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(71) Applicant: HALLIBURTON ENERGY SERVICES, INC. [US/US]; 3000 N. Sam Houston Parkway E., Houston, Texas 77032 (US).

(72) Inventors: ABDULRAZZAQ, Waseem; Plot# 18&21 King Abdullah Science, Al-Khobar, 31261 (SA). **ALBURAIKAN, Reem**; AlBuraikan, Al-Khobar, 31261 (SA).

(74) Agent: GARDNER, Jason D. et al.; Kilpatrick Townsend & Stockton LLP, Suite 1400, 4208 Six Forks Road, Raleigh, North Carolina 27609 (US).

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(54) Title: DETERMINING BOND STRENGTH OF MATERIALS USED IN WELLBORE OPERATIONS

(57) Abstract: Described herein are methods and apparatus for testing bond strength of a chemical-sealant, lost circulation material to a subterranean sample. An apparatus can include a body defining walls of a chamber to receive a subterranean core sample from a wellbore, the body being sized to receive a chemical-sealant lost-circulation material (CS-LCM) to an area of the chamber between the core sample and the body, and a removable insert mateable to the body, the insert defining a base of the chamber, where the test apparatus is positionable to transfer a force to the core sample to decouple the core sample from the CS-LCM such that a shear bond strength of the CS-LCM to the core sample is determinable based on a maximum amount of force used to decouple the core sample from the CS-LCM.

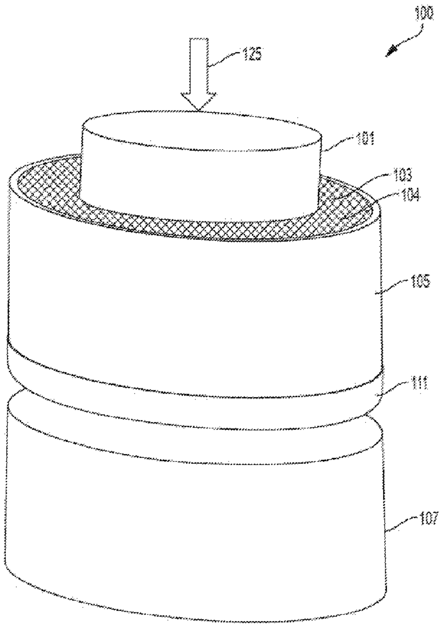

FIG. 2

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DETERMINING BOND STRENGTH OF MATERIALS USED IN WELLBORE OPERATIONS

Field

[0001] The present disclosure relates generally to testing bond strength of materials used in wellbore operations. More specifically, but not by way of limitation, this disclosure relates to determining the bond strength of lost-circulation materials that can be used as chemical sealants.

Background

[0002] During drilling operations, a drilling fluid can be used to cool the drilling bit, control pressure within the wellbore, and suspend and transport drill cuttings from the wellbore to the surface. The drilling fluid can be circulated downwardly through the interior of a drill pipe and upwardly through the annulus, which is located between the exterior of the pipe and the wall of the subterranean formation. Once a casing is in place, a cement slurry may be positioned in the annulus to attach the casing to the walls of the wellbore and seal the annulus. During drilling and cementing, permeable zones in the subterranean formation may be encountered. The permeable zones may be, for example, vugs, voids, naturally occurring fractures, or induced fractures that occur when weak zones have fracture gradients exceeded by the hydrostatic pressure of the drilling fluid or the cement slurry. These permeable zones may result in the loss of the drilling fluid circulation in the wellbore during the drilling operation and can cause the drilling operation to be delayed. During the cementing operation, a portion of the cement slurry may also be lost to the permeable zones in the subterranean formation, which may not permit the cement slurry to fill the annulus completely, or the strength of the cement may be compromised by dehydration of the cement, causing additional delays.

[0003] Plugging materials can be used to seal the permeable zones in the subterranean formation and to prevent the loss of circulation of drilling fluids and cement slurries during the wellbore creation. The bond strength of the plugging materials with the subterranean formation can indicate whether the materials will successfully seal the permeable zones within the wellbore and prevent fluid loss. But the bond strength can depend on the geological makeup of the subterranean formation.

Determining the bond strength of a particular material with respect to a particular subterranean formation can be difficult.

Brief Description of the Drawings

[0004] FIG. 1 is an illustrative schematic of the drilling of a wellbore through a subterranean formation during which a plugging material is employed to seal the formation to prevent the lost-circulation of the drilling fluid according to one example of the present disclosure.

[0005] FIG. 2 is a schematic of an assembled testing apparatus for testing plugging materials according to one example of the present disclosure.

[0006] FIG. 3 is a perspective view of a body of the testing apparatus according to one example of the present disclosure.

[0007] FIG. 4 is a side view of a body, insert, and seat of a testing apparatus according to one example of the present disclosure.

[0008] FIG. 5 is a perspective view of a support cell of the testing apparatus according to one example of the present disclosure.

[0009] FIG. 6 is a side view of an assembled testing apparatus with the seat and insert unscrewed according to one example of the present disclosure.

[0010] FIG. 7 is a side view of an assembled apparatus with the seat and insert installed according to one example of the present disclosure.

[0011] FIG. 8 is perspective view of a core sample prior to testing in a testing apparatus according to one example of the present disclosure.

[0012] FIG. 9A is a perspective view of a test sample prior to uniaxial load testing according to one example of the present disclosure.

[0013] FIG. 9B is a bottom view of the test sample of FIG. 9A according to one example of the present disclosure.

[0014] FIG. 10A is a perspective view of a test sample subsequent to uniaxial load testing according to one example of the present disclosure.

[0015] FIG. 10B is a bottom view of the test sample of FIG. 10B subsequent to uniaxial load testing according to one example of the present disclosure.

[0016] FIG. 11 is a chart of compressive strain with respect to load of core sample A, obtained from load testing according to one example of the present disclosure.

[0017] FIG. 12 is a chart of compressive strain with respect to load of core sample B, obtained from load testing according to one example of the present disclosure.

[0018] FIG. 13 is a chart of compressive strain with respect to load of core sample C, obtained from load testing according to one example of the present disclosure.

[0019] FIG. 14 is a perspective view of test samples A, B, and C subsequent to load testing according to one example of the present disclosure.

Detailed Description

[0020] Certain aspects and features of the present disclosure relate to determining a shear bond strength of a plugging material and a core sample. Plugging materials, such as a chemical-sealant, lost-circulation material (CS-LCM), can be used to bond to a subterranean formation, seal the permeable zones in a wellbore, and prevent the loss of circulation of drilling fluids and cement slurries through permeable zones within the formation. In some examples, a shear bond strength of a CS-LCM and a core sample may be determined using a test apparatus. A subterranean core sample from a wellbore, along with a CS-LCM can be inserted into the test apparatus. A force can be applied to the core sample, and a shear bond strength of the material to the core sample can be determined. The bond strength can be determined by a uniaxial load test, in which the applied force moves the core sample toward an end of the test apparatus.

[0021] The bond strength of the CS-LCM and the subterranean formation is a factor in the ability of the particular material to seal the permeable zones within a wellbore and prevent fluid loss. The selection of a CS-LCM may depend on the geological makeup of the subterranean formation. The shear bond strength between a CS-LCM and a core sample determined from the use of a test apparatus can be used in the selection of a plugging material. The shear bond strength can be correlated with, and may forecast, the bonding strength of the CS-LCM to a formation in a wellbore. Testing the bond strength external to the wellbore can aid in the selection of an optimal sealant material for use in a given wellbore, thereby reducing downtime for wellbore operations. By selecting a plugging material with high bond strength, the permeable zones within the wellbore can be effectively plugged, allowing wellbore operations to continue.

[0022] These illustrative examples are given to introduce the reader to the general subject matter discussed here and are not intended to limit the scope of the disclosed concepts. The following sections describe various additional features and examples with reference to the drawings in which like numerals indicate like elements, and directional descriptions are used to describe the illustrative aspects but, like the illustrative aspects, should not be used to limit the present disclosure.

[0023] FIG. 1 illustrates a wellbore **44** being drilled through a subterranean formation 42, during which a plugging material may be used to seal the subterranean formation **42**. A drill rig **40** can be used for drilling the wellbore **44**. A drill bit **50** may be mounted on the end of a drill string **52** that includes multiple sections of drill pipe. The wellbore **44** may be drilled by using a rotary drive at the surface to rotate the drill string **52** and to apply torque and force to cause the drill bit **50** to extend through wellbore **44**. The drilling fluid may be displaced through the drill string **52** using one or more pumps **54**. The drilling fluid may be circulated past the drill bit **50** and returned to the surface through the annulus of wellbore **44,** as indicated by arrows **46**, thereby removing drill cuttings (e.g., material such as rock generated by the drilling) from the wellbore **44**. Although not shown, additional conduits besides drill string **52** may also be disposed within wellbore **44**.

[0024] The subterranean formation **42** may contain permeable zones through which the drilling fluid may migrate from wellbore **44** into the subterranean formation **42**. These permeable zones may be, for example, fractures, fissures, streaks, voids, vugs, and the like. The presence of such permeable zones in the subterranean formation **42** may cause the circulation of the drilling fluid in wellbore **44** to be lost such that the fluid does not flow back to the surface of the earth. To maintain good circulation of the drilling fluid in wellbore **44,** a plugging material may be passed into wellbore **44** and allow to set downhole. The strength of the plugging material may increase with time after placement downhole.

[0025] In some examples, plugging material for use in sealing a subterranean formation can include for example, hydraulic cements, diesel oil bentonite mud mixes (DOB), diesel oil bentonite mud mixes with cement added to increase strength (DOBC), sorrel cement mixed in viscosified oil (clay and viscosifier), resins, and thixotropic slurries. The plugging materials can include a chemical-sealant, lost-circulation material (CS-LCM).

[0026] While co-mingling or meeting with the favorable downhole conditions, the free flowing plugging material can set irreversibly hard into permeable zones. The favorable conditions can include, for example, pH, salinity, water, temperature, and shear rate. And, the plugging material may rapidly develop compressive strength. The plugging material can quickly set into a rigid sealing mass that is substantially impermeable to fluid (i.e., no fluid or only a small amount of fluid can pass through the mass). After setting, the plugging material may not be able to be washed out of the permeable zones, and the circulation of the drilling fluid through wellbore **44** may be resumed without concern that the drilling fluid may escape from wellbore into the subterranean formation.

[0027] After drilling the wellbore **44** is completed, drill string **52** and drill bit **50** may be removed from wellbore **44**. And a casing can be positioned in wellbore **44.** Primary cementing may then be performed by pumping a cement slurry down the casing and into the annulus between the casing and the wall of wellbore **44.** The set-plugging material can block the permeable zones and prevent the cement slurry from flowing into the subterranean formation. As a result, the hydrostatic pressure of the cement slurry may be maintained such that the slurry returns to the surface and forms a relatively strong cement column in the annulus of the wellbore.

[0028] One factor in selecting a plugging material in a wellbore application is the ability of a plugging material to bond to a formation and sufficiently plug the permeable zones within the formation. The bond strength of the material and the formation can be used to predict the feasibility of using a particular plugging material. Conditions in the wellbore can emulated to determine a bond strength of a material and a formation. In some cases, a core sample from a geologic formation may have permeable zones that require plugging to continue wellbore operations. Bond strengths for various plugging materials may be determined and aid in the selection of a plugging material that can form a strong bond with a specific formation. In some cases, bond strength data for chemical-sealant, lost-circulation materials (CS-LCMs) and representative core samples from a geologic formation may be determined.

[0029] In some examples, the conditions within the wellbore can be emulated to facilitate the selection of a suitable plugging material. For example, the test apparatus may be heated to substantially cure the CS-LCM. In some cases, a core sample from a specific wellbore formation may be used in the apparatus to determine the bond strength of a potential CS-LCM to the formation. The bond strength determination can forecast the ability of the selected plugging material to block permeable zones within the wellbore. The bond strength can also forecast the ability of the selected plugging material to withstand hydrostatic pressures within the wellbore. In some examples, a CS-LCM maybe placed in contact with a core sample and the bond strength of the CS-LCM and the core sample tested under compressive force.

[0030] In some examples, a test apparatus may include a body that defines walls of a chamber to receive a subterranean core sample from a wellbore. The body can be sized to receive a chemical-sealant, lost-circulation material (CS-LCM) to an area of the chamber between the core sample and the body. The apparatus may include a removable insert that can be mated to the body, where the insert defines a base of the chamber. The test apparatus can be positioned in a test instrument to determine the strength of the bond between the core sample and CS-LCM. The instrument may apply a force to the core sample to decouple the core sample from the CS-LCM. Once the force applied exceeds the bond strength, the core sample may decouple from the CS-LCM and the core sample may travel in the apparatus in the direction of the applied force. The shear bond strength of the CS-LCM to the core sample can be determined based on a maximum amount of force used to decouple the core sample from the CS-LCM.

[0031] An assembled testing apparatus **100** according to some examples is shown in FIG. 2. The apparatus includes a body **105**, an insert **111** and a support cell **107**. A core sample **101** is located substantially in the center of a chamber **103,** defined by a body **105** of a test apparatus. A CS-LCM **104** can be added to an area of the chamber **103** between the core sample **101** and the body **105** such that the CS-LCM **104** and the core sample **101** can at least partially bond together. A compressive force

125 can be applied to the core sample **101** to decouple the core sample **101** from the CS-LCM **104**.

[0032] FIG. 3 shows a perspective view of a body **105** of the testing apparatus **100** according to one example. The one-piece body **105** has female grooves **106** on an interior surface of a distal end of the body **105**. In some examples, the body **105** has sidewall approximately two inches in height. In FIG. 3, the body **105** is a ring, but other geometries can be used.

[0033] FIG. 4 shows a side view of a body **105**, insert **111**, and seat **109** of a testing apparatus **100** according to one example. The insert **111** has male grooves **112** on an exterior surface of a proximal end of the insert **111**, where the male grooves **112** of the insert **111** are mateable to the female grooves **106** (shown in FIG. 3) of the body **105**. The insert **111** further includes a passageway defined by the thickness of the insert **111**. In some cases, the insert **111** can include female grooves **113** on a surface defining a portion of the passageway. The passageway may be sized to allow the core sample **101** to move at into at least a portion of the passageway when a compressive force is applied to the core sample **101**. The apparatus can include a seat **109** having male grooves **110** on a side wall, the grooves **110** of the seat **109** being mateable with the female grooves **113** of the passageway of the insert **111**. Mating of the apparatus components is made with grooves or threads in some cases. In other cases, the components can be mated using pins and notches or other connection means known to one of skill in the art. The testing apparatus **100**, including the body **105**, insert **111**, and seat **109**, may be substantially circular in shape; however, other geometries may be used.

[0034] FIG. 5 is a perspective view of a support cell **107** of the testing apparatus **100** according to one example. The support cell **107** includes a passageway having a diameter greater than the diameter of a core sample **101**. During testing, the assembled body **105** and insert **111** can rest upon the support cell **107**. The top surface of the support cell **107** may be substantially planar to hold the mated body **105** and insert **111** in a stacked configuration. The size of the passageway of the support cell **107** allows for the core sample **101** to move toward the support cell **107** when the compressive force **125** is applied to the core sample **101**. In some cases, the core sample **101** may partially enter the passageway of the support cell **107**.

[0035] FIG. 6 is a side view of an assembled testing apparatus **100** according to one example with the seat **109** and insert **111** unscrewed. FIG. 7 is a side view according to one example of the assembled testing apparatus **100** with the seat **109** and insert **111**.

[0036] FIG. 8 is perspective view of a core sample **101** from a wellbore prior to testing in a testing apparatus according to one example of the present disclosure. A core sample **101** can be cylindrical in shape; however, other geometries can be used. A cylindrical shape can allow for substantially uniform shear stress to be exerted about the core sample **101**. In some examples, the height of the core sample **101** can be larger than the height of the body **105**. For example, the core sample can be approximately three inches in height for use in a body of two inches in height.

[0037] FIGS. 9A and 9B show a test sample prior to uniaxial load testing according to one example described herein. FIG. 9A is a perspective view, while FIG. 9B is a bottom view. The core sample **101** is placed in the assembled testing apparatus **100** such that the core sample **101** is substantially centered within a chamber **103** defined by the body **105** of the test apparatus. The core sample **101** rests upon the seat **109** at the distal end of the testing apparatus **100** while the CS-LCM **104** is added to an area of the chamber **103** between the core sample **101** and the body **105**. In some cases, the seat **109** can position a depth of the core sample **101** within the testing apparatus **100**. In a prepared sample, the plugging material, CS-LCM **104**, substantially fills the area of the chamber **103** between the body **105** and the core sample **101** and contacts the core sample **101**. As shown in FIG. 9B, the seat **109** can be removed from the insert **111** once the CS-LCM **104** has cured and is at least partially bonded together with the core sample **101.** In some examples, the testing apparatus **100** may be heated to substantially cure the CS-LCM **104**. Once the seat **109** is removed, the end of the core sample **101** is located above the female grooves **113** of the insert **111**.

[0038] FIGS. 10A and 10B show a test sample subsequent to uniaxial load testing according to one example of the present disclosure. FIG. 10A is a perspective view and FIG. 10B is a bottom view. During a bond strength test using a uniaxial compression instrument, a compressive force **125** is placed on the core sample **101**. Once the compressive force **125** exceeds the bond strength of the CS-LCM **104** to the core sample **101**, the core sample **101** will decouple from the CS-LCM **104** and the core sample **101** will move toward the distal end of the testing apparatus **100**, through the passageway of the insert **111**. The core sample **101** may move to the distal end of the body **105**, covering the female grooves **113** of the insert **111** once the core sample **101** decouples from the CS-LCM **104** due to the compressive force **125** applied. In some cases, the core sample **101** may be pushed downward into a portion of the support cell **107** beneath the testing apparatus **100**. The test apparatus **100** can be decoupled to aid in removing the CS-LCM **104** and core sample **101** once the test has been completed. The insert **111** may decouple from the body **105**, which provides full access to the body **105** to remove the CS-LCM **104**.

[0039] The resulting bond strength can be determined from the maximum compressive force or load applied to the core sample and the area of the bond, which is the interface between the core sample and CS-LCM. The CS-LCM fills a chamber around the core sample. For cylindrical samples, the area of the bond can be calculated from interface of the CS-LCM and the core sample using the height of the chamber and the circumference of the core sample according to Equation 1:

$$Bond\ area\ = d \cdot h \cdot \pi \quad \text{(Eq. 1)}$$

where d is the diameter of the core sample and h is the height of the CS-LCM in the chamber in contact with the core sample. The bond strength is then calculated according to Equation 2.

$$Bond\ Strength\ = (Maximum\ Load/Bond\ area) \quad \text{(Eq. 2)}$$

[0040] The bond strength measured relates directly to the strength of the interface of the CS-LCM with the core sample. The design of the apparatus with a simple one-piece body, removable insert, and seat that aligns the core sample until the CS-LCM has substantially cured can help ensure reproducibility and reduce testing error.

[0041] In some examples, a system for determining bond strength of a plugging material and a core sample may include an instrument that can apply a force to a subterranean core sample from a wellbore. The core sample may be positioned within a test apparatus and placed in contact with a CS-LCM. In some cases, the instrument can be controlled by a computer and can include extensometers configured to measure the compressive strain of the core sample during testing. The shear bond strength of the material to the core sample can be determined from the maximum forced applied and the size of the core sample. In some cases, the system may further include a heating unit configured to substantially cure the material in the test apparatus.

Example

[0042] Three samples were prepared according to a method described herein. A body, insert, and seat were assembled to prepare the apparatus for testing. A pocket was created for the core sample to sit in by exposing an outer portion of the threads of the seat. A core sample approximately three inches in height and 1.5 inches in diameter was obtained to simulate the wellbore. The core sample was placed in the apparatus, with a portion of the core sample resting in the pocket of the seat, which helped to align the core sample in the center of the apparatus. A chemical-sealant, lost-circulation material (CS-LCM) was added to the chamber of the apparatus and heated according to manufacturer's specification to substantially cure the CS-LCM. Once the CS-LCM had substantially cured, the seat was removed from the apparatus, exposing the end of the core sample through the passageway of the insert.

[0043] The test sample was placed upon a support cell on a lower plate of a uniaxial compression instrument. A top plate of the instrument was set touching the end of the core sample. The compression test was initiated and the top plate moved downward toward the stationary bottom place until the force exceeded the bond strength of the sample, the CS-LCM decoupled from the core sample, and the core sample moved to a lower portion of the apparatus, extending into the support cell. The testing method was repeated for the remaining samples. The maximum force applied by the instrument was recorded for each sample tested. FIGS. 11, 12, and 13 show the load-strain plots for Samples A, B, and C respectively. Sample A exhibited a much higher maximum load than Samples B and C.

[0044] The conditions for sample preparation and testing and resulting bond strength results are provided in Table 1.

Table 1

Test Information	Sample A	Sample B	Sample C
CS-LCM Density, ppg	9.5	9.5	10.5
CS-LCM Activator	77pcf WBM	77pcf WBM	shear dependent

CS-LCM Curing Condition	Wet	Wet	Wet
CS-LCM Curing Temp, °F	190	190	175
CS-LCM Curing Time, hrs	2	2	24
Load rate, inch/s	0.0001	0.0001	0.0001
Core used	Brea sandstone	Brea sandstone	Brea sandstone
Core Diameter, inch	1.5	1.5	1.5
CS-LCM height, inch	2	2	2
Bond Strength, psi	130	8.75	8.4

[0045] A visual inspection of the core samples was also made after testing. FIG. 14 shows three core samples **101** after uniaxial compression testing. Each of the samples (A, B, and C) had residual CS-LCM **115** attached to the core sample **101.** However, Samples B and C had more CS-LCM remaining along the length of the core sample, which may correlate to the lower bond strength result.

Illustrative embodiments of suitable fluids and methods.

[0046] As used below, any reference to methods, products, or systems is understood as a reference to each of those methods, products, or systems disjunctively (e.g., "Illustrative embodiment 1-4 is understood as illustrative embodiment 1, 2, 3, or 4.").

[0047] Illustrative embodiment 1 is a test apparatus comprising a body defining walls of a chamber to receive a subterranean core sample from a wellbore, the body being sized to receive a chemical-sealant lost-circulation material (CS-LCM) to an area of the chamber between the core sample and the body and a removable insert mateable to the body, the insert defining a base of the chamber, wherein the test apparatus is positionable to transfer a force to the core sample to decouple the core sample from the CS-LCM such that a shear bond strength of the CS-LCM to the core sample is determinable based on a maximum amount of force used to decouple the core sample from the CS-LCM.

[0048] Illustrative embodiment 2 is the apparatus of any preceding or subsequent illustrative embodiment, wherein the body comprises grooves at an interior surface of a distal end of the body and the insert comprises grooves on an exterior surface of a proximal end of the insert, the grooves of the insert being mateable to the grooves of the body.

[0049] Illustrative embodiment 3 is the apparatus of any preceding or subsequent illustrative embodiment, wherein the insert further comprises a passageway defined by a thickness of the insert, the passageway being sized to allow the core sample to move into at least a portion of the passageway when the force is applied to the core sample.

[0050] Illustrative embodiment 4 is the apparatus of any preceding or subsequent illustrative embodiment, further comprising a seat mateable to the insert, the seat positioning a depth of the core sample within the body.

[0051] Illustrative embodiment 5 is the apparatus of any preceding or subsequent illustrative embodiment, wherein the thickness of the insert defining the passageway comprises grooves and the seat comprises grooves on a sidewall.

[0052] Illustrative embodiment 6 is the apparatus of any preceding or subsequent illustrative embodiment, wherein the seat is substantially the same size as the passageway of the insert.

[0053] Illustrative embodiment 7 is the apparatus of any preceding or subsequent illustrative embodiment, further comprising support cell having a planar surface to hold the mated body and insert and a passageway defined by a thickness of the support cell, the passageway being sized to allow the core sample to move into a portion of the passageway when the force is applied to the core sample.

[0054] Illustrative embodiment 8 is the apparatus of any preceding or subsequent illustrative embodiment, wherein the core sample is substantially cylindrical in shape allowing for substantially uniform shear stress to be exerted about the core sample.

[0055] Illustrative embodiment 9 is a method comprising adding a subterranean core sample from a wellbore to a chamber defined by a body of a test apparatus, adding a chemical-sealant, lost-circulation material (CS-LCM) to a volume of the chamber between the core sample and the body such that the CS-LCM and the core sample contact one another and at least partially bond together, applying a force to the core sample to decouple the core sample from the CS-LCM, and determining a shear bond strength of the CS-LCM to the core sample based on a maximum amount of the force used to decouple the core sample from the CS-LCM.

[0056] Illustrative embodiment 10 is the method of any preceding or subsequent illustrative embodiment, wherein applying the force to the core sample includes causing the core sample to move toward a distal end of the test apparatus.

[0057] Illustrative embodiment 11 is the method of any preceding or subsequent illustrative embodiment, further comprising heating the test apparatus to substantially cure the CS-LCM prior to applying the force to the core sample.

[0058] Illustrative embodiment 12 is the method of any preceding or subsequent illustrative embodiment, further comprising placing the core sample upon a removable seat to align the core sample within the body of the test apparatus and removing the seat prior to applying force to the core sample.

[0059] Illustrative embodiment 13 is the method of any preceding or subsequent illustrative embodiment, wherein the core sample is substantially cylindrical in shape.

[0060] Illustrative embodiment 14 is the method of any preceding or subsequent illustrative embodiment, wherein the CS-LCM substantially fills the volume of the chamber between the core sample and the body.

[0061] Illustrative embodiment 15 is the method of any preceding or subsequent illustrative embodiment, further comprising placing the test apparatus on a support cell comprising a passageway prior to applying the force to the core sample, the passageway being defined by a thickness of the support cell.

[0062] Illustrative embodiment 16 is the method of any preceding or subsequent illustrative embodiment, wherein a diameter of the support cell passageway is larger than the diameter of the core sample.

[0063] Illustrative embodiment 17 is a system comprising an instrument to apply a force to a subterranean core sample from a wellbore and that is positioned within a test apparatus and in contact with a chemical-sealant, lost-circulation material (CS-LCM), to determine a shear bond strength of the CS-LCM to the core sample.

[0064] Illustrative embodiment 18 is the system of any preceding or subsequent illustrative embodiment, wherein the instrument is controllable by a computer.

[0065] Illustrative embodiment 19 is the system of any preceding or subsequent illustrative embodiment, further comprising a heating unit to substantially cure the CS-

LCM in the test apparatus prior to the instrument applying the force to the subterranean core sample.

[0066] Illustrative embodiment 20 is the system of any preceding or subsequent illustrative embodiment, wherein the test apparatus comprises a body defining walls of a chamber to receive a subterranean core sample from a wellbore, the body being sized to receive a chemical-sealant lost-circulation material (CS-LCM) to an area of the chamber between the core sample and the body, and a removable insert mateable to the body, the insert defining a base of the chamber.

Definitions and Descriptions

[0067] The terms “disclosure,” “the disclosure,” “the present disclosure,” “embodiment,” “certain embodiment” and the like are used herein are intended to refer broadly to all the subject matter of this patent application and the claims below. Statements containing these terms should be understood not to limit the subject matter described herein or to limit the meaning or scope of the patent claims below. The terms “comprising,” “having,” “including,” and “containing” are to be construed as open-ended terms (i.e., meaning “including, but not limited to”) unless otherwise noted. It is further noted that, as used in this specification, the singular forms “a,” “an,” and “the” include plural referents unless expressly and unequivocally limited to one referent.

[0068] Notwithstanding that the numerical ranges and parameters setting forth the broad scope of the present disclosure are approximations, the numerical values set forth in the specific examples are reported as precisely as possible. Any numerical value, however, inherently contains certain errors necessarily resulting from the standard deviation found in their respective testing measurements. Moreover, all ranges disclosed herein are to be understood to encompass any and all subranges subsumed therein. For example, a stated range of “l to 10” should be considered to include any and all subranges between (and inclusive of) the minimum value of 1 and the maximum value of 10; that is, all subranges beginning with a minimum value of 1 or more, e.g. 1 to 6.1, and ending with a maximum value of 10 or less, e.g., 5.5 to 10. Additionally, any reference referred to as being “incorporated herein” is to be understood as being incorporated in its entirety.

[0069] Various embodiments of the present disclosure have been described herein. It should be recognized that these embodiments are merely illustrative of the present disclosure. Variations of those preferred embodiments may become apparent to those of ordinary skill in the art upon reading the foregoing description. The inventors expect skilled artisans to employ such variations as appropriate, and the inventors intend for the present disclosure to be practiced otherwise than as specifically described herein. Accordingly, this disclosure includes all modifications and equivalents of the subject matter recited in the claims appended hereto as permitted by applicable law. Moreover, any combination of the above-described elements in all possible variations thereof is encompassed by the present disclosure unless otherwise indicated or otherwise clearly contradicted by context.

[0070] It is to be understood that the present description illustrates aspects of the disclosure relevant to a clear understanding of the present disclosure. Certain aspects of the disclosure that would be apparent to those of ordinary skill in the art and that, therefore, would not facilitate a better understanding of the disclosure have not been presented in order to simplify the present description. Although the present disclosure has been described in connection with certain embodiments, the present disclosure is not limited to the particular embodiments disclosed, but is intended to cover modifications that are within the spirit and scope of the disclosure.

WHAT IS CLAIMED IS:

1. A test apparatus comprising:

a body defining walls of a chamber to receive a subterranean core sample from a wellbore, the body being sized to receive a chemical-sealant lost-circulation material (CS-LCM) to an area of the chamber between the core sample and the body; and

a removable insert mateable to the body, the insert defining a base of the chamber,

wherein the test apparatus is positionable to transfer a force to the core sample to decouple the core sample from the CS-LCM such that a shear bond strength of the CS-LCM to the core sample is determinable based on a maximum amount of force used to decouple the core sample from the CS-LCM.

2. The test apparatus of claim 1, wherein the body comprises grooves at an interior surface of a distal end of the body and the insert comprises grooves on an exterior surface of a proximal end of the insert, the grooves of the insert being mateable to the grooves of the body.

3. The test apparatus of claim 1, wherein the insert further comprises a passageway defined by a thickness of the insert, the passageway being sized to allow the core sample to move into at least a portion of the passageway when the force is applied to the core sample.

4. The test apparatus of claim 3, further comprising a seat mateable to the insert, the seat positioning a depth of the core sample within the body.

5. The test apparatus of claim 4, wherein the thickness of the insert defining the passageway comprises grooves and the seat comprises grooves on a sidewall.

6. The test apparatus of claim 4, wherein the seat is substantially the same size as the passageway of the insert.

7. The test apparatus of claim 1, further comprising support cell having a planar surface to hold the mated body and insert and a passageway defined by a thickness of the support cell, the passageway being sized to allow the core sample to move into a portion of the passageway when the force is applied to the core sample.

8. The test apparatus of claim 1, wherein the core sample is substantially cylindrical in shape allowing for substantially uniform shear stress to be exerted about the core sample.

9. A method comprising:

adding a subterranean core sample from a wellbore to a chamber defined by a body of a test apparatus;

adding a chemical-sealant, lost-circulation material (CS-LCM) to a volume of the chamber between the core sample and the body such that the CS-LCM and the core sample contact one another and at least partially bond together;

applying a force to the core sample to decouple the core sample from the CS-LCM; and

determining a shear bond strength of the CS-LCM to the core sample based on a maximum amount of the force used to decouple the core sample from the CS-LCM.

10. The method of claim 9, wherein applying the force to the core sample includes causing the core sample to move toward a distal end of the test apparatus.

11. The method of claim 9, further comprising heating the test apparatus to substantially cure the CS-LCM prior to applying the force to the core sample.

12. The method of claim 9, further comprising placing the core sample upon a removable seat to align the core sample within the body of the test apparatus and removing the seat prior to applying force to the core sample.

13. The method of claim 9, wherein the core sample is substantially cylindrical in shape.

14. The method of claim 9, wherein the CS-LCM substantially fills the volume of the chamber between the core sample and the body.

15. The method of claim 9, further comprising placing the test apparatus on a support cell comprising a passageway prior to applying the force to the core sample, the passageway being defined by a thickness of the support cell.

16. The method of claim 15, wherein a diameter of the support cell passageway is larger than the diameter of the core sample.

17. A system comprising an instrument to apply a force to a subterranean core sample from a wellbore and that is positioned within a test apparatus and in contact with a chemical-sealant, lost-circulation material (CS-LCM), to determine a shear bond strength of the CS-LCM to the core sample.

18. The system of claim 17, wherein the instrument is controllable by a computer.

19. The system of claim 17, further comprising a heating unit to substantially cure the CS-LCM in the test apparatus prior to the instrument applying the force to the subterranean core sample.

20. The system of claim 17, wherein the test apparatus comprises:

a body defining walls of a chamber to receive a subterranean core sample from a wellbore, the body being sized to receive a chemical-sealant lost-circulation material (CS-LCM) to an area of the chamber between the core sample and the body; and

a removable insert mateable to the body, the insert defining a base of the chamber.

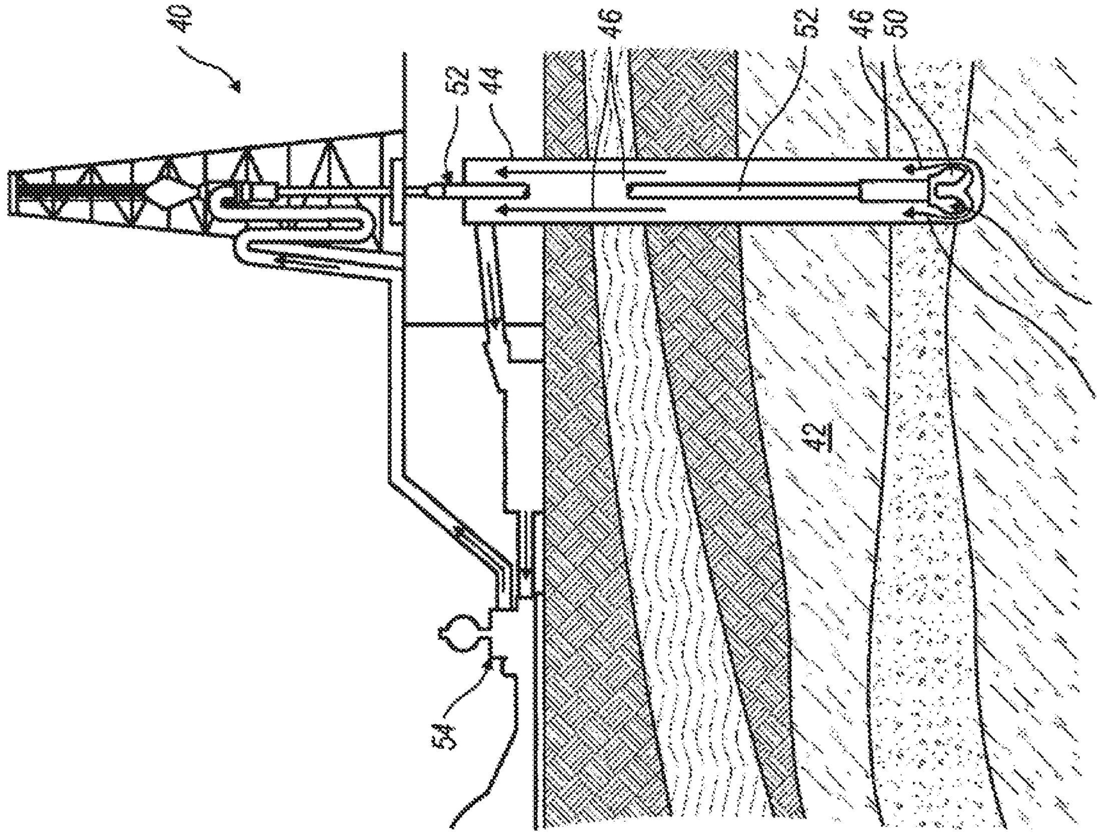

FIG. 1

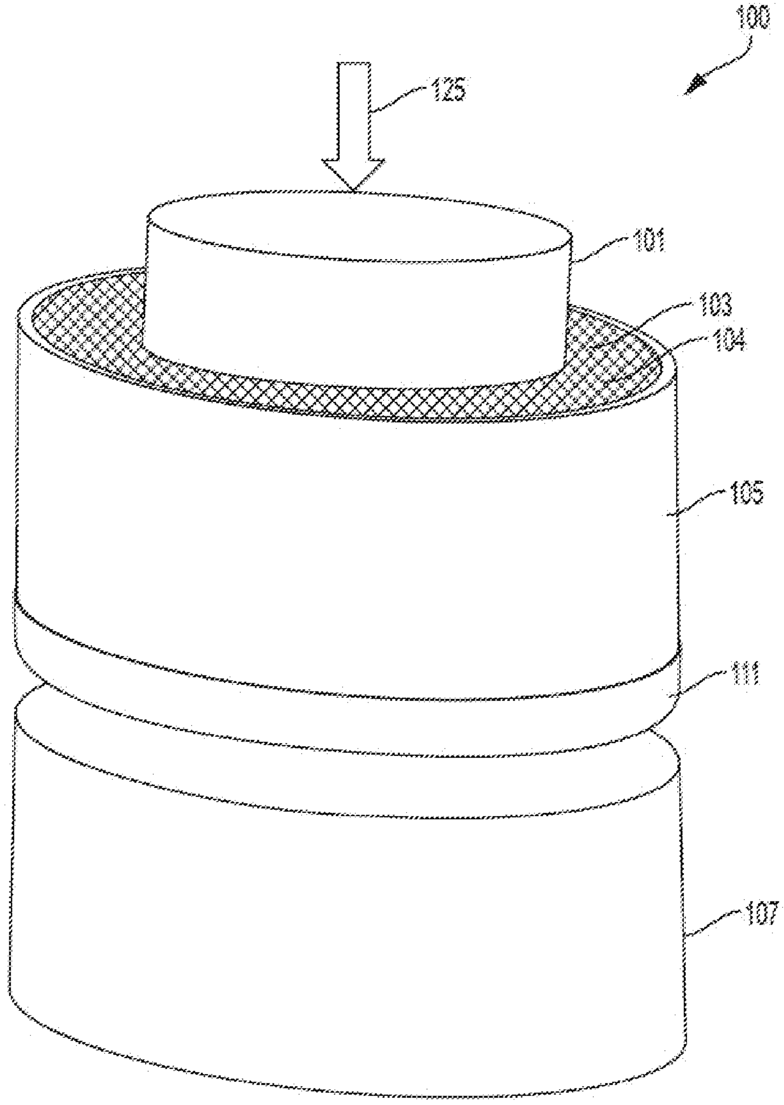

FIG. 2

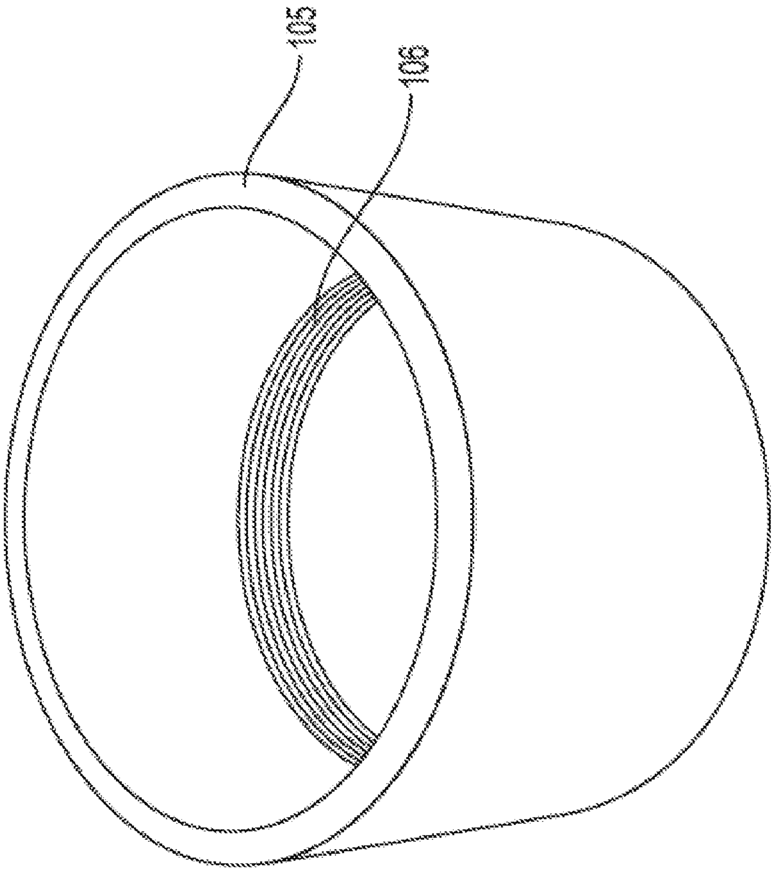

FIG. 3

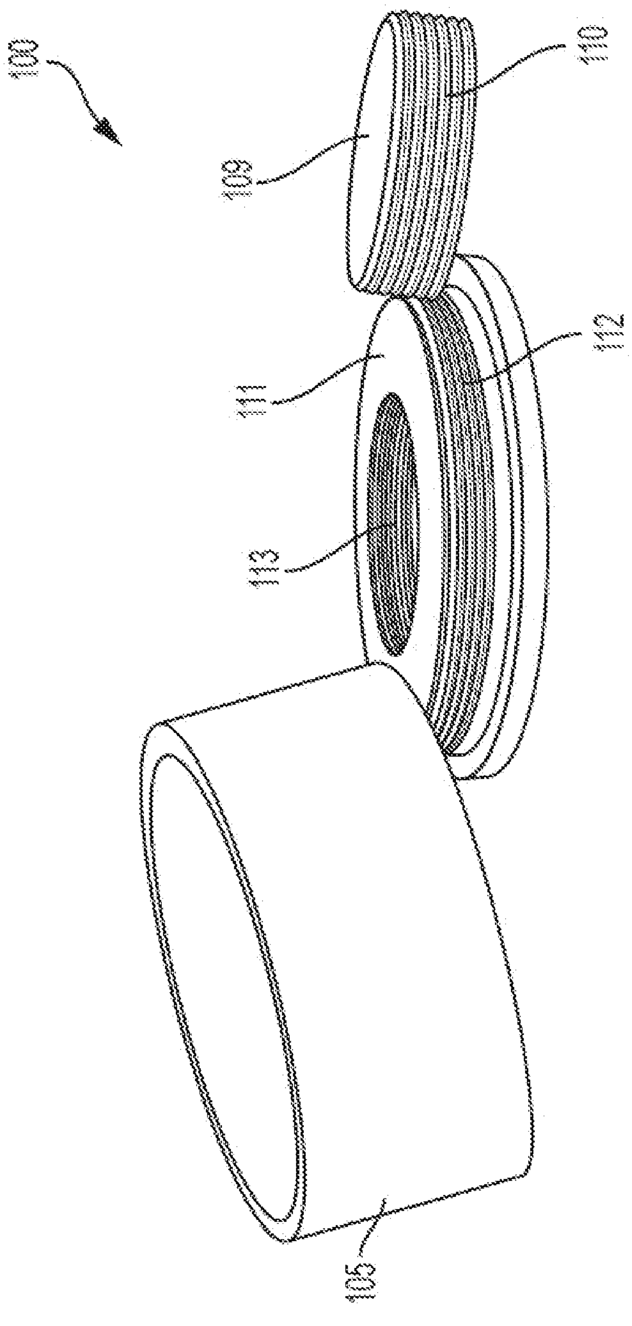

FIG. 4

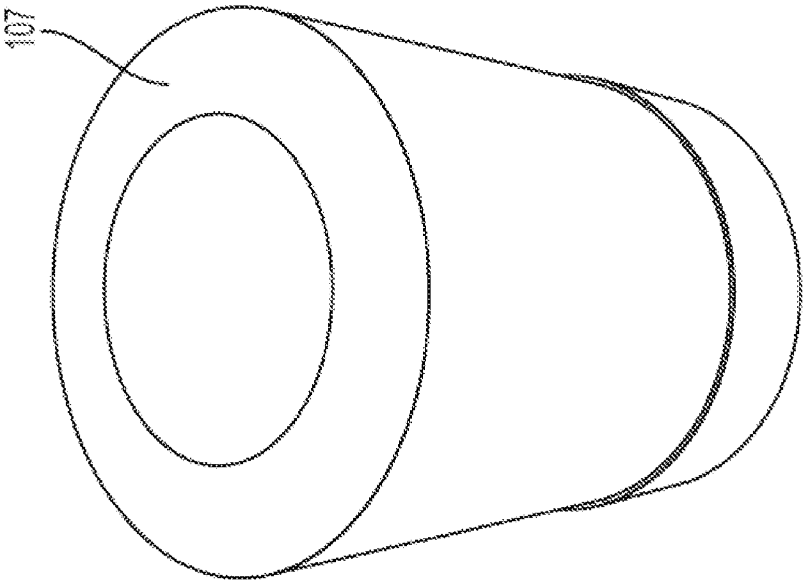

FIG. 5

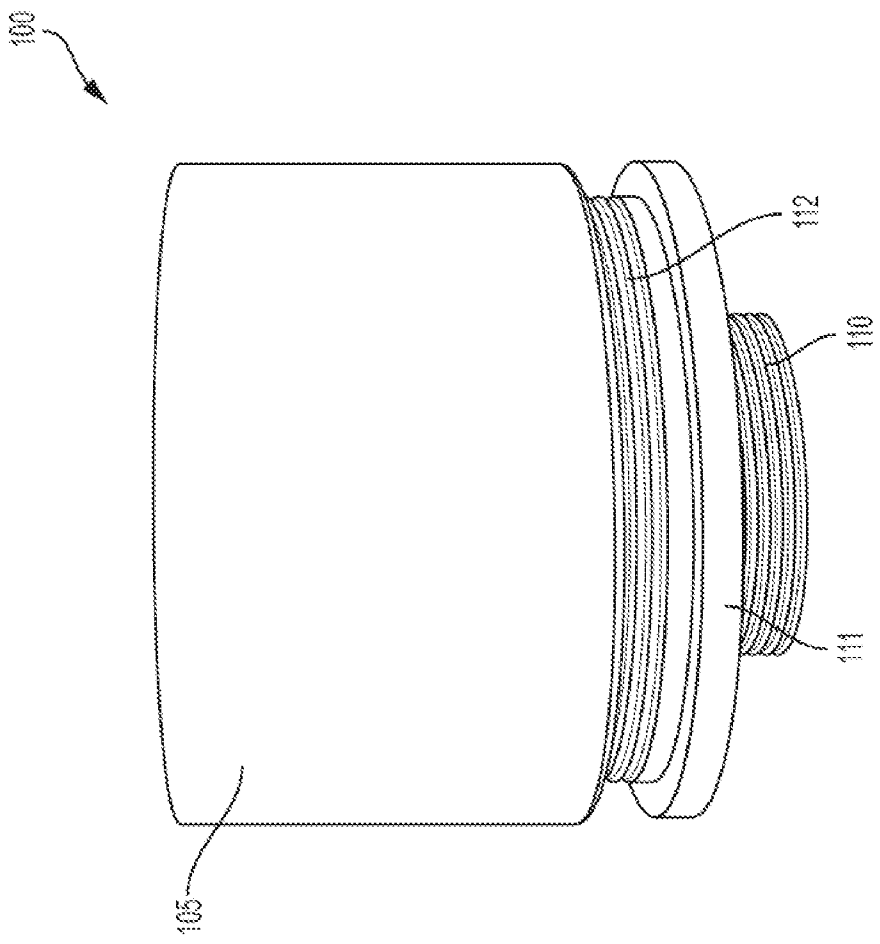

FIG. 6

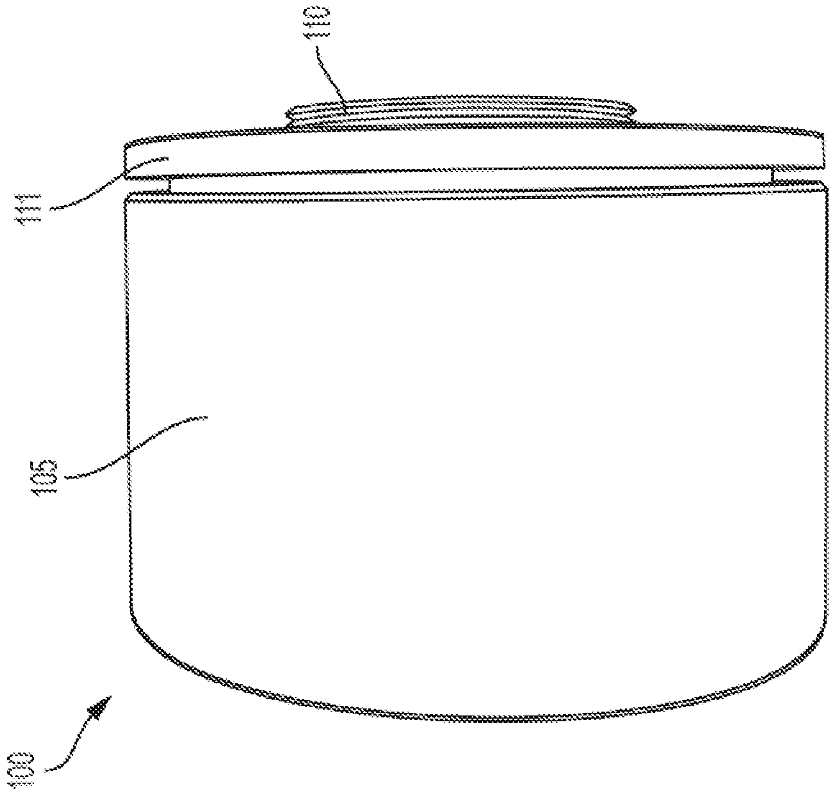

FIG. 7

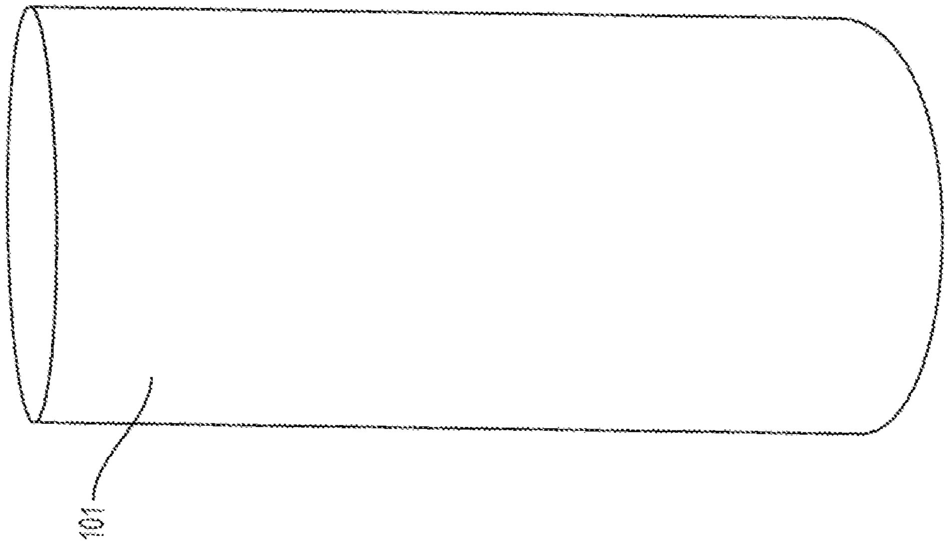

FIG. 8

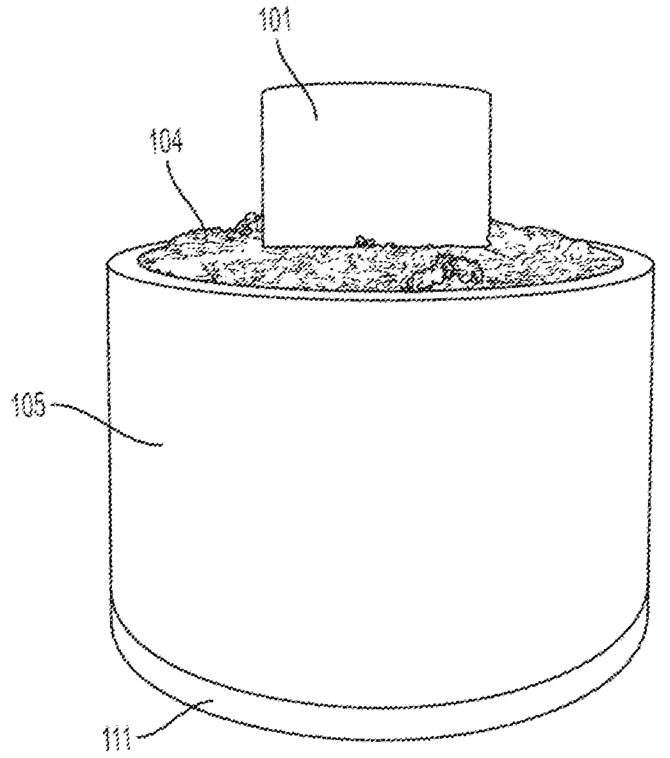

FIG. 9A

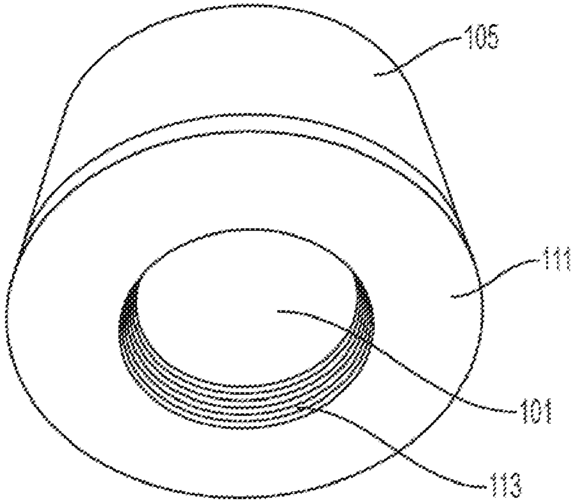

FIG. 9B

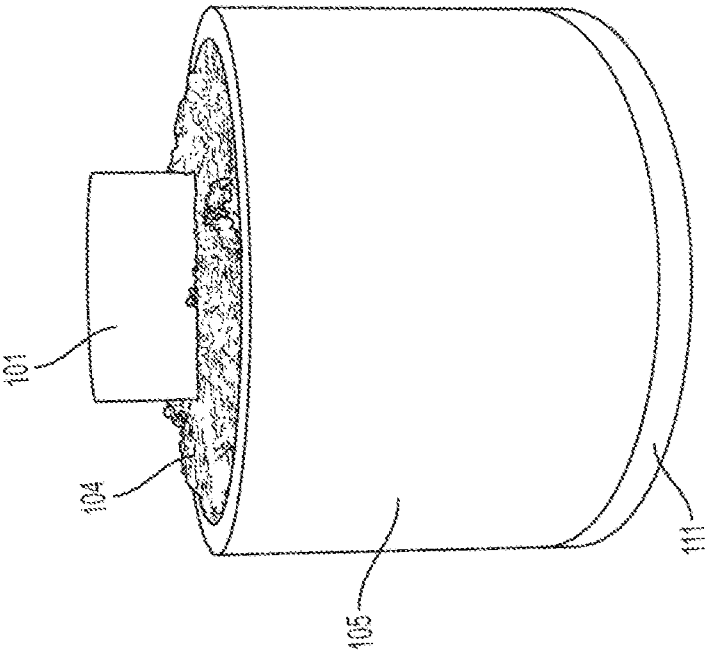

FIG. 10A

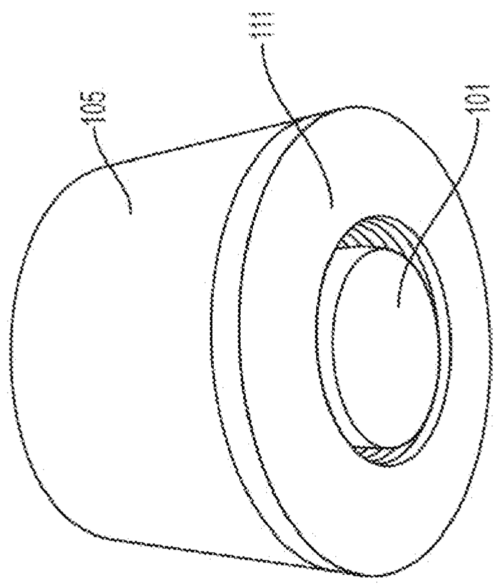

FIG. 10B

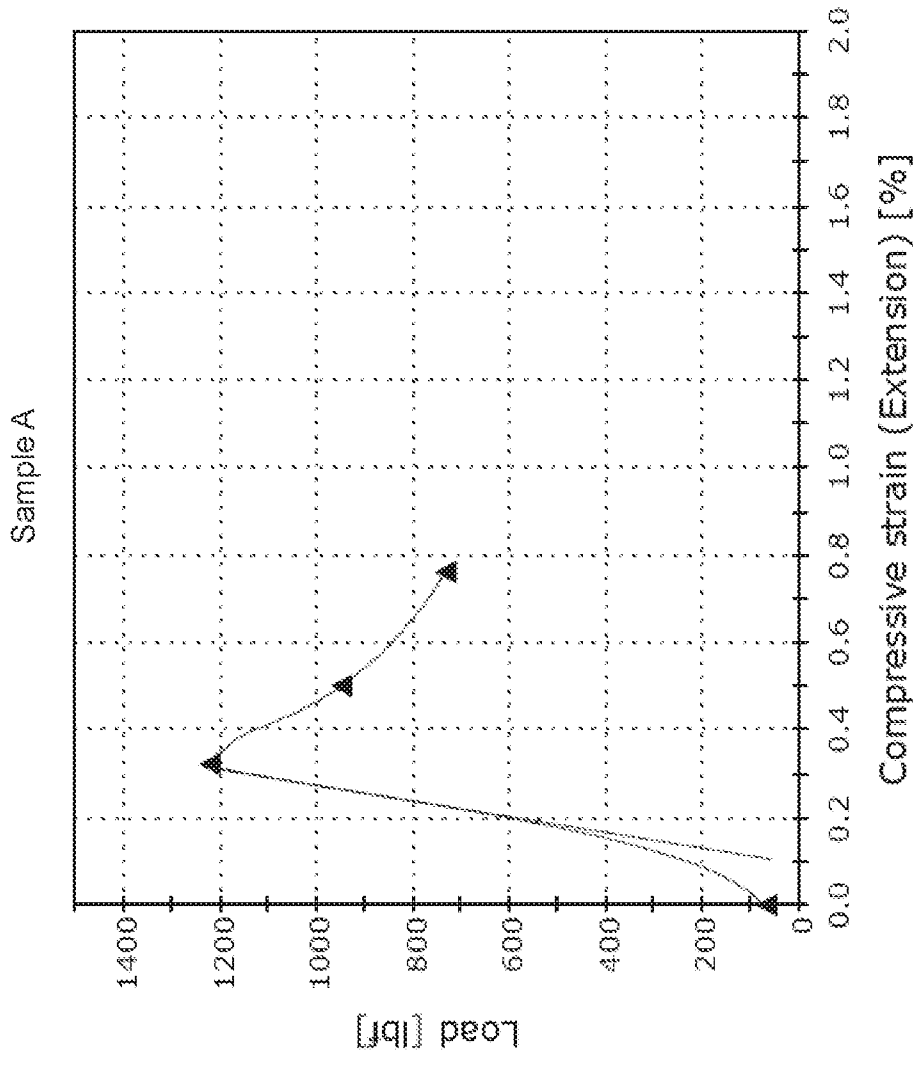
Sample A
Load [lbf]
0
200
400
600
800
1000
1200
1400
0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
Compressive strain (Extension) [%]

FIG. 11

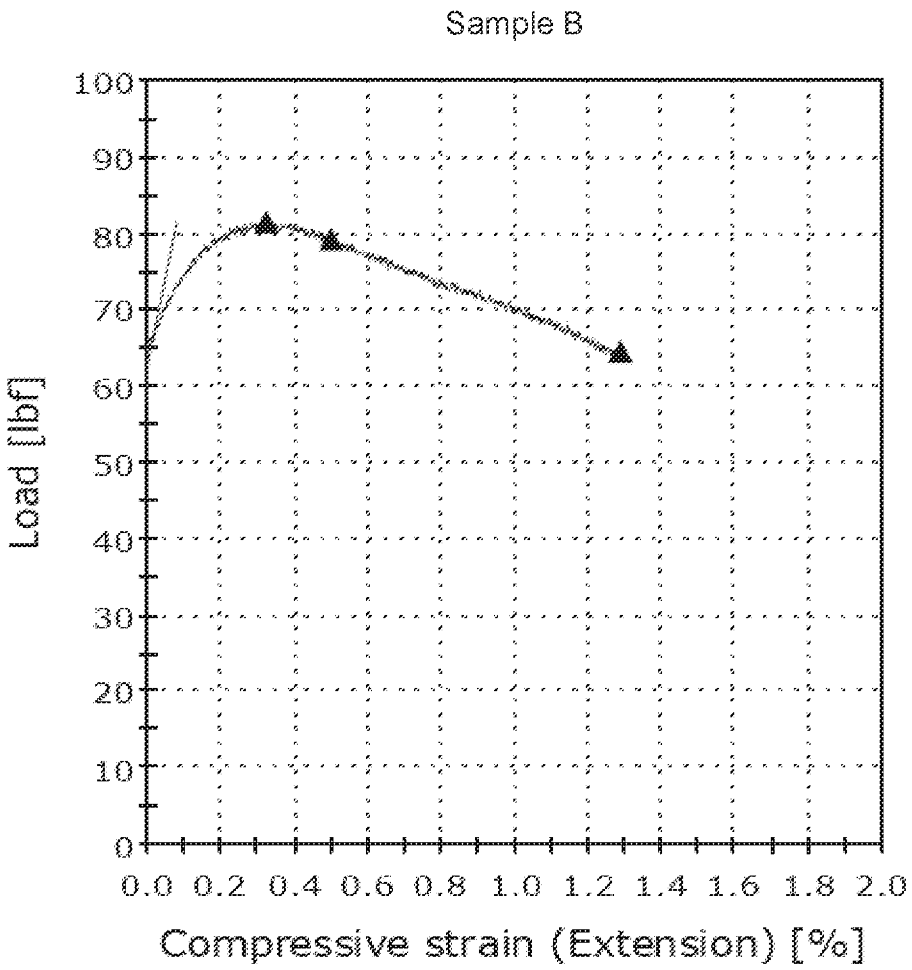
Sample B

FIG. 12

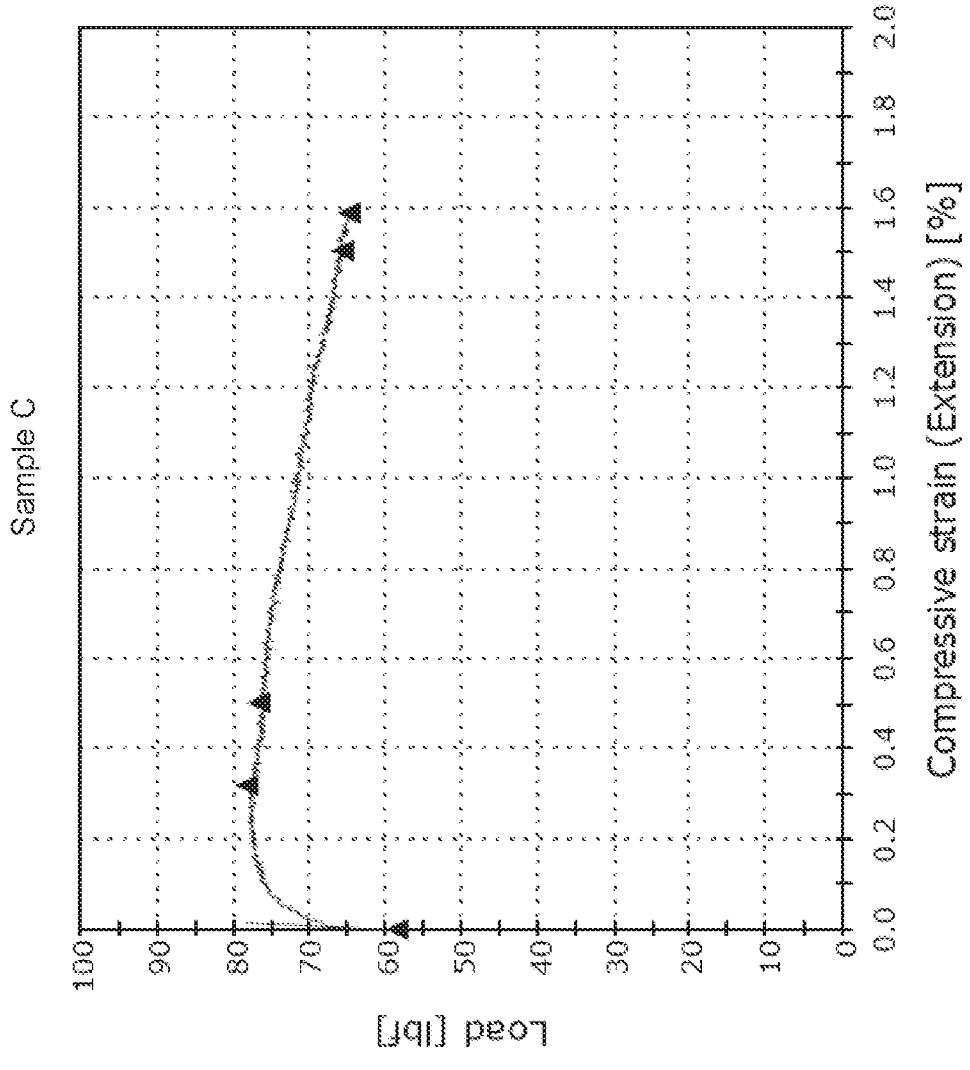
Sample C
Load [lbf]
100
90
80
70
60
50
40
30
20
10
0
0.0
0.2
0.4
0.6
0.8
1.0
1.2
1.4
1.6
1.8
2.0
Compressive strain (Extension) [%]

FIG. 13

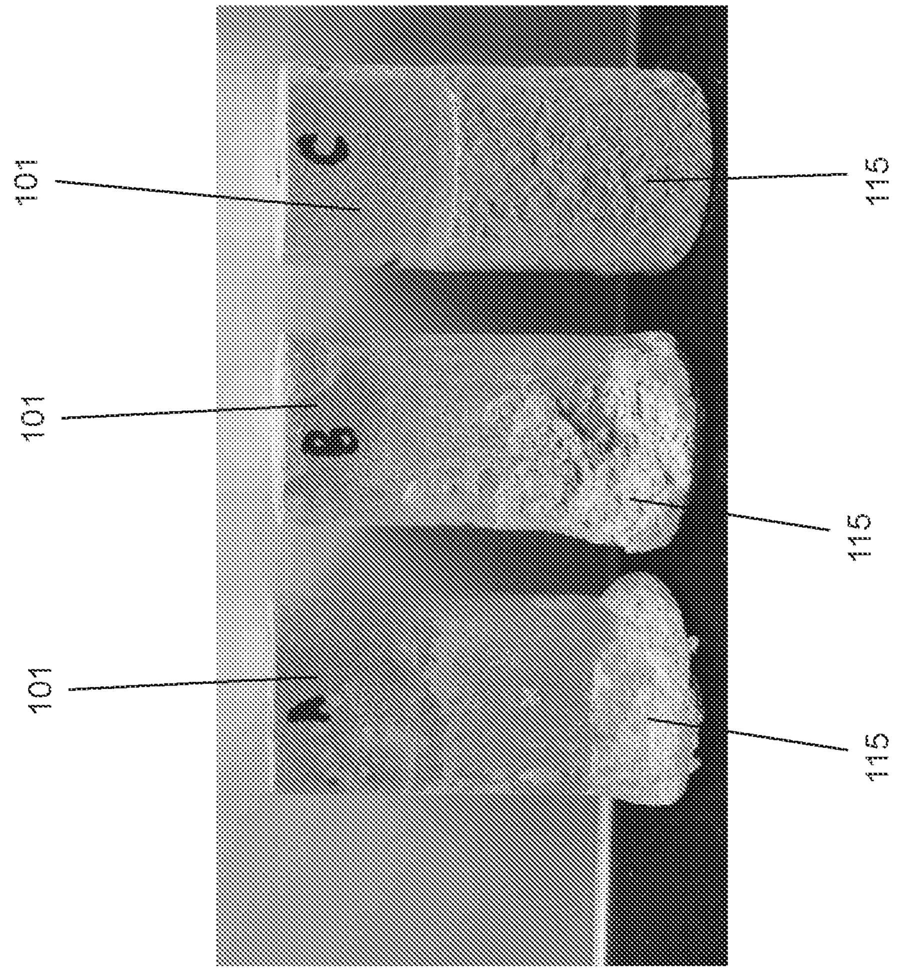

FIG. 14

INTERNATIONAL SEARCH REPORT

International application No.
PCT/US2018/045791

A. CLASSIFICATION OF SUBJECT MATTER

G01N 19/04(2006.01)i, G01N 3/08(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)
G01N 19/04; C09K 8/508; E21B 33/138; G01N 33/38; G01N 3/08

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched
Korean utility models and applications for utility models
Japanese utility models and applications for utility models

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)
eKOMPASS(KIPO internal) & keywords: bond strength, force, formation, test apparatus, insert, core sample, plugging material

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 2014-0174192 A1 (BAKER HUGHES INCORPORATED) 26 June 2014 See paragraphs [0026]-[0046], claims 27, 33 and figures 1-5.	1-6,8-14,17-20
Y		7,15,16
Y	CN 105403505 A (ANTON OILFIELD SERVICES (GROUP) LTD.) 16 March 2016 See paragraphs [0057]-[0062] and figures 1, 3, 4.	7,15,16
A	WO 2004-023111 A1 (SINGAPORE INSTITUTE OF MANUFACTURING TECHNOLOGY) 18 March 2004 See page 10, line 16 - page 11, line 4 and figures 2, 7.	1-20
A	CN 105547999 A (YINBANG CLAD MATERIAL CO., LTD.) 04 May 2016 See claim 1 and figure 1.	1-20
A	US 8703659 B2 (DALRYMPLE et al.) 22 April 2014 See claim 1 and figures 1, 2.	1-20

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:

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"O" document referring to an oral disclosure, use, exhibition or other means

"P" document published prior to the international filing date but later than the priority date claimed

"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

"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

"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

"&" document member of the same patent family

Date of the actual completion of the international search 03 May 2019 (03.05.2019)	Date of mailing of the international search report **03 May 2019 (03.05.2019)**
Name and mailing address of the ISA/KR International Application Division Korean Intellectual Property Office 189 Cheongsa-ro, Seo-gu, Daejeon, 35208, Republic of Korea Facsimile No. +82-42-481-8578	Authorized officer LEE, Hun Gil Telephone No. +82-42-481-8525

INTERNATIONAL SEARCH REPORT

Information on patent family members

International application No.

PCT/US2018/045791

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
US 2014-0174192 A1	26/06/2014	US 9228993 B2	05/01/2016
CN105403505 A	16/03/2016	None	
WO 2004-023111 A1	18/03/2004	AU 2002-337581 A1	29/03/2004
CN 105547999 A	04/05/2016	CN 105547999 B	24/08/2018
US 8703659 B2	22/04/2014	AR 053663 A1	16/05/2007
		AR 053664 A1	16/05/2007
		AU 2010-300581 A1	17/05/2012
		AU 2010-300581 B2	07/02/2013
		CA 2790096 A1	09/09/2011
		CA 2790096 C	30/08/2016
		EP 2483364 A1	08/08/2012
		GB 2438123 A	14/11/2007
		GB 2438123 B	21/07/2010
		GB 2440053 A	16/01/2008
		GB 2440053 B	28/07/2010
		MX 2007008863 A	13/03/2008
		MX 2012003760 A	12/06/2012
		MX 2012010159 A	03/10/2012
		MX 346399 B	16/03/2017
		RU 2007132014 A	27/02/2009
		RU 2007132015 A	27/02/2009
		RU 2012117213 A	10/11/2013
		RU 2400517 C2	27/09/2010
		RU 2413064 C2	27/02/2011
		RU 2564708 C2	10/10/2015
		US 2006-0162930 A1	27/07/2006
		US 2006-0167133 A1	27/07/2006
		US 2006-0234871 A1	19/10/2006
		US 2010-0036017 A1	11/02/2010
		US 2011-0214866 A1	08/09/2011
		US 2012-0279708 A9	08/11/2012
		US 7267174 B2	11/09/2007
		US 8343896 B2	01/01/2013
		US 9284478 B2	15/03/2016
		WO 2006-077371 A2	27/07/2006
		WO 2006-077371 A3	27/03/2008
		WO 2006-077374 A1	27/07/2006
		WO 2007-083109 A1	26/07/2007
		WO 2011-041540 A1	07/04/2011
		WO 2011-107743 A1	09/09/2011