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GEOTHERMAL FRACTURE STIMULATION TECHNOLOGY

Volume 2. High Temperature Proppant Testing

By
Maurer Engineering

July 1980

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Republic Geothermal, Incorporated
Santa Fe Springs, California

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GEOTHERMAL FRACTURE STIMULATION TECHNOLOGY

VOLUME II

HIGH TEMPERATURE PROPPANT TESTING

July 1980

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Contract No. DE-AC-32-79AL10563

GEOTHERMAL RESERVOIR WELL STIMULATION PROGRAM

By

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I. SUMMARY AND CONCLUSIONS

Available data in the literature on proppants only gives properties and strengths under triaxial stress at low temperatures. Data in this report was obtained from a newly built proppant tester, operated at actual geothermal temperatures. The short term test results show that most proppants are temperature sensitive, particularly at the higher closure stresses. Long-term effects on the proppants may also be of significance; and long-term tests will be reported in Volume IV to quantify the effects of the geothermal fluids on each type of proppant.

Many materials have been tested using a standard short-term test, i.e., fracture-free sand, bauxite, and a resin-coated sand retained good permeability at the high fluid temperatures in brine over a range of closure stresses. The tests were designed to simulate normal closure stress ranges for geothermal wells which are estimated to be from 2,000 to 6,000 psi. Although the ultra high closure stresses in oil and gas wells need not be considered with our present geothermal resources, there is a definite need for chemically inert proppants that will retain high permeability for long time periods in the high temperature formations.

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II. INTRODUCTION

After 30 years of development and use, hydraulic fracturing is a standard stimulation technique in the oil and gas industry. Proppants are an important aspect of hydraulic fracturing since they help retain fracture conductivity created by the injected high-pressure fluids.

In a hydraulic fracturing treatment, a fluid is injected down the casing or tubing at rates higher than the reservoir matrix will accept. This rapid injection produces a buildup in wellbore pressure until the pressure is large enough to overcome compressive earth stresses and rock tensile strength. At this pressure, failure occurs allowing a crack (fracture) to be formed. Continued fluid injection increases the fracture's length and width.

In order to achieve stimulation, the fracture conductivity (permeability times width) after the well is returned to production must be larger than the reservoir permeability. To obtain high conductivity, a large granular proppant is injected along with the fracturing fluid and deposited in the fracture. This material must be strong enough to maintain a high permeability when subjected to the compressive earth stresses which we call closure stresses. For geothermal wells, the proppant must be inert so that it retains permeability in the high temperature fluid under closure stress.

The amount of stimulation a fracture treatment will produce depends on packed fracture length, reservoir permeability, fracture conductivity, and amount of permeability damage in the formation near the wellbore. Design of an effective fracturing treatment must consider all these factors.

Wells are fractured for two reasons: (1) to increase the rate of fluid production; and (2) to decrease the pressure drop around the wellbore. To predict the production increase from a fracturing treatment, one must know the physical characteristics of the propped fracture, fracture orientation (horizontal, vertical, and azimuth), and the behavior of the proppant under closure stress and temperature.

Well stimulation by fracturing can be achieved either by providing a flow path through a damage zone about the wellbore or by altering the flow pattern in the reservoir. Small-volume fractures overcome wellbore damage and restore productivity to a well by removing the zone of low permeability (or high-pressure drop) from the flow pattern. Large treatments provide a much larger surface for fluid production, thereby stimulating a well by altering the reservoir flow pattern into linear flow.

The key to reservoir stimulation by fracturing is altering the flow pattern in the reservoir from radial to one which approaches linear flow. McGuire and Sikora¹ conducted an analog simulation of the productivity of a vertically fractured well. Their results (Figure 1) are the basis for predicting the stimulation achievable with vertical fractures. Figure 1 shows that the important variables are the ratio of fracture length to drainage radius, L/r_e , and the ratio of fracture conductivity to formation permeability, WK_f/k which is set by proppant permeability.

How proppant is distributed in a fracture and how much is used determines proppant bank geometry. Model studies have shown that proppants in thin fluids form a proppant bank that grows away from the wellbore with time. Under constant flow conditions the bank eventually reaches an equilibrium height where the rate of deposition is in equilibrium with the rate of erosion.² The primary variables in sand

deposition are the ratio of the injection velocity to the equilibrium settling velocity of the proppant, and the fluid velocity above the sandbank at which equilibrium bank height will be reached.

Figure 1. Increases in Production from Vertical Fractures

The first variable can be reduced to an interaction between injection rate into the fracture, density difference between the particle and fracture fluid, and fracturing fluid viscosity. As the density difference decreases or viscosity increases, particle settling velocity decreases and proppant is swept farther down the fracture before being deposited in a bank. This gives a long sandbank that requires a long time to build to its equilibrium height. In the limit of extremely high fluid viscosity or perfect suspending fluid, an equilibrium bank is never reached during the time sand is injected. In

this case, an extremely long sandbank is formed because the sand is held in a slurry. The drawback is that very small fracture widths are created which may decrease fracture conductivity. This happens in very viscous or high gel treatments that are currently used in low temperature oil and gas wells. In general, for geothermal applications thin fluids will be the rule because of cost and the extremely high temperatures that degrade the gelled polymer fluids.

Proppant permeability is extremely important to the effective stimulation by fracturing. Experiments show that proppant permeability in the fracture is a strong function of the type of proppant, stresses placed on the proppant, proppant size, and particle size distribution. Also, the improper use of fluid-loss additives and other plugging mechanisms can be detrimental to the proppants' permeability in the fracture.

Permeability decreases as closure stress increases and also as temperature increases with certain proppants. Data will be presented in the succeeding sections to show the available low temperature data and also data taken on all proppants at geothermal temperatures.

At closure stresses above 10,000 psi, no proppant (except possibly metallic shot or coated or uncoated bauxite) has adequate permeability to effectively stimulate a well when placed as a multilayer. In this situation, it would be desirable to place a partial monolayer of a deformable proppant which would result in a narrow fracture with high porosity and permeability. Treatments that try to use partial monolayers have not been very successful. However, some successes in the oil and gas industry have been claimed in treatments where an oil-soluble particle is injected along with the proppant and deposited as a multilayer. Subsequent dissolution of these particles, in theory, leaves only the proppant monolayer. Results from these treatments, however, can be explained by other mechanisms as well as partial

monolayer. One explanation is that a multilayered proppant pack is placed with a fracture channel left open above the proppant pack.

In geothermal wells, closure stress should be much lower than the equivalent depth oil and gas wells. The actual value of closure stress will vary, but they should be on the order of the water gradient which is about 0.5 psi/ft or from 2,000 to 6,000 psi in wells from 4,000 to 12,000 ft in depth.

III. TYPE OF PROPPANTS

A. Historical Proppants

While there are many materials available for use as a hydraulic fracturing proppant, only sand has been widely used over the last 30 years. The reason is its widespread availability and low cost. Also, sand is fairly easy to store and grade into several different mesh sizes.

Several years ago, glass beads appeared on the market and were widely and unquestionably accepted. However, their beautiful appearance and perfect roundness was found to have its drawbacks since the roundness caused stress concentrations and internal stresses caused catastrophic failure in water and brine at elevated closure stress and temperature. Recent publications⁶ show that the supposed benefit of glass beads at higher closure stresses did not exist. Because of the published test data and their premium price, glass beads have essentially been withdrawn from the market.

One other proppant that has been used is walnut hulls. In low temperature shallow wells, walnut hulls would hold up for awhile; but as the formation temperatures increased, the walnut hull would continue to deform and eventually lose conductivity in the fracture. Today, walnut hulls are used for fluid loss control during drilling but not as proppants.

B. Research on Proppants

Research began on proppants as early as people realized that hydraulic fracturing was going to be successful. In the 1950s, sand, glass beads, and walnut hulls were tested and considered. During the 1960s, in-depth studies were made on many materials that exhibited the desired properties of high strength under load.

Deformability under load was considered to be an important advantage in a proppant. While walnut hulls were deformable under low temperatures and load, they did not work at higher temperatures or loads. In 1961, Kern, Perkins, and Wyant of Atlantic Richfield published a paper³ on aluminum particles as proppants. They claimed that aluminum pellets worked better than sand; however, because of cost, availability, and corrosion, the industry never used aluminum pellets widely.

Recently, high-strength materials have dominated the research efforts on proppants. Many kinds of metal pellets^{3,4} were tested. Glass beads were being used to a much larger extent and metallized coatings⁵ were considered on glass beads. Mined garnet was considered and many types of high-strength plastics, nylon and fiber glass reinforced materials were tested.⁵ Several had very good results in certain situations; but for general use there were too many drawbacks and disadvantages.

During the 1970s, the industry got into high gear and turned out several new proppants for high-closure stress fracturing operations. The following list shows a product and the originating and/or manufacturing companies.

Table 1
1970-1979 Proppants

<u>Product</u>	<u>Origination</u>	<u>Manufacturer</u>
Fluidized Coke ⁷	Exxon Research	- - - - -
Uncured Resin Coated Sand ⁸	Exxon Research	Santrol Products, Inc.
Sintered Bauxite ⁹	Exxon Research	Norton and Carborundum
Ceramic Oxide ¹⁰	Union Carbide	Union Carbide
Zirconium Oxide ¹¹	Institut Francais Du Petrole	- - - - -

Also, much more attention began to be paid to where the proppant went in the fracture. Computer models of proppant transport,¹² technical papers^{2,13} and films¹⁴ were made to illustrate the travels and accumulation of proppant in the fracture. These were used to help maximize the results from a particular fluid system and/or proppant. Work by Daneshy¹² and Prokop and Babcock² are still used today to determine propped length in the fracture and proppant distribution.

C. Properties of Proppant

The main proppant parameters needed for hydraulic fracture design are density, size, strength, deformability and inertness. There are many tests, some of them quite simple, that illustrate these parameters. Density can be easily measured in water using Archimedes' prin-

ciple of fluid displacement. Single grain strength can be measured by a crush test where a load (L) can be withstood by a grain of diameter (D). These points are calculated by the ratio L/D^2 to compare different grain sizes.

Permeability versus closure stress is easily measured in a specially designed linear flow cell (see Appendix) that is pressed between two platens on a heated, hydraulic press. This puts any proppant into a simulated downhole environment which includes the correct temperature, pressure and triaxial stress on the proposed proppant. Temperature and closure stress are the main effects on proppant permeability.

IV. PREVIOUS AND NEW PROPPANT DATA

Much of the previous data on proppant permeability and fracture conductivity has been carried out at low temperatures, many times using gas or air as the "fluid." The test methods vary from company to company but, in general, linear flow cells have been used to measure proppant permeability under varying closure stress. One company routinely uses a radial flow system because of the speed and number of tests that are required. Many companies are now gearing up to do more realistic tests in the actual frac fluids or brines and at actual reservoir conditions. The results are showing temperature sensitivity and destruction of some of the more standard proppants at high temperatures. For example, sand tested in brine at higher temperatures is less permeable than when tested in air or oil. Glass beads disintegrate in high temperature brine under low to medium closure stress.

A. Matrix of Proppant Properties

To more fully explore the higher temperature effects on proppants, Maurer Engineering has set up a modified linear flow cell able to withstand internal pressures up to 1,000 psi. This is equivalent to water temperatures of over 500°F. By using water preheating and a heated Dake Hydraulic Press, actual geothermal reservoir conditions are simulated. While the proppants are being tested, they are monitored by varying flow and closure stress. A complete description and diagram of the Geothermal Proppant Test System is shown in Appendix A of this report.

Most of the tests reported in Volume II on the proppants are short-term tests to screen out the unsuitable proppants. Tests still need to be run to assess long-term effects of proppants used for the field treatments.

The actual data from the tests is shown in the next section; however, a matrix of previously known and new data will quickly summarize the properties of proppants for our use in geothermal well stimulation. The proppants are subjectively arranged by their performances in geothermal triaxial testing with the best suited proppants (regardless of cost) at the top of the matrix and the most unsuitable proppants are down at the bottom of the matrix. Other properties shown are the proppants' physical strength (single grain), solubility, specific gravity, availability and cost. The matrix in Table No. 2 shows the ranking for all 350°F tests of usable proppants in the 20/40 mesh range. Table No. 3 shows all other sizes of particles at 350°F. Table No. 4 shows the matrix of properties of all proppants at 500°F.

TABLE NO. 2
MATRIX OF GEOTHERMAL PROPPANT PROPERTIES*

Geothermal Proppant Type	Permeability Retention Under 5000 psi Load (%)	Solubility in High Temperature Geothermal Water	Specific Gravity
Resin-Coated Bauxite	100%	Slightly Soluble above 500°F Insoluble below 500°F	3.37
Resin-Coated Sand	100%	Slightly Soluble above 500°F Insoluble below 500°F	2.53
Sintered Bauxite	82.5%	Insoluble	3.60
Ottawa Sand	68%	Slightly Soluble Temperature + pH Sensitive	2.65
Brady Sand	34%	Slightly Soluble Temperature + pH Sensitive	2.65
Glass Beads	23%	Slightly Soluble Temperature + pH Sensitive	2.41
Colorado Sand	15%	Slightly Soluble Temperature + pH Sensitive	2.65

*This chart is based on short-term 350°F tests and 20/40 mesh materials.

TABLE NO. 2A
AVAILABILITY/COSTING OF PROPPANTS

Availability	Cost ¢/lb (approx for 1980)	Proppant Type
Special Order: Santrol Products and Service Companies	100	Resin-Coated Bauxite
Excellent: Santrol Products and Service Companies	28	Resin-Coated Sand
Excellent: Norton, Carborundum and General Abrasives and Service Companies	80	Sintered Bauxite
Excellent: Unimin, Bellrose, Ottawa and Service Companies	6	Ottawa Sand
Excellent: PGS, Texas Mining and Service Companies	5+	Brady Sand
No Longer Available Except by Special Order	95	Glass Beads
Excellent: Fountain Sand, and Some Service Companies	5+	Colorado Sand

TABLE NO. 3
MATRIX OF VARIOUS SIZE PROPPANTS AT 350°F

Geothermal Proppant Type	Permeability Retention Under 5000 psi Load (%)	Solubility in High Temperature Geothermal Water	Specific Gravity
Resin-Coated Sand 10/20	100	Slightly Soluble above 500°F; Insoluble below	2.53
Corundum 20/40	100	Insoluble	4.0
Alumina 20/40	100	Insoluble	4.0
Millcreek Sand	85	Soluble in Unsaturated Brine	2.65
Chert 10/20	36.2	Slightly Soluble; pH and Temperature Sensitive	2.65
Ottawa Sand 20/40	36	Slightly Soluble; pH and Temperature Sensitive	2.65
Brady Sand 10/20 Mesh	34	Slightly Soluble; pH and Temperature Sensitive	2.65
Brady Sand 8/12	16	Slightly Soluble; pH and Temperature Sensitive	2.65
Colorado Sand 20/20 and 20/40	8	Slightly Soluble; pH and Temperature Sensitive	2.65

TABLE NO. 4
MATRIX OF ALL PROPPANTS AT 500°F

Permeability Geothermal Proppant Type	Retention Under 5000 psi Load (%)	Solubility in High Temperature Geothermal Water	Specific Gravity
Alumina 20/40	100	Insoluble	4.0
Corundum	100	Insoluble	4.0
Resin-Coated Bauxite 20/40	92	Slightly Soluble above 500°F Insoluble below 500°F	3.37
Resin-Coated Sand 20/40	90	Slightly Soluble above 500°F Insoluble below 500°F	2.53
Sintered Bauxite 20/40	90	Insoluble	3.65
Mill Creek 100 Mesh	72	Soluble in Unsaturated Brine	2.65
Ottawa 20/40	33	Slightly Soluble; pH and Temperature Sensitive	2.65
Brady Sand 20/40 Mesh	27	Slightly Soluble; pH and Temperature Sensitive	2.65
Brady Sand 10/20 Mesh	23	Slightly Soluble; pH and Temperature Sensitive	2.65
Glass Beads	12.5	Slightly Soluble; pH and Temperature Sensitive	2.61
Brady Sand 8/12 Mesh	10	Slightly Soluble; pH and Temperature Sensitive	2.65

B. Physical Strength Properties

The apparent physical strength of each of the proppants is measured by an additional test. This is a less important parameter than those shown on the matrix since multigrain or multipoint contacts can cause early failure and also stress corrosion cracking cannot be adequately simulated as it is in the triaxial (permeability retention) cell. Table No. 5 shows the physical strength of a single grain of all the major proppants at optimum conditions. This value is calculated in pounds by dividing the load to crush by the single particle's diameter squared, (L/D^2) .

TABLE NO. 5
APPROXIMATE PHYSICAL STRENGTH OF A SINGLE GRAIN

Type of Proppant	L/D^2 (lb)	Conditions
Resin-Coated Bauxite	60,000	All tests in air at room temperature except where specified
Resin-Coated Sand	21,500 20,000	~ 350°F 75°F
Sintered Bauxite	21,000	
Ottawa Sand	14,000	
Brady Sand	9,000	
Glass Beads	56,000	
Colorado Sand	8,000	

V. GEOHERMAL PROPPANT TESTING

A. Geothermal Tests on Permeability Retention Under Load

Short-term tests at geothermal temperature and closure stress on the linear flow cell described in Appendix A are presented in this section. The test data starts on Figure 2 with the effects of temperature and closure stress on four types of sand. Then, glass bead data is discussed in Figure 9. Figures 10 through 12 show sintered bauxite, corundum and alumina data, and Figures 13 through Figure 17 present the resin-coated high strength proppants. A discussion and comparison of all the proppants data are made in Figures 18 and 19. Finally, a short discussion of miscellaneous products that were considered for proppants and other miscellaneous data is shown in Figure 20.

1. Sand

a. Brady Frac Sand

The most widely used proppant in oil and gas fracturing is Brady (Texas) sand. Millions of tons of the Brady sand have been used over the past 30 years. It is a well sorted sand, has medium roundness, and has good cleanliness. In testing, it retains high permeability at low temperature and low closure stress. Crushing starts below 4,000 psi at room temperature and begins between 2,000 and 3,000 psi at elevated temperatures. At 10,000 psi closure stress, only a fine powder is left and this can damage a high closure stress well rather than stimulate it. Figures 2, 3, and 4 show the Brady sand permeability retention at 75°F, 350°F and 500°F and illustrate the crushing of the sand at elevated temperature and stress levels. Each of the above figures looks

at a different mesh size. It should be mentioned that 10/20 Brady sand (Figure 3) retains more permeability under load than the larger 8/12 Brady sand. This is because of the weakness and natural flaws or fractures of the larger sand particles.

b. Ottawa Frac Sand

The Ottawa sands come from Illinois, Minnesota, and Wisconsin. It is widely sought after for frac sand use, but until recently it was in limited supply. The Ottawa deposit is a wind blown sand and is much rounder and clearer (fracture free) than Brady sand. Also, the surface is free of iron oxides that commonly appear on river type sand. Although other mesh sizes are now available, the 20/40 mesh has been the most widely used in fracturing operations. Figure 5 shows the behavior of the Ottawa sand at 75°F, 350°F and 500°F. Crushing starts at room temperature between 4,000 and 6,000 psi while elevated temperature tests at 350°F show crushing starting below 4,500 psi and below 3,000 psi at 500°F. This sand appears usable in the lower temperature geothermal wells (below 350°F) when the closure stress remains below 4,000 psi. Long-term site specific tests will give more definite units and exact information on the practicality of using Ottawa sand as a proppant.

c. Colorado Sand

Figures 6 and 7 show the temperature effects on Colorado sand. It is a river type sand with a high feldspar content and low roundness. It crushes easily even at room temperature. Crushing starts at 2,000 psi or less. Larger size sand such as the 10/20 mesh (Figure 7) shows less permeability retention than the 20/40 mesh sand. It will not be recommended for geothermal stimulation work.

d. Millcreek Sand

This fine mesh sand averages 100 mesh and is from Millcreek, Oklahoma. It has long been used for glass making because of its size, purity and roundness. Recently, however, it has been used as a lead-in proppant and fluid loss additive in hydraulic fracturing. It is only slightly temperature sensitive and retains a large percent of permeability under load due to its many support points allowed by the small grain size. Figure 8 shows the Millcreek data which averages between 5 and 10 darcies at all closure stresses.

*K equals X 1000

Figure 2. Permeability Retention for Brady Sand (20/40)

Figure 3. Permeability Retention for Brady Sand (10/20)

Figure 4. Permeability Retention for Brady Sand (8/12)

Figure 5. Permeability Retention for Ottawa Sand (20/40)

Figure 6. Permeability Retention for Colorado Sand (20/40)

Figure 7. Permeability Retention for Colorado Sand (20/40 and Other)

Figure 8. Permeability Retention for Millcreek Sand

2. Glass Beads

Glass beads are a manufactured product and were used in the 1960s and '70s. They are no longer used because of catastrophic failure behavior under combined temperature and load. Figure 9 shows how glass beads respond to load and temperature. Crushing a single bead in air requires a very high pressure; however, in multilayer proppant tests at room temperature (in water or brine) crushing begins at 4,000 to 5,000 psi. At elevated temperatures, crushing begins between 2,000 and 4,000 psi. Failure, when it occurs, is catastrophic or complete. The failure mechanism is brittle failure along with stress corrosion cracking by the high temperature brine which acts as a corrosive fluid and attacks the glass bead. Glass beads are not acceptable for geothermal well stimulation.

Figure 9. Permeability Retention for Glass Beads

3. Sintered Bauxite

During the 1970s, higher strength proppants have been sought. One of the successful types invented by Exxon is sintered bauxite. It is many small particles of bauxite sintered together at high temperature to allow some deformation before crushing. It has a specific gravity of 3.65 versus 2.65 for sand. Figure 10 shows how well it holds up to high temperatures and closure stress. Crushing starts usually above 8,000 psi and is not severe until above 12 to 14,000 psi. While there is no well defined temperature sensitivity, there is some loss of permeability due to loose particle repacking and slight crushing.

It is a suitable proppant for geothermal if economical considerations allow for its high per pound cost and higher specific gravity.

Figure 10. Permeability Retention for Sintered Bauxite

4. Corundum and Alumina

Corundum and alumina are aluminum oxide materials and are quite strong and temperature resistant for a fracturing proppant. The specific gravity of 4.0 is even higher than the sintered bauxite, and some crushing does occur above 10,000 psi. It is less available and in less demand than the sintered bauxite. Figures 11 and 12 show the corundum and alumina proppant tests, respectively.

Figure 11. Permeability Retention for Corundum

Figure 12. Permeability Retention for Alumina

5. Resin-coated Sand

In 1975, Exxon Production Research invented an uncured resin-coated particle for sand control and as a high strength proppant. The uncured resin-coated sand (available as Super Sand) has a deformable coating that polymerizes at elevated temperatures under closure stress to form a cohesive high strength layer of permeable sandstone. It has cohesiveness between grains and shows no tendency to crush or lose permeability up to 10,000 psi closure stress at elevated temperatures. Figure 13 shows the resin-coated sand at 350°F and 500°F. Within the experimental error, the resin-coated sand and sintered bauxite materials are comparable in the 20/40 mesh size. However, tests on larger mesh sizes of the resin-coated sand (Figure 14) show that there is not enough support points or a thick enough resin coating, and above 7,000 psi a permeability decrease is measured.

This is a most suitable and probably the most cost effective proppant for geothermal wells because of its insensitivity to temperature, protective coating of inert resin, and low density.

Figure 13. Permeability Retention for Resin-coated Sand (20/40)

Figure 14. Permeability Retention for Resin-coated Sand (10/20)

6. Resin-coated Sintered Bauxite

Also covered by Exxon's 1975 patent is an uncured resin coating for sintered bauxite. This is the best proppant ever tested and retains a higher permeability than any other proppant of its size tested. While fairly expensive, it would be quite suitable for geothermal applications. Figure 15 shows its 350°F and 500°F behavior and its high retained permeability under load.

a. Cost Comparisons

A comparison of the resin-coated materials is made in Figures 16 and 17. Interestingly, the absolute permeability of the resin-coated sand is about 50 to 60 percent of the resin-coated bauxite regardless of temperature or closure stress. The cost comparison, however, is \$1.00/lb for the resin-coated bauxite versus \$.28/lb for the resin-coated sand. Adjusting for specific gravity differences (3.6 vs. 2.6) the resin-coated sintered bauxite is almost 5 times more expensive. On the basis of cost in cents per lb per darcy in the fracture at 500°F, the resin-coated bauxite costs 0.434¢/lb/darcy at 5,000 psi closure stress (Figure 17). The cost for the resin-coated sand in cents per darcy is only 0.2¢/lb/darcy at the same closure stress and temperature.

Figure 15. Permeability Retention for Resin-coated Sintered Bauxite

Figure 16. Permeability Retention for Resin-coated Proppants (350°F)

Figure 17. Permeability Retention for Resin-coated Proppants (500°F)

7. Available Proppants

Figures 18 and 19 show comparison data of all available geothermal proppants at 350°F and 500°F, respectively. In their order of decreasing permeability at 350°F, the resin-coated bauxite is the highest with about 300 darcies at 5,000 psi stress. Resin-coated sand is next at 180 darcies. Sintered bauxite follows the resin-coated sand at 168 darcies. The Brady sand and glass beads are much lower at 72 and 62 darcies, respectively based on these 4 to 6 hour tests.

At 500°F the sintered bauxite and resin-coated sand change places with permeability values of 180 and 138 darcies, respectively. Otherwise, the list is the same as above; however, all values are 23 percent lower in permeability with the exception of the sintered bauxite which was essentially constant.

8. Miscellaneous Proppants

Figure 20 shows other materials which have been used from time to time as proppants and were tested in our linear flow, triaxial test cell. A 10/20 mesh chert was tested only at 350°F. It showed that crushing began around 3,000 psi and became severe at higher closure stresses. It is quite similar to Brady Sand in its testing and chemical characteristics; however, there is presently a supply problem of buying well sorted chert particles.

20/40 plastic pellets were tested at 75°F and while they deform under load without crushing, they do lose much of their permeability at higher closure stresses. Elevated temperatures cause more rapid deformation and permeability loss. Finally, 20/40 fiberglass reinforced nylon pellets were tested at 75°F. While deformable, they quickly lose permeability at higher loads and extruded at higher temperatures.

Figure 18. Permeability Retention of Available Proppants at 350°F

Figure 19. Permeability Retention of Available Proppants at 500°F

Figure 20. Miscellaneous Proppants

B. Temperature and Chemical Effects

Tests on proppants show that most proppants are sensitive to some chemical and temperature effects. These effects are intensified when the proppants are put under closure stress. The silicon-based materials show an increased sensitivity to brine at points of contact. With a silica unsaturated brine, dissolution of the surface is possible at higher temperatures (350°F or higher) and if crushing occurs new silica surfaces are exposed which are immediately attacked by the hot brine.

In short-term tests the chemical effects are noticed, but the significance and extent of the chemical interaction is difficult to quantify. In preliminary longer term tests, up to 50 hours, the permeability continues to decrease to quite low values with simulated down-hole conditions of 350°F and 5,000 psi. For this reason many long-term tests are planned on the proppants reported in this volume. Long-term test results will be reported along with scanning electron microscope pictures of the proppants in Volume IV of the Stimulation Technology series.

VI. LITERATURE SURVEY

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VII. APPENDIX

A. Geothermal Proppant Test System

The reliability of data is dependent upon several factors, a few being test procedures, test apparatus, and sample quality. Therefore, it is essential that an active and ongoing screening process or quality assurance program is maintained to not only insure the quality of samples (proppants) being tested but also to insure that proper test procedures are used and that test apparatus is properly maintained.

Sample quality is of main importance and proppants are subjected to critical inspection and evaluation prior to testing. Upon the receipt of a known proppant from a supplier or manufacturer, it is visually examined and scrutinized to determine if it is a "clean sample," free of foreign matter. It then undergoes a standard sieve analysis and the large particulates and small fines are removed insuring a particular sample size (10/20, 20/40, etc.). The sample is once again inspected, cataloged, and is then available for testing.

Proppant permeability is determined by the use of a laboratory model utilizing linear flow while also employing the use of a heated-platen hydraulic press to simulate as much as possible actual downhole conditions. The use of the press also allows for direct control over temperature and closure stress and has made possible the testing of proppants at such temperatures as 350°F and 500°F with closure stresses ranging from 2,700-9,700 psi and 2,200-9,200 psi, respectively.

The model (see Figure A-1) consists of a steel cell connected to a liquid source (H_2O) pressurized by nitrogen. The cell is actually two interchangeable plates between which the proppant is placed. As previously stated, load and/or heat is applied to the proppants by means of

a 50-ton hydraulic press. The pressure drop across the pack is determined by monitoring flow at both ends of the pack while the flow of liquid (H_2O) through the proppant is determined by collecting a timed volume, which can be adjusted by use of a micrometering valve. At each closure stress, the height between cell plates is measured and is used in calculating the area of the pack. Permeability of the pack can then be calculated by use of the standard Darcy equation (Eq. 1) with the data obtained.

$$k = \frac{Q_{UL}}{A\Delta P} \quad (1)$$

Upon completion of the screening/cataloging process, the proppant is loaded within the test cell occupying a height of approximately .250 inches. Next a metal shim followed by an EPR (high-temperature elastomer) rubber gasket is installed. The top plate containing a sealing O-ring is then placed within the bottom plate and the cell is complete. The completed unit is then positioned on the hydraulic press and test apparatus calibrated. Following calibration, the cell is connected to the model lines and a starting closure stress is applied (i.e., 2,700 psi @ room temperature, 2,700 psi/350°F, and 2,200 psi/500°F), along with pressurizing the test fluid (H_2O). Heat, if applicable is also applied at this time.

Figure A-1. Schematic of Geothermal Proppant Tester

TEST PROCEDURE

B. Test System Operations Manual

1. Pre-Test Checks
 - a. Accumulator
 - b. Nitrogen Cylinder
 - c. CO₂ Cylinder
 - d. Coolant Tank
 - e. High Vac Pump Oil
2. Loading the Test Cell
 - a. Clean
 - b. Load Proppant
 - c. Level Proppant
 - d. Install Proper Seals
 1. Buna-N, below 350°F
 2. EPR, 350°F or above
3. Calibration--Part I
 - a. Initializing the Zero Setting
 - a. Setting the Span (Gain)
4. Removing Water from Lines
 - a. Before the Test Cell
 - b. After the Test Cell
 - c. Transducer Area
5. Filling the Lines with Water & Calibration--Part II
 - A. Evacuation
 - B. CO₂ Purge
 - C. Evacuation
 - D. Fill Lines with Water
 - E. Finalize the Zero Setting

6. Taking Data

- A. Turn on Heated Platens
- B. Set Closure Stress
- C. Stabilize Flow Rate
- D. Take Data
 - 1. psi
 - 2. flow rate
 - a. volume
 - b. time
 - 3. height right and left
 - 4. press reading
 - 5. line pressure
 - 6. proppant type
 - 7. gasket thickness
- E. Reset Closure Stress & Repeat C, D

7. Shutting Down Test

- A. Open Transducer Equilizer Valve
- B. Shut Off Gas Cylinders
- C. Relieve Nitrogen Pressure
- D. Relieve Line Pressure
- E. Shut Off and Cool Press
- F. Remove Test Cell

1. PRE-TEST CHECKS

- a. Check top accumulator water level. See "Filling the Accumulator with Water."
- b. Check nitrogen tank pressure. It should be at least 200 psi in excess of the line pressure required by the test temperature.
- c. Check carbon dioxide tank pressure. It should be at least 150 psi.
- d. Check the water level in the coolant tank. The cooling coil should be completely covered.
- e. Check the oil in the high vacuum pump. If the oil is cloudy, replace it with new refrigeration oil. This will be necessary every 4-5 tests.

FILLING THE ACCUMULATOR WITH WATER

If more than a quart of water is used in the previous test, the accumulator should be filled.

1. Fill the water cylinder halfway with clean water.
2. Open valve 14 to relieve nitrogen pressure. (See Figure A-1 for valve location.)
3. Valve 2 should be pointing toward the water cylinder.

Figure 1. Schematic of Pressurized Water System

4. Valve 3 should be in the "On" position. (Red lever points in the same direction as the line.)

Figure 2. Valve Position for Accumulator Pump System

5. Hook up the high vacuum pump to the top of the water cylinder and activate the pump.
6. Open Valve 1. Note: The water level in the cylinder will rise because the vacuum is pulling water from the top accumulator. Do not allow the water to come any closer than 2 inches from the top of the cylinder or excessive water vapor will be drawn into the vacuum pump. Remove water if necessary.
7. Allow the pump to run for 30-45 minutes and periodically tap the sides of the cylinder to free the air bubbles clinging to the side of the cylinder.
8. At this time, proceed to "Loading the Test Cell" while waiting for the water to deaerate, then return to Step 9 below.
9. Slowly disconnect the vacuum pump line from the top of the water cylinder and shut off the pump.

10. Hook up the compressor side of the diaphragm compressor/vacuum pump to the water cylinder and turn on the pump. This will force the water into the accumulator.

Figure 3. Top View of Diaphragm Vacuum Pump

11. When the water level stops dropping (indicating a full accumulator) or if the water level drops to within 1 inch of Valve 1, turn off the pump and close Valve 1.
12. Slowly disconnect the compressor line at the compressor outlet to relieve cylinder pressure.
13. Close Valve 14.

2. LOADING THE TEST CELL

- a. Clean the test cell and lines with water. Use a garden hose to clear the lines.

Figure 4. Test Cell

- b. Install screen strips into end slots.

Figure 5. Inlet Screen on Test Cell

- c. Load with proppant to a depth of approximately .25 inches.
- d. Level the proppant carefully. Check by installing the cell piston without seals and measure the distance from the cell body to the plate on top of the piston in several places to determine where additional proppant is needed.

HEIGHT MEASUREMENT

Figure 6. Proppant Pack Height Measurement

- e. After the proppant is level, install the stainless steel shim, the flat rubber gasket, and the cell piston with O-ring seal. Use Buna-N gaskets and seals below 350°F, and EPR gaskets and seals for tests 350°F and above.

Figure 7. Cross Section of Test Cell

3. CALIBRATION --PART I--INITIALIZING ZERO AND
SETTING THE SPAN

- a. Open the equalizer valve between the calibration tubes to give approximately the same height in each tube.

Figure 8. Pressure Calibration Equipment

- b. Close the equalizer valve.
- c. Vent both sides of the transducer to atmosphere by opening Valves 7, 8, and 12.
- d. Open the bleed screws on the side of the transducer 1-1/2 to 1-5/8 turns. DO NOT remove completely.

Figure 9. Differential Pressure Transducer

- e. Quickly and simultaneously flip both transducer valves so that the levers point upwards.

Figure 10. Pressure Transducer Valves

- f. Water will now be draining out of the bleed holes. Allow this to continue for at least 10 seconds, then close the bleeders. Check the clear lines which go to the calibration tubes for air bubbles. If any are found, loosen the swage lock fitting at the bottom of the clear line to flush the bubble out.
- g. Add or remove water as necessary to the calibration tubes so that both sides are of equal height ± 0.01 inches.

Figure 11. Zero Pressure Calibration

- h. Set the zero knob on the readout unit with range in low position.
- i. Place range switch in high position with full scale pressure applied to the transducer and adjust span control for a 100.0% indication.
- j. Note: Recheck calibration
- k. Quickly and simultaneously flip both transducer valves so that the levers point downward (the opposite of the position shown in Step 5).
- l. Cover the top of the calibration tubes to prevent debris from entering.

4. REMOVING WATER FROM LINES

There are 3 main areas that need to have the water removed: (1) before the test cell, (2) after the test cell, and (3) the transducer area.

NOTE: The test cell hasn't been installed yet.

Before the Test Cell

- a. Hook up the diaphragm vacuum pump to the evacuation port (by Valve 12).
- b. Open Valves 9, 11, and 12. NOTE: Opening Valve 11 allows the vacuum to be monitored.
- c. Close (or check, as some should already be closed) Valves 8, 10, and 15.
- d. Start the vacuum pump and allow to run for 3-4 minutes (leave pump on and proceed to Step 5).
- e. Close Valve 9.

After the Test Cell

- a. Close Valve 13.
- b. Open Valve 10.
- c. After 3-4 minutes, close Valve 10.

Transducer Area

- a. Close Valves 5 and 6.
- b. Open Valve 7. IMPORTANT - RECHECK
- c. Open Valve 4
- d. Open Valve 8 slowly, stop opening if the readout unit reads over 15.00.
- e. After 3-4 minutes, close Valve 4.
- f. Open Valve 5.
- g. After 3-4 minutes, close Valve 5.
- h. Open Valve 6.
- i. After 3-4 minutes, close Valve 8, RECHECK.
- j. Remove the bleed screws completely from the transducer. Take extreme care not to drop the bleed screws and store them safely.
- h. Slowly open Valve 8.
- j. Close Valve 6.
- k. After 1-2 minutes, close Valve 8.
- l. Carefully replace the transducer bleed screws and snug them lightly.
- m. Shut off and disconnect the vacuum pump.

5. FILLING THE LINES AND CALIBRATION--PART 2

- a. Install the test cell in the center of the platens and load with 5 tons.
- b. Hook up the lines to the test cell.
- c. Hook up the high vacuum pump to the evacuation port.
- d. Open Valves 4, 5, 6, 7, 8, 9, 10, 11, and 12.
- e. Turn on the vacuum pump.
- f. After 3-4 minutes, close Valve 12, close valve 11.
- g. Slowly open the main valve on the CO₂ cylinder. The regulator should be set at 80-100 lbs. Open valve 11 slowly, then close the CO₂ cylinder.
- h. Open Valve 12 slightly to allow the vacuum pump to evaluate the CO₂ at a rate of no more than 1 psi/second. Caution must be taken to avoid pressuring the vacuum pump.
- i. When full vacuum is attained, close Valves 1, 10, 11 and 12.
- j. Turn off and unhook the vacuum pump.
- k. Open the main valve on the top of the nitrogen cylinder.
- l. Adjust the T-handle on the regulator so that the desired line pressure is indicated on the regulator gauge. The line pressure is given in the "Test Temperature vs. Zero Setting" table.
- m. Slowly start turning Valve 2 so that it will point in the direction of the line gauge. The water will flow from the upper accumulator into the lines and will make an audible hiss as the flow begins.

Stop turning the valve approximately 10° after the hiss starts. This will allow the lines to fill slowly. After the hissing stops, turn the valve completely toward the line to the line gauge.

- n. Close valves 6, 8, 9, and 10.

Figure 12. Charging of Pressurizing System

- o. Adjust the nitrogen regulator so that the line gauge indicates the desired line pressure. If the desired pressure is exceeded, bleed off the excess at the nitrogen line vent (Valve 14).
- p. Open both bleed screws on the transducer 1/2 to 5/8 turns. Watch the readout unit carefully while opening the bleeders to make sure the differential indicated does not exceed 19.99. Allow water to run out of the transducer for 5 to 10 seconds. Then close the bleeders snugly.
- q. Adjust the zero setting to read "0.00 ±.02".

6. TAKING DATA

- a. Turn on the upper and lower platens if desired test temperature is above ambient.

Figure 13. Hydraulic Heated-Platen Press

- b. Close Valve 7, RECHECK.
- c. Set the load at 15 tons.
- d. Open Valve 13 slightly while watching the readout unit. The desired flow rate is 1 to 5 drops/second, but it will be necessary to adjust the valve several times because the flow will drop substantially before it stabilizes. When opening the valve, DO NOT allow the indicated reading to exceed 19.99. The reading is considered stabilized when it varies no more than 0.1 in a 15 second period.

- e. While waiting for the flow rate to stabilize, take height measurements on the test cell's right and left midpoints. Also record the press tonnage and line pressure.

Figure 14. Height Measurement Points on Test Cell

- f. After the flow rate has stabilized, use a 10 ml graduated cylinder to capture the flow for 15 seconds. Record the volume and the duration of the flow. Also record the average indicated pressure drop from the readout unit. Repeat 2 more times at intervals of 2-3 minutes.
- g. Increase the press tonnage by 5 tons and repeat Steps 6, 7, and 8 until a 50 ton load is reached.

NOTE: If raising the press tonnage results in a pressure drop that continues to rise and does not stabilize, allow 20-30 minutes between raising the tonnage and taking data. The continual rise in pressure drop indicates that the proppant is still crushing (time effect of static load). This effect is most noticeable at high temperatures and high closure stresses.

7. SHUTTING DOWN AFTER THE TEST

- a. Turn off the platens. DO NOT shut off the coolflow unit.
- b. Open Valve 7.
- c. Shut off the main nitrogen cylinder valve and back out the regulator T-handle until the spring tension is relieved.
- d. Check that Valve 1 is closed.
- e. Turn Valve 2 so that it points toward the water cylinder.
- f. Open Valve 13 to relieve the lines of water pressure.
- g. If shutting down the system for the day or if it will be necessary to add water to the accumulator for the next test, open Valve 14 to bleed the accumulator bladder pressure.
- h. Remove the test cell.
- i. When the platen thermometer indicates that the platen heat is below 200°F, shut off the coolflow unit.

C. Physical Strength Test Procedure - Single Grain

In this test, a cross section of a representative sample is used to determine the average stress a single grain can tolerate prior to crushing. This is accomplished by using a hand-operated hydraulic pump from which a load (pounds) is applied to a proppant grain by pressurizing the cylinder which contains a movable piston. When pressurized a platen extends and exerts a direct load on the grain. The load is steadily increased and monitored (1 pound/minute) until the crush point is reached and the value recorded. This value is then used

to calculate the apparent strength from the equation, $S = \text{Load/square of grain diameter}$.

D. Specific Gravity Test Procedure

1. Specific gravity and density of a solid by Archimedes Principle.

A sample is weighed in air and then carefully immersed in water. The apparent loss in weight of the solid when immersed in water is equal to the weight of liquid displaced by the solid. From careful measurements of water displaced and actual weight readings specific gravity and density can be determined. Complications can occur if the surface of the unknown solid is not fully wetted by the calibrating fluid; however, for proppant specific gravity enough accuracy for comparative purposes is attained by using water as the calibrated fluid.

2. Materials.

- a. triple-beam balance
- b. 2 graduate cylinders or beakers
- c. 50°C thermometer
- d. distilled H₂O

3. Procedure.

- a. Using a weighted graduate cylinder or beaker, add 1 cm³ (1 ml) of sample. The resultant weight is (total weight less graduate cylinder weight) is the weight-density of the sample in air.

- b. In a graduate cylinder or beaker place a known volume of distilled H_2O and record its temperature.
- c. Immerse the sample in H_2O and determine the weight of H_2O displaced.
- d. Refer to a table of water density and determine the density of H_2O at the observed temperature.
- e. Compute the specific gravity of the sample relative to the water used with the equations given below;

$$\begin{aligned}
 \text{Sp. gr.} &= \frac{\text{Density of Sample}}{\text{Density of } H_2O} \\
 &= \frac{\text{Weight of Sample in Air}}{\text{Weight of } H_2O \text{ Displaced}}
 \end{aligned}$$