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GEOTHERMAL FRACTURE STIMULATION TECHNOLOGY

Volume 1. Fracturing Proppants and Their Properties

By
Mauer Engineering

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Republic Geothermal, Incorporated
Santa Fe Springs, California

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GEOTHERMAL FRACTURE STIMULATION TECHNOLOGY

VOLUME I

FRACTURING PROPPANTS AND THEIR PROPERTIES

July 1980

Prepared for

U.S. Department of Energy

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GEOTHERMAL RESERVOIR WELL STIMULATION PROGRAM

by

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I. SUMMARY

Volume I of the Geothermal Fracture Stimulation Technology series is a review of previously published literature on proppant permeability. This data will be used in the subsequent phases of the geothermal stimulation project. Much information comes from the oil and gas industry which has tested various proppants during the past thirty years over a range of different closure stresses at the lower temperatures found in oil reservoirs. At high temperatures, testing as described in Volume II is required to extend present day data to that which is needed for geothermal wells. Historically, very little temperature and/or chemical dependence has been investigated for fracturing proppants.

This report summarizes and reviews the historical development of proppants, and presents a variety of data on proppants found in today's literature. Also included are several standard test procedures and equipment setups used in measuring proppant properties and in proppant testing.

II. INTRODUCTION TO USE OF PROPPANTS

A. Their Role in Hydraulic Fracturing

Proppants play an important role in the use of hydraulic fracturing for well stimulation.¹ Hydraulic fracturing occurs when a fluid is pumped down the wellbore at a rate higher than can be accepted by the matrix permeability of the formation. The formation cracks in an effort to reduce the stresses caused by the fluid pressure. The fracture continues to grow and is held open by the fluid pressure. When fluid pressure is taken off the formation (pumps are stopped), the two walls of the fracture will close or "heal" unless the fracture contains matter such as spalled formation, sand, or other type of proppant. Figure 1 shows a schematic of a propped fracture.

Proppants need to be strong and permeable to allow the fluids from the formation to be produced through the proppants and prevent the closure of the fracture. Many people think of this propped fracture as a pipeline from the underground reservoir to the wellbore. The selection of the type of proppants is based upon their strength, permeability retention under load, cohesiveness and other properties. Sand has been the most popular type of proppant used in the thirty years of oil and gas well stimulation by hydraulic fracturing. Sand is not an ideal proppant, however, because it is brittle and it tends to crush under high loads. The fines created when sand crushes can be very damaging to valves and the tubular goods in the well. Proppants have been tried that are deformable, that is, they take load and deform without being brittle. Many of these types of proppants have been invented, tried and discarded due to loss of permeability with deformation. An ideal proppant² is one which will fully support the closure stress or earth overburden that is imposed on it. It can retain its permeability during production operations, remain inert, and be chemically nonreactive to the produced fluid and formation.

Figure 1. A Schematic of a Proppant Filled Fracture

B. History of Proppants

Hydraulic fracturing, as a stimulation tool, has a thirty-year history for the oil and gas industry. Sand was used as a proppant in the very earliest fracturing jobs because it was available at very low costs. Sometime during the 1950's, further research into what constituted a good proppant took place. During this time, glass beads and walnut hulls were investigated and used in small quantities as proppants. Glass beads had a spherical shape, were of uniform size, were clean, and looked much better than sand as a proppant. For high priced and first class operations, glass beads were sometimes recommended. On the other hand, an important property lacking in both sand and glass beads was a property called deformability. Since neither sand nor glass beads are deformable, they are classified as brittle materials which means that they tend to crush under high load. Walnut hulls have been found useful in shallow applications where the closure stresses are not too high because walnut hulls deform (made of a cellulosic wood). In deeper and hotter applications, walnut hulls tend to degrade and disintegrate. The use of walnut hulls continued throughout the 50's and early 60's when many new products began to appear. In the 1960's super strong proppants came into vogue and the use of aluminum pellets and steel pellets were considered. The main disadvantages that steel and aluminum pellets³ have as proppants are their high density and high cost per volume. Also, these proppants are susceptible to corrosion under the acidic environments of the producing formations.

In the 1960's, the use of partial monolayer proppant technology began to be developed.² These packs are formed by using spacers (inert, or dissolvable particles) mixed in with the regular proppant to fill the fracture, and then, after treatment the spacer dissipates to allow a sparsely propped fracture. Theoretically, this type of propped fracture has the desirable effect of allowing flow or production to return

through an open channel in the hydraulically created fracture. This type of pack can result in an order of magnitude increase in fracture conductivity. Figure 2 shows the effect of proppant concentration on fracture conductivity. Figure 3 shows the area open to flow in the three possible cases of partial and full monolayer and multilayer pack. Unfortunately, the use of monolayer or partial monolayer of proppants is difficult to achieve under field conditions. Very few documented cases of successful field treatment using partial monolayer proppant packs have ever been reported.

During the 1970's, many new products were introduced. Some of these are unfamiliar to us today because they briefly appeared on the scene and quickly left when one or more disadvantages became apparent. Some of these that are mentioned are not documented anywhere except in the archives of the oil companies' libraries. Some of the interesting ones are aluminum³ and brass coated glass beads, exotic plastics, garnet, different forms of aluminum oxide, and ceramic pieces or pellets. For most of these proppants the cost and expense to manufacture or process became the overriding consideration of why they could not be produced in large quantities. In the middle 70's the French succeeded in producing a round ceramic or zirconium oxide based particle¹⁰ which has had usage in Europe. It is known to be very strong but cost and availability are not known.

At Exxon Production Research Company two important patents were issued in 1975, both of them on high strength proppants. The first is sintered bauxite.⁹ It is made by sintering many small pieces of bauxite together into a round proppant. It combines a fairly uniform size with very high strength behavior under closure stress. It is slightly deformable and is insensitive to most normal downhole fluids.

The second proppant that was invented by Exxon Production Research Company in 1975 was an uncured resin-coated sand (Super Sand).⁸ The

resin coating is a thermosetting coating which allows full protection of the sand or other propping material from the corrosive fluid environment and spreads the load by deforming under load to keep the sand from crushing or breaking. Today, in the oil and gas industry the three proppants in widespread use are sand, sintered bauxite, and the uncured resin-coated sand.

Figure 2. Effect of Particle Concentration on Fracture Conductivity

Figure 3. Area Open to Flow in a Propped Fracture

III. PROPPANT PROPERTIES

The following data represent an intensive literature search of published and unpublished proppant-permeability data. The report is broken into sections depicting the different types of proppants which are: Section A reviews the use of sand; Section B--glass beads; Section C--ceramic or bauxite pellets; Section D--resin-coated particles; and Section E covers other miscellaneous proppants. The last two Sections, F and G, cover published data on the amount of permeability reduction that is obtained when fluid loss additives are used in fracturing the well or when mixed sands are used, and on cost comparisons of the available proppants, respectively.

A. Sand as a Proppant

Sand is the oldest and most widely used proppant. Advantages of sand as a proppant are cost and availability. Fracturing sand quality has been upgraded through better cleaning and sieving practices of most sand suppliers. Table I lists minimum and maximum specifications for fracturing sand. It is important that these specifications be met, since a small percentage of fine-mesh sand can greatly reduce the permeability of a larger-mesh sand, as shown in Section F.

TABLE I
NORMAL FRAC SAND SPECIFICATIONS

Type: Well-rounded, single-grain, medium-to-coarse, white or colored quartz having high particle strength.

	<u>Minimum</u>	<u>Maximum</u>
Silica Content: Wt. %	98.0	100.0
Size Distribution:	80.	
Wt.% within specified range	80.0	90.0
Wt.% passing specified coarser screen but not passing specified finer screen	99.0	100.0
Roundness: Ratio of radii (a)	0.6	0.8
Sphericity: (b)	0.85	0.89
Soft Particle Content: Wt. %	0.50	0.10
Silt and Clay Content: Wt. %	0.10	none

(a) Roundness is defined as the ratio of the average radius of curvature of the several corners to the radius of curvature of the largest inscribed circle on the projected sand grain image.

(b) Sphericity is defined as the cube root of the ratio of particle volume to circumscribed sphere volume.

Fracturing sand is normally referred to by a mesh size range. The mesh size may be given as U.S. or Tyler sieve number. Table II compares the two systems with a typical sand used for proppants.

TABLE II
SAND SIZE SPECIFICATIONS
(Typical Sand Used for Proppants)

U.S. Sieve No.	Tyler Sieve No.	Sieve Opening In.	% Retained on Sieve					
			8-12	10-20	10-30	16-30	20-40	40-60
8	--	0.0937	3					
10	--	0.0790	47					
12	10	0.0661	40	1				
16	14	0.0469	10	17	13	7		
18	16	0.0394		29		46		
20	20	0.0331		35		37	2	
30	28	0.0232		14	81	10	70	
35	32	0.0197				1		
40	35	0.0165		4	6		24	31
45	42	0.0138					2	
60	--	0.0098					1	52

1. Size and distribution

A larger sand grain size in the fracture provides a more permeable pack at low closure stress conditions. The type of proppant used has to be evaluated in relation to the type of formation being propped. Dirty formations or those subject to significant fines migration are poor candidates for large-size props as the fines tend to invade the prop pack resulting in partial plugging and rapid reduction of proppant permeability. In this situation, smaller sizes can better resist the invasion of fines. Although smaller sizes of proppant offer less conductivity initially, the average fluid conductivity over the life of the well is higher and more than offsets the initial higher productivity associated with larger proppants which experience a rapid decline.

Larger grain sizes are generally not considered in deeper wells because of greater susceptibility to crushing and placement problems.

Grain size distribution and prop quality are closely related. A high percentage of smaller grains mixed with larger grains can have the same effect on proppant pack permeability as the invading formation fines. Figures 4 and 5 show that the particle-size distribution¹⁴ and proppant quality¹⁴ are less important in shallower wells with lower closure stress but become more important with depth and higher overburden stress.

Figure 4. Effect of particle size on fracture conductivity. Uniform size grains distribute the load more evenly and withstand higher stresses.

Figure 5. Effect of prop quality on conductivity. These curves represent sand containing feldspars as impurities. Any fines tend to reduce permeability of prop pack under closure stresses.

2. Proppant density

Density is a physical property that can be important under certain well conditions and is often associated with proppant transport problems. An explanation of the requirements of frac fluids for fracture penetration and proppant transport will help define the relationship of proppant and fluid properties needed for effective stimulation.

Stimulation of low permeability reservoirs requires creation of deeply penetrating, high conductivity fractures. This requires the placement of a large volume of propping agents, long distances from the wellbore, and usually at low injection rate or fluid velocities in the fracture.

The factors that affect proppant placement are fluid velocity, viscosity, density, efficiency, and propping agent size and density.

The requirements for good proppant placement are to:

- (a) Create sufficient width for particle-size placement.
- (b) Maintain sufficient fluid velocity within the fracture to carry the proppant to the desired distance.
- (c) Maintain sufficient fluid viscosity for proppant placement. Proppant dropout-type fluids or proppant support-type fluids are used to develop the propped fracture width, conductivity, and propped length for maximum productivity.

Thus, proppant density is an integral part of fluid design. High density proppants are more difficult to suspend and transport in the fracture. High density fluids and highly viscous fluids can transport the heavier proppant, but may also present difficulty in dropping out or placing the proppant where desired. Treatment design then becomes a compromise between density and placement. In other words, a fluid might need to be designed to provide proppant placement in a manner that would provide adequate, but less than optimum, flow capacity.

Proppant size is subject to a similar compromise. Large size proppants tend to settle faster than smaller sizes due to weight, and face similar placement problems as the high density proppants. In addition, large proppants require wider fractures which could be a problem where well conditions or fluids dictate a narrower fracture width.

3. Roundness factor

Figure 6 shows the effect of closure stress¹⁴ on the permeability of various mesh sands, depending on the roundness factor of the sand grain. Stresses in the sand grains of a similar size are more evenly distributed when the grains are more rounded. The grains will also offer higher resistance to a high load before grain failure occurs.

Figure 6. Effect of Roundness on Fracture Conductivity. Stresses are more evenly distributed in uniformly round grains and the grains will resist a higher load before failure.

4. Other properties of brittle proppants (sand)

In general, brittle or "crushable", particles used for proppants are approximately spherical crystalline (sand) or amorphous (glass) materials. These particles have moderate to high strength when a single particle is loaded between flat plates, but much lower strength when point loaded in a multilayer pack. The following proppants were studied:

Sand - The most commonly used propping agent, sand, gives fair fracture permeability up to closure stresses of about 6000 psi and is relatively inexpensive. Most sands give similar permeability load data; however, "Ottawa" or "Heart of Texas" sand should be used when available because of its uniform size and high degree of roundness.

Figure 7 shows the permeability of various size sands at a closure stress of 2000 psi. Closure stress is equal to the frac gradient times depth minus the bottom-hole pressure. Figure 7 shows that a smaller size sand has a smaller permeability.

Figure 7. Permeability of "Heart of Texas" Sand as a Function of Mean Proppant Diameter (2000 psi Closure Stress)

Permeability tests were run on various size sands under load versus time. The long term effects appeared to be the same for all sands when a dimensionless permeability was used. Figure 8 shows the dimensionless permeability (or permeability retention) versus time for a "Heart of Texas" sand.

Figure 8. Dimensionless Permeability of "Heart of Texas" Sand vs Time

From Figure 8 a conversion factor or multiplier was calculated which is shown in Figure 9 to give the permeability of a sand under a given closure stress after one year at room temperature. At higher temperatures these fractions will be much less.

Figure 9. Correction of Instantaneous Permeability to Permeability at One Year of Stress

In Figures 10 through 15 all of the various sands were tested with linear flow (using room temperature gas) to obtain permeability measurements from 1 to 10,000 darcies under various closure stresses to 20,000 psi.

Figure 10. Instantaneous and Corrected Permeability for 8-12 Mesh "Heart of Texas" Sand

Figure 11. Permeability--Closure Stress Curves for "Heart of Texas" Sand

Figure 12. Permeability--Closure Stress Curve for "Ottawa" Sand

Figure 13. Permeability--Closure Stress Curves for "Gopher State" Sand

Figure 14. Permeability--Closure Stress Curves for Chert

Figure 15. Permeability--Closure Stress Curves for Arkhola Sand

Figures 16 through 20 show fracture conductivity versus the proppant concentration. The various closure stresses are plotted on each figure to show the effect of decreasing fracture conductivity at increasing closure stress. The areas of partial and full monolayers and the area of a packed (multilayer) fracture can easily be seen in these figures. A radial flow cell was used to measure the proppant's permeability under load. Most of this data has been published by the oil field service companies such as Halliburton, BJ Hughes, Dowell and the Western Company.

Figure 16. Fracture Conductivity vs Proppant Concentration at Various Closure Stress Levels, 20-40 Round Sand, Soft Formation

Figure 17. Fracturing Conductivity vs Proppant Concentration at Various Closure Stress Levels, 20-40 Round Sand, Hard Formation

Figure 18. Fracture Conductivity vs Proppant Concentration at Various Closure Stress Levels, 20-40 Round Sand, Very Hard Formation

Figure 19. Flow Capacity vs Proppant Sand Concentration at Various Closure Stress Levels, Soft Formation

Figure 20. Flow Capacity vs Proppant Sand Concentration at Various Closure Stress Levels, Hard Formation

The next set of figures (Figure 21 through 25) show how increased temperatures influence the permeability retention of the sand pack. All of the data⁷ have been taken in a linear flow cell using actual wellbore fluids.

Figure 21. Effect of Stress, Fluid, and Temperature on the Permeability of 8-12 Mesh Sand.

Figure 22. Effect of Stress, Fluid, and Temperature on the Permeability of 10-20 Mesh Sand.

Figure 23. Effect of Stress, Fluid, and Temperature on the Permeability of 20-40 Mesh Sand.

Figure 24. Effect of Stress, Fluid, and Temperature on the Permeability of 40-60 and 60-120 Mesh Sand.

Figure 25. Time Effect on 10-20 Mesh Sand, 250°F Brine, 5000 psi Stress.

B. Glass Beads

Glass beads were once thought to be the strongest proppant available as the first tests were made in air at room temperature. However, in brine at elevated temperatures, researchers⁹ found that glass beads are sensitive to stress corrosion cracking and are chemically attacked by high temperature brine. In this environment the glass beads crumbled to a fine powder with high closure stress. As a result of the later tests, they are no longer used today. The reason that the glass beads looked good was that they are well rounded and sorted. They were, however, expensive to manufacture and process. Figure 26 shows the early low temperature air data that initially gave glass beads an edge over sand.

Figure 26. Permeability of 8-12 Mesh Sand and Glass Beads

Figures 27 through 29 show the permeability of various sizes of glass beads under closure stresses to 20,000 psi.

Figure 27. Instantaneous and Corrected Permeability for 8-12 Mesh "UCAR" Proppant.

Figure 28. Permeability of 20-40 Mesh "Ottawa" Sand, 10% 12-20 Mesh Glass Bead and 90% 20-40 Mesh "Ottawa" Sand

Figure 29. Permeability--Closure Stress Curves for Glass Beads--
"UCAR"

Figures 30 and 31 show how proppant concentration affected fracture conductivity (in air). The various closure stresses are shown as multiple curves on each figure. This data has been used to support the need for partial monolayer stimulation treatments.

Figure 30. Fracture Conductivity vs Load Pressure 12-20 UCAR Props, Very Hard Formation.

Figure 31. Fracture Conductivity vs Load Pressure 8-12 UCAR Props, Very Hard Formation

Figure 32 by Cooke⁹ of Exxon Production Research investigated the early claims of superiority by glass beads over sand and found that glass beads tested in hot water or hot brine were not as good as sand packs in retaining permeability under load.

Figure 32. Effect of Stress and Fluid on the Permeability of Glass Beads.

C. Ceramic or Bauxite Pellets

1. Sintered bauxite

This material is manufactured by the sintering of bauxite ore into fine-grained particles having a specific gravity in the range of 3.4 to 3.8. (By comparison, sand has a density of 2.6.) The material is available in a wide range of mesh sizes including all the common sizes usually employed in fracturing.

When subjected to high closure stresses, the fine grained structure of the sintered bauxite particles enables the particles to deform under pressure rather than crush. The particles resist crushing when placed in a multilayer propping pattern in the fracture, even when exposed to the presence of brine.

SINTERED BAUXITE FRACTURE CONDUCTIVITY CHARTS

Work has been completed on fracture conductivity measurements of Norton 20-40 bauxite proppant. The results are shown on the attached charts (Figures 33 through 35) and illustrate the superior strength and conductivity of bauxite at closure stresses over 6000 psi. Using the perfect support fluids available, we should be able to tailor fracturing treatments using the bauxite proppants to their greatest advantage.

PHYSICAL PROPERTIES

<u>Mesh Size</u>	<u>Percent Retained on Screen</u>	<u>Krumbein Roundness</u>	<u>Acid (HCL) Solubility</u>	<u>Specific Gravity</u>
20	-	-	-	-
20	37.4	-	-	-
40	60.5	0.5	0.07	3.5
40	2.1	-	-	-

Figure 33.

Figure 34

Figure 35

Additional data on fracture conductivity of sintered bauxite from a different source⁹ is shown in Figure 36 below.

Figure 36. Effect of Proppant Concentration and Stress on the Conductivity of 10-12 Mesh Sintered Bauxite Particles Between Sandstone Slabs.

Figure 37, from an Exxon patent, shows the permeability of several ceramic particles, sintered bauxite, and sand versus closure stress. The bauxite appears to hold up under high closure stresses while sand and glass beads disintegrate.

Figure 37. Permeability Versus Closure Stress for Various Proppants.

2. Aluminum oxide

Another form of bauxite is purified aluminum oxide. With a hardness of nine, it is mineralogically similar to the ruby and sapphire. The material is hard and strong. Figure 38 shows the permeability behavior⁹ under closure stress to 8000 psi.

Figure 38. Effect of Stress on the Permeability of Aluminum Oxide.

3. Ceramic oxide of aluminum

Union Carbide published several papers on their ceramic proppant, oxide of aluminum.¹¹ Because of a dispute and patent litigation all work and discussion in literature of this proppant has stopped. To our knowledge this product is not available; however, some interesting data has been published.

Figures 39 through 42 show pictures of the sintered bauxite and the ceramic proppant illustrating the visual and cross-sectional view of the proppant. Figures 43 through 48 show the fracture flow capacity versus fracture closure stress of the ceramic proppants as compared to sand, sintered bauxite, and other proppants. Ceramic props appear to be as good as or better than sintered bauxite. This material was sold as white colored spheres of ceramic and is sometimes found in certain areas mixed with black sintered bauxite.

Figure 39. 30 Grit Sintered Bauxite Particles

Figure 40. 20 x 40 Mesh Oxide Ceramic Prop.

Figure 41. Microstructure of Sintered Bauxite Particles. 55X.

Figure 42. Microstructure of 20 x 40 Mesh Oxide Ceramic Prop. 55X.

Figure 43. Effect of Hydrochloric Acid and Closure Pressure on the Fracture Flow Capacity of Proppants.

Figure 44. Effect of Proppant Concentration and Closure Pressure on the Fracture Flow Capacity of 12 x 20 Mesh Oxide Ceramic Prop. Brine @ 65°C.

Figure 45. Effect of Core Type on the Fracture Flow Capacity of 20 x 40 Mesh Oxide Ceramic Prop. Brine @ 65°C.

Figure 46. Effect of Closure Pressure on the Fracture Flow Capacity of 12 x 40 Mesh Proppants. Brine @ 65°C.

Figure 47. Effect of Closure Pressure on the Fracture Flow Capacity of 20 x 40 Mesh Proppants. Brine @ 65°C.

Figure 48. Effect of Closure Pressure on the Fracture Flow Capacity of 20 x 40 Mesh Proppants. Nitrogen @ 25°C.

4. Zirconium oxide beads

The structure of zirconium oxide beads (types) are semicrystalline. A well crystallized baddeleyite skeleton (monoclinic ZrO_2) is embedded in a fused silica matrix. A chemical analysis expressed in terms of equivalent oxide weight shows that the components are: ZrO_2 + HfO_2 (68.7%), SiO_2 (31.2%) and TiO_2 + Fe_2O_3 (0.1%) . There are also small quantities of calcium, magnesium, and aluminum.

The material proposed for hydraulic fracturing¹³ purposes consists of beads. Their mean density is 3.76. A multilayered bead pack has a specific gravity of 2700 kg/m³ (~0.1 lb_m/in³) corresponding to a 30% porosity. This average porosity slightly depends on the mesh size and roundness of the beads.

Hardness (Vickers microhardness) of the beads solid components is 10⁴ MPa (14 x 10⁵ psi) for baddeleyite and 7 x 10³ units (10⁶ psi) for the silicon matrix.

The average crushing strength of an individual bead is 720N (160 lb_f) for a 2-mm (0.08-in.) diameter and 240N (53 lb_f) for a 1-mm (0.04-in.) diameter. Fragments of zirconium oxide are several times larger than fragments of glass.

Percentages of broken beads have been measured in multilayered packs under stresses of 100 MPa (14,000 psi) and 274 MPa (38,360 psi). Several materials have been tested: zirconium oxide beads (20-40 mesh), high-strength vitro-ceramic beads (30-40 mesh), Ottawa sand (20-30 mesh). Under 100 MPa, 5% zirconium oxide are broken instead of 26% and 61%, respectively, for the other materials. Under 274 MPa, these percentages are, respectively, 38% for zirconium oxide beads and 71% for 24% PbO/Crown glass in the 16-18 mesh size.

A new type of zirconium bead (Type N) has been tested in the laboratory. Its chemical composition is slightly different and has been formulated in order to obtain an individual strength greater than the standard beads strength (1000 Newtons instead of 730 Newtons for a 2-mm diameter bead). This kind of material may see use only as a monolayer proppant because of its high cost.

Fracture conductivity of the zirconium oxide beads versus closure stress is shown in Figures 49 through 52. This data compares favorably with sintered bauxite although no cost comparisons were available.

Figure 49. Conductivity of 10-20 Mesh Zirconium Oxide Beads

Figure 50. Influence of Mesh Size on Zirconium Oxide Beads Conductivity

Figure 51. Influence of Sphericity on 10-20 Mesh Zirconium Oxide Beads.

Figure 52. Effect of Concentration on the Conductivity of 20-40 Mesh Beads.

D. Resin-Coated Particles

The addition of an uncured resin coating to a particle of sand or other brittle particle adds deformability to the particle.⁸ This resin-coated particle is stronger, retains higher permeability under load and protects the coated material from acid, chemical attack, or hot brine.

This resin is only sensitive to temperature and it cures (polymerizes) by thermosetting. The phenolic formaldehyde resin turns into a bakelite coating when cured downhole which then makes the particle resistant to acids and high temperature brines.

1. Resin-coated sand

Resin-coated sand is a new, high strength proppant now available for use. Called Super Sand, it has properties that make it an ideal replacement for sand and glass beads in formations where there is high-closure stress or the need for sand control. It is ordinary frac sand which is coated with a resin which remains dry and hard at ordinary temperatures. It mixes readily with frac fluids and pumps easily. The heat of the formation inside the fracture softens it to a glassy state, then it polymerizes (cures) and hardens. It fills the fracture with a strong, cohesive "rock" that retains high permeability at stresses up to 10,000 psi (69 MPa).

Laboratory tests and field experience indicate that the resin-coated sand has excellent long-term stability since the resin coating is inert to most common oil field chemicals, acids, and high-temperature water.

A comparison of properties of resin-coated sand, sand and sintered bauxite is shown in Table III. Following the comparison table is a technical data specification sheet to explain the exact makeup of the resin-coated material and the time required for thermosetting.

TABLE III
COMPARISON OF PROPPANT PROPERTIES

	Resin-Coated Sand	Sand	Glass Beads	Bauxite Pellets
Proppant Strength	Excellent to 10,000 psi (69 MPa) Closure Stress	Good below 4,000 psi (27.6 MPa) Poor above 4,000 psi (27.6 MPa) Closure Stress	Excellent below 5,000 psi (34.5 MPa) Poor above 5,000 psi (34.5 MPa) Closure Stress	Excellent to 10,000 psi (69 MPa) Closure Stress
Permeability Retention under Closure Stress	Excellent, no plugging by fines	Fair, partial plugging with crushed sand	Fair, partial plugging with crushed beads	Good to Excellent
Low Temperature Limits	130° F (54.4° C) minimum (Natural or Artificial)	None	None	None
High Temperature Limits	500° F (260° C) 600° F (315° C)	Silica Solubility above 300° F (148.7° C)	Silica Solubility above 300° F (148.7° C)	Not known
Self-Cohesion	Excellent	None	None	None
Minimizes Embedment	Yes	No	No	No
Cost	Intermediate	Low	High	High

TECHNICAL DATA SHEET FOR SUPER SAND II

95% Base Material: Ottawa high-purity crystalline silica sand produced from the air blown deposits found in the Jordan sandstone formation.

5% Uncured Resin Coating: A phenolic formaldehyde thermosetting resin with additives to promote uniform behavior under temperature, fluid and closure stress. Securely bonded to clean sand grain surface so that it cannot come off during operations. On 20/40 mesh sand the deformable resin layer is only about 0.001 inch thick. Cures in the formation to a smooth acid-resistant material similar to Bakelite.

Technical Specifications

Krumbein Roundness and Sphericity: both 0.9

Acid Solubility: less than 2 percent by weight

Physical Strength(L/D^2) 20,000 to 24,000 psi

Permeability Retention for 20/40 mesh: 100% of low stress "K" at 5000 psi
78% of low stress "K" at 10,000 psi

Temperature Stability: Good to 500°F

Compressive strength of core of Super Sand II: 3000 psi uniaxial

(thermoset with closure stress at temperature)

Recommended Set Times:	(Hours)	(days)
(In Brine)		
130°F	200	8
150°F	48	2
170°F	24	1
190°F	12	$\frac{1}{2}$
220°F	8	-
250°F	4	-
300°F	2	-
(and above)		

Note: Above 190°F shut-in or set times should closely relate to the normal gel break times used by the service companies. In oil recommended set times should be doubled.

a. Temperature, time and cohesion

The temperature-time relationship to thermoset the resin-coated sand to a uniaxial strength value of 2500 psi (17.25 MPa) is given in Equation 1. Continued buildup in the uniaxial strength depends on reservoir temperature, but the strength is believed to increase linearly with time up to a maximum measured value of about 3000 psi (20.7 MPa).

$$T = (\theta)^{-10.8} \times 10^{26} \quad (1)$$

where

T = Time, Hours

θ = Temperature, °F

The resin-coated sand is much stronger than indicated by the uniaxial test (L/D^2) because it withstands much higher stresses when loaded. Triaxial strength is indicated by the permeability retention versus applied stress as shown in Figures 53, 54 and 55. The uniaxial strength is a more stringent test of a material and indicates its cohesiveness. None of the other proppants listed in Table III has any cohesive strength, so resin-coated sand has a unique advantage over other proppants.

Cohesiveness prevents proppant movement or migration in the fracture and prevents return of sand or crushed fines to the wellbore and the surface. In current field tests of the resin-coated sand used in sand control applications, none has been produced back to the wellbore. Cohesiveness and uniaxial strength at temperature for 4 to 90 days have been measured in the lab.

Resin-coated sand's long-term permeability under load is excellent. To date no deterioration has been found. Test data for a

90-day period at 200°F (93.2°C) show it to be stable (Figure 55A). To maximize long-term productivity in a fracture, a proppant must retain its permeability and strength for years. The resin coating is insoluble after thermosetting in the fracture and is not affected by most surfactants, chemicals, or acids.

b. Advantages

Fracture conductivity is maximized to optimize the results of any stimulation treatment; resin-coated sand has properties that can retain stimulated production. For example, it has:

- 1) Superior fracture conductivity retention (permeability) under load, when compared with sand or glass beads.
Application: high-closure stress wells.
- 2) Minimum crushing because of a strong resin coating that prevents point-to-point loading. The result is constant permeability under high loads.
- 3) Minimum embedment in softer formations because of solid formations face against the sheet of solid resin-coated sand. This results in application to many types of formation due to the cohesive proppant and its solid structure in the fracture.
- 4) Cohesive strength that no other proppant has. This cohesiveness prevents fines migration and forms a consolidated structure in the fracture which retains high conductivity.

It can be used for the entire proppant system or it can be slugged in with other proppants. In either case, resin-coated sand solves many of the problems that have been inherent in hydraulic fracturing for years. Lab tests continue and more design ideas are evolving as experience is gained in the field.

The following figures (Figures 53 through 55) show the permeability versus closure stress of resin-coated sand and compare it to sand, glass beads, and sintered bauxite. The main information to glean from these

curves is that it holds its permeability as well as or better than sintered bauxite and is much superior to sand or glass beads. The other advantage, which is not shown, is its cohesiveness in the fracture to control sand or fines flow back. The lower specific gravity of Super Sand allows much less proppant per frac volume.

Figure 53. Super Sand and Sand Permeabilities Versus Closure Stress

Figure 54. Super Sand, Sintered Bauxite and Sand Permeability Versus Closure Stress

Figure 55. Super Sand, Sintered Bauxite and Sand Permeability Versus Closure Stress.

Figure 55A. Permeability Retention Under Closure Stress

2. Other resin-coated particles

Sintered bauxite and glass beads, or other materials can also be coated with the uncured resin to improve permeability under load and to add cohesiveness to the untreated proppant. The resin coating protects the proppant from temperature or chemical reactions to the natural fluids in the well or to fracturing fluids and at the same time prevents flowback, embedment, or any fines movement.

E. Miscellaneous Proppants

1. Deformable proppants

This category of proppants is the deformable, or "noncrushable," proppants. In general, these materials are ductile and deform rather than break or crush. The following deformable proppants were studied:

a. Steel or iron shot

Shot gives the best permeability load characteristics of any proppant studied and is under consideration for use in high-closure-stress wells. Steel shot cost approximately the same as glass beads on a volume basis and for this reason has the same economic restrictions as glass beads.

b. U.S.S. Max-Well particles

These particles are a steel-base proppant produced by U.S. Steel with permeability characteristics similar to glass beads. At the present time this proppant is not commercially available.

c. Walnut hulls

Walnut hulls have been suggested for use in monolayer packs. Since monolayers are not too practical, multilayer properties are usually considered. Data indicate that multilayer packs of walnut hulls have an extreme loss in permeability under load, with resulting low fracture permeabilities. Both temperature and water exposure on the walnut hulls are detrimental to its properties.

Steel or iron shot would be the ideal fracture proppant, except for high cost and corrosion problems. Tests indicate corrosion can be controlled with inhibitors, but cost cannot be greatly altered.

When stressed in a pack, steel shot do not fail but are only slightly deformed at points of contact with neighboring particles. Therefore, a very high permeability is retained at all closure stresses. For example, Figure 56 shows a comparison of the permeability versus closure stress of steel shot; 8-12 mesh, 12-18 mesh and 20-40 mesh sizes. Steel proppant has almost constant permeability, regardless of the closure stress.

Although U.S.S. Max-Well particles are not commercially available, four samples have been tested to allow comparison with available proppants. (Data for the four samples are given in Figure 57.)

Figure 56. Permeability--Closure Stress Curves for Steel Shot

Figure 57. Permeability--Closure Stress Curves for U.S.S. Max-Well Particles

d. Aluminum Pellet

Reported by Kern³ in 1960, aluminum pellets were designed for partial monolayer use. Corrosion was one major factor in their non-use along with an extremely high cost. Figure 58 gives fracture conductivity versus formation stress to allow for comparison with the other available proppants.

Figure 58. Comparison of Calculated and Experimental Conductivities for Cracks Sparsely Propped with Aluminum.

2. Nondeformable pellets

Many available minerals were tested to find out if they had desirable properties. The bauxites, aluminum oxide, and zirconium oxide have been reported in Section C. One other material that was considered for use as a proppant was garnet.

Common garnet grains are mined in states like Idaho and Maine; however, they are usually only available with long lead times. They do have about the same permeability as sand but availability usually prevails in the selection process. Figure 59 shows the permeability versus closure stress for garnet in air tests.

Figure 59. Permeability--Closure Stress Curves for Garnet

F. Permeability Reduction By Fluid Loss Additives And Fines

To evaluate the effects of fluid-loss additives on proppant permeability, a series of tests was performed in which a suspension of fluid-loss additives were circulated through a proppant pack and the proppant pack permeability recorded. Tests were run using 10-20 and 20-40 mesh "Heart of Texas" sand at closure stresses of 1000 and 3000 psi. In each sand the effect was evaluated by circulating an oil with 20 pounds of Adomite Mark II per 1000 gallons and a gelled brine containing 20 pounds of silica flour per 1000 gallons. Results are shown in Figure 60 and Figure 61. (In these figures a dimensionless permeability of 1.0 represents the permeability of the sand pack to the circulation fluid without additive.)

Data taken at 1000 psi (Figure 60) show that permeability of a 20-40 mesh sand pack is severely reduced by Adomite Mark II and that silica flour can produce a significant permeability reduction. However, 10-20 mesh sand suffers only a very slight permeability decrease with either fluid-loss additive. At 3000 psi closure stress (Figure 61) severe plugging occurs in 20-40 mesh sand with any additive. Again, plugging is not severe with 10-20 mesh proppant.

These tests do not model what occurs in a fracture, rather they are intended to show what can and probably will occur in some instances. These data suggest that the following precautions be taken:

- Do not use 20-40 mesh proppant with fluid-loss additives if larger proppant is available.
- Following a fracture treatment, backflow the well at a rate such that the closure stress is not larger than 3000 psi if 10-20 mesh or larger proppant is used, or 1000 psi if 20-40 mesh proppant must be used. Maintain this rate until at least the volume of fluid injected into the formation has been recovered.

Figure 60. Fracture Permeability Reduction by Fluid-Loss Additives--
1000 psi Closure Stress.

Figure 61. Fracture Permeability Reduction by Fluid-Loss Additives--
3000 Psi Closure Stress

Figure 62 shows the effect of 2000 psi closure on a mixed proppant of 10-20 mesh and 20-40 mesh particles. Also it should be noted that the addition of small quantities of a larger mesh, high-strength proppant to a lower strength sand does not improve the sand permeability.

Figure 62. Effect of 20-40 Mesh Particles on Permeability of 10-20 Mesh Proppant.

To illustrate the effect of a poor particle-size distribution, a series of experiments was performed in which varying amounts of 20-40 mesh particles were added to 10-20 and 8-12 mesh "Heart of Texas" sand. Typical permeability closure stress data are presented in Figure 63 for 100%, 90%, and 80% 10-20 mesh sand. These data show that if the sand contains 10% fine material, permeability is reduced by about 15%; with 20% fines, permeability is reduced by about 45%.

Results of all experiments to determine permeability decrease with increasing fines concentration can be combined by plotting the data as a dimensionless permeability rather than absolute permeability. In this plot (Figure 64) the dimensionless permeability is written as:

$$k_d = \frac{k_{mix} - k_{fin}}{k_{snd} - k_{fin}} \quad (2)$$

where:

k_d = dimensionless permeability

k_{fin} = permeability of the fine particles alone (in these experiments $k_{fin} = k_{20-40}$)

k_{snd} = permeability of the large sand without any fines (expected sand permeability).

The dimensionless permeability will vary between 1.0 and 0.0 in most instances, with a value of 1.0 indicating the mixture has the permeability for large sand alone. A value of $k_d = 0.0$ indicates the combined proppant permeability is equal to the permeability of the fines alone, and negative value for k_d indicates a mixed permeability lower than the permeability of the fine particles alone.

Figure 63. Permeability of Mixtures of 10-20 and 20-40 Mesh "Heart of Texas" Sand

Figure 64. Proppant Permeability Decline with Increasing Fines

G. Cost Comparisons

The cost of proppants is a significant part of the total cost of the well stimulation treatment. Larger and larger quantities of proppant are being used per treatment because of the popularity of massive treatments. During 1979, sand costs rose 5 to 6 cents per pound. Since enormous quantities of sand are being used for stimulation treatments, some spot shortages have appeared during the last several years.

The best way to compare cost is to assign sand a value of 1.0 based on 6¢/lb for 1979-1980 and then ratio present day costs of all proppants to the most current cost for sand. These values are given below in Table IV.

TABLE IV
COMPARISON OF PROPPING AGENTS FOR FRACTURING

<u>Material</u>	<u>Specific Gravity (SG)</u>	<u>Unit Cost per Pound</u>	
		<u>Actual</u>	<u>Adjusted for SG</u>
Sand	2.65	1.0	1.0
Walnut Hulls	1.28	5.3	2.5
High-Strength Glass Beads	2.65	24.0	24.0
Aluminum Pellets	2.65	37.0	37.0
Oil-Soluble Spacer	1.10	40.0	16.6
Iron or Steel Shot	7.82	7.5	22.0
Resin-Coated Sand	2.6	4.6	4.5
Sintered Bauxite	3.65	15.0	20.7

Other cost comparison graphs are shown in Figures 65 and 66 where relative costs unadjusted for specific gravity were based on 1977-1978 prices.

Figure 65. Cost Comparisons 1978--by Sinclair

Figure 66. Cost for Fracturing Conductivity at Varying Closure Stresses.

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IV. REFERENCES AND BIBLIOGRAPHY

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V. APPENDIX--TEST METHODS AND CONDITIONS

The earliest methods of measuring permeability under load was to push gas through linear and radial models which used a hydraulic jack for the varying closure stress. All data was taken with air or nitrogen at ambient conditions. Figures A-1 and A-2 show a radial test cell and system, respectively.

Figure A-1. Core Mounting Assembly

Figure A-2. Nitrogen Flow System for Measuring Fracture Capacity.

Later models of linear and radial flow added fluids of low temperature and heated platen hydraulic presses which more closely matched downhole conditions. Figure A-3 shows a schematic of a linear flow system to measure proppant permeability under load. The experimental procedure is explained in detail below.

Figure A-3. Apparatus for Measurement of Proppant Permeability and Fracture Flow Capacity.

The laboratory apparatus employed for determination of the permeability and fracture flow capacity of the various proppants is shown schematically in Figure A-3. The apparatus consists of a steel cell connected to a nitrogen or liquid source. The cell, inside dimensions 1.5 in. wide by 4 in. long, contains interchangeable plates between which the proppant is placed. The load to the proppant pack is applied by means of a 50-ton hydraulic press. The pressure drop across the proppant bed is determined by means of pressure taps at either end of the proppant bed or by maintaining a constant liquid head. In the case of gas permeability, a correction is made to the pressure drop to account for the compressibility of the gas. The flow of gas through the pack is determined by a flow meter having a range of 0 to 1300 cc/min. and the flow of liquid by collecting a timed volume. A micrometer gauge indicates the hydraulic cam travel to 0.001 in., from which the pack thickness was determined at each compaction pressure.

The procedure followed for determining the data presented was as follows. The proppant to be tested was weighed to the nearest 0.1 gm. and placed between the plates in the cell. Silicone rubber and/or Teflon was used to seal around the plates to prevent leakage under pressure and to assure that all fluid passage was through the proppant pack. The loaded cell was positioned in the hydraulic press and a pressure of 2000 psi was slowly applied. The fluid inlet and outlet fittings then were attached to the appropriate gas or liquid lines. The pressure was held for a minimum of 15 minutes and the fluid flow (Q), pressure drop (ΔP), and proppant pack thickness (W_f) recorded. The pressure then was increased incrementally and the same data recorded. The pack permeability was calculated from the standard Darcy equation, shown below.

$$K = \frac{Q\mu L}{A\Delta P} \quad (A-1)$$