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(71) Demandeur/Applicant:
BJ SERVICES COMPANY, US

(72) Inventeur/Inventor:
GUPTA, D.V. SATYANARAYANA, US

(74) Agent: BERESKIN & PARR

(54) Titre : METHODE D'UTILISATION DE POLYAMIDES LEGERS DANS LES OPERATIONS DE CONTROLE DE FRACTURATION ET D'ELIMINATION DU SABLE

(54) Title: METHOD OF USING LIGHTWEIGHT POLYAMIDES IN HYDRAULIC FRACTURING AND SAND CONTROL OPERATIONS

(57) **Abrégé/Abstract:**

Lightweight polyamide particulates may be used in treatment of subterranean formations, including hydraulic fracturing and sand control methods, such as gravel packing. The polyamide particulates typically have an apparent specific gravity (ASG) between from about 1.05 to about 2.0 and are stable at temperatures up to 500° C. The polyamide particulates may be used in combination with a filler which further serves to increase the strength and temperature stability of the resulting composite. Fracture conductivity may be increased by the placement of the low density polyamide particulates as a partial monolayer.



ABSTRACT OF THE DISCLOSURE

Lightweight polyamide particulates may be used in treatment of subterranean formations, including hydraulic fracturing and sand control methods, such as gravel packing. The polyamide particulates typically have an apparent specific gravity (ASG) between from about 1.05 to about 2.0 and are stable at temperatures up to 500° C. The polyamide particulates may be used in combination with a filler which further serves to increase the strength and temperature stability of the resulting composite. Fracture conductivity may be increased by the placement of the low density polyamide particulates as a partial monolayer.

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APPLICATION FOR PATENT**INVENTORS D. V. SATYANARAYANA GUPTA****:****TITLE: METHOD OF USING LIGHTWEIGHT POLYAMIDES IN
HYDRAULIC FRACTURING AND SAND CONTROL OPERATIONS****SPECIFICATION****Field of the Invention**

This invention relates generally to methods for treating subterranean formations, such as hydraulic fracturing treatments and sand control. In particular, this invention relates to use of lightweight polyamides as proppant material in hydraulic fracturing treatments or as particulate material in sand control methods such as gravel packing, frac pack treatments, *etc.*

Background of the Invention

Stimulation procedures often require the use of well treating materials having high compressive strength. In hydraulic fracturing, such materials must further be capable of enhancing the production of fluids and natural gas from low permeability formations. In a typical hydraulic fracturing treatment, fracturing treatment fluid containing a solid proppant material is injected into the wellbore at high pressures. Once natural reservoir pressures are exceeded, the fluid induces fractures in the formation and proppant is deposited in the fracture, where it remains after the treatment is completed. The proppant material serves to hold the fracture open, thereby enhancing the ability of fluids to migrate from the formation to the wellbore through the fracture. Because fractured well productivity depends on the ability of a fracture to conduct fluids from a formation to a wellbore, fracture conductivity is an important parameter in determining the degree of success of a hydraulic fracturing treatment. Choosing a proppant is critical to the success of well stimulation.

Proppants used in the art include sand, glass beads, walnut hulls, and metal shot as well as resin-coated sands, intermediate strength ceramics, and sintered bauxite; each

employed for their ability to cost effectively withstand the respective reservoir closure stress environment. The relative strength of these various materials increases with their corresponding apparent specific gravity (ASG), typically ranging from 2.65 for sands to 3.4 for sintered bauxite. Unfortunately, increasing ASG leads directly to increasing degree of difficulty with proppant transport and reduced propped fracture volume, thereby reducing fracture conductivity.

More recently, ultra lightweight (ULW) materials have been used as proppants since they reduce the fluid velocity required to maintain proppant transport within the fracture, which, in turn, provides for a greater amount of the created fracture area to be propped. Exemplary of such proppants are significantly lighter deformable particles. Such ULW proppants, like conventional heavier proppants, have the capability to effectively withstand reservoir closure stress environments while increasing fracture conductivity.

Materials of various specific gravities have been disclosed for use as deformable particulates for downhole conditions. For example, successful deformable particles include modified ground walnut hulls manufactured by impregnating closely sized walnut particles (i.e. 20/30 US mesh) with epoxy or other resins. These impregnated walnut hull particles are then coated with phenolic or other resins. Further exemplary of deformable particles are polystyrene divinylbenzene (PSDVB) deformable beads.

In addition to having low specific gravity, ULW proppants must also be of sufficient strength to withstand the rigors of high temperatures and high stresses downhole. ULW proppants, while offering excellent compressive strength, often soften and lose their compressive strength especially at high temperature and high pressure conditions. For instance, ULW proppants composed of resinous materials have been observed to deform at elevated temperatures to the extent that under a 5,000 psi stress load at temperatures greater than 250° F, the permeability of the ULW proppant pack is deformed beyond the limits of its commercial utility even though the melting point of the resin is at a temperature of well greater than 300° F.

Thus, alternative materials which exhibit high particle strength at high temperatures are needed for utilization in those applications which require high temperature and high pressure downhole conditions.

Summary of the Invention

Lightweight polyamide particulates are useful in treatment of well penetrating subterranean formations. In a preferred embodiment, lightweight polyamide particulates are used in sand control methods, including gravel packing, as well as hydraulic
5 fracturing.

The lightweight polyamide particulates are characterized by an apparent specific gravity (ASG) between from about 1.05 to about 2.00 and are stable at temperatures up to 500° C. In addition, the polyamide particulates exhibit crush resistance under conditions as high as 10,000 psi closure stress.

10 The polyamide particulates may be used in combination with one or more fillers which further serve to increase the strength and temperature stability of the resulting composite. Suitable as fillers are minerals, such as finely divided minerals or finely divided minerals and/or fibers, clay, hematite, inorganic salts, kaolin, molybdenum disulfide, oil, alumina, hematite, aluminum flake, stainless steel, silicone,
15 polytetrafluoroethylene, cement, carbon black, carbon Buckminster fullerenes, carbon nano-tubes, polyhedral oligomeric silsesquioxane, metals, metallic oxides, phosphates, borates, calcium carbonate, calcium chloride, calcium bromide, barium sulfate, aluminum flakes, a modified naturally occurring material, crushed nut shells, ground or crushed seed shells, ground or crushed fruit pits, processed wood and organic polymeric
20 materials. Filled polyamides containing such fillers render higher permeability factors than unfilled polyamides.

When used in hydraulic fracturing, a suspension of lightweight and/or substantially neutrally buoyant polyamide particulates in a carrier fluid may be injected into a subterranean formation in conjunction with a hydraulic fracturing treatment or
25 other treatment at pressures sufficiently high enough to cause the formation or enlargement of fractures or to otherwise expose the aggregates to formation closure stress. Such other treatments may be near wellbore in nature (affecting near wellbore regions) and may be directed toward improving wellbore productivity and/or controlling the production of fracture proppant or formation sand. Particular examples include
30 gravel packing and frac-packs.

Fracture conductivity may be increased by use of the low density polyamide proppant as a partial monolayer.

Brief Description of the Drawings

5 In order to more fully understand the drawings referred to in the detailed description of the present invention, a brief description of each drawing is presented, in which:

FIG. 1 compares the permeability differences between a filled and an unfilled polyamide proppant used in hydraulic fracturing.

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Detailed Description of the Preferred Embodiments

The lightweight and/or substantially neutrally buoyant polyamide proppant or sand control particulates for use in the treatment of well penetrating subterranean formations, in accordance with the invention, have low specific gravity and are stable at
15 temperatures up to 500° C.

The lightweight polyamide particulates exhibit crush resistance under conditions as high as 10,000 psi closure stress, API RP 56 or API RP 60, generally between from about 250 to about 8,000 psi closure stress. For instance, the polyamide particulates may be employed as proppant/sand control particulates at temperatures as high as, for
20 example, 700°F and pressures up to about 8,000 psi.

The lightweight polyamide particulate are characterized by an apparent specific gravity (ASG) (API RP 60) which is substantially less than the ASG of a conventional particulate material employed in hydraulic fracturing or sand control operations. Since polyamides, such as nylon, exhibit low molecular weight, the ASG of the proppant is
25 typically between from about 1.05 to about 2.00. In a preferred embodiment, the polyamide particulate has an ASG less than or equal to 1.75, most preferably less than or equal to 1.50.

The thermoplastic polyamide particulates are further non-reactive in that they are not curable at in-situ temperatures or elevated heat conditions. Being thermoplastic, the
30 polyamide particulates for use in the invention may be employed in the absence of a cold set resin, binder or cementitious agent. Preferred polyamides include polyamide-6, 6 as

well as polyamide 6, such as Technyl® polyamides from Rhodia Engineering Plastics. Further suitable polyamides are polyamide 6,10; polyamide 6,12; polyamide 4,6, polyamide 11 and polyamide 12.

5 The polyamide particulates may be of any size or shape suitable for hydraulic fracturing and sand control methodology. In addition to being substantially spherical, such as being beaded, or pelleted, the particulates for use in the invention may be non-beaded and non-spherical such as an elongated, tapered, egg, tear-drop or oval shape or mixtures thereof. For instance, the particulates may have a shape that is cubic, bar-shaped (as in a hexahedron with a length greater than its width, and a width greater than
10 its thickness), cylindrical, multi-faceted, irregular, or mixtures thereof. In addition, the particulates may have a surface that is substantially roughened or irregular in nature or a surface that is substantially smooth in nature. Moreover, mixtures or blends of particulates having differing, but suitable, shapes for use in the disclosed method further be employed.

15 In one embodiment, when the particulates have a cylindrical shape or an elongated beaded shape with a substantially uniform diameter, the particulates may have a maximum length aspect ratio equal to or less than about 5. As used herein, "maximum length based aspect ratio" means the maximum aspect ratio that may be obtained by dividing the length of the particulate by the minimum (or shortest) dimensional value that
20 exists along any other axis (other than the length axis) taken through the center of mass of the particulate.

The lightweight and/or substantially neutrally buoyant polyamide particulates may be formed from materials that are chipped, ground, crushed, or otherwise processed to produce particulate material having the desired particle size. In one exemplary
25 embodiment, particle sizes include, but are not limited to, sizes ranging from about 4 mesh to about 200 mesh, alternatively from about 12 mesh to about 50 mesh.

Particle size of the disclosed particulate materials may be selected based on factors such as anticipated downhole conditions and/or on relative strength or hardness of the particulate material/s selected for use in a given application. In this regard, larger
30 particle sizes may be more desirable in situations where a relatively lower strength particulate material is employed. For example, 12/20 mesh polyamide particulates may

be desirable for use where closure stresses of up to about 1500 psi are anticipated. Smaller particle sizes may be more desirable in situations where a relatively higher strength particulate material is employed. For example 20/40 mesh polyamide particulates may be desirable for use where closure stresses of up to about 4000 psi.

5 The polyamide particulates deform with stress and yet are sufficiently strong to be used on their own at high pressures. Since breakage is unlikely to occur, the polyamide particulates supporting the fracture walls do not generate fines. They may further be used in conjunction with other well treatment agents including more conventional proppant materials, such as sand.

10 The polyamide particulates may be used in conjunction with one or more fillers to form a well treating composite. Such fillers are non-reactive and further serve to increase the strength and temperature stability of the polyamide composite as well as to modify the density of the particulate.

 The presence of such fillers further render a well treating composite having a
15 ASG different than the ASG of the polyamide particulate. Volume proportions of filler may be selected so as to control crush strength and temperature stability.

 Typically, the particle size of the filler range from about 100 nm to about 200 μm .

 Suitable as fillers are minerals (such as finely divided minerals or finely divided minerals and/or fibers) optionally bound by a suitable organic or inorganic binder.
20 Suitable minerals include fly ash, silica and sand (including fumed silica, quartz sand, and silica flour), alumina, mica, silicates, such as orthosilicates and metasilicates, aluminum silicate and calcium silicate, kaolin, talc, zirconia, boron and glass, such as glass spheres (especially glass microspheres), glass powder, glass beads, glass bubbles, ground glass, borosilicate glass and fiberglass. Suitable fibers include mineral fibers,
25 glass fibers, ceramic fibers, carbon fibers, polymeric fibers, coated fibers (such as nickel coated carbon fibers) and synthetic fibers. Further, suitable fillers include clay, hematite, alkali metal salts, molybdenum disulfide, oil, aluminum flake, stainless steel, silicone, polytetrafluoroethylene, cement, inorganic salts, carbon black, carbon Buckminster
30 fullerenes, carbon nano-tubes, polyhedral oligomeric silsesquioxane, metals, metallic oxides (such as trimanganese tetraoxide), metallic salts (including alkali metal salts), phosphates, borates, calcium carbonate, calcium chloride, calcium bromide, barium

sulfate, aluminum flakes, a modified naturally occurring material, crushed nut shells, ground or crushed seed shells, ground or crushed fruit pits, processed wood and organic polymeric materials. Further, the filler may contain a cation selected from the group consisting of alkali metals, alkaline earth metals, ammonium, manganese, and zinc and an anion selected from the group consisting of a halide, an oxide, a carbonate, nitrate, sulfate, acetate and formate.

The amount of filler(s) in the composite is such as to impart to the well treating composite the desired ASG. Typically, the amount of filler in the well treating composite is between from about 1 to about 85, more typically between from about 25 to about 60, percent by volume of the well treating composite. The amount of filler and polyamide particulate may be adjusted to tailor the composite to achieve the desirable physical properties, including particle density, bulk density, crush strength, etc.

The well treating composites are generally prepared by blending the polyamide particulate with the selected filler(s), in a molten state, and forming a composite of the desired size and physical properties. This can be achieved by known techniques in the powder/particulate industry such as melt processing, cryogenic grinding, etc.

The polyamide particulates may be employed with carrier or treatment fluids in order to facilitate placement of the composite to a desired location within the formation.. Any carrier fluid suitable for transporting the particulate into a well and/or subterranean formation fracture in communication therewith may be employed including, but not limited to, carrier fluids including a completion or workover brine, salt water or brine, fresh water, potassium chloride solution, a saturated sodium chloride solution, liquid hydrocarbons or a gas or liquefied gas such as nitrogen or carbon dioxide.

The fluids may be gelled, non-gelled or have a reduced or lighter gelling requirement as compared to carrier fluids employed with conventional fracture treatment/sand control methods. The latter may be referred to as "weakly gelled", i.e., having minimum sufficient polymer, thickening agent, such as a viscosifier, or friction reducer to achieve friction reduction when pumped downhole (*e.g.*, when pumped down tubing, work string, casing, coiled tubing, drill pipe, *etc.*), and/or may be characterized as having a polymer or viscosifier concentration of from greater than 0 pounds of polymer per thousand gallons of base fluid to about 10 pounds of polymer per thousand gallons of

base fluid, and/or as having a viscosity of from about 1 to about 10 centipoises. The non-gelled carrier fluid typically contains no polymer or viscosifier.

Gelling agents for proppant carrier fluids may provide a source of proppant pack and/or formation damage, and settling of proppant may interfere with proper placement
5 downhole. The resulting suspension preferably forms a pack of particulate material that is permeable to fluids produced from the wellbore and substantially prevents or reduces production of formation materials from the formation into the wellbore.

The carrier or fracturing fluid may further contain one or more conventional additives to the well service industry such as a gelling agent, crosslinking agent, gel
10 breaker, surfactant, biocide, surface tension reducing agent, foaming agent, defoaming agent, demulsifier, non-emulsifier, scale inhibitor, gas hydrate inhibitor, polymer specific enzyme breaker, oxidative breaker, buffer, clay stabilizer, acid, buffer, solvent or a mixture thereof and other well treatment additives known in the art. The addition of such additives to the carrier fluids minimizes the need for additional pumps required to add
15 such materials on the fly.

The polyamide particulates may be advantageously pre-suspended as a substantially neutrally buoyant particulate and stored in the carrier fluid (*e.g.*, brine of near or substantially equal density), and then pumped or placed downhole as is, or diluted on the fly.

The term “substantially neutrally buoyant” refers to polyamide particulates that
20 have an ASG sufficiently close to the ASG of the selected ungelled or weakly gelled carrier fluid (*e.g.*, ungelled or weakly gelled completion brine, other aqueous-based fluid, slick water, or other suitable fluid) which allows pumping and satisfactory placement of the proppant/particulate using the selected ungelled or weakly gelled carrier fluid. For
25 example, polyamides having an ASG of from about 1.25 to about 1.35 may be employed as a substantially neutrally buoyant proppant/particulate in completion brine having an ASG of about 1.2.

In a preferred embodiment the lightweight polyamide proppant or particulates are suspended in a carrier fluid and introduced into the subterranean formation at a pressure
30 above a fracturing pressure of the subterranean formation. In this method, at least a portion of the particulate material is substantially neutrally buoyant in the carrier fluid.

Further, the lightweight polyamide and/or substantially neutrally buoyant proppant or particulate is used in a sand control method for a wellbore penetrating a subterranean formation and may be introduced into the wellbore in a slurry with a carrier fluid; the particulate material being placed adjacent the subterranean formation to form a fluid-
5 permeable pack that is capable of reducing or substantially preventing the passage of formation particles from the subterranean formation into the wellbore while at the same time allowing passage of formation fluids from the subterranean formation into the wellbore. In this method at least a portion of the individual particles of the particulate material may be substantially neutrally buoyant in the carrier fluid.

10 The polyamide particulates are easy to place within a targeted zone due to lessened settling constraints. The reduced mass of the polyamide particulates is generally required to fill an equivalent volume than is required with conventional sand control particulates used, for example, for gravel packing purposes.

When employed in well treatments, the lightweight and/or substantially neutrally
15 buoyant particulates may be introduced into the wellbore at any concentration deemed suitable or effective for the downhole conditions to be encountered. For example, a well treatment fluid may include a suspension of proppant or sand control aggregates.

The low ASG of the polyamide particulate and/or substantially neutrally buoyant polyamide particulate may result in a larger fracture or frac pack width for the same
20 loading (*i.e.*, pound per square foot of proppant) to give much larger total volume and increased width for the same mass. Alternatively, this characteristic allows for smaller masses of proppant or sand control particulates to be pumped while still achieving an equivalent width.

The use of a non-gelled carrier fluid eliminates a source of potential proppant
25 pack and/or formation damage and enhancement of well productivity. Elimination of the need to formulate a complex suspension gel may further mean a reduction in tubing friction pressures, particularly in coiled tubing and in the amount of on-location mixing equipment and/or mixing time requirements, as well as reduced costs. In one
30 embodiment employing a substantially neutrally buoyant polyamide particulate and a brine carrier fluid, mixing equipment need only include such equipment that is capable of

(a) mixing the brine (dissolving soluble salts), and (b) homogeneously dispersing in the substantially neutrally buoyant polyamide particulate.

When used in hydraulic fracturing, the composite may be injected into a subterranean formation in conjunction with a hydraulic fracturing fluid at pressures sufficiently high enough to cause the formation or enlargement of fractures.

The polyamide particulates may be mixed with the carrier fluid in any manner suitable for delivering the mixture to a wellbore and/or subterranean formation. In one embodiment, the disclosed particulates may be injected into a subterranean formation in conjunction with a hydraulic fracturing treatment or other treatment at pressures sufficiently high enough to cause the formation or enlargement of fractures, or to otherwise expose the particles to formation closure stress. Such other treatments may be near wellbore in nature (affecting near wellbore regions) and may be directed toward improving wellbore productivity and/or controlling the production of fracture proppant or formation sand. Particular examples include gravel packing and "frac-packs." Moreover, such particulates may be employed alone as a fracture proppant/sand control particulate, or in mixtures in amounts and with types of fracture proppant/sand control materials, *e.g.*, conventional fracture or sand control particulate.

The composites of the invention are particularly effective in hydraulic fracturing as well as sand control fluids such as water, salt brine, slickwater such as slick water fracture treatments at relatively low concentrations to achieve partial monolayer fractures, low concentration polymer gel fluids (linear or crosslinked), foams (with gas) fluid, liquid gas such as liquid carbon dioxide fracture treatments for deeper proppant penetration, treatments for water sensitive zones, and treatments for gas storage wells. The polyamide particulates demonstrate strong stability in liquid carbon dioxide.

The diameter of the proppant pack is substantially similar to the width of the created fracture. Fracture conductivity may be increased by the placement of the low density polyamide particulates as a partial monolayer to support the fracture. Fractures containing polyamide partial monolayers exhibit vacant areas around and between particulates which thereby increases the relative conductivity of the propped fracture. The polyamide monolayer is created when the propped fracture has a width that is equal to one particle diameter with no remaining voids into which additional particles may be

placed. While the packing of a complete monolayer of proppant is 2 pounds per square foot, the packing of a partial monolayer of proppant is typically between from about 0.02 to about 0.8 lbs. per sq. ft for a polyamide proppant with ASG between 1.1 and 1.5. Such packing causes an increase in porosity of the fracture. The resulting partial monolayer of polyamide particulate as proppant exhibits greater conductivity than that evidenced with the complete monolayer. For instance, at 4000 plus psi, the permeability of a formation has been seen to be much higher than a 1 lb per square foot pack polyamide proppant. Testing protocols for determination of proppant packing are set forth in Harold D. Brannon et al., "Maximizing Fracture Conductivity with Proppant Partial Monolayers; Theoretical Curiosity or Highly Productive Reality?", Paper SPE 90698, presented at the 2004 SPE Annual Technical Conference and Exhibition, Houston, Texas, September 26-29, 2004, herein incorporated by reference.

The polyamide particulates may be employed to simplify hydraulic fracturing treatments or sand control treatments performed through coil tubing, by greatly reducing fluid suspension property requirements. When placed downhole, the polyamide particulates exhibit a much reduced propensity to settle (as compared to conventional proppant or sand control particulates), particularly in highly deviated or horizontal wellbore sections.

In this regard, the polyamide particulates may be advantageously employed in any deviated well having an angle of deviation of between about 0° and about 90° with respect to the vertical. However, in one embodiment, the polyamide particulates may be advantageously employed in horizontal wells, or in deviated wells having an angle with respect to the vertical of between about 30° and about 90°, alternatively between about 75° and about 90°.

The polyamide particulate may be introduced as part of a treating fluid into a well down wellbore tubulars (*e.g.*, tubing, workstring, casing, drill pipe) or down coiled tubing, for example at concentrations of about 0.25 to about 15 pounds per gallon of treating fluid. In one exemplary embodiment, specific gravity of the polyamide particulates may be about 1.5, and therefore they may be used as a substantially neutrally buoyant proppant or sand control particulate in light or heavy brines, thus eliminating the need for complex crosslinked fracturing or sand control carrier fluids.

The polyamide particulates may further be utilized as particulate/proppant material at more severe or demanding downhole conditions (*e.g.*, at higher downhole temperatures and/or under higher downhole conditions of closure stress) than the conditions under which conventional particulates are suitably employed. For example, in
5 one exemplary embodiment, the polyamide particulate may be formulated as described herein and introduced or pumped (*e.g.*, as neutrally buoyant proppant and/or sand control particulate) in any suitable carrier fluid (*e.g.*, saturated sodium chloride solution carrier fluid or other completion or workover brine) into a well for exposure to downhole temperatures of greater than or equal to about 300°F.

10 In one exemplary embodiment, a gravel pack operation may be carried out on a wellbore that penetrates a subterranean formation to prevent or substantially reduce the production of formation particles into the wellbore from the formation during production of formation fluids. The subterranean formation may be completed so as to be in communication with the interior of the wellbore by any suitable method known in the art,
15 for example by perforations in a cased wellbore, and/or by an open hole section. A screen assembly such as is known in the art may be placed or otherwise disposed within the wellbore so that at least a portion of the screen assembly is disposed adjacent the subterranean formation. A slurry including the lightweight and/or substantially neutrally buoyant particulates and a carrier fluid may then be introduced into the wellbore and
20 placed adjacent the subterranean formation by circulation or other suitable method so as to form a fluid-permeable pack in an annular area between the exterior of the screen and the interior of the wellbore that is capable of reducing or substantially preventing the passage of formation particles from the subterranean formation into the wellbore during production of fluids from the formation, while at the same time allowing passage of
25 formation fluids from the subterranean formation through the screen into the wellbore. It is possible that the slurry may contain all or only a portion of the lightweight and/or substantially neutrally buoyant particulates. In the latter case, the balance of the particulate material of the slurry may be another material, such as a conventional gravel pack or sand control particulate.

30 As an alternative to use of a screen, the sand control method may use the lightweight polyamide and/or substantially neutrally buoyant particulates in accordance

with any method in which a pack of particulate material is formed within a wellbore that it is permeable to fluids produced from a wellbore, such as oil, gas, or water, but that substantially prevents or reduces production of formation materials, such as formation sand, from the formation into the wellbore. Such methods may or may not employ a gravel pack screen, may be introduced into a wellbore at pressures below, at or above the fracturing pressure of the formation, such as frac pack, and/or may be employed in conjunction with resins such as sand consolidation resins if so desired.

Examples

The following examples will illustrate the practice of the present invention in its preferred embodiments. All parts are given in terms of weight units except as may otherwise be indicated. Other embodiments within the scope of the claims herein will be apparent to one skilled in the art from consideration of the specification and practice of the invention as disclosed herein. It is intended that the specification, together with the Examples, be considered exemplary only, with the scope and spirit of the invention being indicated by the claims which follow.

Example 1:

A polyamide composite containing 40 weight percent kaolin was obtained from Rhodia Engineering Plastics. The ASG of the composite was 1.46 and the bulk density was 0.87 g/cc and the size was about a 36/60 mesh and the shape was substantially spherical.

Conductivity tests were performed according to a modified API RP 61 (1st Revision, Oct. 1, 1989) using an API conductivity cell with Ohio sandstone wafer side inserts to simulate the producing formation. A multilayer pack of the composite containing about 31.5 g of proppant was then loaded between the sealed sandstone wafers to increase the propped width. The proppant pack exhibited a density of about 1 lb/ft².

The conductivity cell was then placed on a press while stress was applied at 100 psi/minute until the target temperature was reached. Fluid was then allowed to flow through the test pack maintaining Darcy flow. The differential pressure was measured across 5 inches of the pack using a "ROSEMOUNT" differential pressure transducer (#3051C). Flow was measured using Micromotion mass flow meters and data points

were recorded every 2 minutes for 50 hours. An Isco 260D programmable pump applied and maintained effective closure pressure.

Experimental parameters and results are set forth in Table I below:

5

Table I

Temperature: 53.4° C
 Closure Pressure (psi): 2000-6000
 Fluid pressure (psi): 500

<u>Time</u>	<u>Width</u> <u>mm</u>	<u>Conductivity</u> <u>md-ft</u>	<u>Permeability</u> <u>Darcies</u>	<u>Closure</u> <u>Psi</u>
0	6.32	4722	228	2110
10	5.97	4897	250	1949
20	5.97	4687	239	2030
30	5.97	4699	240	2030
40	6.17	4559	333	2029
50	6.17	4509	330	2024
0	5.84	3757	196	2601
10	3.77	1597	129	4016
20	3.77	1600	129	4016
30	3.77	1599	129	4016
40	3.74	1459	119	4012
50	3.72	1408	115	4012
60	3.71	1348	111	4009
70	3.70	1339	110	4005
80	3.70	1335	110	4005
90	3.70	1328	109	4005
100	3.69	1339	110	4006
0	3.07	595	59	6006
10	3.07	322	32	6006
20	3.42	291	26	6034
30	3.38	238	22	6033
40	3.38	392	35	6046
50	3.37	350	32	6038
60	3.43	170	15	6178
70	3.42	119	11	6054
80	3.37	117	11	6052

10

As may be seen from the results of this example, introduction of a polyamide composite achieves the requisite fracture conductivity. Further, Table I illustrates high

conductivity at increasing stress levels. As shown, significant strength is imparted by the polyamide proppants across the closure range.

Example 2:

- 5 Conductivity tests were performed on the composite of Example 1 according to the recited procedure at varying temperatures. Experimental parameters and results are shown in Table II below:

Table II

10

Closure Pressure (psi): 5000

Fluid Pressure (psi): 500

<u>Time</u> <u>Hours</u>	<u>Temp</u> <u>°C</u>	<u>Closure</u> <u>Psi</u>	<u>Conductivity</u> <u>md-ft</u>	<u>Width</u> <u>mm</u>	<u>Permeability</u> <u>darcies</u>
0	24.4	1019	174988	5.11	10447
0	65.5	1024	38164	5.03	2313
10	65.2	1029	24054	4.98	1473
20	64.9	1024	22784	4.95	1402
0	65.1	5091	1793	4.01	136
10	64.8	5096	1176	3.86	93
20	65.2	5096	1123	3.86	89
0	74.5	5091	639	3.76	52
10	79.4	5096	529	3.73	43
20	79.5	5018	480	3.73	39
30	79.5	5018	472	3.73	38
40	79.5	5013	385	3.73	31
50	79.6	5018	448	3.73	37
60	79.6	5013	502	3.73	41
70	79.3	5018	423	3.71	35
0	93.5	5013	233	3.68	19
10	93.6	5018	173	3.68	14
20	93.4	5013	192	3.68	16
30	93.5	5013	168	3.68	14
40	93.4	5013	186	3.68	15
50	93.5	5013	169	3.68	14
60	93.7	5018	180	3.68	15
70	93.7	5018	165	3.68	14

Table II illustrates high conductivity at increasing temperatures while stress is maintained at around 5,000 psi.

Example 3:

5 The polyamide composite of this Example was obtained from pilot run samples. The polyamide composite was prepared as set forth in Example 1 but on a production scale. A partial monolayer proppant pack was created by loading 1.3 grams of proppant as a pack between the sealed sandstone wafers. The proppant pack exhibited a loading of about 0.041 lb/ft². Conductivity tests were then run as described in Example 1.
10 Experimental parameters and results for the test are shown in Table III below:

Table III

15 Closure Pressure (psi): 1000-6000
Fluid Pressure (psi): 500

<u>Time</u> <u>Hours</u>	<u>Temp</u> <u>°C</u>	<u>Closure</u> <u>Psi</u>	<u>Conductivity</u> <u>md-ft</u>	<u>Width</u> <u>mm</u>	<u>Permeability</u> <u>darcies</u>
0	65.6	1073	430904	1.08	121381
10	113.0	1078	101466	0.884	34988
0	110.0	2028	37598	0.884	13349
10	110.0	2028	43136	0.818	16075
20	110.2	2033	43085	0.800	16413
24	111.2	2024	38225	0.782	14893
0	110.3	4018	27210	0.696	11917
10	109.8	4023	19153	0.691	8450
20	109.2	4018	17119	0.681	7665
24	110.2	4018	17426	0.676	7861
0	110.4	6096	10465	0.368	8661
10	110.3	6086	1368	0.351	1189
20	110.4	6086	1209	0.338	1095
24	109.9	6091	1185	0.323	1120

20 Table III shows the effect of varying the closure pressure on the permeability of the pack. As may be seen from the results of this example, use of a partial monolayer of polyamide renders higher permeability than that seen with the conventional polyamide of

Example 1. Note, for instance, that at 4000 plus psi, the permeability of the formation is much higher than a 1 lb per square foot pack at lower width.

Example 4:

- 5 Polyamide beads of nylon-6, 6 were obtained from Rhodia Engineering Plastics. The ASG of the polyamide beads was 1.12 and the bulk density was 0.69 g/cc and the size distribution was 8 to 12 mesh. Conductivity tests were performed using 31.5 g of the unfilled polyamide 6, 6 in accordance with the procedures set forth in Example 1. The test parameters and results are set forth in Table IV below.

10

Table IV

Closure Pressure (psi): 1000-4000
Fluid Pressure (psi): 500

<u>Time</u> <u>Hours</u>	<u>Temp</u> <u>°C</u>	<u>Closure</u> <u>Psi</u>	<u>Conductivity</u> <u>md-ft</u>	<u>Width</u> <u>mm</u>	<u>Permeability</u> <u>darcies</u>
0	26.0	1032	18627	8.05	706
10	26.7	1033	15073	6.909	665
24	26.7	1029	12705	6.858	565
0	25.5	4007	1670	5.639	90
5	24.9	4008	1445	5.588	79
0	37.0	4008	694	5.334	40
5	37.0	4008	672	5.334	38
10	50.9	4014	464	5.232	27
20	51.0	4014	421	5.232	25
30	65.0	4008	294	4.343	21
40	65.0	4014	285	4.343	20
50	65.0	4012	278	4.318	20
60	81.8	4013	261	4.801	17
70	79.5	4013	190	4.775	12
80	79.5	4010	184	4.775	12
90	93.2	4009	126	4.978	8
100	93.0	4011	121	4.978	7
110	121.7	4009	59	4.928	4
120	121.0	4013	55	4.902	3

15

As illustrated in Table IV, introduction of polyamide into a formation achieves requisite fracture conductivity.

5 Example 5:

Conductivity tests were conducted at 37°C with the 31.5 g of the unfilled polyamide beads of Example 4 and 31.5 g of the filled polyamide composite of Example 1. The tests were conducted in accordance with the procedures set forth in Example 1. The results are set forth in Table V below.

10

Table V

Closure Pressure (psi): 1000–5000

Fluid Pressure (psi): 500

Unfilled polyamide

15

<u>Time</u> <u>Hours</u>	<u>Closure</u> <u>Psi</u>	<u>Conductivity</u> <u>md-ft</u>	<u>Width</u> <u>mm</u>	<u>Permeability</u> <u>Darcies</u>
0	994	16674	6.12	830
10	991	11458	6.05	577
0	1991	4093	5.56	225
10	1997	2701	5.23	157
20	1997	2263	5.23	132
30	1994	1995	5.21	117
40	1996	1808	5.18	106
50	1994	1669	5.18	98
60	1989	1564	5.18	92
70	1992	1476	5.18	87
80	2002	1391	5.11	83
90	2005	1321	5.11	79
0	2963	898	4.93	56
10	2967	739	4.88	46
20	2965	692	4.85	43
30	2963	652	4.85	41
40	2964	630	4.83	40
50	2960	614	4.78	39

Filled Time, Hours	polyamide Closure, Psi	Conductivity, md-ft	Width, Mls	Permeability, Darcies
0	1045	11702	5.11	698
10	1036	7686	4.97	471
0	2007	2535	4.56	169
10	1995	2035	4.50	158
20	1998	2005	4.11	149
30	1998	2261	4.10	168
40	2016	2200	4.10	164
50	2027	2194	4.09	163
0	2936	1387	3.98	106
10	2946	785	3.93	61
20	2937	740	3.92	58
30	2949	780	3.91	61
40	2977	745	3.91	58
50	2985	737	3.90	58
0	4007	506	3.81	40
10	4005	407	3.78	33
20	4002	387	3.77	31
30	4003	377	3.70	31
40	3999	365	3.66	30
50	3999	359	3.66	30
0	5009	283	3.61	24
10	5009	224	3.56	19
20	5009	217	3.55	19
30	5007	211	3.55	18
40	5006	209	3.54	18
50	5007	208	3.54	18

Table V illustrates that higher permeability is obtained using filled polyamide composites versus unfilled polyamide. The distinction between filled and unfilled polyamide is graphically displayed in FIG. 1.

From the foregoing, it will be observed that numerous variations and modifications may be effected without departing from the true spirit and scope of the novel concepts of the invention.

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