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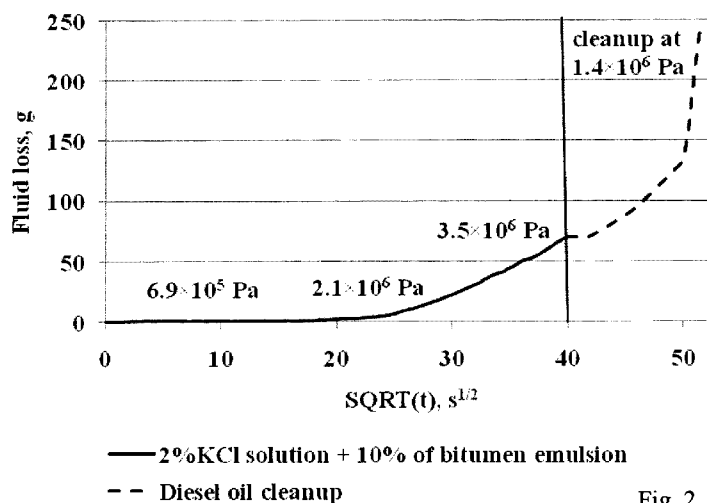


Fig. 2

(57) Abstract: A method is disclosed in which an aqueous bitumen-internal emulsion is injected into a wellbore to treat a subterranean formation. The emulsion is then typically inverted, by natural means or by an emulsion inversion triggering agent. The bitumen then performs one or more of a variety of functions, such as fluid loss control, bridging and plugging control required for diversion, fines migration control, and flowback control. The bitumen may optionally be removed.

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BITUMEN EMULSIONS FOR OILFIELD APPLICATIONS

Background of the Invention

The invention is related to well stimulation for increasing oil and gas production. There are different approaches to well stimulation, and traditionally those technologies are sensitive to the formation properties such as porosity, temperature, stress, and chemical composition. Every stimulation case requires fine tuning of the composition stimulation material and there is still a need for more options.

Summary of the Invention

One embodiment of the invention is a method for treating a subterranean wellbore, fracture, fluid flowpath and/or formation involving injecting an aqueous fluid containing a bitumen-internal emulsion into a well. The emulsion is typically stabilized by a surfactant. The emulsion is typically inverted in the inverted in the wellbore, fracture, fluid flowpath and/or formation.

Inversion of the emulsion is normally triggered by adding an emulsion inversion triggering agent. In various embodiments, the emulsion inversion triggering agent is injected before the injection of the fluid comprising the emulsion, or is injected at the same time as the injection of the fluid comprising the emulsion, or is injected in a second fluid which is injected with or after injection of the fluid comprising the emulsion. Optionally, the triggering agent is in the emulsion and/or is encapsulated. In other embodiments, the emulsion inversion triggering agent adheres to a subterranean surface, or is in a coating on a solid particulate in the fluid, or is itself a solid particulate. Optionally the fluid

containing the bitumen-internal emulsion contains a precursor of the emulsion inversion triggering agent.

In yet other embodiments, the fluid contains a proppant, fibers, or particulates or any two or all three of these. Optionally, the fibers or particulates or both are degradable.

In a further embodiment the bitumen deposited during or after the emulsion inversion is later removed by injecting an organic solvent for the bitumen.

In yet a further embodiment, the bitumen emulsion is added to only a portion of the injected fluid (for example to the portion containing proppant when proppant is injected in slugs).

Another embodiment is a method for treating a subterranean formation involving injecting an oil-based fluid containing a bitumen emulsion.

Brief Description of the Drawings

Figure 1 is a schematic of the laboratory fluid loss testing apparatus.

Figure 2 shows results of a test of a bitumen emulsion used for fluid loss control followed by cleanup with diesel oil in a sandstone core.

Figure 3 is a schematic of the laboratory apparatus and the slots used for bridging tests.

Figure 4 shows results of a test of bridging of a bitumen emulsion - fiber slurry in a 4 mm slot.

Figure 5 is a schematic of the laboratory apparatus used for testing emulsion sealing (plugging) of proppant-fiber packs.

Detailed Description of the Invention

Although some of the following discussion emphasizes diversion and fluid loss control in fracturing, the bitumen emulsions and methods of the invention may be used in many other downhole operations. The invention will be described in terms of treatment of vertical wells, but is equally applicable to wells of any orientation. The invention will be described for hydrocarbon production wells, but it is to be understood that the invention may be used for wells for production of other fluids, such as water or carbon dioxide, or, for example, for injection or storage wells. It should also be understood that throughout this specification, when a concentration or amount range is described as being useful, or suitable, or the like, it is intended that any and every concentration or amount within the range, including the end points, is to be considered as having been stated. Furthermore, each numerical value should be read once as modified by the term “about” (unless already expressly so modified) and then read again as not to be so modified unless otherwise stated in context. For example, “a range of from 1 to 10” is to be read as indicating each and every possible number along the continuum between about 1 and about 10. In other words, when a certain range is expressed, even if only a few specific data points are explicitly identified or referred to within the range, or even when no data points are referred to within the range, it is to be understood that the inventors appreciate and understand that any and all data points within the range are to be considered to have been specified, and that the inventors have possession of the entire range and all points within the range.

We describe here the use of bitumen emulsions, either commercially available or prepared for the purpose, for a variety of

downhole applications. As a stimulation fluid additive, a bitumen emulsion provides advantages as it interacts with other materials in the fluid or in the formation, or interacts with the formation.

We have found that bitumen emulsions, for example when used as additives to fracturing fluids, provide many benefits for downhole applications. The emulsion may optionally be inverted downhole, for example by chemicals (in the fluid, in another injected fluid, or in the formation,) by heat, by shear or by contact with a rough or convoluted surface. As one example of use in the invention, bitumen is readily obtained in commercial emulsions in the water-continuous phase as soft bitumen particles or emulsion droplets, and when the emulsion inverts the bitumen adheres to the pores of a formation, providing fluid loss control. We have also found that proppant/fiber/bitumen emulsion systems provide better bridging and more stable plugging of natural and artificial fractures and perforations, enhanced by the tendency of the bitumen to adhere to other surfaces. Inverted bitumen emulsions increase the adhesion of fibers to one another, providing a downhole fiber network that prevents proppant and fines migration and increases bridging. Since they are easily soluble in oil, bitumens precipitated from emulsions provide a reversible or temporary effect and may be removed by contact with oil or other organic solvents during flowback or in other treatment stages.

Bitumen emulsions are well known materials used extensively, for example, in heavy oil lifting *via* a steam injection technique, as emulsion fuels, in road cold pavements, and in building water-proofing. In some cases, heavy oil transport difficulties have been solved by delivery in emulsion form from the place of origin to other locations. An example is ORIMULSION[®], an oil-in-water emulsion of 70 percent Orinoco

bitumen and 30 percent water (and a small amount of surfactant) produced by or for PDVSA in Venezuela. The bitumen occurs naturally, but to deliver this oil to processing plants, the bitumen is turned into a much less viscous oil-in-water emulsion. Note that a water-in-oil emulsion, on the other hand, would be expected to demonstrate a high viscosity.

The concentration of commercially available bitumen emulsions varies with the source, but is typically from about 40 to about 80 weight percent. (Of course, a commercially prepared bitumen need not be used; the operator may prepare the bitumen himself.) The preferred range for use in the present invention is from about 50 to about 75 percent. The initial bitumen emulsion is added to other components to make up the final fluid for oilfield use, for example a fracturing fluid or diversion fluid. The final bitumen concentration is typically from about 1 to about 40 weight percent, preferably from about 3 to about 30 weight percent. Laboratory testing should be done to ensure that the bitumen and the other fluid components are compatible with one another and do not affect the ability of each to serve its purpose; adjustments to the choice or concentration of components should be made accordingly.

Since surface applications of bitumen emulsions are quite extensive, they have become common, large-scale, inexpensive, well-defined and well-characterized products, commercially available almost everywhere in the world. We have found that, for downhole applications, the droplets of bitumen suspended in water influence a number of fluid properties, for example the ability to carry proppant or other solids. We have also found that the ability of a bitumen-in-water emulsion to invert into a bitumen-external emulsion upon physical or chemical triggering is

useful, for example, for surface modification, aggregation of particulates, fluid loss control, and bridging and plugging for fluid diversion.

Normally the term “bitumen” is defined as a mixture of organic fluids that is highly viscous, black, sticky, entirely soluble in carbon disulfide (CS_2), and having a high amount of condensed polycyclic aromatic hydrocarbons. Naturally occurring crude bitumen is produced, for example, from tar sands, and it must be heated or diluted for separation from the sand. Another source of bitumen is oil refinery processes; refined bitumen is the residual (bottom) fraction obtained by fractional distillation of crude oil. Typically, the lower boiling point is about 525 °C. Here we also include as bitumen a mixture of heavy hydrocarbons (of any origin) that becomes solid-like and sticky at room temperature. In U.S. terminology, this refined residue is generally known as asphalt (or asphalt cement). Similarly, in the technical literature “bitumen emulsion” and “asphalt emulsion” mean the same thing. These emulsions contain up to about 80 weight percent bitumen and typically from about 1 to about 2 weight percent chemical additives. There are two main types of emulsions with different affinities for aggregates: cationic and anionic. Heavy oil and bitumen are characterized by high viscosities (i.e. resistance to flow measured in cP) and high densities measured in API gravity compared to conventional oil. The World Petroleum Congress defines heavy oil as oil whose gas-free (dead) viscosity is between 100 cP and 10,000 cP at reservoir temperature. Heavy oil is slightly less dense than water with an API gravity between 10° and 20°. Heavy oil can flow in some reservoirs at downhole temperatures and/or with in situ solution gas, but, at the surface, it is a thick, black, gooey fluid. Bitumen has a viscosity greater than 10,000 cP. Bitumen is predominantly defined as those crude oils or portions of crude oils with a

dead-oil viscosity $>10,000$ cP. If no viscosity data are available, then crude oils having an API gravity of $<10^\circ$ are sometimes referred to as bitumen. Extra-heavy oil is that heavy crude with an API gravity of $<10^\circ$ and a dead-oil viscosity of $<10,000$ cP. For comparison, oil with an API gravity of $<10^\circ$ is denser than water. (It should also be noted that the term “Oil Sands” was created for incentive tax purposes in Canada for those heavy crude oils found above a certain latitude where the infrastructure was almost non-existent.)

The fundamentals of bitumen droplet stability have been studied extensively. The key role played by electrostatic repulsion of micron-sized bitumen droplets suspended in KCl water solution, has been demonstrated by using a colloidal particle scattering (CPS) technique; the droplets in the bitumen emulsions used in the invention are believed to be from about 1 to about 100 microns. U. S. Patent No. 6,613,720 disclosed a wide range of controlled release techniques using emulsions. This patent disclosed controlled release of chemical or biological agents by stabilizing the active ingredients in the discontinuous phase of an emulsion, which was then destabilized by a number of different triggers. The only materials disclosed as the discontinuous phase were diesel and heptanes, and were described as solvents. In the present invention, the discontinuous phase is the agent.

The use of heavy oil in heavy oil-in-water (HO/W) emulsions for downhole operations was described in SPE 110754 “Innovative Gas Shutoff Method Using Heavy Oil-In Water Emulsion” (2008). Note that this was not bitumen, but a heavy oil having viscosities of ~ 10 and 34 MPa-s at 20°C and shear rates > 1 sec $^{-1}$.] Laboratory tests were conducted for sealing of pores in model cells and porous cores with HO/W emulsions with different sizes of droplets and different rheologies.

Emulsion flow through porous media is a complex process that depends both on droplet properties and the parameters of the solid matrix. The emulsion parameters (for example, emulsifier concentration) were adjusted to the properties of the matrix to achieve breaking of the emulsion (droplet coalescence) in a sample with known pore parameters. Droplets may coalesce and produce larger droplets when the surfactant concentration in solution drops to the minimum level. Another mechanism described was coalescence of droplets because of high shear forces rupturing the interface film while droplets were being forced toward each other. The application and effectiveness of a heavy oil-in-water emulsion to block (plug) a porous sample was demonstrated for a variety of porous media. Sealed porous media withstood pressure gradients as high as 42 MPa per 1 m (1800 psi for 29 cm of porous medium). A porous medium was completely blocked by heavy oil after injection of 10 pore volumes of an emulsion (only emulsions containing up to 13 per cent heavy oil were used). Usually the plugging effect of more viscous oils was higher compared to those with lower viscosity, due to the combined effect of capillarity and the viscosity of the fluid injected. The depth of emulsion penetration depended on the droplet-to-throat size ratio and on the pre-flush treatment of the core. Water wettability of the solid matrix provided deeper penetration of heavy oil/water emulsions into the porous formation.

Fluid loss control

Fluid loss control is often an important requirement for successful formation hydraulic fracturing. Effectiveness is primarily a function of the viscosity and cake forming properties of the hydraulic fracturing fluid and the permeability and porosity of the formation. If the permeability and porosity of the formation are high, fluid loss is typically controlled by

increasing the viscosity or the cake forming properties of the hydraulic fracturing fluid by adding polymers or appropriately sized particles. Fluid loss control additives vary. For example, additives include carbonates, salts, minerals, and hard or elastic oil-soluble resins. Fluid loss to the formation rock may be prevented by using additives that are made from a deformable and/or hydrolysable material. A typical problem often associated with solid fluid loss additives is the risk of plugging equipment, perforations or formation. Also, once downhole, hard solid particles may not fit well into pore spaces, thus reducing the effectiveness of fluid loss control. Reversible formation sealing may be achieved by using degradable additives; most require water or hydrocarbon and temperature to degrade.

Bridging and plugging

Reversible isolation of a well interval (or flow diversion from that interval) by generating barriers made with hydrolysable polymer fibers have been described. The use of these fibers as mud loss preventing additives is also known. U. S. Patent No. 7,275,596 described the use of fibers for transport of proppant or gravel for hydraulic fracturing. A commercial application of this patent involves adding fibers to low viscosity fluids to prevent rapid settling of the proppant and allow uniform proppant placement in the fracture. The patent also described fibers capable of hydrolyzing, after the completion of the treatment, to natural products that do not settle in aqueous solutions. The fibers and their decomposition products are compatible with fluids used downhole. However, providing a high-quality downhole barrier remains a challenge; it is difficult selectively to place downhole a barrier having a permeability that is low enough to lower the injectivity; it is also difficult to control the permeability of the barrier. Using temporary barriers downhole has been

reported (see Ali *et al.* "Combined Stimulation and Sand Control," *Oilfield Review*, vol. 14, No. 2, pp. 30-47) in which a method is described for controlling sand production with simultaneous formation of a temporary barrier containing fibers when producing hydraulic fractures). Degradable materials, for example in the form of fibers, are also known for downhole diversion or isolation. For effective plugging, the fibers should be pumped at a high loading. This causes risks of plugging surface or downhole equipment and therefore significantly limits their application. Additionally, the need to mix and pump the fiber-laden slurry constrains certain properties of the fiber (for example the length) to values that are sub-optimal for plugging large fractures. These limitations reduce the scope of fiber application for bridging/plugging of fractures, fissures and wormholes.

U. S. Patent No. 3,917,535 disclosed the use of aqueous-based (water external) emulsions for acid fracturing of formations. There are also techniques that use water-in-oil emulsions for well treatments. U. S. Patent No. 4,233,165 disclosed preparation of water-in-oil emulsions (the continuous phase was light hydrocarbons) in which the aqueous phase (water droplets with sizes less than 10 microns) carried acid for acid treatment or carried reactive solids (for example, carbonates of zinc for reaction with hydrogen sulfide in the production fluid). The overall hydrocarbon phase concentration in the dispersion may be less than 25%. However, this type of emulsion is unsuitable for the goals described in the present invention. This hydrocarbon-based dispersion cannot selectively plug the target zones.

Another patent, U. S. Patent No. 6,939,832 disclosed a method of controllable inversion of water-in-oil emulsions for well treatments. The pumped fluid was a W/O emulsion with a dispersible chemical and a

demulsifier, (in this case a surfactant with a cloud point temperature above 40 °C (or above 60° C)). Alternatively, a W/O emulsion and a demulsifier were mixed within the formation. For the examples shown, the emulsion with a demulsifier lost stability (inverted) at temperatures above 100 °C, while the same composition remained stable for several days under ambient conditions.

Oil-in-water emulsions have been used as treating fluids (for example for formation plugging. Examples include those described in U. S. Patent Nos. 7,231,976 and 7,392,844; U. S. Patent Application Publication Nos. 2008/0217012 and 2009/0078417; and PCT Patent Application Publication Nos. WO 2001/94742, and WO 1994/28085). None of these disclosed the use of bitumen. An oil-in-water emulsion, because of its high viscosity, by itself tends to seal off a formation until it is subsequently broken. In addition, the emulsion serves as an excellent carrier fluid for particulate materials such as diverting or plugging agents. The use of emulsions allows reduction of the concentration of expensive gelling agents.

Specially prepared bitumen emulsions (O/W type) now available commercially (including properly selected surfactants and other stabilizing agents) remove many of the obstacles to field usage, since these types of emulsions are stable, inexpensive, and available.

Bitumen emulsions are droplets of bitumen suspended in water and stabilized with one or more surfactants. The surfactants may be anionic, but cationic stabilized emulsions are the most widely available. Bitumen emulsions were introduced as an alternative to hot bitumen for road and building construction or coating. Emulsion of the bitumen allows working at ambient temperatures and under humid conditions. Often bitumen emulsions are used with solid particles like gravel, crashed

stones, sand, etc. Upon mixing and drying, bitumen precipitates from emulsions onto solid particles, thus gluing particulates together. Cationic bitumen emulsions (stabilized with cationic surfactants) invert easily on contact with anionic surfaces, such as those of carbonates, gravel and other solid materials often found or placed downhole.

A bitumen emulsion itself may be the discontinuous phase of a water-based or oil-based fluid. For example, an oil-based continuous phase may contain water droplets, which in turn contain droplets of bitumen; this is called an O/W/O double emulsion. In this way bitumen emulsions may be used in oil-based fluids as well as in water-based fluids as is described in the rest of the present discussion. Other suitable double emulsions are W/O/W emulsions. Similarly, triple or quaternary emulsions such as W/O/W/O, O/W/O/W, W/O/W/O/W and O/W/O/W/O emulsions may be used. Double, triple, etc. emulsions may also be stabilized with surfactants and used as are ordinary bitumen emulsions.

Emulsion inversion downhole may be triggering by several methods, or even by combinations of methods. Any such method may be used, for example contact of the bitumen droplets with porous media, interaction with triggering agents (for example a surfactant having a charge opposite to that of the surfactant stabilizing the emulsion), formation temperature, and a change in pH or shear. Those skilled in downhole treatments are familiar with many methods. The conditions and chemicals for triggering bitumen emulsion inversion are also known to those skilled in the art of heavy oil demulsification.

The bitumen-in-oil emulsions used in the various methods of the invention are transported to and optionally may be stored at the job site. They may then be added to a fracturing or other fluid alone or together

with other additives, including proppant and fiber, depending upon the purpose of the job.

In addition to fracturing, fluids containing bitumen emulsions are useful as additive to treatment fluids for drilling and in a variety of downhole applications. Examples of uses include fluid loss control, bridging and plugging control required for diversion, fines migration control, matrix acidizing (including naturally fractured formations), and flowback control.

Bitumen emulsions may be added to fracturing and other well-treatment fluids at concentrations of from about 0.1 to about 90 weight percent, preferably from about 0.5 to about 20 weight percent. To prevent fluid loss through formation pores, about 1 to about 20 weight percent of a bitumen emulsion is typically added to a fracturing or drilling fluid. If the intent is to prevent fluid loss through fissures, wormholes or fractures, a bitumen emulsion is typically pumped at a concentration of from about 0.1 to about 20 weight percent together with a supporting material or materials such as particulates of various materials (for example minerals, polymers, etc), often including fibers.

It is known that flocculation of fibers plays an important role in effective bridging and plugging. A bitumen emulsion may improve fiber flocculation, thus providing better bridging and/or plugging in fissures, wormholes or fractures. This in turn may provide fluid diversion downhole.

Bitumen emulsions may be used for fluid loss control and diversion as obtained or prepared, or they may be used with a triggering agent. The triggering agent depends upon the nature of the surfactant

used for emulsion stabilization. If a cationic surfactant is used, the triggering agent should be anionic, and *vice versa*. Thus, for cationic surfactant stabilized bitumen emulsions, carbonates, silicates, hydroxides, etc. are used as triggers. For anionic surfactant stabilized bitumen emulsions, calcium, magnesium, zinc and other cation derivatives (salts, hydroxides, complexes, etc.) are used. The triggers may be water soluble or may have limited solubility. They also may be solid particulates (for example calcium carbonate). Triggers must be separated from immediate contact with the bitumen emulsion by encapsulation, physical separation of the emulsion and triggering stages, trigger surface modification, or over-stabilization of the emulsion (adding more surfactant, or stabilization with particulates, etc.), to provide triggering after a desired time after pumping, according to the job design.

Bitumen emulsions may be added to fluids on the fly or may be added when fluids are batch mixed. Bitumen emulsions may be added to the entire treatment fluid volume (for example for general fluid loss control), or to certain stages only (for example to a diversion stage or stages). For flowback control, a bitumen emulsion is added to the tail-in stage fluid.

Adding bitumen emulsion to proppant loaded slurry with subsequent inversion is used for proppant agglomeration. The emulsion may be added to proppant slugs if proppant slugs are used in a job; alternatively, pulses of emulsion may be added periodically, while proppant is pumped constantly throughout a job. In each case emulsion triggering results in proppant agglomeration by the bitumen. In the first case dispersion is prevented. In the second case, agglomerated proppant will preferably be placed closer to the wellbore, in contrast to non-

agglomerated proppant, which will be delivered further into the fracture. Agglomeration should occur in the fracture, after the fluid has passed through the perforations. When proppant and bitumen emulsion are pumped together, the fluid may contain from about 0.1 to about 90 weight percent bitumen emulsion and from about 24 to about 1200 g/L (about 0.2 to about 10 ppa (pounds proppant added)) proppant. A suitable proppant in such cases is mica, and preferred amounts when mica is used are from about 1 to about 20 weight percent bitumen emulsion and from about 60 to about 240 g/L (about 0.5 to about 2 ppa) proppant.

In those situations in which a triggering agent stage is pumped before or immediately after a bitumen emulsion stage, an inert spacer (such as fracturing fluid, slick water, etc.) is required to prevent undesirable non-timely inversion, unless immediate inversion is part of the job design.

After treatment, the undesired inverted bitumen emulsion residues formed from the bitumen emulsion is removed by the produced oil, or by a solvent pumped after treatment. Suitable solvents that dissolve bitumen include oil, plant oils, diesel, kerosene, etc.

Bitumen emulsions are preferably used at temperatures in the range of from about 0 to about 160 °C.

The present invention can be further understood from the following examples.

In this work a bitumen emulsion manufactured by Sibavtoban, PLC (Novosibirsk, Russia) was used. It is a commercially available cationic-type stabilized emulsion designated BEC-2 by Sibavtoban, and has a bitumen content of about 60%.

Example 1:

Fluid loss control

Addition of the bitumen emulsion to a linear gel or to other fracturing fluids significantly improved fluid loss control in the laboratory. No additional triggering (inverting agent) was required. Bitumen emulsion breaking was observed in the pores of a core simulating a formation, resulting in bitumen particles precipitated in the core. During flowback or cleanup, bitumen was removed from a formation with oil (for example during flowback) or was flushed with one or more organic solvents typically used in the industry (such as toluene, diesel oil, and naphtha.)

Figure 1 is a schematic of the laboratory fluid loss testing apparatus. A pump pushed a piston in a cylinder that contained fluid that was forced through a core. The pressure of the fluid above the piston was measured. Ten weight percent of the BEC-2 bitumen emulsion was added to a 2 weight percent solution of KCl in deionized water. The mixture was placed in the piston and pumped through a sandstone core (having a permeability of 137 mD; a diameter of 17.5 mm, and a length of 23 mm). The sandstone core had been flushed with a 2 weight percent solution of KCl. The tests were conducted at various different constant

pressures from 100 to 500 psi (0.68 to 3.4 MPa). The mass of effluent was measured at the outlet of the core. Cleanup was with diesel oil at 200 psi (1.36 MPa). Again, the mass of fluid passed through the core was measured.

Figure 2 shows the results of the test of the bitumen emulsion used for fluid loss control followed by cleanup with diesel oil in the sandstone core. It can be seen that significant fluid loss control was achieved even in this short core. The barrier created by the deposition of bitumen was easily removed by cleaning with diesel.

Example 2:

Bridging

Bridging is an important phenomenon for fluid diversion; we define it as the blocking of a channel open to flow by simultaneous arrival of stable particles whose sizes are smaller than the channel. Furthermore, for a bridging agent (solid particles) to be effective once placed, the particles must not be displaced by the flowing carrying fluid and must generate an effective barrier to flow, which is referred to as a “plug”. Generally, the initial barrier formed at the location of the bridging must be sealed with additional smaller and/or deformable particulates and compacted under pressure to generate a plug. Two important characteristics of plugs are the differential pressure they are able to withstand, and their permeability.

To achieve bridging in slots or fractures of different geometries, proppant and/or fibers may be used. However, when the fracture or slot width significantly exceeds the particulate size, bridging does not occur. Also, even for small fractures, high concentrations of particulates should

be pumped, which may result in plugging of surface equipment, or constricted sections of the completion. In addition, bridging is a function of the slurry velocity. The lower the rate, the better the bridging ability. However, for various reasons, limiting the rate is not always desirable (or achievable) in fracturing treatments.

For example, published laboratory tests (see for example SPE 119636, 19-21 January, 2009) have shown that a 2 mm wide slot may be bridged by degradable fibers 6 mm in length and 14 microns in diameter a concentration of 0.5 weight per cent in a linear guar gel having a viscosity of 20 cP at 170 s^{-1} at a fluid and a velocity of 1.2 ft/s (0.37 m/s). A 4 mm slot may be bridged under the same conditions at a fiber concentration of 1.6%. However, to bridge a 6 mm slot with that fiber concentration, it is necessary to reduce the flow rate to about 0.4 ft/s (0.12 m/s). An 8 mm slot cannot be bridged at all with those fibers under those conditions.

Bridging and sealing wide fractures in a controllable manner is a challenge for which no good solution currently exists. Ideally, bridging should occur at low particulate concentrations in a controllable manner, and be insensitive to the fracture width (which is often a parameter that is not well known when an operator is pumping a fracturing treatment). We have found that a bitumen emulsion, when added to a fracturing or other fluid, increases the bridging ability of fiber slurries.

The bridging ability of fibers in the presence of a bitumen emulsion, inverted with carbonate anion, was studied using the laboratory apparatus shown in Figure 3 and channels of different geometries (slot and hole). The procedure was based on the one described in SPE 119636 referenced above.

For a linear slot: Inversion of a slurry made up with 480 ml of a linear guar gel having a viscosity of 53 cP at 170 s^{-1} , 15 ml of the BEC-2 bitumen emulsion, 9 g of polylactic acid fiber (150 ppt (pounds per thousand gallons), 1.8 weight percent) was triggered by 10 ml of 2% NaHCO_3 solution to release the bitumen. 10 minutes after addition of the trigger solution, the slurry was pumped through a 2 mm to 9 mm flat slots at a rate of 30 ml/min. Bridging was obtained in all cases and the pressure jumped up to more than 450 psi (3.1 MPa) whereupon automatic pump safety shut down occurred. Even for the 9 mm slot and for other conditions (for examples lower rates (which normally enhance bridging), higher fiber concentrations, and lower fluid viscosities), there was no plug formation without the added bitumen emulsion). A typical pressure jump during pumping through a cell is demonstrated in Figure 4. A similar experiment without an emulsion resulted in bridging failure. Lowering the concentration of the added emulsion (to 10 ml in the above slurry) or lowering the fiber concentration (to 1.2%) resulted in bridging failure.

For a cylindrical hole: The same procedure was used, but with a hole that had a diameter of 10.4 mm. Bridging occurred with the following slurry content: 310 ml of linear gel, 13.2 ml of bitumen emulsion, 100 ppt (1.2%, 3.72 g) of fiber, and 6.6 ml of 2% NaHCO_3 as a triggering agent for the bitumen emulsion inversion. No bridging was achieved without the bitumen emulsion.

The increase of fiber mutual adhesion on-the-fly in the presence of triggered or untriggered bitumen emulsion when pumping a slurry of fibers and emulsion downhole provides flowback control. An untriggered emulsion can provide control because of an increase in fiber

mutual adhesion once bitumen droplets are between fiber filaments (because of the high viscosity of bitumen).

Figure 1 shows the results of a test of bridging of a slurry of bitumen emulsion and fibers in a 4 mm slot.

Plugs, created with a slurry of degradable fiber and bitumen emulsion later degrade in water (the fiber component of the bridge) and/or in oil (the bitumen component of the bridge) thus providing reversibility of fracture/wormhole/fissure/perforation plugging.

Example 3:

Plugging

A bitumen emulsion added to a fluid not only improves bridging but effectively seals by reducing the permeability of the plug. The plugging ability of a bitumen emulsion was tested in the laboratory apparatus shown in Fig. 5. A proppant/fiber pack was made by packing a slurry of linear guar gel (viscosity 120 cP at 170 s^{-1}), 40 ppt fiber (0.48 weight per cent, 4.8 g/L), 4 ppa proppant (pounds proppant added) (480 g/L, size 16/20 mesh) and 3 ppt (0.36 g/L) of CaCO_3 (as a solid trigger for the bitumen emulsion) in a steel tube (12 cm in length, 1.1 cm in diameter, 11 ml volume). The proppant/fiber pack was heated in a bath to 80 °C and heated deionized water (at 80 °C) was fed through the pack at a rate of 10 ml/min (the pressure drop was 5 psi (0.034 MPa)) for 1 minute and then at 3 ml/min. Then a 50:50 by weight mixture of water and BEC-2 bitumen emulsion was injected into the pack. The emulsion was not heated before injection; when it was pumped into the pack (representative of a high-temperature formation or fracture) inversion was triggered by the temperature and the bitumen precipitated and sealed the

pack. Within a minute, the pressure built up significantly; when the pressure exceeded 750 psi (5.2 MPa) a relief valve opened. The permeability of the proppant pack immediately changed from very high (estimated at 780 D by the pressure drop during the water injection) to negligible (no flow was observed at the pressure drop of 750 psi (5.2 MPa)).

Having thus described our invention, we claim:

1. A method for treating a subterranean formation comprising injecting an aqueous fluid comprising a bitumen-internal emulsion.
2. The method of claim 1 wherein the emulsion is stabilized by a surfactant.
3. The method of claim 1 wherein the emulsion is inverted in the formation.
4. The method of claim 3 wherein the emulsion inversion is triggered by adding an emulsion inversion triggering agent.
5. The method of claim 4 wherein the emulsion inversion triggering agent is injected before the injection of the fluid comprising the emulsion.
6. The method of claim 4 wherein the emulsion inversion triggering agent is injected at the same time as the injection of the fluid comprising the emulsion.
7. The method of claim 6 wherein the emulsion inversion triggering agent is injected in a second fluid.
8. The method of claim 6 wherein the fluid comprising the emulsion also comprises the emulsion inversion triggering agent.
9. The method of claim 4 wherein the emulsion inversion triggering agent is injected after the injection of the fluid comprising the emulsion.
10. The method of claim 4 wherein the emulsion inversion triggering agent adheres to a subterranean surface.
11. The method of claim 4 wherein the emulsion inversion triggering agent is in a coating on a solid particulate in the fluid.

12. The method of claim 4 wherein the emulsion inversion triggering agent is a solid particulate.
13. The method of claim 4 wherein the emulsion inversion triggering agent is encapsulated.
14. The method of claim 4 wherein the fluid comprises a precursor of the emulsion inversion triggering agent.
15. The method of claim 1 wherein the fluid further comprises proppant.
16. The method of claim 1 wherein the fluid further comprises fibers or particulates or both.
17. The method of claim 16 wherein the fibers or particulates or both are degradable.
18. The method of claim 1 wherein the fluid further comprises proppant and fibers.
19. The method of claim 1 wherein deposited bitumen is later removed by injecting an organic solvent for the bitumen.
20. The method of claim 1 wherein the bitumen emulsion is part of a triple or quaternary emulsion.
21. The method of claim 1 wherein the bitumen emulsion is added to only a portion of the fluid.
22. A method for treating a subterranean formation comprising injecting an oil-based fluid comprising a bitumen emulsion.

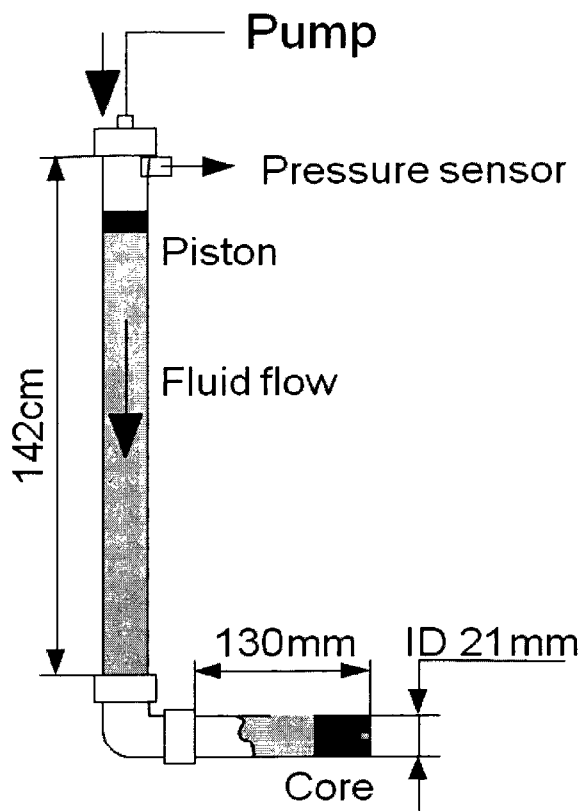


Fig. 1

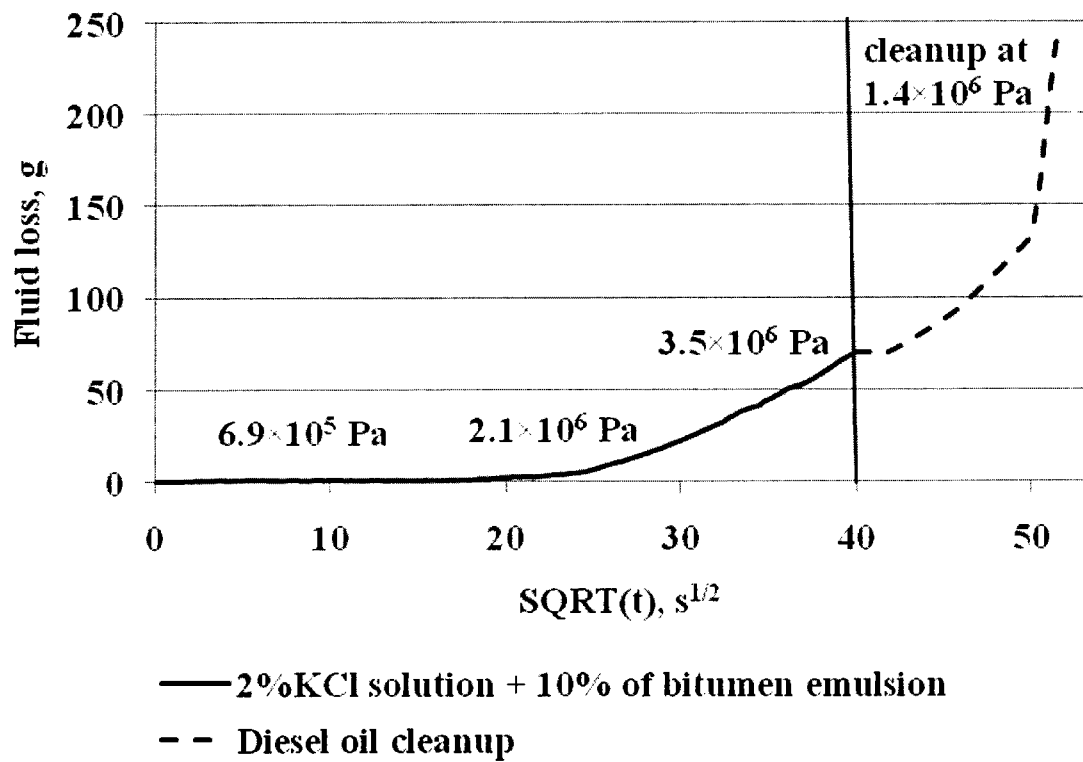
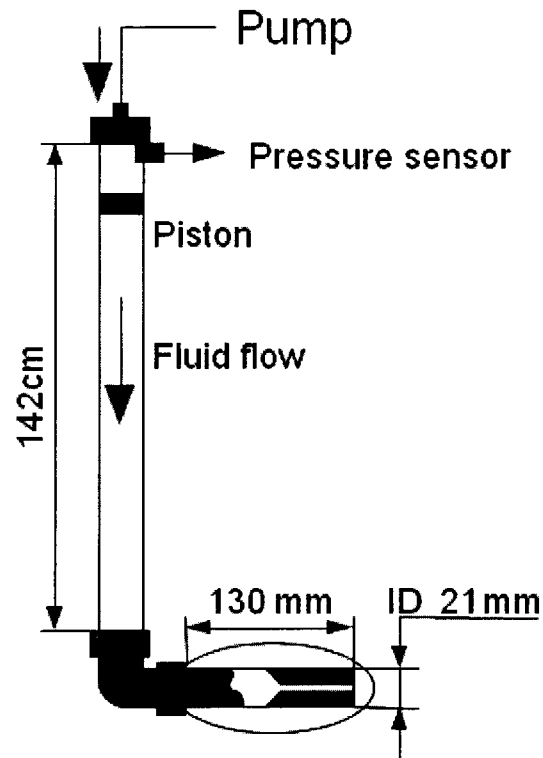
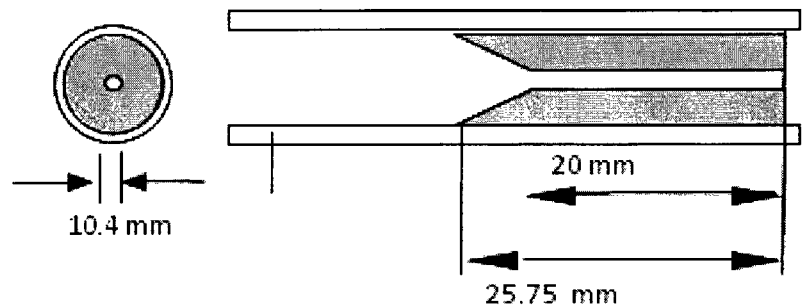


Fig. 2



Wormhole geometry



Fissure/Fracture Geometry

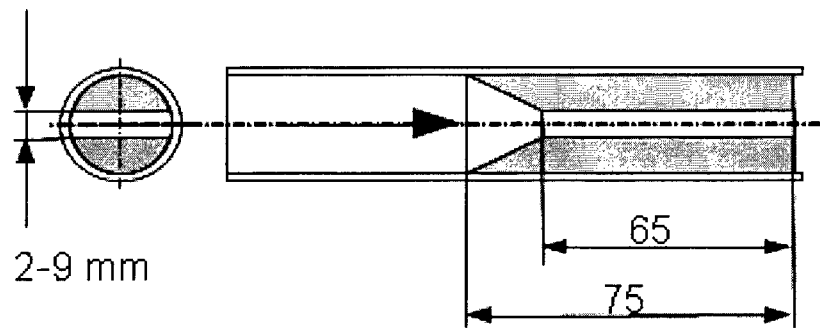


Fig 3

3/4

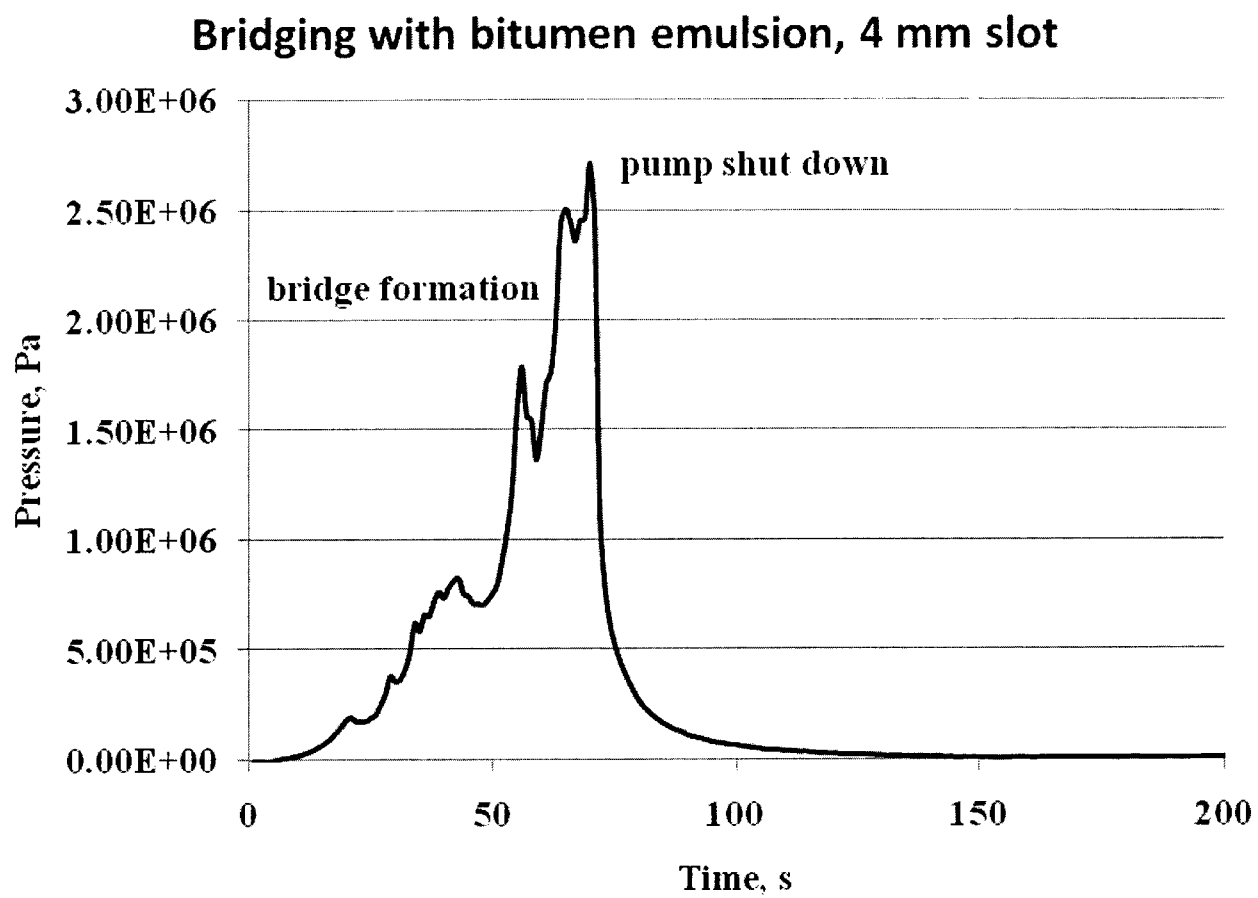


Fig. 4

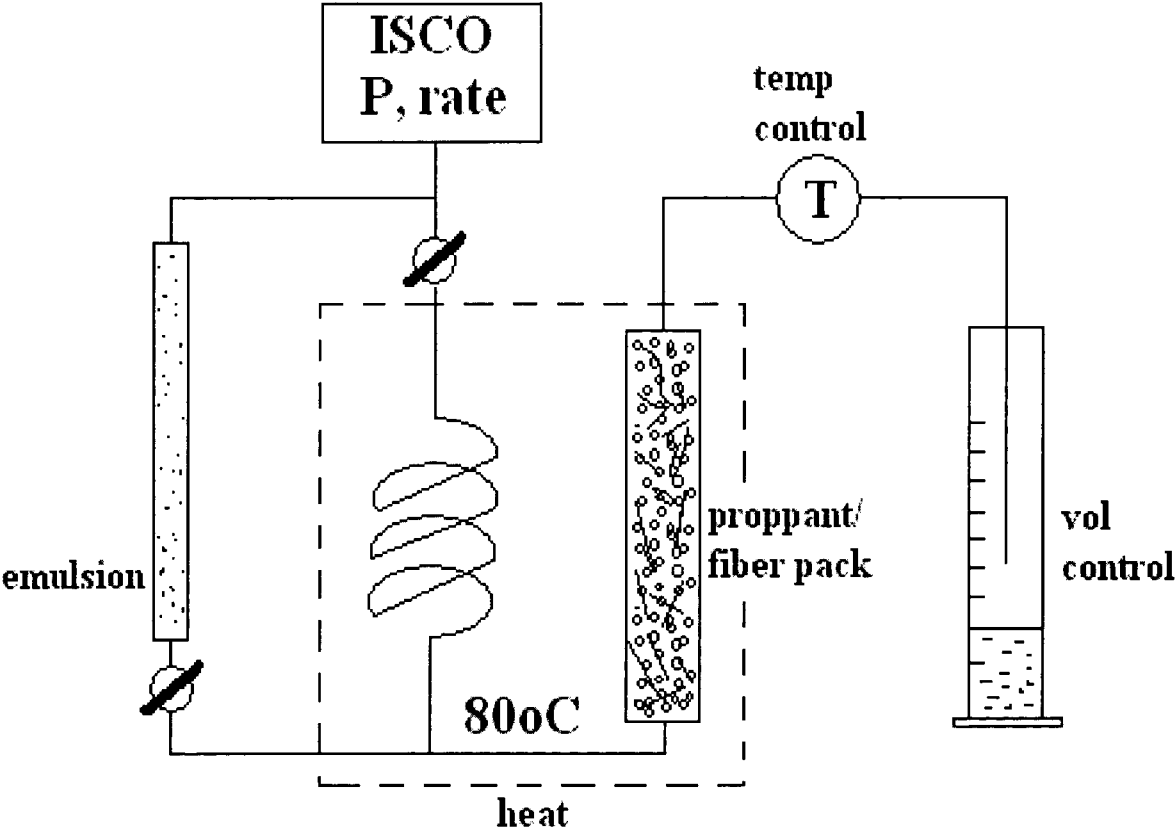


Fig. 5

INTERNATIONAL SEARCH REPORT

International application No.

PCT/RU 2011/000098

A. CLASSIFICATION OF SUBJECT MATTER		<i>C09K 8/506 (2006.01)</i> <i>C09K 8/80 (2006.01)</i> <i>E21B 43/22 (2006.01)</i> <i>E21B 43/267 (2006.01)</i>																				
According to International Patent Classification (IPC) or to both national classification and IPC																						
B. FIELDS SEARCHED																						
Minimum documentation searched (classification system followed by classification symbols)																						
C09K 8/00-8/94, E21B 21/00-21/14, 33/00-33/138, 43/00-43/32																						
Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched																						
Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)																						
RUPAT, RUPAT_OLD, RUPAT_NEW, RUABRU, PatFT, EAPATIS, PAJ, Esp@cenet, PatSearch																						
C. DOCUMENTS CONSIDERED TO BE RELEVANT																						
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.																				
X	US 3974877 A (TEXACO EXPLORATION CANADA LTD.) 17.08.1976, col. 1, lines 7-12, col. 4, lines 3-16, col. 5, lines 9-32, claims	1-4, 7, 9, 15, 16, 19, 21																				
Y		5, 6, 8, 10-14, 17-18, 20																				
Y	RU 2001107412 A (OOO NAUCHNO-ISSEDOVATELSKY INSTITUT "SIBGEOTEH") 20.02.2003, claims 1-4	5, 6																				
Y	RU 2230900 C2 (ZAO "HIMEKO-GANG") 20.06.2004 [online] Retrieved from DB esp@cenet, abstract, p. 4, col. 1, line 52, example 2	8																				
Y	RU 2260673 C1 (OBSHESTVO S OGRANICHENNOY OTVETSTVENNOSTIU "LUKOIL-PERMNEFTEORGSIINTEZ") 20.09.2005, abstract, p. 4, lines 22-25, 35-36, example 1	18																				
☒ Further documents are listed in the continuation of Box C. ☐ See patent family annex.																						
* Special categories of cited documents: <table border="0"> <tr> <td>"A"</td> <td>document defining the general state of the art which is not considered to be of particular relevance</td> <td>"T"</td> <td>later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</td> </tr> <tr> <td>"E"</td> <td>earlier document but published on or after the international filing date</td> <td>"X"</td> <td>document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</td> </tr> <tr> <td>"L"</td> <td>document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</td> <td>"Y"</td> <td>document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</td> </tr> <tr> <td>"O"</td> <td>document referring to an oral disclosure, use, exhibition or other means</td> <td>"&"</td> <td>document member of the same patent family</td> </tr> <tr> <td>"P"</td> <td>document published prior to the international filing date but later than the priority date claimed</td> <td></td> <td></td> </tr> </table>			"A"	document defining the general state of the art which is not considered to be of particular relevance	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention	"E"	earlier document but published on or after the international filing date	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone	"L"	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	"Y"	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art	"O"	document referring to an oral disclosure, use, exhibition or other means	"&"	document member of the same patent family	"P"	document published prior to the international filing date but later than the priority date claimed		
"A"	document defining the general state of the art which is not considered to be of particular relevance	"T"	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention																			
"E"	earlier document but published on or after the international filing date	"X"	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone																			
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"P"	document published prior to the international filing date but later than the priority date claimed																					
Date of the actual completion of the international search		Date of mailing of the international search report																				
02 November 2011 (02.11.2011)		10 November 2011 (10.11.2011)																				
Name and mailing address of the ISA/ FIPS Russia, 123995, Moscow, G-59, GSP-5, Berezhkovskaya nab., 30-1		Authorized officer T. Melnikova																				
Facsimile No. +7 (499) 243-33-37		Telephone No. (495) 730-7641																				

INTERNATIONAL SEARCH REPORT

International application No.

PCT/RU 2011/000098

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
Y	US 2005/0261138 A1 (IAN D. ROBB et al.) 24.11.2005, claims 11, 12	11, 13
Y	US 2008/0119374 A1 (DEAN M. WILLBERG et al.) 22.05.2008, paragraphs [0010]-[0016]	10, 12, 14, 17
Y	US 2005/0194141 A1 (A. RICHARD SINCLAIR et al.) 08.09.2005, paragraphs [0015], [0017], [0033]	17
X	US 6284714 B1 (BAKER HUGHES INCORPORATED) 04.09.2001, abstract, col. 2, lines 5-43, col. 3, lines 9-38, col. 4, lines 46-48, col. 5, lines 17-26	22
Y		20