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3,438,440

**ELECTROLESS METAL BONDING OF UN-
CONSOLIDATED FORMATIONS INTO
CONSOLIDATED FORMATIONS**

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No Drawing. Continuation-in-part of application Ser. No.
579,223, Sept. 14, 1966. This application Dec. 22, 1967,
Ser. No. 692,686

Int. Cl. E21b 33/13, 43/27

U.S. Cl. 166—292

11 Claims

ABSTRACT OF THE DISCLOSURE

A method of consolidating an incompetent formation having a temperature greater than 125° F. by metalizing or metal plating the formation by an electroless metal plating process using an acidic metal plating solution.

**CROSS-REFERENCE TO RELATED
APPLICATION**

This application is a continuation-in-part of copending patent application Ser. No. 579,223, filed Sept. 14, 1966, now Patent No. 3,393,737.

This invention relates to treating incompetent or unconsolidated formations such as unconsolidated subsurface formations and more particularly to treatment of loose or incompetent earth formations surrounding well bores so as to consolidate said formations into a permeable, thermally and hydrolytically resistant consolidation formation for improved and efficient recovery of fluids therefrom.

BACKGROUND OF THE INVENTION

It is well known that many difficulties are encountered in producing or recovering fluids from incompetent earth formations due to collapsing or sloughing of the well bore walls. Numerous means have been employed to alleviate this and among the methods and devices used to prevent collapsing and sloughing of unconsolidated formations is the use of perforated pipe liners, gravel packing or tubular screens or by injecting resin forming materials such as phenol-formaldehyde resins or epoxide resins which function as bonding and consolidating agents for weak formations. Another method employed involves subjecting the incompetent formations to elevated temperatures so as to cause fusion of constituents therein, e.g., silica sand particles, to provide bonding agents. Still another means is to form carbonized or coked materials which act as binders to hold the formation as an integral consolidated mass. Essentially these methods and means for consolidating incompetent earth formations have serious limitations as, for example, the mechanical devices mentioned tending to become plugged and generally are incapable of preventing fine particles from entering the production well. Also, these devices require cleaning and constant attention. The use of resin consolidating materials requires special equipment and a treatment process necessitating the presence of a drilling rig and the process is generally time consuming and costly. In essence the same applies to thermal means of consolidating formations as mentioned above or other similar means known to the art. Thus conventional thermal and chemical means of consolidating loose or incompetent formations are generally inefficient, ineffective, costly and generally cause a decrease in permeability of the formation, and they lack desired resistance to changes in stresses, strains, pressure and temperature conditions normally encountered in producing effluent from such formations.

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An object of the present invention is to provide an improved method of consolidating loose or incompetent subsurface formations.

Another object of the present invention is to consolidate loose formations with a high temperature metallic binding agent which is resistant to hydrolysis and is capable of withstanding great pressures, strains and stresses.

Still another object of the present invention is to bind the grains of loose formations, at elevated temperatures greater than about 125° F. with a polyvalent metallic binding agent which is resistant to corrosion and is not effected by hot fluids such as water, steam and the like and is also resistant to high thermal temperatures caused by combustion drives and the like.

Still another object of the present invention is to form a metallic consolidated subsurface earth formation having good permeability and good compressive strength, for the recovery of hydrocarbon fluids therefrom particularly when using thermal drives such as hot water or steam in the recovery process.

Still another object of this invention is to metalize formations so as to consolidate them and protect natural concentration materials against dissolution, particularly at high temperatures.

Still other objects and advantages will be apparent from the description and examples illustrating the present invention.

SUMMARY OF THE INVENTION

It has now been discovered that incompetent formations in which high temperatures are encountered and which are generally penetrated by wells, can be effectively consolidated and completely protected from said dissolution by penetrating such formations at high temperatures with an acidic electroless metal-deposition solution having a pH of from about 2.5 to about 5, preferably between 3 and 4.5, capable of plating (at a controlled reaction rate) on the unconsolidated sand grain particles, a metal coating which protects and binds together the grains of the formation into a permeable consolidated form. The electroless metal-deposition and consolidation of the formations is effectively accomplished by first penetrating an unconsolidated formation with an activator fluid capable of activating or catalyzing the grains of the formation, and thereafter contacting the activated or catalyzed formation at an elevated temperature with an acidic metal-deposition solution containing chemicals inclusive of polyvalent metal ions and a reducing agent, and effecting a chemical-reduction deposition of metal within the formation.

In consolidating oil-bearing loose formations having high reservoir temperature (above 125° F. or higher, e.g., 150–200° F.) for the recovery of hydrocarbon fluids therefrom by means of wells completed therein, it is preferable that prior to penetrating such formations with an activator and acidic metal-deposition solutions as mentioned above, that such formations be pretreated with an acidizing fluid and/or preflushing fluid so as to displace oil and connate-water in areas desired to be consolidated by injecting therein suitable acidizing solutions and/or preflushing solvents. During the activating of the components of loose formations, e.g., fine and/or coarse sand particles, by an activating or catalyzing solution and the subsequent consolidation of the formations with an acidic metal-deposition solution at a high temperature, the solutions can be injected into the loose formations with or without spacer fluids between them. It is preferable that the formations be pretreated with a suitable acidizing solution or solvent, prior to penetrating the loose formations with the catalyzing or activator fluid, followed by displacing the activator fluid with an inert fluid such as air or water or by a liquid composition containing a re-

ducing agent and finally metal coating the loose formations with acidic metal-deposition solution by electroless means as will be fully described below.

Metals deposited by the process of this invention on loose sand grains in unconsolidated earth formations form excellent binding agents which consolidate the loose sand grains into stable permeable integral formations capable of sustaining great compressive forces and resisting damage to the formations caused by thermal drives. Also, the metal coatings on the grains form impermeable layers that protect the grains from destruction by hot fluids such as hot water and/or steam. The metal coating of siliceous components in earth formations also prevents dissolution of the silica that is contacted by hot aqueous fluids when such fluids are flowed through the earth formations, for example, in recovery of hydrocarbon fluids therefrom.

An unconsolidated mass of sand grains is consolidated by the process of this invention by impregnating the mass first with an activator liquid and then with an acidic metal-deposition solution containing chemicals inclusive of metal ions, a lower carboxylic acid, e.g., formic or acetic acid or alkali metal salts thereof, and a reducing agent so as to chemically deposit within the mass a metal coating which consolidates the mass. The amount and disposition of the deposited metal are sufficient to bind the sand grains into a consolidated mass capable of sustaining compressive forces of many hundreds of pounds per square inch. Also, the sand grains are coated with an impermeable layer of metal that protects them from being dissolved by hot aqueous fluids.

For the most effective results it is desirable to flow a plurality of pore volumes of both the activator solution and acidic metal-plating solution through the interval of the formation into which the well is opened and preferably flow pore volumes of each of said solutions through generally shaped zones, e.g., cylindrical or spherical zones having a diameter of from about 1 to about 5 feet around to open portions of a well borehole.

The results of effecting a chemical-reduction deposition of metal within a porous earth formation that surrounds the borehole of a well are such that this is a particularly advantageous process for treating such an earth formation. Where the earth formation is unconsolidated, the metal deposition provides a method of consolidation in which the chemical costs are no more than those of sand consolidation procedures which have proven to be economically advantageous. Where the well is to be employed in the injection or production of hot fluids, the metal deposition provides a treatment that (a) consolidates any unconsolidated portions of the earth formation; (b) metal plates any siliceous components and prevents the dissolution of silica that tends to occur whenever a hot aqueous fluid is flowed through a siliceous earth formation that was naturally consolidated or was consolidated by a conventional sand-consolidation procedure; (c) metal plates and improves the stability of any intergranular bonding material that has been formed within the earth formation; and (d) reduces the heat loss that occurs within the tubing string of production wells that extend into communication with the earth formation by depositing on the tubing strings a reflective metal plating that reduces the thermal emissivity of the tubing string.

In general, a chemical-reduction deposition of metal within a porous mass of earth-formation material is an advantageous procedure for improving the strength and stability of the mass. The electroless acidic metal-deposition treatment provides a convenient and relatively economical procedure for binding a sand into a mold in which to solidify a molten material, such as a molten metal, for increasing the thermal or electrical conductivity of a mass of earth-formation material or for dispersing and fixing metals that are to be utilized as catalysts, activators, property indicators, or the like, within such a porous mass, etc.

PREFERRED EMBODIMENT OF THE INVENTION

The process of metal plating unconsolidated earth formations into consolidated form can be effected by the following sequential steps for consolidating a zone around a borehole:

Sand formation consolidation at temperatures
above 125° F.

(1) Inject if necessary an acidizing fluid such as a mud acid and thereafter wash the formation with several pore volumes of a solvent such as isopropyl alcohol to achieve the desired injection rate;

(2) Preflush the formation also if necessary or desirable with conventional liquids such as several pore volumes of hydrocarbon oil, e.g., diesel oil, and/or solvent such as isopropyl alcohol;

(3) Inject activator solutions which are capable of activating or catalyzing the sand grains such as a colloidal palladium solution;

(4) Inject an acidic metal plating solution;

(5) Inject a spacer fluid which is preferably an ammoniacal buffer solution;

(6) Inject water; and

(7) Consolidation of formations by metalization is thereafter effected.

In permeating a porous mass by the process of the present invention with an activator or catalytic liquid, each element of the mass is preferably contacted with at least several pore volumes of the liquid. Palladium-activator solutions or stannous-activator solutions should also contain reducing agents such as hydrazine or sodium hypophosphate or lower aldehyde, e.g., formaldehyde. Such activator solutions can be palladium chloride and/or stannous chloride solutions or corresponding bromide, nitrate or sulfate solutions or preferably an acidic aqueous palladium chloride-hydrazine solution. Other such activator fluids can be aqueous solutions containing gold, ruthenium, rhodium, platinum or any of the so-called metallic hydrogenation catalysts and a reducing agent such as hydrazine with or without the presence of protective colloids, e.g., soluble gums such as gum arabic tragacanth; proteins, e.g., gelatin, albumin, starch, glucosides or the like. The porous mass can be first treated with an acid solution such as sulfuric or hydrochloric acid solutions alone or in conjunction with the activator solutions. The pH of the activator solution should be in the range of 3-5 and can be controlled by the presence of lower acids such as formic or acetic acids and salts thereof and mixtures thereof.

The formation can be pretreated with a mud acid (410 cc. concentrated HCl+590 cc. H₂O+32 grams NH₄F+2 cc. amine corrosion inhibitor) or any suitable acidizing fluids such as described in U.S. Patents 3,215,199; 3,236,305; 3,249,536 and 3,251,415.

The activator liquid may be displaced prior to injecting the acidic metal plating solution at an elevated temperature (100-200° F.) by an inert fluid, for example, by air where the liquid is drained from a mold, or by a liquid containing a reducing agent. In treating a subsurface earth formation it is preferable to precede the above steps by a conventional oil- and connate-water-displacing procedure such as described in U.S. Patent 3,294,166 for sand consolidation with epoxy resin. Since this procedure generally displaces oil and connate-water films from the tubing string, such a pretreatment ensures that some metal deposition will occur in the injection tubing string when the metal-deposition solution is injected into the treated porous mass through said tubing string in the well bore in communication with the porous mass, and therefore some thermal-emissivity reduction can be provided in respect to the thermal properties of the tubing string.

The acidic metal-containing solution can be in the pH range of 2.5-5, preferably 3-4.5. The pH regulators can be aqueous acidic solutions containing lower carboxylic

acids, e.g., formic and/or acetic acids, and their salts with strong bases and mixtures thereof. Regulation and control of the pH of the solution is essential in controlling the reaction rate of metal plating so as to prevent heavy metal deposition at the inlet face of the formations to be consolidated. Also, by this means more controlled and uniform metal deposition through the formation is accomplished and to greater depths. The metal deposition on the surfaces to be consolidated is most effective when the pH of the solution is about 3 or 4.5 and the temperature is in the range of from about 150° F. to about 200° F.

The acidic metal plating compound can be a polyvalent metal compound of which preferred compounds include nickel, cobalt and copper compounds and mixtures thereof, e.g., nickel and/or cobalt chloride and/or sulfate. These metal compounds are reduced by such reagents as hypophosphorous acid, hypophosphites, e.g., sodium hypophosphite or alkaline solution of molybdenate, formate and/or hydroxy carboxylates, e.g., hydroxyacetate. The concentration of the metal-containing compounds and the reducing agents in aqueous solutions can be varied over a wide range such as from 1 to 50%, respectively, and preferably from 5 to 40% each.

To keep the hydrogen evolution to a minimum during the reaction, the reducing agents in the metal-plating solutions should be kept at a minimum, generally not in excess of 10% of the total electroless metal plating solution. Also, hydrogen evolution can be effectively suppressed and the life of the metal-plating reaction increased by addition to such aqueous solutions buffering and chelating agents such as hydroxy carboxylic acids and polycarboxylic acids and their salts, e.g., citric, tartaric, maleic, gluconic and succinic acids or ammonium or alkali metal salts of said acids such as sodium citrate, sodium succinate and the like. However, the gas can be effectively eliminated from the area being metalized by applying pressure of 200 pounds or more on the system.

To promote wetting of the surfaces to be metalized by the electroless process of the present invention, wetting agents can be used such as reaction products of alkyl-phenol and alkylene oxide, e.g., nonyl phenoethylene oxide reaction product wherein the number of ethylene oxide units in the molecule ranges from 4 to 20; sulfated alcohols, sulfonate of fatty acids having from 12 to 18 carbon atoms, e.g., sulfonated oleic acid, sulfonated mineral oil fractions and the like.

Also, when using hydrophosphates as the reducing agent their concentration should be controlled since depending in part on the phosphorus content of the solution the metal being plated can be in the form of an alloy of metal-phosphorus nickel phosphide. High concentrations such as above 10% of hypophosphite in the metal plating solution tend to form these alloys.

The acidic metal-plating consolidation process of the present invention can be also used to improve earth formations which have been previously consolidated by various resins or plastics such as epoxy resins or various other types by forming on the resin coated surface a metal coating that renders the consolidated formation resistant to hydrolysis at elevated temperatures, such as those encountered when hot water and/or steam is injected into such systems for secondary recovery of hydrocarbon fluids such as petroleum oil. The metalization of resin or plastic consolidated formations is effectively accomplished by the process of the present invention. This is particularly desirable in cases where resins used to consolidate formations are thermally stable but are hydrolytically unstable and tend to disintegrate on prolonged exposure to steam or hot water. The same applies to formations consolidated with quartz or other types of consolidators.

To illustrate the use of metal plating in sand consolidation the following illustrative examples are set forth.

EXAMPLE I

Fine sand, Delta Block 24 Field, Louisiana—
about 7,000 feet

Conditions

- (1) Formation temperature, 160° F.
- (2) Absolute pressure, 5,000 p.s.i.
- (3) Injection rate during plating, ¼ bbl./min. per perforated well bore foot (1.3 pore volumes per minute)
- (4) Time of plating, 60 minutes
- (5) N₄ sands S/L 998 No. 30 (16 perforations—4 per foot)

Procedure

- (1) Remove all debris and other fine material from the perforations by injecting about 500 gallons of mud acid (12% HCl, 3% NH₄F, .01% Inhibitor A-109 at rate of 1 bbl./min.) (spot heavy brine solution below perforation to avoid loss of solutions down rat hole).
- (2) Remove crude oil and water by flushing formation with about 8 barrels of diesel oil and about 8 barrels of anhydrous isopropyl alcohol.
- (3) Activate with about 15 barrels of the following solution:

- (a) 15 barrels filtered drinking water (Venice, La.)
- (b) 0.634 pounds gum arabic
- (c) 3.836 liters hydrazine hydrate (85%)
- (d) A solution composed of:
 - 153 grams PdCl₂
 - 989 cc. 30% HCl (reagent grade)
 - 9 liters water
- (e) 12 liters glacial acetic acid or sufficient to adjust pH to 4.2
- (f) 53 pounds NiSO₄·6H₂O
- (4) Use as spacer about 2 barrels of following solution:
 - (a) 2 barrels filtered drinking water
 - (b) 12 gallons activator solution (3) as described above
 - (c) Adjust pH to 4.2 with glacial acetic acid
 - (d) 7 pounds NiSO₄·6H₂O
- (5) Consolidate with about 60 barrels of the following:
 - (a) 1,613 gallons filtered drinking water
 - (b) 1,770 pounds NiSO₄·6H₂O
 - (c) 2,520 pounds NaH₂PO₂·H₂O
 - (d) 1,890 pounds Na acetate·3H₂O
 - (e) 630 pounds Na succinate·6H₂O
 - (f) 504 gallons glacial acetic acid
- (6) Displace spent plating solution in formation with 5 barrels of 5% NaCl solution.

Results

Well produced sand free for about 4 months and well productivity after consolidation was much higher than the field average.

EXAMPLE II

Coarse sand, Delta Block 24 Field, Louisiana—
about 8,000 feet

Conditions

- (1) Formation temperature, 170° F.
- (2) Absolute pressure, 5,000 p.s.i.
- (3) Injection rate during plating, 0.32 pore volumes per minute
- (4) Time of plating, 235 minutes
- (5) O₂ Sands S/L 998 No. 30D (8 perforations—4 per foot)

Procedure

- (1) Same as Example I, step (1).
- (2) Same as Example I, step (2).
- (3) Same as Example I, step (3).
- (4) Consolidation is achieved by flowing approximately 75 pore volumes of the following solution through the activated sand:
 - (a) 84 grams/l. NiSO₄·6H₂O

- (b) 120 grams/l. $\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$
 (c) 30 grams/l. Na succinate $\cdot 6\text{H}_2\text{O}$
 (d) 45 grams/l. Na formate
 (e) 53 cc./l. 90% formic acid
 (f) 827 cc./l. water

Results

Well produced sand free and well productivity after consolidation was much higher than the field average.

Other useful electroless basic metal plating solutions are shown in Table 1.

TABLE 1.—PLATING SOLUTIONS

Component	III	IV	V	VI	VII	VIII	VIIIa	IX	X	XI	XII	XIII	XIV	XV
H_2O , cc./l. (gal./bbl.)	878 (36.9)	690 (28.6)	878	800	680	775	790	640	660	740	725	838	855	800
$\text{NiSO}_4 \cdot 6\text{H}_2\text{O}$, grams/l. (lb./bbl.)	42 (14.7)	42 (14.7)	42	42	42	42	42	84	84	84	84	84	84	84
$\text{NaH}_2\text{PO}_2 \cdot \text{H}_2\text{O}$, grams/l. (lb./bbl.)	68 (13.8)		68			50	68	120	120	120	120	120	120	120
Na Citrate $\cdot 2\text{H}_2\text{O}$, grams/l. (lb./bbl.)						20								
Na Acetate $\cdot 3\text{H}_2\text{O}$, grams/l. (lb./bbl.)	60 (21.0)		60	60	60	60	60	90	90					
Na formate, grams/l.												45	45	
Na acetate, grams/cc.		60 (21.0)												
Na succinate $\cdot 6\text{H}_2\text{O}$, grams/l.								15	30	30	30	30	30	30
Na glycolate, 98%, grams/l.									30					
NaCH_3 , grams/l.										26.4	26.4			
Glacial acetic acid, cc./l. (gal./bbl.)	37.5 (1.58)	240 (10.1)	37.5	120	120	150	120	200	175					18
100% formic acid, cc./l.										143				
90% formic acid, cc./l.											159	50	10	
pH	4.35	3.52	4.35	3.82	3.52	3.9		4.0	4.0	2.9	2.9	3.4	4.3	5.0

Acidic nickel systems using some of the compositions were subjected to the Pipe Nipple Test to determine the effectiveness of these compositions to consolidate a West Lake Verret Field (WLVB), La., formation and its compressive strength in p.s.i. after consolidation are noted in Table 2 and the Pipe Nipple Test procedure is as follows:

Prepare the sandpack by clamping the outlet cap in a vise with the pipe nipple threads up. Center circular sleeve and disc, then screw pipe nipple in tight, using Teflon thread tape to prevent leaks. Add the fine screen (stainless steel) and push the O-ring through the pipe nipple. If sample is coarse, uniform sand without fines, a little at a time is added with taping and bouncing of the assembly to pack the sand tightly. If the sample is a mixture of coarse and fine sand or clay, or if the sample is moist, a little at a time is added and tamped thor-

If the sand is oil-free and preferentially water-wet, water may be pumped through the pack to remove any air present. If the sand is nonwater-wet or if oil is present, this must be washed out. To accomplish this, flush with 4 or 5 PV each of isopropyl alcohol, xylene, isopropyl alcohol, respectively. The final displacement of isopropyl alcohol with H_2O will provide an oil-free pack. These washings will be facilitated if all air is first removed by flowing liquid through the pack at atmospheric pressure and at 600 p.s.i. alternatively.

After all air and oil are removed, reduce the outlet pres-

sure to 1 atmosphere and measure the pressure drop across the sandpack for some convenient water flow rate. The pressure drop will be used to measure any changes in the fluid conductivity of the pack during subsequent operations.

The consolidation test experiment is carried out by passing the various activating and plating solutions through the sandpack under the desired conditions. Finally, the pack is flushed with water, which is used to remove all gas as described above. Outlet pressure is reduced to 1 atmosphere and the inlet pressure measured as before. The difference in the initial pressures (corrected for any differences in flow rate) and the final pressure is used to calculate the loss of permeability in the sandpack. The sandpack is then removed from the bath and opened for inspection and evaluation of the consolidation.

TABLE 2.—PIPE NIPPLE TEST

[Condition: 0.02% Triton X-100 added to plating solution (for laboratory tests only)]

Run No.	Sand	Composition	Type ¹	pH	T, ° F.	t, min	F, PV/ min	Comp. Str. $\times 10^{-3}$, p.s.i.		Fractional loss of permeability
								Inlet ²	Outlet ³	
2	No. 5	XI	Acetic-Succ.	5.0	130	84	0.52	2.0	3.8	0.21
3	No. 5	XIV	Form (90)-Succ.	4.3	162	80	0.74	2.0	4.4	
4	No. 5	XIII	Form (90)-Succ.	3.3	180	82	0.64	1.8	2.8	
5	No. 5	XII	Form (90)-Succ.	2.8	200	88	0.85	4.0	2.2	
6	No. 5	XI	Form (100)-Succ.	2.9	180	89	0.45	0.5	1.3	
10	No. 5	I	Acetic-Succ.	4.0	180	60	1.22	8.0	2.0	
11	No. 5	I	Acetic-Succ.	4.0	180	61	1.23	4.0	4.0	
15	1.1	X	Acetic-Gly	4.0	144	37	2.60	5.0	1.9	0.0
17	No. 5	VIIIa	$\frac{1}{2}$ Acetic-Succ.	4.0	142	53	2.5	Good		0.45
18	No. 5	VIII	Cit.-Succ.	3.8	144	42.5	1.88	16.0	12.0	0.77
19	Sidewall samples	I	Acetic-Succ.	4.0	142	65	1.13	Good		0.69
21	Acid washed	Ia	do.	4.0	150	167	0.68	Good		

¹ Cit.=citrate; Form (90)=90% formic acid; Form. (100)=100% formic acid; Gly.=glycolate; Succ.=succinate.

² Not extrapolated to inlet face, represents approximate middle of consolidation.

³ Middle of outlet half of consolidation.

⁴ Poor depletion.

⁵ 2 mud acid treatments (8 PV) before plating.

oughly with a flat rubber stopper to avoid segregation. When the pipe nipple is filled to the proper height, add the inlet fine screen and push the O-ring down tight but not tight enough to bow the screen up in the center of the pipe. Then add 20–40 mesh Ottawa sand to fill the volume enclosed by the O-ring. Position the coarse screen and disc. Place the spring in position so that when the inlet cap is screwed on tight (with Teflon thread tape) the spring is compressed just enough to keep the sandpack tight. The inlet volume of the tubing and holder from the pump to the sand inlet face should be about 80 cc. for a suitable system.

The sandpack is connected to the inlet and outlet tubing and immersed in the bath at the desired temperature.

A number of consolidated sand samples have been exposed to flowing hot water in the 400° to 600° F. range. The results are given in Table 3.

Table 3.—Hot water resistance

65	Conditions: (1) Nickel consolidation, (2) No. 5 sand
	Consolidation on control sample: Composition I
	T, ° F. ----- 180
	Comp. Str., ¹ p.s.i. ----- 4,000
70	After hot water exposure of test sample:
	Time (days) ----- 11
	T, ° F. ----- 575
	Comp. Str. ² p.s.i. ----- 4,000

¹ Approximate middle of consolidation.

² No visible degradation of the deposit or loss of sand.

Notable features of the present invention are the control of the reaction rate of the electroless metal process so that greater depths of consolidation are achieved and also the metal plating process aids in reducing corrosion and heat loss of tubing strings used in the bore wells for when injecting the binding and activating fluids into the underground production areas the tubing strings are metalized by the process of the present invention. As these fluids are injected into the loose formations the tubing strings are also metalized with such materials as nickel or cobalt or nickel phosphide or cobalt phosphide or nickel-iron protective metal coatings, as well as other parts of the equipment and apparatus with which said metalizing fluids come in contact.

I claim as my invention:

1. A method of consolidating at a controlled reaction rate an incompetent formation penetrated by a well, said formation having a temperature of greater than about 125° F. comprising:

- (a) injecting through the well and into the formation an acidic aqueous solution containing an activating agent capable of activating the surface of the incompetent formation;
- (b) injecting through the well and into said formation an acidic aqueous solution containing a metal-plating compound and a reducing agent; and
- (c) injecting enough of said acidic metal-plating containing solution to displace said activating agent solution and flow a plurality of pore volumes of the acidic metal-plating compound solution through the interval of said formation into which the well is opened to achieve consolidation of the formation.

2. The method of claim 1 wherein the activating agent is a compound selected from the group consisting of palladium, platinum, gold, ruthenium, rhodium and stannous chloride, bromide, nitrate and sulfate and mixtures thereof, the metal-plating compound is selected from the group consisting of nickel, cobalt and copper compounds and mixtures thereof and the reducing agent is selected from the group consisting of hypophosphorus acid, alkali metal hypophosphite, alkali metal molybdenate, a lower aldehyde and mixtures thereof and the metal plating solution is made acidic by the presence of a lower acid selected from the group consisting of formic acid, acetic acid and mixtures of said acids and their alkali metal salts.

3. The method of claim 1 wherein the acidic metal plating is accomplished within an incompetent formation having a temperature between 125° F. and 200° F.

4. The method of claim 1 wherein prior to injecting the activating and metal plating solutions, a solvent or

acidizing solution or mixtures thereof are injected into the incompetent formation to sweep oil and connate water therefrom.

5. The method of claim 1 wherein the pH of each activating solution (a) and metal-plating solution (b) is between 3 and 5.

6. A method of consolidating an incompetent formation having reservoir temperatures above 125° F. and penetrated by a borehole of a well comprising:

- (a) injecting through a tubing string of the well and into the incompetent formation an acidic aqueous solution containing a small amount of palladium chloride and a reducing agent;
- (b) injecting through a tubing string of the well an acidic aqueous solution of nickel chloride and sodium hypophosphite at a temperature of between 125° F. and 200° F.; and
- (c) injecting enough of said nickel plating compound solution to displace said activating agent solution and flow a plurality of pore volumes of the nickel-plating compound solution through the interval of said formation into which the well is opened to achieve consolidation.

7. The method of claim 6 wherein the nickel chloride solution is made acidic by addition of acetic acid.

8. The method of claim 6 wherein the nickel chloride solution is made acidic by addition of formic acid.

9. The method of claim 6 wherein prior to injecting solutions (a) and (b) into the incompetent formation, an acidizing fluid and solvent are injected into the formation.

10. The method of claim 9 wherein the solutions (a) and (b) are separated by a spacer fluid.

11. The method of claim 6 wherein the pH of each activating solution (a) and metal-plating solution (b) is between 3 and 5.

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U.S. Cl. X.R.

166—300; 117—54