost, was imposed periodically across the column and the column's permeability was measured and recorded. Following each permeability measurement, initial flow of nitrogen was restored. Compressive stress measurements were taken and recorded every 15 minutes or oftener throughout each test.

Compressive Stress at Retorting Level

As retorting zones advance in nuclear chimneys, it is likely that compressive stresses at the respective interfaces between retorting zone and hot oil shale immediately ahead will differ at equal depths between rubble columns. These differences, if they should exist, could be attributed to a combination of circumstances derived from two sources--the inherent properties of the initial rubble column, and the state of the processed zone overhead. In the former, the grade of oil shale and the amount of induced porosity affect stress.

Factors which could influence stress at different levels in the processed zone include the following: (1) The presence of raw or partly retorted oil shale or both, (2) the amount of carbonaceous matter deposited on the mineral constituents, and (3) the extent of mineral carbonate decomposition. There is, however, no established information as to whether or not some of these factors might occur in the processed zone, or in what combination and extent they might occur, or how they may vary with depth.

Table 1 presents calculated compressive stresses at successive 100-foot intervals to a depth of 800 feet for each of the four oil shales. Calculations were based on the following assumptions: (1) That the oil shale within the 800-ft section was of uniform grade, and (2) that the nuclear explosion induced 27.5 percent porosity, which was fairly evenly distributed throughout the rubble column. These data are presented to associate depth within a rubble column with the three stress levels used in this study. For example, compressive stress of 200 psi (calculated according to table 1) would occur at approximately the 300-ft level in a rubble column of 34.5-gal/ton raw oil shale and at the 400-ft level after the oil shale was retorted.

TABLE 1. - Compressive stresses at successive 100-foot intervals, psi

Grade and state of oil shale	Depth, feet							
	100	200	300	400	500	600	700	800
27.0-gal/ton:								
Raw oil shale, unbroken column............	95	190	285	380	475	570	665	760
Raw oil shale, rubble column[1].............	69	138	207	276	344	413	482	551
Retorted oil shale[1] [2].....................	58	116	174	232	291	349	407	465
Retorted oil shale, mineral carbonate free[1]	45	89	134	179	224	269	313	358
34.5-gal/ton:								
Raw oil shale, unbroken column............	90	180	270	360	450	540	630	720
Raw oil shale, rubble column[1].............	65	131	196	261	326	392	457	522
Retorted oil shale[1] [2].....................	52	104	157	209	261	313	365	418
Retorted oil shale, mineral carbonate free[1]	41	80	121	161	202	241	282	322
45.5-gal/ton:								
Raw oil shale, unbroken column............	84	168	252	336	420	504	588	672
Raw oil shale, rubble column[1].............	61	122	183	244	305	365	426	487
Retorted oil shale[1] [2].....................	45	90	134	179	224	269	314	359
Retorted oil shale, mineral carbonate free[1]	35	70	104	138	173	207	242	277
63.5-gal/ton:								
Raw oil shale, unbroken column............	75	150	225	300	375	450	525	600
Raw oil shale, rubble column[1].............	54	109	163	218	272	326	381	435
Retorted oil shale[1] [2].....................	34	69	103	138	172	206	241	275
Retorted oil shale, mineral carbonate free[1]	26	53	79	106	133	159	186	212

[1] Initial induced porosity of 27.5 percent.
[2] Oil shale free of organic matter.

RESULTS AND DISCUSSION

Compressive Strength of Raw Oil Shales

Compressive strengths, perpendicular to the bedding plane of the four raw oil shales (27.0-, 34.5-, 45.5-, and 63.5-gal/ton) were, to the nearest 50 psi, 13,500, 11,300, 10,000, and 9,850 psi, respectively. These values, about twice those of high-strength concrete, far exceed the support capability required to withstand the stresses incurred in a nuclear chimney. Compressive strengths, either perpendicular or parallel to the bedding plane, do not differ greatly in uniformly consolidated oil shales (17).

Organic content had a marked effect on the mode of structural failure. Rich oil shale exhibited elastic properties, whereas low-grade oil shale behaved as a brittle material. Specimens from the richest oil shale averaged 14.5 percent compressive strain prior to structural collapse by fragmentation. As the specimens were undergoing deformation they stored elastic strain energy. When stress was relieved, prior to structural collapse, strain was reversible as the specimens exhibited gradual elastic rebound; with time they regained almost 100 percent of their initial height. From the time the specimens were initially stressed to the time of structural collapse, no audible sounds were noted. In contrast, specimens from the low-grade oil shale exhibited no pronounced strain. Structural failure occurred through brittle rupture which progressed very rapidly, and once started could not be stopped. Failure was accompanied by a loud explosive noise. Structural failure in all four oil shales was attributed to a conical-type fracture.

Response of Stress-Free Cores to Heat

Structural deformation, either constrictive or expansive, is not easily induced in the initial rock by mechanical means. However, most oil shales in a stress-free environment will undergo expansive deformation simply by application of heat. Deformation, a function of both organic content and temperature, becomes extensive in rich oil shales, whereas in lean oil shales, which yield less than 12 gal/ton, deformation is minor (17).

Figures 3B and 3C illustrate structural deformation in the four oil shales after they were heated stress free in an inert atmosphere to 725° and to 825° F. These photographs clearly show the extent of deformation when compared with the untreated specimens shown in figure 3A. Considerable deformation occurred after the specimen had reached 725° F. In all instances, deformation occurred perpendicular to the bedding plane with no significant deformation along the specimens' minor axes. Many fractures shown in the photographs were visible and quite pronounced by the time the specimens reached 350° to 450° F. The precursors of these fractures were likely flaws or microcracks present in the raw oil shales or minute tension fractures which developed at ambient temperatures after the oil shales were removed from their initial environment, or combinations of both. As temperature increased the microfractures enlarged and propagated along their major axes until either a free surface was reached or their progress was blocked. Once the microfractures were formed, other factors, besides heating, that probably influenced their growth were swelling of the organic matter and formation of gas within the specimens.

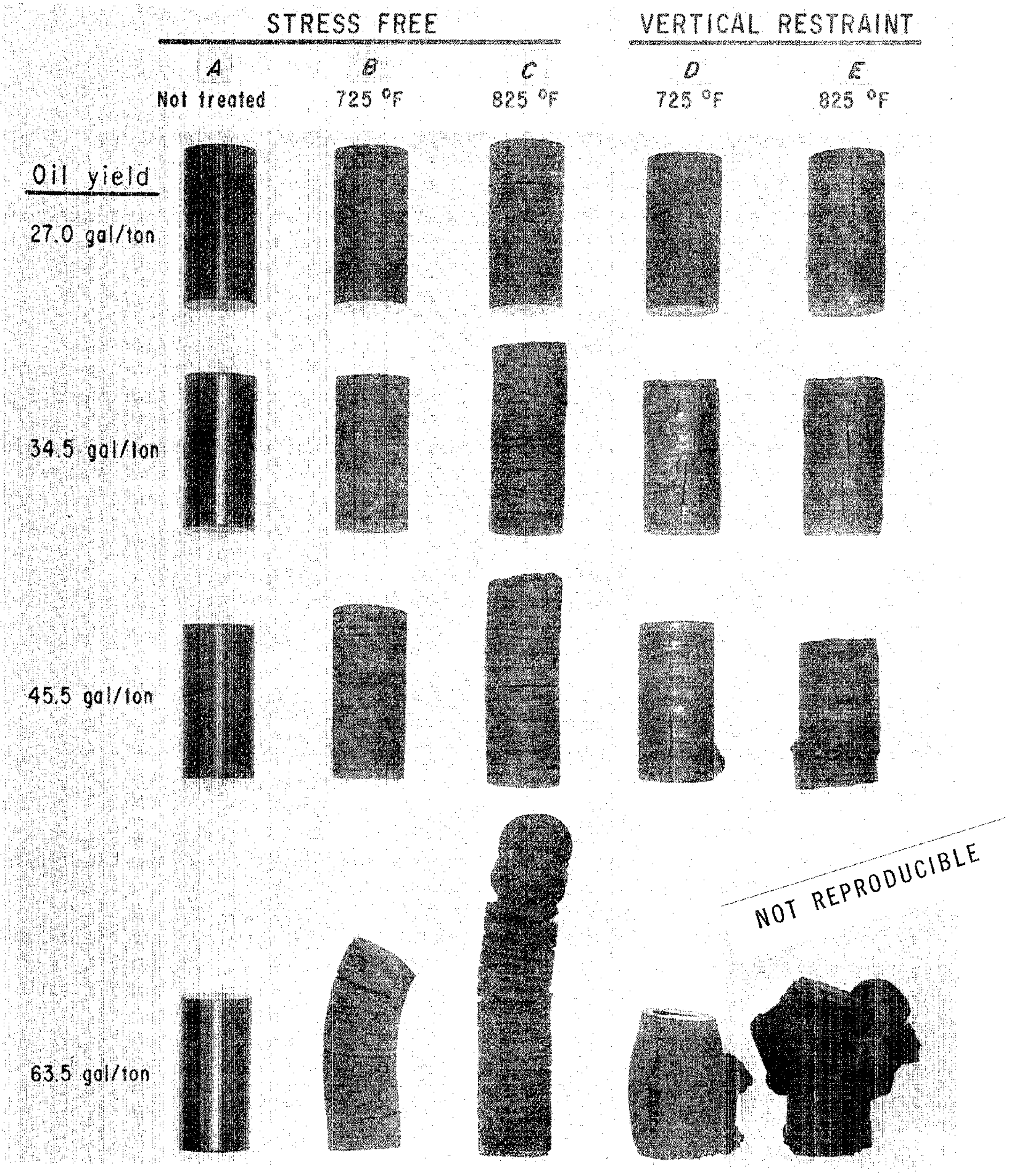

FIGURE 3. - Structural Response of Oil-Shale Cores to Heat in an Inert Atmosphere.

Figures 3D and 3E illustrate structural response to heat where movement perpendicular to the bedding plane was restrained. Although stress free initially, these specimens incurred increasing compressive stress in their attempt to expand under the influence of heat. At some temperature, compressive stress in each specimen exceeded its mechanical strength and structural failure ensued in all four oil shales. During these tests two distinct events occurred below 725° F. First, as compressive stress exceeded each specimen's mechanical strength, fractures developed perpendicular to the bedding plane. The number of vertical fractures appeared to be a function of organic content--the richer the oil shale, the greater the number of fractures. Some fractures extended the entire length of the specimen. Secondly, the specimens from the two richest oil shales exhibited viscous thermoplastic flow (figs. 3D and 3E). As heating was continued, these specimens became so pliable that they began to deform under their own weight. The 45.5-gal/ton oil shale (fig. 3E) probably represents the borderline where some free flow can be expected to begin in stressed oil shales. Free flow in the 63.5-gal/ton oil shale heated to 825° F was so extensive that the specimens failed to remain upright in the viselike frame. Where free flow occurred, the mineral constituents moved freely along with the organic matter.

Extensive structural breakdown in rich oil shales, brought about by heat alone, points to mechanically weak mineral matrices. This form of structural behavior indicates the following: (1) The mineral constituents are loosely bound, (2) the organic matter is the continuous phase, and (3) the properties of rich oil shales are primarily those of the organic matter. Volume-percents of organic matter in the four oil shales, calculated according to Smith (15), were 32.3, 39.6, 48.5, and 60.0 percent, respectively. Equal volumes of organic and mineral matter occur in a 48.5-gal/ton oil shale.

Structural breakdown or fracturing at subretorting temperatures, which in effect is a form of size reduction ahead of the retorting zone, should be an asset to retorting large pieces since this behavior provides new entrances for hot gases. If oil shale in a column of rubble behaves similarly to the small specimens, fractures due to heat alone can be expected to occur in oil shales which yield as low as 25 gal of oil per ton. The following surface-creating events, or combination of events, may occur depending on depth: (1) Rubble with free surfaces exposed to void spaces could fracture and expand, particularly if the free surfaces are perpendicular to the bedding plane, as illustrated in figures 3B and 3C; (2) fracturing may be induced in hot oil shales when the overburden stresses exceed their mechanical strength, as illustrated in figures 3D and 3E; and (3) free flow will likely occur in very rich oil shales to increase surface area, as shown by the behavior of the 63.5-gal/ton oil shale (fig. 3E).

Response of Cores to Heat and Uniaxial Stress

Yield temperatures of the specimens from the four oil shales heated under uniaxial compressive stress (80, 200, and 325 psi) are presented in table 2. These values denote the temperatures at which the specimens failed to support the applied load and they began to undergo compressive strain. In most tests the yield temperatures for a given oil shale could be repeated within

1° to 3° F from the averages given in table 2. All yield temperatures are considerably below those required for rapid pyrolysis of the organic matter, 825° to 900° F. Loss of organic matter ranged from 3.5 to 7.5 weight-percent of the specimens' initial content. These losses were probably bitumen (benzene-soluble material) instead of thermally degraded kerogen.

TABLE 2. - Yield temperatures of uniaxially stressed cores, ° F

Stress, psi	Core grade, gal/ton			
	27.0	34.5	45.5	63.5
80.............	750	737	727	721
200.............	713	709	704	686
325.............	698	690	685	658

When uniaxial stress was maintained on the specimens at their yield temperature, they would, in time, undergo structural collapse. Instead of using the yield temperature as the reference temperature to determine elapsed time to structural collapse, fresh specimens from the four oil shales were all heated to 725° F under the three stress levels, 80, 200, and 325 psi. In all tests the uniaxially stressed cores, except for the specimens from the 27.0-gal/ton oil shale stressed to 80 and 200 psi, sustained the applied loads for only short periods of time before they underwent structural collapse. Figure 4 shows the configuration of these cores just prior to structural collapse by fragmentation.

All specimens from the 27.0-gal/ton oil shale sustained stress levels of 80 and 200 psi for 48 hours without structural collapse except for one core that collapsed after 24 hours at 200 psi. This core is shown in figure 4C. Specimens that did not collapse, however, underwent structural breakdown as fractures developed parallel to the main axis of stress. Some fractures extended nearly the full length of the core as noted in figures 4B and 4C. The specimens which did not collapse at either 80 or 200 psi had undergone compressive strain equivalent to 2.5 and 4.3 percent of their respective initial height. All specimens that were stressed to 325 psi collapsed by fragmentation within 2 to 4 hours after they reached 725° F, with an average loss of 25.6 weight-percent organic matter.

The three rich oil shales behaved quite differently under heat and uniaxial stress than did the 27.0-gal/ton oil shale. Immediately after they reached their yield temperatures, compressive strain was slow; however, it accelerated with time. As the specimens began to compress, many minute fractures developed parallel to the stress axis. These fractures, in turn, propagated and enlarged as cohesive forces were overcome. Finally, compressive strain became very rapid and the specimens collapsed by fragmentation.

At the yield temperature the specimens were pliable and they could easily be sliced with a knife either parallel or perpendicular to their bedding plane. Each specimen exhibited considerable elastic rebound over a wide temperature range. This elastic rebound was determined by repeatedly reducing and

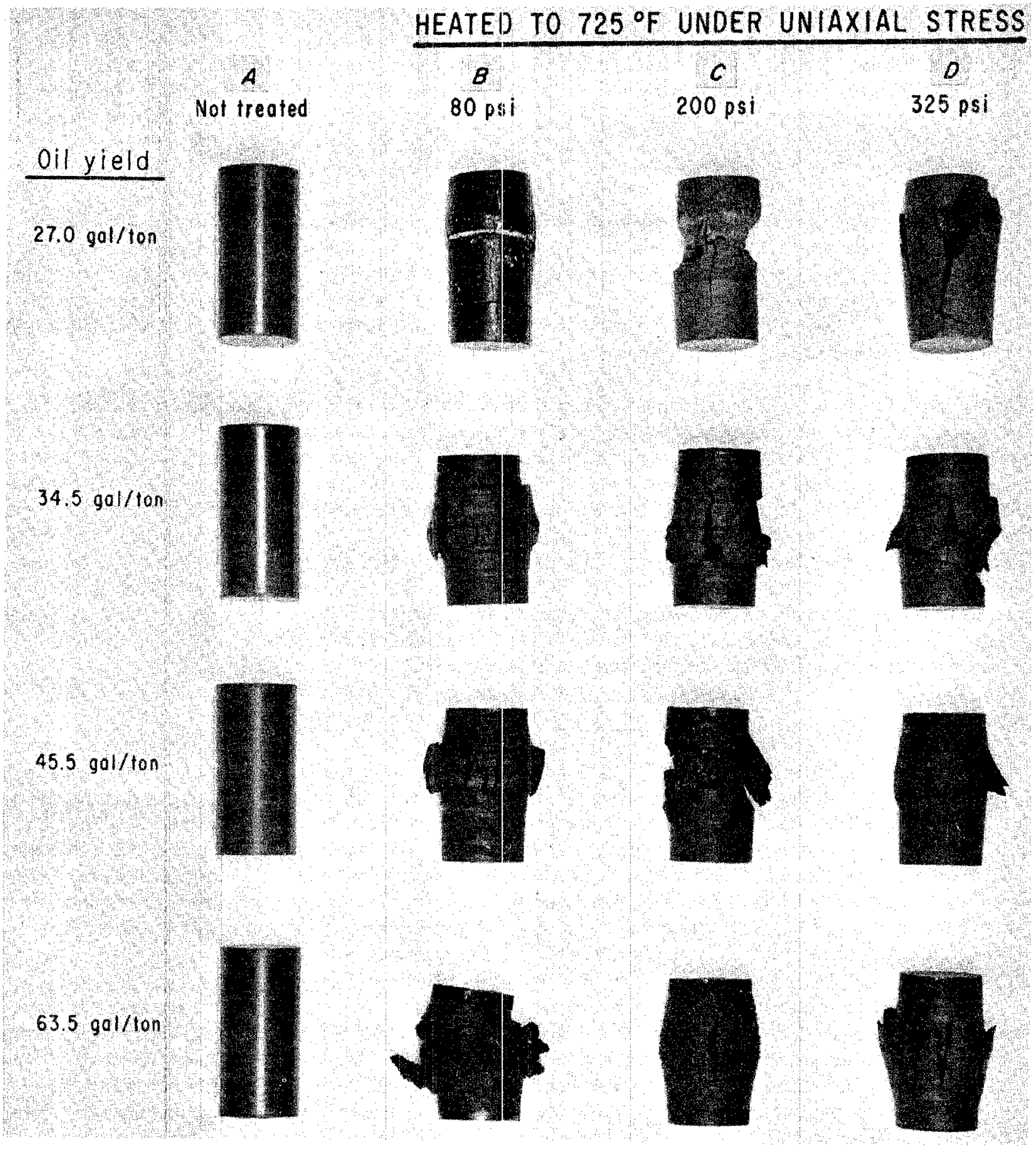

FIGURE 4. - Structural Response of Oil-Shale Cores to Heat and Compressive Stress.

NOT REPRODUCIBLE

reapplying uniaxial stress. The specimens appeared to exhibit simultaneously, in varying degrees, elastic and viscoelastic properties along with plastic deformation. Cores which were cooled to ambient temperature after they reached their yield temperatures, regained considerable mechanical strength with corresponding loss of pliability and resilience. After structural collapse the fragments also withstood considerable stress without deformation when cooled, indicating again that the organic matter lost its pliability.

The maximum and minimum time intervals to structural collapse after the specimens reached 725° F were as follows: (1) Two hours and 15 minutes for the 34.5-gal/ton oil shale stressed to 80 psi and (2) 12 minutes for the richest oil shale stressed to 325 psi. Respective losses of organic matter were 22.3 and 11.6 weight-percent. These losses indicate no extensive pyrolysis of the organic matter.

The following factors strongly indicate that the organic matter is both the continuous phase and the predominant contributor to rich oil shales' mechanical properties and structural response to heat and stress: (1) The large differences in compressive strengths between the oil shales at ambient temperature and at 725° F (about 10,000 to less than 325 psi); (2) the ease with which the specimens could be sliced at 725° F, either perpendicular or parallel to the bedding plane; (3) the fairly high degree of elastic rebound over a wide temperature range; (4) the extensive structural breakdown in a stress-free environment owing to heat, as illustrated in figure 3; and (5) the high recovery of mechanical strength after specimens heated to their yield temperature are cooled to ambient temperature.

Response of Fragments to Heat and Stress

The confined fragments' structural response to heat and stress is presented in compressive strain-time curves (figs. 5-9). Specimens in figures 5 through 8 were heated to 725° F and those in figure 9 were heated to 825° F. In addition to compressive strain-time plots, figures 6, 7, 8, and 9B through 9D include permeability time plots which show the effect of compressive strain on the three rich oil shales' induced permeability. Each figure also presents a plot of the thermal treatment given the specimen, the applied stress, and the induced porosity for each column of fragments.

Once the columns of fragments at ambient temperature reached equilibrium with compressive stress, compressive strain became negligible with respect to time. However, as heating commenced, compressive strain (in most tests) began within the first hour and at temperatures below 150° F. Strain continued until the temperature reached 350° to 400° F at which time the columns of fragments ceased to yield and they resisted the applied stress as noted by the onset of the first plateau in the compressive strain-time curves (figs. 5-9). Strain which occurred prior to each plateau, a function of both organic content of the fragments and applied stress, varied from essentially zero in the 27.0-gal/ton oil shale stressed to 80 psi (fig. 5A) to 10 percent of the initial column height in the 63.5-gal/ton oil shale stressed to 325 psi (fig. 8C). Stress that occurred at low temperatures likely resulted from reorientation of the fragments and not to any appreciable loss in the individual fragments'

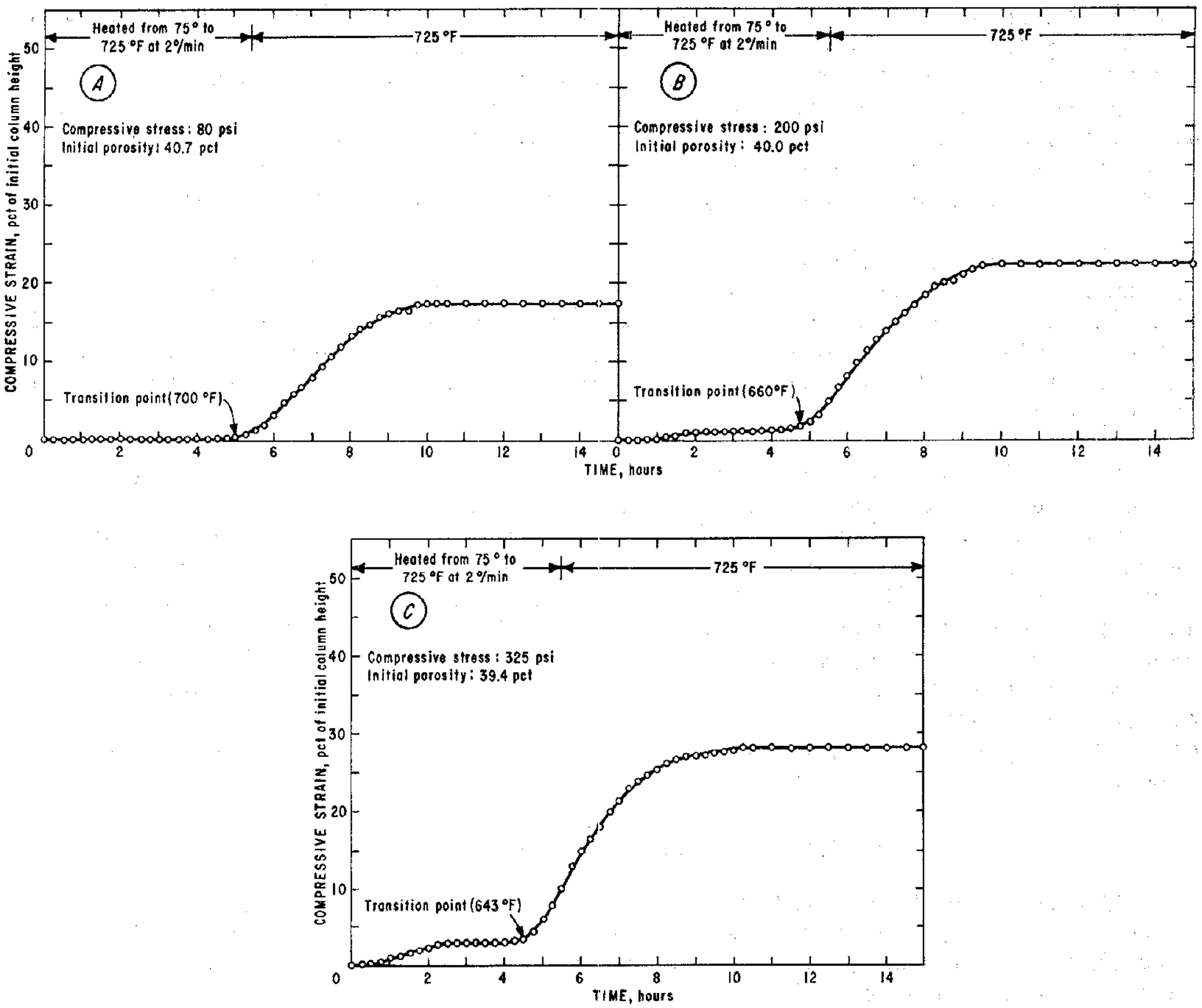

FIGURE 5. - Compressive Strain-Time Curves for 27.0-Gal/Ton Oil-Shale Fragments Heated to 725° F.

mechanical strength. A plausible reason for reorientation at low temperatures may be that as the bitumen and kerogen became warm they served as lubricants to reduce the initial coefficient of friction between the fragments' points of contact, which resulted in slippage, reorientation, and partial compaction. Swelling may also have influenced reorientation.

Following onset of each plateau, the columns of fragments sustained the applied stresses from 2 to 3 hours, at which time the fragments reached their yield temperature or transition point where they again failed to support the applied stress. At this point the rate of compressive strain increased rapidly as noted by the slopes of the compressive strain-time curves (figs. 5-9). The well-defined transition points reflect a pronounced change or changes in the oil shales' properties. At this point ductility or toughness changed abruptly. Yield temperature, a function of organic content and of applied

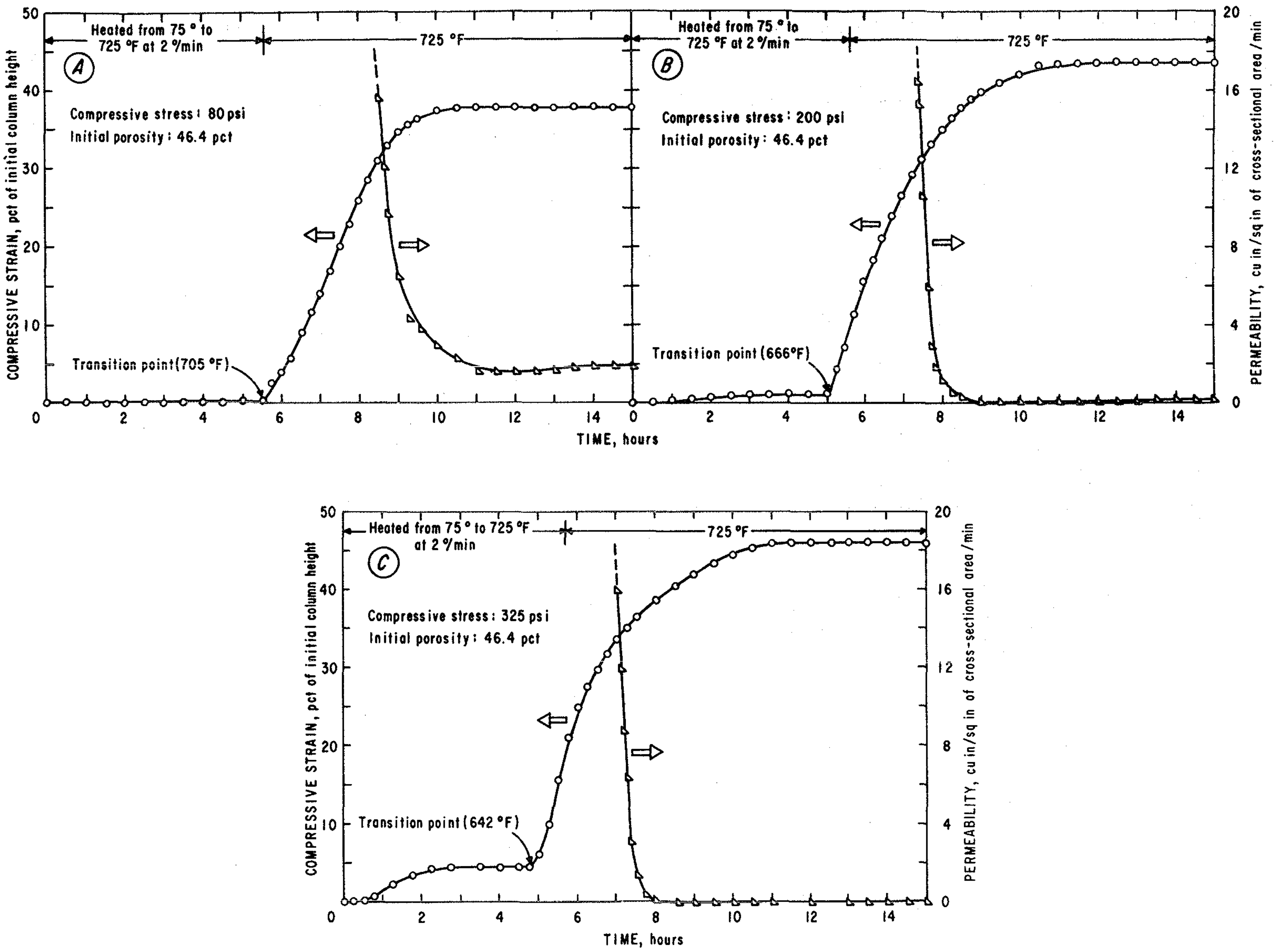

FIGURE 6. - Compressive Strain-Time and Permeability-Time Curves for 34.5-Gal/Ton Oil-Shale Fragments Heated to 725° F.

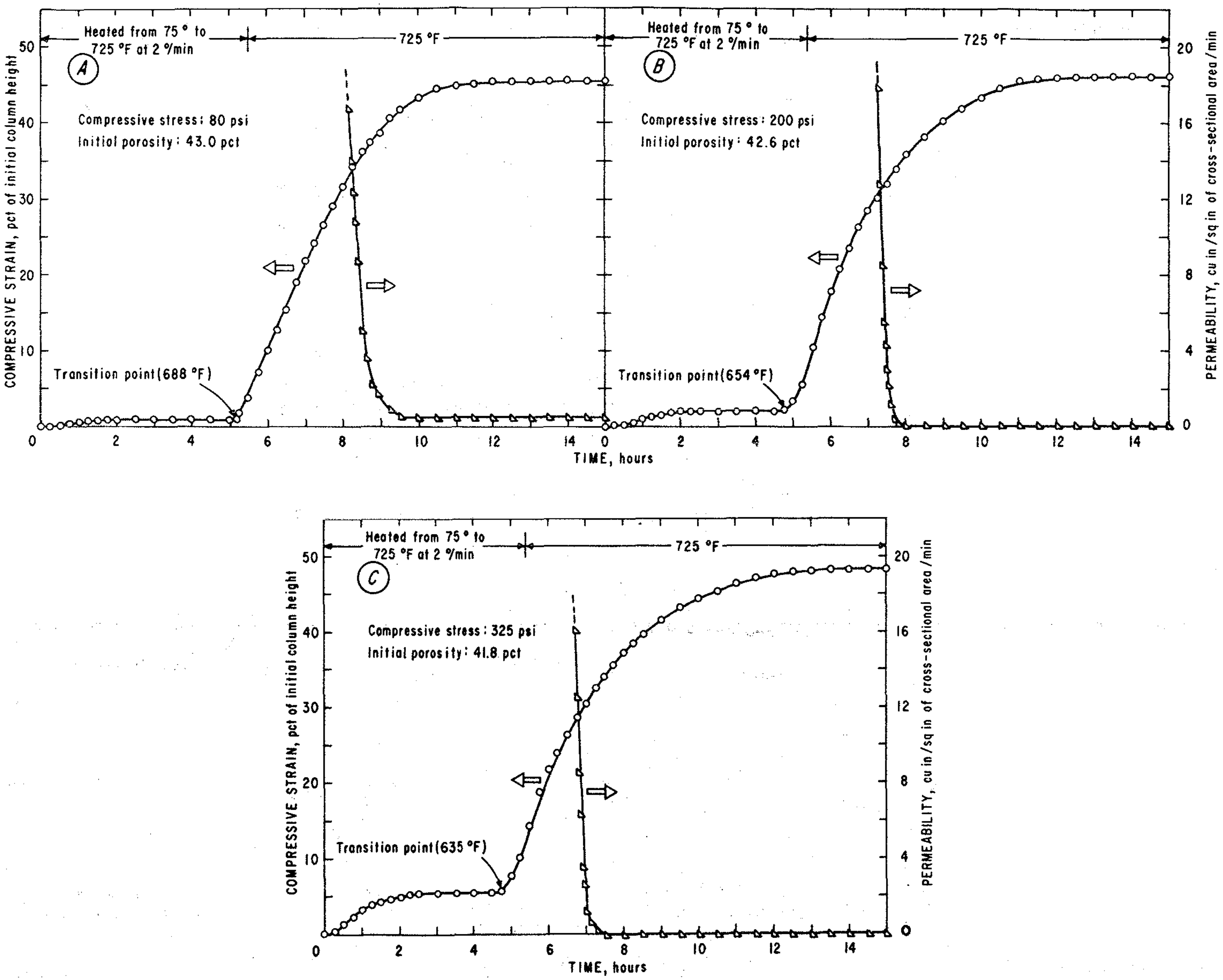

FIGURE 7. - Compressive Strain-Time and Permeability-Time Curves for 45.5-Gal/Ton Oil-Shale Fragments Heated to 725° F.

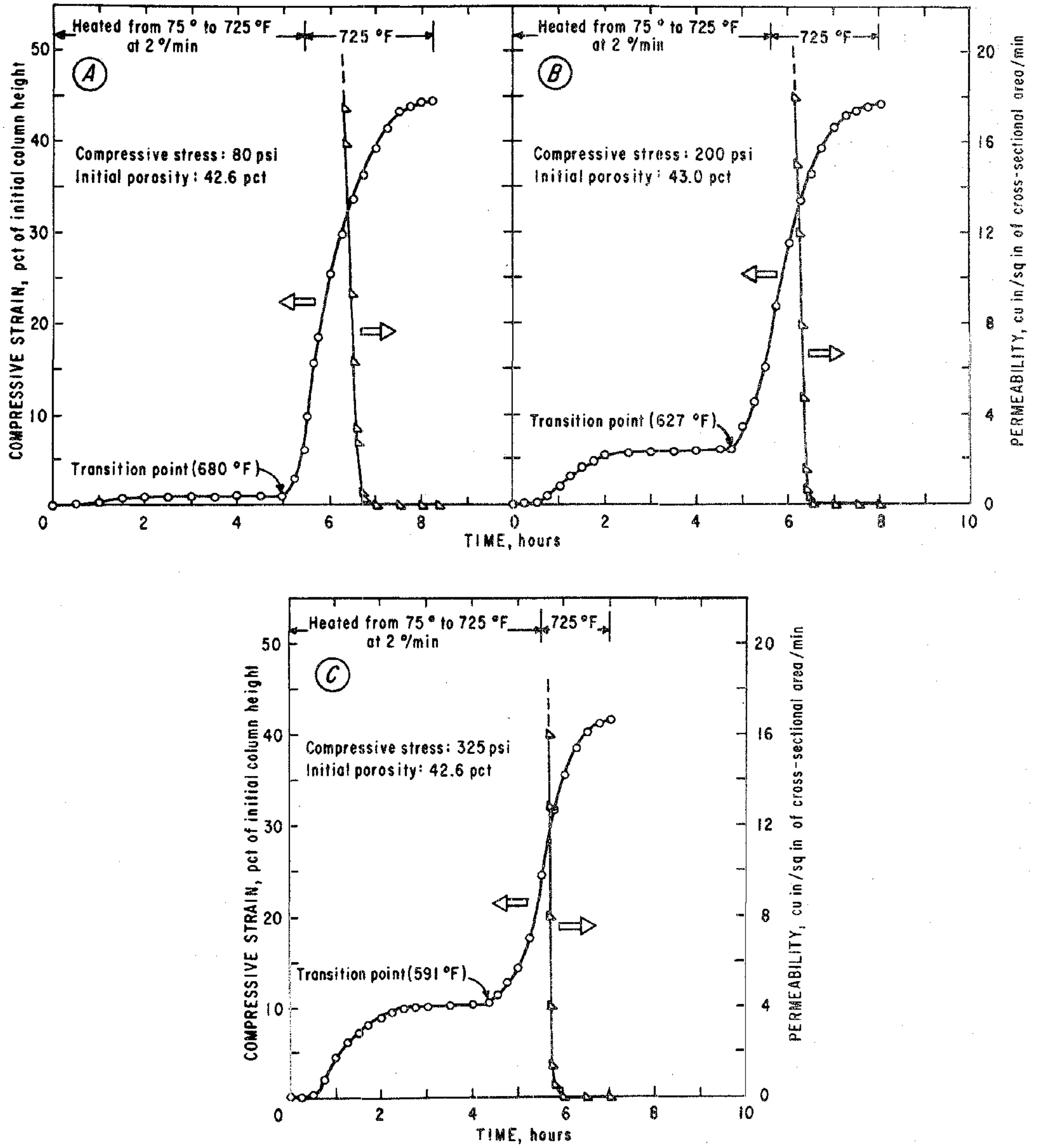

FIGURE 8. - Compressive Strain-Time and Permeability-Time Curves for 63.5-Gal/Ton Oil-Shale Fragments Heated to 725° F.

stress, ranged from 700° F in the 27.0-gal/ton oil shale that was stressed to 80 psi (fig. 5A) down to 591° F in the 27.0-gal/ton oil shale that was stressed to 325 psi (fig. 8C). These two temperatures are well below those

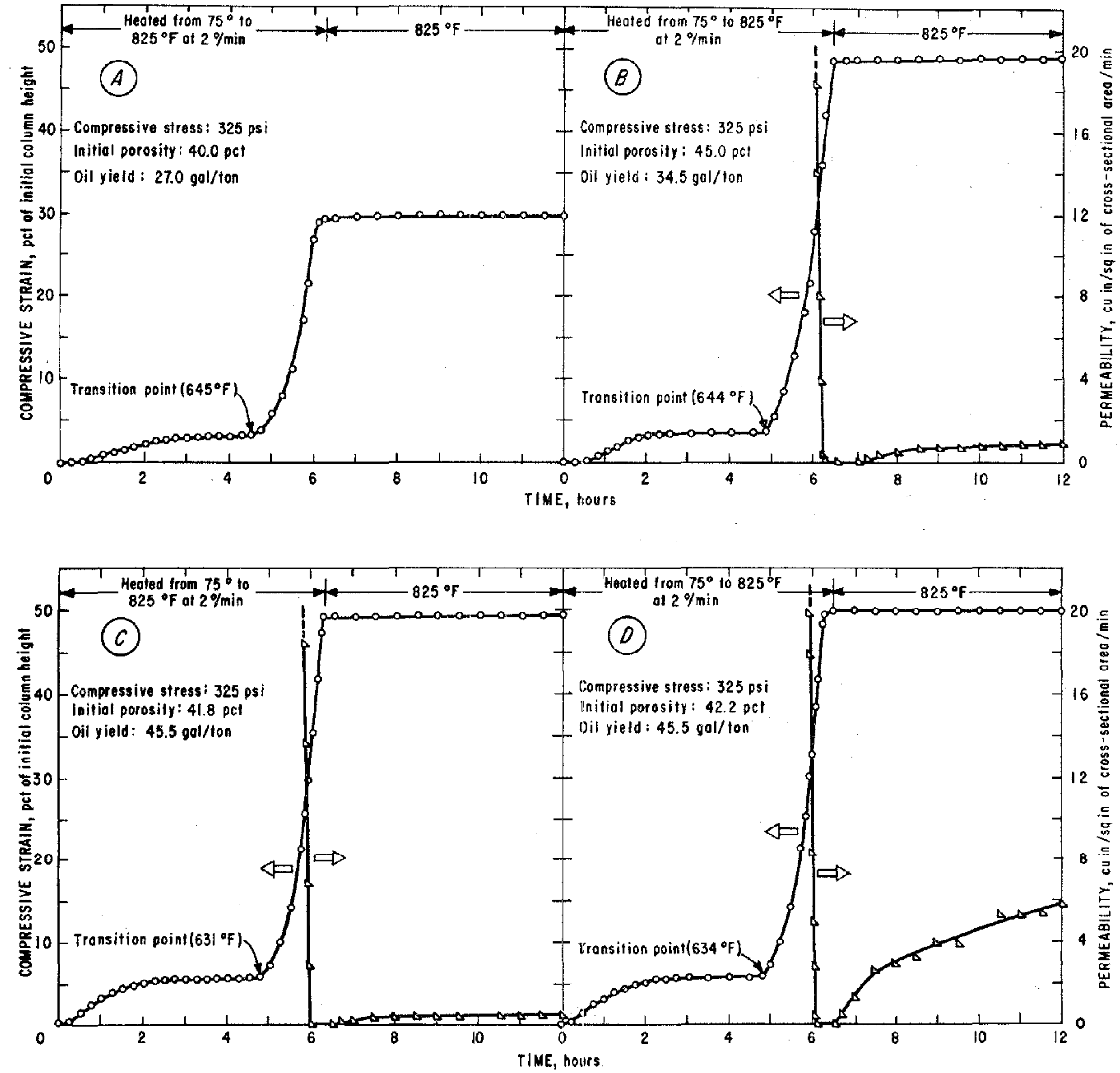

FIGURE 9. - Compressive Strain-Time Curve for 27.0-Gal/Ton Oil Shale and Compressive Strain-Time Plus Permeability-Time Curves for 34.5- and 45.5-Gal/Ton Oil-Shale Fragments Heated to 825° F.

required for rapid pyrolysis. The following three factors strongly suggest that the phenomenon which occurred at each transition point was primarily due to physical rather than chemical changes: (1) the small loss of organic matter; (2) the pliability of the hot specimens; and (3) the high recovery of mechanical strength once the specimens are removed from their yield-temperature environment and again cooled to ambient temperature. These factors indicated that kerogen degradation had not become extensive at the specimens' yield temperature. On repeated tests, yield temperatures for one set of operating conditions were within a narrow range, especially for the rich oil shales. Repeated tests using fresh specimens gave yield temperatures within 2° to 3° F from the averages.

As noted by the compressive strain-time curves, all specimens, once they reached their transition point, underwent rapid compression (figs. 5-9). Comparison of the respective strain-time curves for the specimens heated to 725° F under a load of 325 psi (figs. 5C, 6C, and 7C) with those heated to 825° F under the same load (figs. 9A-9D) clearly show the occurrence of three events with respect to the higher temperature:

1. After the transition point was reached, the rate of compressive strain was much faster.

2. The time required for maximum strain to occur, after the transition point, was greatly reduced (6 to 9 hours for the specimens heated to 725° F as compared to only 1-1/2 hours for those heated to 825° F).

3. The maximum strain occurred essentially simultaneous with maximum temperature in the specimens heated to 825° F. In contrast, strain continued to occur at a rapid rate for several hours after the specimens in figures 5C, 6C, and 7C reached their maximum temperature of 725° F.

The stressed confined fragments, prior to extensive kerogen degradation, exhibited elastic rebound and viscoelastic properties similar to those of the stressed cores. Maximum strain, for the specimens from the first three oil shales heated to 725° F during the 15-hour tests, was obtained with the 45.5-gal/ton oil shale stressed to 325 psi (48.2 percent of the initial column height, fig. 7C). On heating to 825° F, under the same stress level, strain increased an additional 2 percent (fig. 9D).

Structural response of the 63.5-gal/ton oil shale was such that all tests had to be terminated in less than 8-1/2 hours; therefore, comparison with the strain data obtained for the three oil shales stressed for 15 hours could not be made. It was expected that the 63.5-gal/ton oil shale should have undergone the highest compressive strain for the following reasons: (1) It contained the highest amount of organic matter (60 volume-percent); (2) its equally dispersed and loosely bound mineral particles contributed no significant mechanical strength which, in turn, presumably permitted the fragments to deform and completely occupy the induced porosity; and (3) it should have undergone the highest strain through greater loss of organic matter by degradation had the tests lasted for 15 hours.

As noted in figure 8, strain in the specimens from the 63.5-gal/ton oil shale increased very rapidly after they reached their respective transition points. Depending on stress, rapid strain continued for 2 to 3 hours during which time the confined fragments were continually undergoing deformation and compaction with progressive elimination of the columns' induced porosity. Much of the strain occurred after the specimen reached 725° F. At the time the tests were terminated, porosity appeared to be completely eliminated. Comparison of initial porosity with compressive strain data (fig. 8) supports this observation. After complete loss of porosity, a second plateau appeared in the strain-time curves which indicated that the oil shale would again support the applied loads. However, within less than 1 hour from onset of the second plateau, each specimen failed to support its respective load, and both organic matter and mineral constituents extruded almost instantly past the piston and the cell wall (clearance about 1 mm). Loss of organic matter at this point, which was stress dependent, ranged from 28.9 to 34.4 weight-percent of the initial amount. Sudden collapse of the specimens after porosity was eliminated, suggesting that the kerogen had undergone a second transition point, one of conversion from a semisolid (or viscoelastic) material to a viscous fluid. Time required for total collapse after the specimens reached 725° F was stress dependent, 2 hours and 50 minutes and 1 hour and 40 minutes for the specimens stressed to 80 and to 325 psi, respectively (figs. 8_A_ and 8_C_). The strain-time curves suggest that once porosity was eliminated, an induction period of about 30 minutes was required before the hot stressed kerogen underwent rapid conversion from a semisolid to a viscous fluid.

Over 95 weight-percent of the mineral particles in a very rich oil shale had equivalent spherical diameters that were less than 50 microns (_18_-_19_). Therefore, the mineral constituents could readily flow concurrently with the organic matter through the 1-mm clearance cited above.

The long plateaus at the upper region of each compressive strain-time curve (figs. 5-7, and 9) indicate that the specimens heated to 725° and 825° F had reached their maximum strain with respect to time.

Effect of Heat, Stress, and Time on Permeability

As compressive strain increased with time, the columns' pore geometry, porosity, and permeability were concurrently undergoing change. Permeability-time curves for the columns of confined fragments prepared from the 34.5-, 45.5-, and 63.5-gal/ton oil shales are presented in figures 6, 7, 8, and 9_B_ through 9_D_. Permeability is expressed as the amount of nitrogen (stp) through the column of fragments in cubic inches per square inch of cross-sectional area per minute at a gas pressure differential across the column of 3 psi, except in figure 9_D_ where the pressure differential across the column was 14.7 psi. Permeabilities which exceeded 20 cu in/sq in of cross-sectional area were not measured. In most tests, permeability was not seriously impaired until the column of fragments had compressed at least 35 percent of its initial height.

Fragments From 27.0-Gal/Ton Oil Shale

After reaching their transition points, the columns of confined fragments from the 27.0-gal/ton oil shale underwent considerable compressive strain, 17.5, 22.5, and 28.1 percent of their initial column height at 80, 200, and 325 psi, respectively (fig. 5). However, many fragments, although they had reoriented and adhered to each other at the higher stress levels, retained essentially their initial geometric configuration at the end of the tests. No permeability-time curves are presented in figure 5 because throughout all tests permeability exceeded 20 cu in/sq in of cross-sectional area per minute at a gas pressure differential across the column of 3 psi.

Under the most rigorous treatment (825° F and 325 psi), the column of fragments compressed 29.8 percent of its initial height at the end of 12 hours (fig. 9A). Its initial porosity of 40 volume-percent was reduced to 14.7 volume-percent. Extent of compression and loss of porosity obtained with the 27.0-gal/ton oil shale was higher than expected considering that its mineral content occupied 67.7 volume-percent. The specimen was completely retorted. Loss of organic matter was 72.9 weight-percent of the original amount.

Fragments From 34.5-Gal/Ton Oil Shale

Permeability in the specimens stressed to 80 and to 200 psi remained above zero during the 15-hour tests (figs. 6A and 6B). Minimum permeabilities, 1.70 and 0.06 cu in/sq in of cross-sectional area per minute, were reached at the time the specimens had compressed 37.8 and 42.0 percent, respectively (figs. 6A and 6B). At the end of the tests, permeability showed a slight increase. Following the 15-hour test, nitrogen was replaced with air at a gas pressure differential across the column of 3 psi. The existing permeabilities allowed for complete oxidation of the carbonaceous matter within 3 and 9-1/2 hours, respectively. These times were approximated by determining how long it took the column to cool to 725° F, the temperature prior to air injection. Some fragments that were stressed to 80 psi, although they had undergone reorientation, retained essentially their initial geometric configuration during the test.

Fragments that were stressed to 325 psi (fig. 6C) lost their permeability after 8 hours at which time the column had compressed 40.5 percent of its initial height. This test was continued beyond the 15 hours shown in the graph. Sixteen hours after permeability had reduced to zero, flow through the column had recovered to 0.12 cu in/sq in of cross-sectional area per minute. At this point nitrogen was replaced with air at a gas pressure differential across the column of 3 psi and the test continued an additional 12 hours. At the end of the 36-hour test, the carbonaceous matter appeared to have undergone no significant oxidation and permeability had again reduced to zero.

Fragments From 45.5-Gal/Ton Oil Shale

Permeability in the column of fragments stressed to 80 psi (fig. 7A) reached a minimum, 0.43 cu in/sq in of cross-sectional area per minute, at the time the column had compressed 42.0 percent of its initial height. This minimum occurred 9-1/2 hours from the beginning of the test or 3-3/4 hours after the specimen had reached 725° F. Once permeability reached its minimum, it remained almost constant for the following 5-1/2 hours. At this time nitrogen was replaced with air at a gas pressure differential across the column of 3 psi. The existing permeability was sufficient for complete oxidation of the carbonaceous matter.

Permeability in the column of fragments that were stressed to 200 psi (fig. 7B) reduced to zero after the column had compressed 35.2 percent, 8 hours from the beginning of the test or 2-1/2 hours after the specimen reached 725° F. During the next 7 hours permeability remained at zero. A gas pressure differential of 3 psi, applied across the column for an additional

16 hours, failed to restore any flow. Pressure across the column was then increased to 14.7 psi. After an additional 12 hours (36 hours from beginning of test) some permeability was restored, 0.90 cu in/sq in of cross-sectional area per minute.

Permeability in the column of fragments stressed to 325 psi (fig. 7C) reduced to zero after the column had compressed 34.5 percent, which occurred 7-1/2 hours from beginning of the test or 2 hours after the specimen reached 725° F. Permeability was not restored at the end of the 15-hour test nor after the gas pressure differential across the column was raised to 14.7 psi.

In one test, other than that shown in figure 7C, a gas pressure differential of 14.7 psi was applied across the column immediately after permeability reduced to zero. Some flow was restored within 4 hours and after 14 hours permeability had increased to 1.00 cu in/sq in of cross-sectional area per minute. Nitrogen was then replaced with air at the same pressure and within several hours the carbonaceous matter was completely oxidized.

Fragments From 63.5-Gal/Ton Oil Shale

After the specimens reached 725° F, permeability reduced to zero at all three stress levels (fig. 8), and it could not be restored throughout the remainder of each test at a gas pressure differential across the column of either 3 or 14.7 psi. Table 3 presents data relating to compressive strain, permeability, time required for total structural collapse after reaching 725° F, and loss of organic matter at time of collapse. As pointed out previously, the tests were terminated because of sudden conversion of the kerogen from a semisolid material to a viscous fluid which resulted in total collapse of the specimen followed by extrusion between the piston and the cylinder wall.

TABLE 3. - Experimental data relating to 63.5-gal/ton oil-shale fragments heated to 725° F

Stress, psi	Strain at zero permeability, pct[1]	Time elapsed to zero permeability, pct[2]	Time elapsed to structural collapse, min[2]	Maximum strain, pct[1]	Loss of organic matter, wt pct
80..........	37.5	85	170	44.5	34.4
200..........	36.6	55	140	44.1	33.0
325..........	33.8	22	100	41.5	28.9

[1] Values represent percent of initial column height.
[2] Time measurements began at time specimens reached 725° F.

Fragments From 27.0-, 34.5-, and 45.5-Gal/Ton Oil Shales Heated to 825° F

Except for the 27.0-gal/ton oil shale, permeability-time curves are shown for the columns of fragments from the 34.5- and 45.5-gal/ton oil shales heated to 825° F at a stress level of 325 psi (fig. 9). A permeability-time curve is not shown for the 27.0-gal/ton oil shale (fig. 9A) because nitrogen flow through the column exceeded 20 cu in/sq in of cross-sectional area per minute throughout the 12-hour test. In figures 9B and 9C permeability decreased very rapidly, from 20 cu in/sq in of cross-sectional area per minute to zero within 15 minutes. After permeability reduced to zero, a gas pressure differential of 3 psi was placed across the column. Within 1 hour some permeability was restored.

Figures 9C and 9D represent similar tests differing only in the gas pressure differential across the columns, 3 psi versus 14.7 psi, respectively. At the end of the two tests, permeability was about six times greater at 14.7 psi than at 3 psi. Comparing the respective permeability-time curves for the fragments heated to 825° F (figs. 9C and 9D) with those heated to 725° F under the same stress level (fig. 7C), it is noted that considerable permeability was restored at the higher temperature versus zero at the end of the 15-hour test at 725° F. Permeability also measured zero at the end of the 15-hour test when the 45.5-gal/ton oil shale was stressed to 200 psi (fig. 7B).

SUMMARY AND CONCLUSIONS

Results from this investigation indicate that underground retorting of Green River oil shale may be seriously impaired through loss of induced permeability brought about by structural deformation of rich oil shale under the influence of heat and compressive stress.

Green River oil shale is a two-component system which covers a broad spectrum based on its mineral and organic contents. At lower levels of organic content, the oil shale's properties are essentially those of its mineral matrix which forms the continuous phase. The mineral particles are tightly bonded together, and the organic-free mineral matrix possesses high mechanical strength (above 10,000 psi in oil shale which yields less than 15 gal/ton) both at ambient temperature and at retorting temperatures, 825° to 950° F. Conversely, towards the upper end of the spectrum the organic matter forms the continuous phase. Here, the amount of mineral matter is greatly reduced, and the mineral particles are loosely bonded together and provide no significant mechanical strength to the oil shale. Therefore, the oil shale's properties approach those of kerogen. Kerogen has high mechanical strength at ambient temperature; however, its mechanical strength is greatly influenced by heat. As kerogen approaches retorting temperatures, it behaves as a viscous fluid under no compressive stress. The ease and extent of structural deformation are essentially inversely proportional to the oil shale's organic content.

Under the conditions of heat and stress studied, all of the uniaxially stressed cores underwent structural collapse by fragmentation when held at 725° F, except for the 27.0-gal/ton oil shale stressed to 80 and 200 psi.

Columns of confined fragments from all four oil shales underwent extensive deformation. In the 34.5- and 45.5-gal/ton oil shales, induced permeability was either seriously impaired or completely lost through structural deformation at a stress level of 200 psi. Permeability was partially restored in some tests after it had reduced to zero. Induced permeability in the 63.5-gal/ton oil shale was also lost through deformation at the three stress levels investigated. When held at 725° F, the stressed oil shale exhibited viscous flow. Both organic and mineral matter extruded past a 1-mm clearance in the test instrument. Loss of organic matter at the time of conversion to a viscous fluid under a stress of 325 psi was 28.9 weight-percent.

The laboratory results indicate that if oil shale in an underground column of rubble, as presently visualized, behaves similarly to the small specimens, structural deformation can be expected in the hot unretorted oil shale immediately ahead of the retorting zone. As the retorting zone advances, deformation will likely occur at the depth where the hot oil shale ceases to sustain the overhead stress. These data indicate, however, that no serious loss of induced permeability, through deformation, should occur in oil shale which yields less than 30 gal/ton.

Additional engineering data are needed to supplement the laboratory findings, particularly the correlation of rubble size with structural deformation. These data are necessary to determine whether the nature and extent of structural deformation under heat and stress are comparable in similar-grade oil shales irrespective of rubble size. The information is essential to determine whether structural behavior of certain grades of oil shales might impose a limit on column height for effective retorting.