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(54) **Title**: A METHOD OF DIFFERENTIAL ETCHING OF THE SUBTERRANEAN FRACTURE

(57) **Abstract**: The present invention relates to the stimulation of wells penetrating subterranean formations. A method of differential etching of the subterranean fracture wherein nonuniform deposition of a masking material on the fracture surface or face is provided; subsequent treatment by an acid or a reactive fluid generates a heterogeneous etch pattern on the fracture surface, the etch pattern is largely influenced by the placement geometry of the masking material upon closure these irregularities provide mismatch of geometry that leave open conductive channel in the fracture.

A method of differential etching of the subterranean fracture

The present invention relates to the stimulation of wells penetrating subterranean formations. More particularly it relates to acid fracturing and methods of preferentially etching the fracture faces in a heterogeneous pattern. This pattern within the hydraulically and chemically generated fracture will have a geometry that results in a conductive path from the fracture tip to the wellbore. It is through this enhanced etch geometry that one can hope to achieve enhanced fluid flow from the formation to the wellbore.

Patent US20050113263 A1 describes the use of a system that contains both an agent that can dissolve at least once component of the formation and inert solid particles that can inhibit the reaction of the dissolving agent with the fracture faces where it contacts them. The inert particles must be shaped so that, or be deformable into shapes so that, they cover part of the fracture face, rather than having just points or lines of contact.

Patent US20060058197 A1 describes a variety of inert solid particles in combination with a delayed acid system that is primarily composed of a solid acid-precursor.

Patent application PCT/RU2007/000252 disclosures the ideas relate to methods of increasing the conductivity around/between pillars or proppant islands once they are placed. The ideas include: 1. A solvent overflush or an oxidizer/breaker-laden fluid is introduced to the propped fracture pillar network after it has been generated. 2. An overflush of an acidic or otherwise chemically reactive fluid that etches the "open" rock surface that exists between/around the proppant pillars. 3. Coating the proppant with a resin coating that under stress binds the conventional proppant together and forms a mask or barrier on the fracture surface.

Patents US6114410, US6328105B1 describes an improved proppant and a method of increasing fracture conductivity in subterranean formations. The proppant contains a mixture of bondable and removable particles. The bondable particles can be coated with a curable resin. The bondable particles within a subterranean formation adhere to adjacent bondable particles to form a permanent, self-supporting matrix; and the removable particles from the self-supporting matrix provide the ambient fracture conditions. This increases fracture conductivity and the overall productivity of the hydraulic operation.

Patent US20050274523A1 describes methods for the treatment of subterranean wells involving injecting a first fracturing fluid into a formation, and then injecting at least a second fracturing fluid into the formation in order to create extended conductive channels through a formation are described. The fracturing fluids can be similar in density, viscosity, pH and the other related characteristics. Alternatively, the fracturing fluids can differ in their densities, viscosities, and pH, allowing for variations in the conductive channels formed. Propping agents can also be included in one or both of the injected fluids, further enhancing the conductive channels formed. The described methods aid in minimizing proppant flowback problems typically associated with hydraulic fracturing techniques.

The ideas in this patent memo describe several methods that may be employed to decrease the tendency of an acid, or any reactive fluid, from reacting evenly with the fracture face, primarily within carbonate formations. However, these inventions can be applied to any formation which can be etched by means of a reactive fluid. By increasing the heterogeneity of the etch pattern, it is desired that conductive channels along the fracture faces are created. A brief summary of each idea is provided below.

Invention embodiment one. The masking material could placed heterogeneously in the fracture. This material invades the surface of the rock through a process of extrusion or simply leaves a film on the surface of the rock when compressed under closure stress condtions (pressure and/or temperature) when the fracture has closed. Acid is then pumped at fracturing pressures, re-fracing the formation. Even though the bulk of the material may be displaced, the remaining residue would protect the rock, even if this residue on the fracture face or glued film was only present on one side of the fracture face.

Invention embodiment two. The masking material is placed relatively uniformly on the fracture faces. A reactive fluid is pumped into the hydraulic fracture. The acid finds anomalies or weak areas of the film and it begins to penetrate this layer and react with the native rock underneath. Provided that the amount of acid exposure is controlled, the resulting etch pattern can be quite irregular.

Invention embodiment three. A material is pumped that self agglomerates once in the fracture. This agglomeration process could be triggered either through diffusion of a reactive chemical or via physical or chemical changes to the material itself. A reactive fluid (e.g. acid) is then immediately pumped into the fracture. The reactive fluid flows uniformly through the fracture in the near wellbore area, but where it contacts the clusters/agglomerations of material, its flow is impeded. Preferential flow is around and not through these structures. Due to the differential flow patterns, the etch pattern is likewise altered and non-uniform.

Detailed Description of the Invention

In acid fracturing treatments, acid is pumped into a hydraulic fracture, preferentially along the entire length of the structure (i.e. from the tip to the wellbore). These are typically pumped in carbonate formations. The goal of these treatments is to create disparities within the rock such that when the opposing fracture faces close upon one another, the geometry does not match. In the absence of other influences, the differential etching typically results from localized heterogeneities in the native formation. It is desired that these new geometries provide a conductive flow path for produced fluid (or injected fluids as the case may be) along the fracture faces.

A major problem that is encountered during acid fracturing treatments pertains to the reaction rates of the acid with the formation. Oftentimes the acid reacts uniformly with the formation, especially in localized regions of the fracture. When this occurs, the etch pattern is not sufficient to support conductive channels along the fracture face after fracture closure. This often occurs when the delivery rate of the acid to the fracture face is much lower than the rate of the reaction of the acid. Several methods have been used in attempts to alleviate or minimize these problems. One method has been to keep the live acid separated from the formation. This can be done by a variety of methods such as emulsifying or encapsulating the acid and then releasing the acid at a later time when and where it is desired. A second method has been to delay the formation or generation of the acid. Several systems have been described that generate acids once they are downhole, within in the fracture. A third method involves the use of non-acidic fluids and acidic fluids which "finger" through one another to generate differential etching patterns.

Recent Schlumberger patent applications (US20050113263, US20060058197) have described inventions that aim to generate heterogeneity within the acid fracturing operation through the use of inert "masking agents". The additional inventions provided in this patent memo are aimed at adding to and improving on the ideas expressed in these patent applications. The embodiment of this invention does not appear to be expressly stated in the claims of the patent.

The first embodiment pertains to the use of inert materials as masking agents, with particular emphasis on their ability to either a) extrude into the rock formation, or b) cast a film or residue on the rock surface. In previous applications, the inert particle is described as having a shape, structure, or properties such that they conform to one or both faces of the fracture and inhibit reaction of acid with the formation where they conform to the fracture face. These masking agents are placed heterogeneously throughout the fracture, covering a portion of the fracture faces and preventing the acid from reacting with this portion of the fracture faces. The un-reacted fracture faces create a small pillar that is capable of holding open the etched fracture when it closes upon itself. The open area of the fracture is nearly infinitely conductive. An illustration of this concept is shown in Figure 1. In this configuration, the masking material which helped create the pillar may remain or can dissolve. The same will hold true for all further masking agents listed in this patent memo.

In previous description, it was usually inferred that the masking material was a solid inert particle that remained on the surface of the fracture face during the acidizing process.

There is a side view of a hydraulic fracture on the Fig. 2.

Frame A: An inert masking material has been heterogeneously placed into the fracture.

Frame B: The fracture closes upon itself and the inert material is compressed.

Frame C: The fracture is hydraulically pressurized by a reactive fluid (arrow). Most of the inert masking material is displaced from its initial location and pushed further down the fracture. The residue of the masking agents is left behind. In region 1 the residue has invaded the formation. In region II the residue has only left behind a thin film on the surface of the formation. In region III no residue from the masking agent is left behind.

Frame D: The inert residue in regions I and II protects the rock from the reactive fluid (arrows). In the “masked” regions the surface of the rock remains as it was originally. In region III the rock is homogeneously etched by the reactive fluid.

Frame E: When the fracture closes on itself, the protected regions in I and II are now high spots and serve as pillars that support the weight of the fracture and hold it open. In region III the fracture closes upon itself as the region was etched homogeneously.

As illustrated in Figure 2, several scenarios may exist where the bulk of the masking material can be sloughed off or otherwise removed, however the small portion of the material 1 that either extruded into the rock face or left behind a thin film provides adequate protection. In this process of film casting or extrusion, the surface of the masking agent may undergo a phase change (solid to liquid and possibly back to solid again).

The second embodiment pertains to the use of a fluid which deposits a relatively uniform film on the fracture surface. This may be likened to a filter cake, however, its primary function is not to control fluid loss. Rather, it provides a barrier which limits the influence of the acid on the native rock underneath. Due to the challenges in applying a completely uniform film, this structure is likely to have anomalies or weak areas that will allow the inflow of acid. As the acid penetrates the film in distinct locations, the formation underneath will begin to etch the rock. Provided that the length of acid exposure is controlled, this can result in an uneven etching pattern that leads to the development of pillar-like structures. The details of this idea are illustrated in Figure 3.

There is side view of a hydraulic fracture in Figure 3. .

Frame A: The hydraulic fracture is maintained in an open configuration by a standard fracturing fluid.

Frame B: A second fluid is pumped into the fracture and it leaves behind a relatively uniform film or residue on the fracture surface.

Frame C: A reactive fluid (black arrows) is pumped into the hydraulic fracture. Within anomalies or weak areas of the film, the acid will begin to penetrate this layer and react with the native rock underneath

Frame D: The fracture is over flushed with a wash or standard fracturing fluid (white arrow). The yellow film may remain on the surface or it may have been removed.

Frame E: The fracture closes. The etch pattern may be irregular and not match on either side of the fracture, yet a series of high and low regions are created. The resulting structure leaves small channels that remain open even after the closure stress is applied to the rock faces.

Within this invention it should also be considered that or a uniformly placed solid could also serve to achieve this goal. In such a scenario, a solid can be placed within a fracture and then under the influences of heat, time, and pressure, it can be converted to a liquid.

The third embodiment pertains to the use of self-assembling (self-agglomerating particles) for the purpose of changing the flow characteristics within the fracture. The material is pumped as relatively small particles (fibers, ribbons, platelet's, spheres, etc) that can pass through the perforation. However, after passing through the perforations, the material undergoes a transformation which aids in the agglomeration of the material. This agglomeration can be tuned by a number of factors (temperature, fluid chemistry, time, pressure, etc).

One possible use of these self-assembling strips for acid diversion is illustrated in Figure 4. Wellbore, perforations and hydraulic fracture formed during the implementation of the methods of this patent memo.

Frame A: Light grey indicates regions exposed to a standard fracturing fluid. Black structures areas indicate particulate matter. In this example they are strips or coated fibers. The fibers are sent down the wellbore and through the perforations as individual particles. Once through the perforations, the material begins to agglomerate and form clusters. The number and size of agglomerated material may vary.

Frame B: A reactive fluid is pumped into the hydraulic fracture (Black arrows). The fluid flows uniformly through the fracture in the near wellbore area, but where it contacts the clusters/agglomerations of material, it's flow is impeded. Preferential flow is around and not through these structures.

Frame C: The resulting fracture geometry after the treatment is over. The fracture face is etched in many locations (shaded area); however, in the vicinity of where the agglomerated material had been deposited, the amount of etch is limited (black). These structures then serve as pillars to support the fracture and keep it open.

It is desired that these agglomerations may become wedged or otherwise travel at a rate which is slower than that of the bulk fluid. Preferential flow of the fluids will therefore be around and not through these agglomerated masses. If a reactive fluid is passed through a fracture containing such structures the resulting geometry should result in an etch pattern that has high spots (less reacted rock) in the vicinity of these agglomerations. Once again, these high areas can serve as pillars which will serve to hold the fracture open after closure.

Experimental Examples

Experiment for embodiment 1: The surface of an Indiana Limestone block (17.8 x 7.6 x 1.9 cm) was selectively covered with a thin film of silicone gasketing material (Dow corning Q3-1566 Heat resistant sealant). The material was applied liberally to the surface in select regions and then scraped off. Successive applications were applied and scraped off. Later the surface was lightly rubbed by hand in order to remove as much of the gasketting material as possible.

The block was then exposed to 37% HCl at room temperature and ambient pressure for approximately 5 minutes. The rock face was heterogeneously etched with preferential etching in the non-coated areas. The surface of the rock was notably smoother in the gasketed areas.

Experiment for embodiment 2: Blocks of of Indiana limestone (8.5 x 4.1 x 2.6 cm).were covered with a thick coating of standard hot melt glue (ethylene vinyl acetate co-polymer). After being pressed together and heated to 80 deg C for several days, the blocks were removed and separated and excess glue was scraped off the surface. This process was repeated to ensure a uniform coating. One of the uniformly blocks was then dipped into a concentrated solution of 37% HCl for approximately 5 min. Upon removal, it was noticed that the surface etch pattern is irregular, and in some regions where the glue is still present, the etch is minimal.

Experiment for embodiment 3: A film of polylactic acid was cast onto a Teflon surface 2. The adhesive coated substrate was then coated with either glucose 3 or calcium carbonate 4. It was noted that when the glucose-coated strips were dipped in water, the glucose dissolved and the strips were made to be sticky again 5. The calcium carbonate coated strips maintained their coating 6, even after being dipped in water. This is illustrated in Figure 5.

When the loose glucose-coated strips were added to stirred slurry containing a linear guar-based fracturing fluid and sand, the strips quickly agglomerated and strongly adhered to one another. However, when calcium carbonate-coated strips were added to a similar solution, no agglomerations were formed. After 10 minutes of mixing 1 mL of acetic acid was added to the solution to dissolve the calcium carbonate coating. The solution was allowed to stir for an additional 5 minutes and no agglomeration was noted. An additional 3 mL of acetic acid was added to the solution. Two minutes after this last addition the adhesive strips began to adhere to one another, forming an agglomerated mass.

Additional experiment for embodiment 3: Intermediate strength ceramic proppant (20/40 mesh) was coated with an acrylic-based spray adhesive. This coating was then covered with a dusting of calcium carbonate powder. This proppant was no longer sticky towards each other or other objects, even when placed in water. Another batch of the ceramic proppant was coated with an commercially available acrylic-based black spray paint. When completely dry, the painted proppant had no adhesive tendencies. The two proppant were placed into a flow loop containing fresh water. The two proppant were free flowing and were homogeneously distributed. Acid was slowly introduced into the flowloop. When the pH had slightly decreased, the external coating of calcium carbonate of the adhesive-covered proppant began to react and dissolve. This revealed the layer of adhesive on these proppant particles. When two adhesive coated proppant particles came into contact, they would sometimes adhere; however, there was less tendency for an adhesive proppant and a painted proppant to adhere, and no tendency for two painted proppant to adhere. Within several minutes all of the adhesive-covered proppant had agglomerated, while the painted proppant remained free-flowing. The agglomerated particles moved more slowly within the flow loop and the painted particles would pass over and around these agglomerated masses.

Claims:

1. A method of differential etching of the subterranean fracture wherein nonuniform deposition of a masking material on the fracture surface or face is provided; subsequent treatment by an acid or a reactive fluid generates a heterogeneous etch pattern on the fracture surface, the etch pattern is largely influenced by the placement geometry of the masking material upon closure these irregularities provide mismatch of geometry that leave open conductive channel in the fracture.
2. The method of claim 1, wherein the acid treatment is applied to surfaces of hydraulic fractures.
3. The method of claim 1, wherein the said material for the masking covering intrudes or extrudes into the formation surface under the influence of closure stress and or formation temperatures; the said material is transported by the fracturing fluid to the fracture.
4. The method of claim 1, wherein the said material consists of either: inert particles (beads, particles, plates, ribbons, fibers, or mixture of thereof); a liquid or a gel.
5. The method of claim 1, wherein the masking material is deposited as a film with a non-uniform thickness or texture.
6. The method of claim 5, wherein the said film is cast onto the surface of the fracture from solid particles that are then transformed into liquid phase.
7. The method of claim 5, wherein the said film is cast onto the surface of the fracture in the form of a liquid or a gel.
8. A method of differential etching of the subterranean fracture wherein particles which are initially non-tacky/non-adhesive are pumped into the fracture by means of a fracturing fluid or other means of conveyance; at a prescribed time, the said particles exhibit adhesive properties; the interaction of the adhesive particles with each other or the fracture surface leads to the non-uniform

distribution of material within the fracture; the particles of agglomerated material impede/restrict the flow of reactive fluids through them, and require the fluid to flow around them.

9. The method of claim 8 wherein the particles of agglomerated material can be used to create the said heterogeneous pattern of claim 1.

10. The method of claim 8 wherein the particles of agglomerated material move along the treated surface slower than the flow rate of subsequent stages of fracturing fluid, including an acid or a reactive fluid.

11. The method of claim 8 wherein the particles of agglomerated material can behave as described in claims 3, 5, 6, or 7, either separately follow individually or a combination of claims.

12. The method of claim 8 wherein the particles exhibit adhesive properties while at some time after their delivery through the perforation and into the fracture.

13. The method of claim 8 wherein the particles can consist of plates, beads, ribbons, fibers, and mixture of thereof.

14. The method of claim 8 wherein the particles can be consist of layers, or coatings of adhesive and non-adhesive materials onto the core particle.

15. The method of claim 8 wherein the particle exhibits adhesive properties due to a chemical or physical change or transformation at the surface of the particle. This change on the surface can occur via a variety of mechanisms that may include: heat, pressure, solubility kinetics, shear rate, abrasion, and addition of chemical agent.

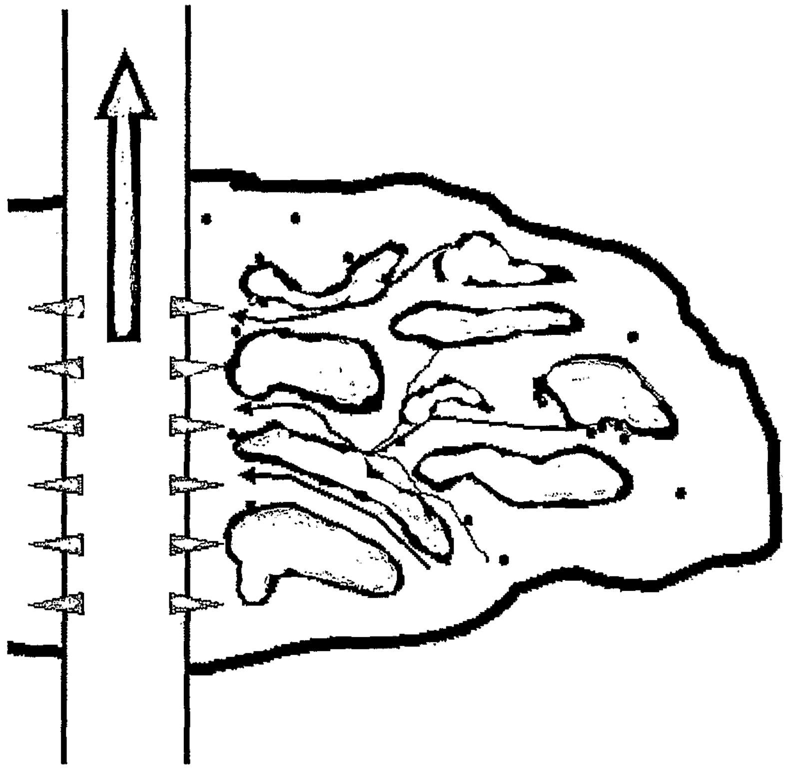

Figure 1

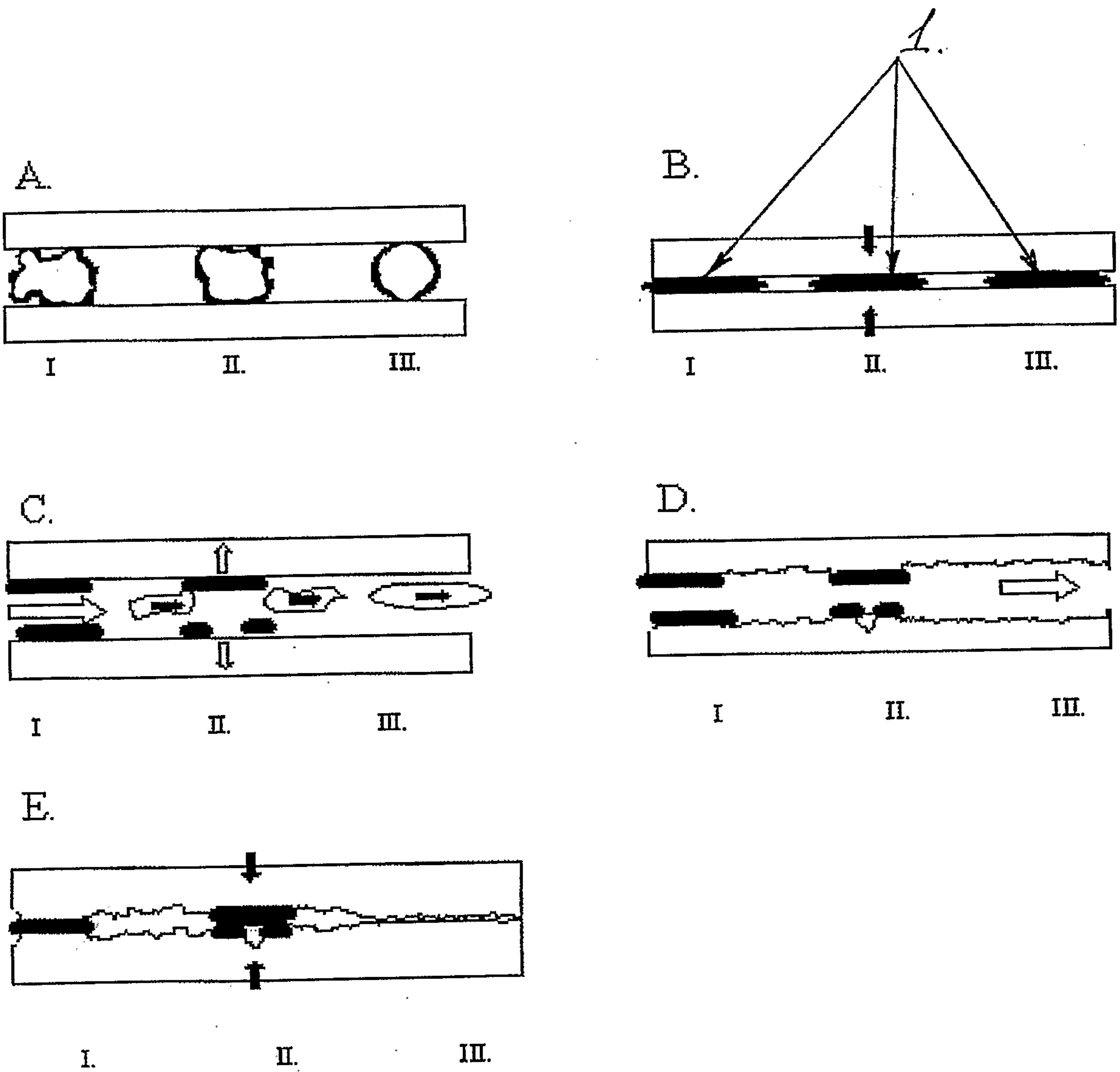

Figure 2

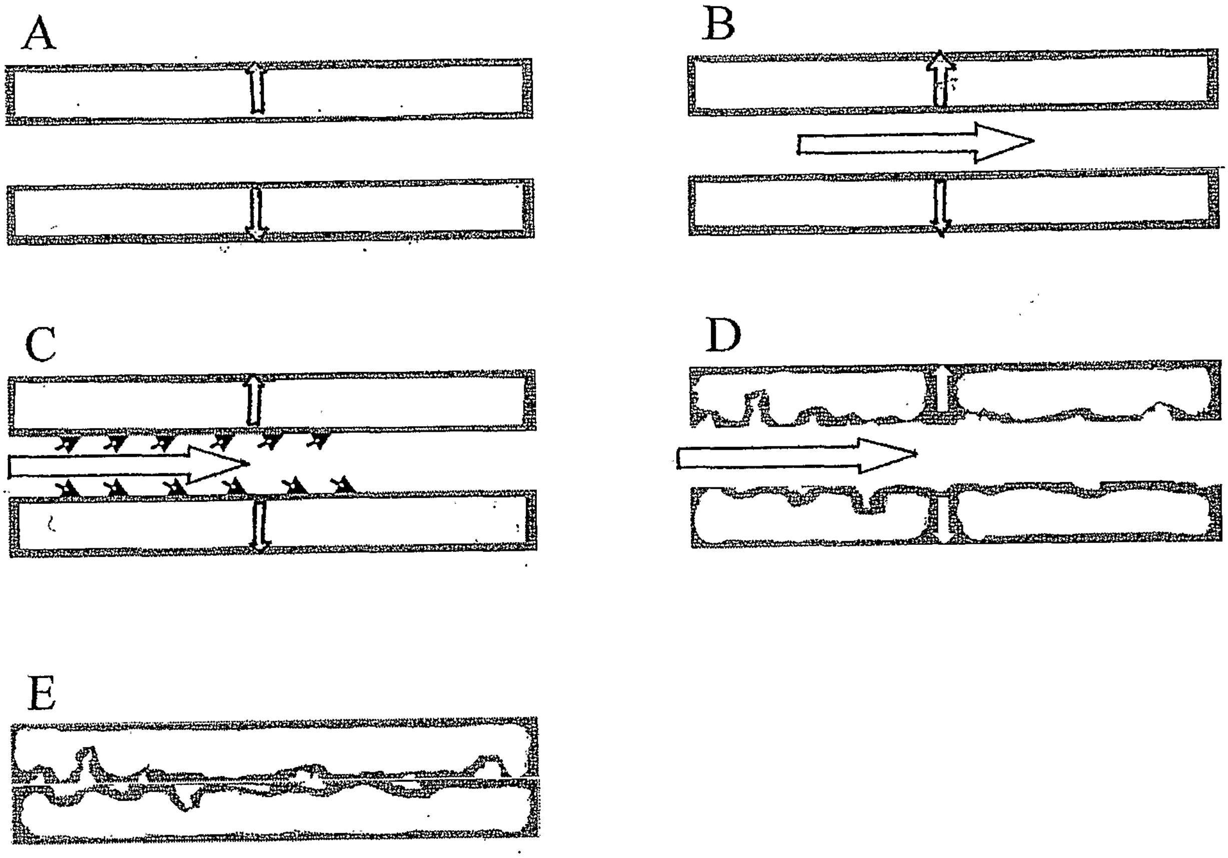

Figure 3

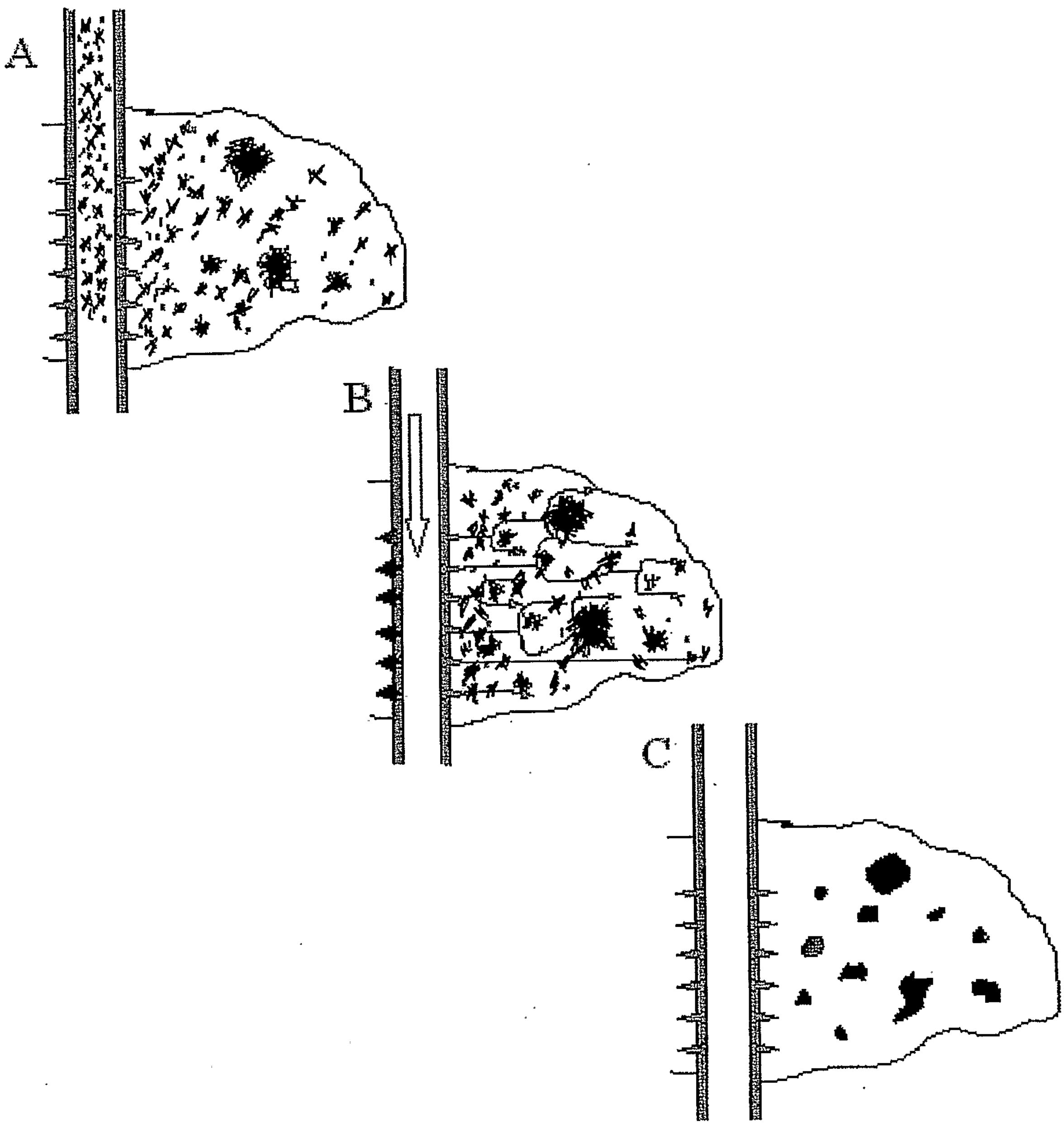

Figure 4

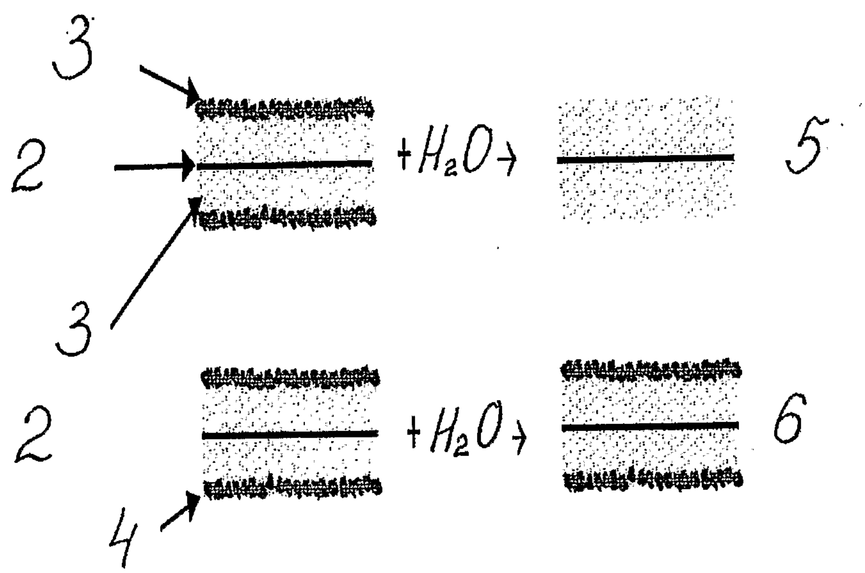

Figure 5

International application No.
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INTERNATIONAL SEARCH REPORT

A. CLASSIFICATION OF SUBJECT MATTER

E21B 43/27 (2006.01)

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)

B01J 2/00, 2/20, B32B 5/00, 5/16, C09K 8/00-8/94, E21B 33/13, 33/138, 43/00, 43/26-43/27

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, PCT, Esp@cenet

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X Y	US 2005/0113263 A1 (J. ERNEST BROWN et al.) 26.05.2005, abstract, paragraph [0003], [0010], [0011], [0018], [0020] - [0024], [0028], [0030], [0031], claims	1-7 8-15
Y	US 6328105 B1 (TECHNISAND, INC.) 11.12.2001, abstract, col. 1, lines 18-45, col. 3, line 17 - col. 4, line 15, claims	8-15
A	US 6632527 B1 (BORDEN CHEMICAL, INC.) 14.10.2003, col. 37, lines 33-40, table 10	1-15
A	WO 2006/032833 A1 (HALLIBURTON ENERGY SERVICES INC. et al.) 30.03.2006, abstract, claims	1-15
A	US 2003/0106690 A1 (CURTIS L. BONEY et al.) 12.06.2003, abstract, claims 4-5	1-15

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

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Date of the actual completion of the international search	Date of mailing of the international search report
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