

Aug. 3, 1965

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3,198,252

PROCESS FOR OVERCOMING LOST CIRCULATION

Filed March 13, 1962

2 Sheets-Sheet 1

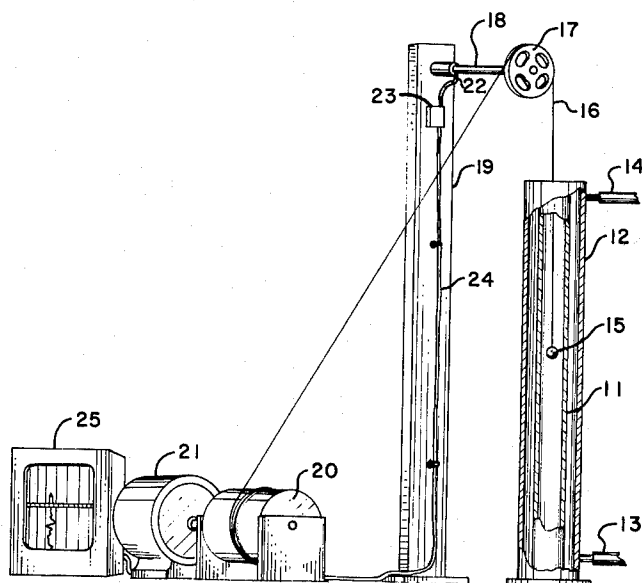


FIG. 1

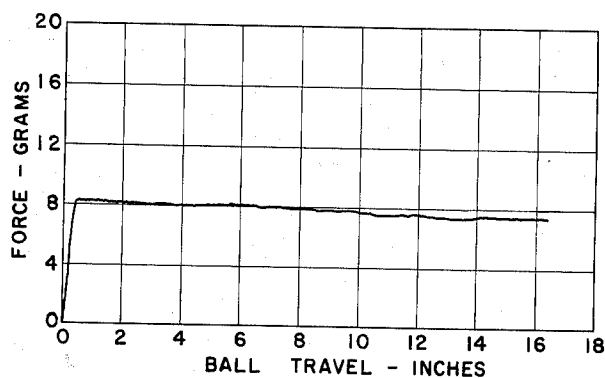


FIG. 2

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2 Sheets-Sheet 2

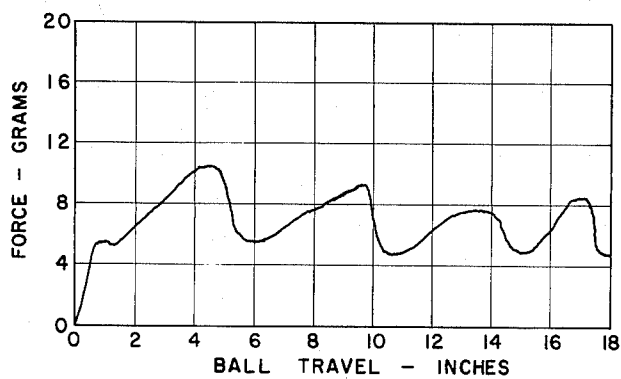


FIG. 3

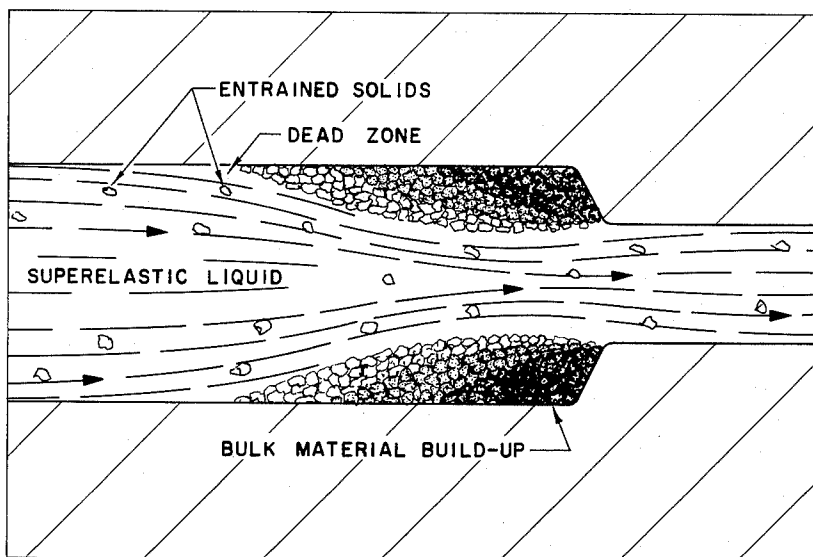


FIG. 4

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PROCESS FOR OVERCOMING LOST CIRCULATION
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8 Claims. (Cl. 166—32)

The present invention is concerned with lost circulation problems encountered in oil wells, gas wells, and similar boreholes and is more particularly concerned with an improved process for closing off lost circulation zones surrounding such boreholes in order to prevent the loss of fluids contained therein. In still greater particularity, the invention is concerned with a process for overcoming lost circulation wherein an organic liquid having "superelastic" properties is injected into a borehole with entrained solids in order to plug vugs and fissures too large to be plugged by conventional means.

It is conventional to employ clays, gums and related materials in drilling muds and similar fluids utilized in the oil and gas industry. Such materials are intended to form a filter cake on the borehole wall and prevent fluid losses to the surrounding porous strata during drilling, completion and workover operations. Although these materials are effective in most instances, there are many cases where fluid losses occur despite their use. Certain limestones and similar formations often contain vugs and fissures which are much too large to be plugged with conventional fluid loss additives. Other formations are easily fractured by pressure in the borehole and may develop fissures of sufficient size to permit the rapid escape of fluid containing clay, gums and the like. Formations which contain such vugs and fissures, whether of natural origin or induced by excessive pressure, are referred to as lost circulation zones because the escape of fluid into the formation normally precludes the maintenance of normal fluid circulation in the borehole. The interruption of normal circulation prevents the entrainment of cuttings and other materials from the borehole, leads to reductions in hydrostatic pressure which may be followed by the influx of high pressure connate fluids, may result in the flooding of producing zones with mud or the like, and may eventually cause the drill string to become stuck in the borehole. Even if circulation is not lost completely and some fluid can be returned to the surface, the fluid flowing into the formation must be replaced continuously and hence the cost of continued operation may become prohibitive. These and other difficulties make it desirable, and in most cases imperative, that lost circulation zones and similar highly porous strata be plugged as rapidly as possible if operations requiring fluid circulation or the maintenance of hydrostatic pressure in the borehole are to be continued.

Lost circulation zones and similar highly porous strata are normally plugged by adding a fibrous, flaky or granular solid to the drilling mud or other fluid and pumping the resulting mixture into the borehole until a bridge or mat forms over the vugs or fissures through which the fluid tends to escape. Plugging materials which have been used for this purpose include hay, excelsior, wood shavings, rice, rubber pulp, cotton, cottonseed hulls, rockwool, granular plastics, mica flakes, feathers, sponges, leather strips and a variety of other materials. The use of such materials frequently permits the plugging of lost circulation zones but success of this method is by no means certain. Despite the pumping of large volumes of fluid containing such solids into the borehole, a bridge or mat may never form over the vugs or fissures. Moreover, the

introduction of mud or similar fluid containing solids in the large quantities required may lead to pressure surges which cause fracturing and result in additional fissures through which fluids can escape.

In lieu of using a solids-laden mud or similar fluid as described above, cement may be pumped into the borehole and allowed to harden in order to plug lost circulation zones. Fibrous, flaky or granular solids may be incorporated into the cement in order to reduce the quantity which escapes into the formation before it sets. Several batches of cement are usually required. Each batch must be pumped into place, allowed to harden, and tested before the next can be placed and hence several days may be consumed in plugging a single zone. The additional time required to drill through the cement after circulation has been restored necessitates a further delay. The cost of plugging a lost circulation zone in this manner is usually high. Expenditures in excess of one hundred thousand dollars for plugging lost circulation zones in a single well are not unusual.

It is therefore an object of the present invention to provide an improved process for plugging lost circulation zones during the drilling, completion and workover of oil wells, gas wells and similar boreholes. A further object is to provide a process which will permit the rapid plugging of lost circulation zones with a small quantity of material. Other objects will become apparent as the invention is described in greater detail hereafter.

In accordance with the invention, it has now been found that vugs and fissures in strata surrounding a well borehole can readily be plugged by injecting solid particles entrained in an organic liquid having "superelastic" properties. Studies have shown that such a liquid will deposit the entrained solids as it passes through vugs and fissures and that openings many times the size of the largest particles used can be quickly plugged as a result of such deposition. The injection of solid particles entrained in organic liquids having "superelastic" properties often permits the restoration of circulation where methods and materials employed in the past are completely unsuccessful. The use of solids entrained in such liquids minimizes the time required for plugging, reduces the quantity of material which might be pumped into the borehole, obviates the necessity of repeated drilling out of cement plugs, avoids fracturing difficulties frequently encountered with solids-laden muds, and permits the restoration of circulation at low cost. These and other advantages make the process of the invention attractive in connection with a variety of oil field operations.

The nature and objects of the invention can be more fully understood by referring to the following detailed description of "superelastic" liquids and their use with entrained solids for controlling lost circulation and to the accompanying drawing, in which:

FIGURE 1 depicts apparatus useful for measuring the "superelastic" properties of organic liquids suitable for carrying out the invention;

FIGURE 2 is a plot illustrating properties of a typical drilling fluid when tested in the apparatus shown in FIGURE 1;

FIGURE 3 is a plot showing the properties of a "superelastic" liquid when tested in the apparatus of FIGURE 1; and

FIGURE 4 is a diagram showing the manner in which solids are deposited by a superelastic liquid as it flows through vugs and fissures in subsurface strata.

The "superelastic" liquids employed for the entrainment of solid particles in carrying out the invention are organic liquids which exhibit elasticity and tensile strength, as well as density and viscosity, and therefore fall into the broad classification of viscoelastic materials. Most liquids which are so classified have extremely low tensile strength

and hence their viscoelastic properties can generally be ignored so far as practical applications of the liquids are concerned. Certain liquids, however, are characterized by high elasticity and pronounced tensile strength and as a result exhibit unique properties during laminar flow. These liquids are referred to herein as "superelastic" liquids.

A "superelastic" liquid can be defined for purposes of the invention as a viscoelastic fluid which satisfies the following requirements at the temperature at which it is to be used:

(1) The liquid must have a recoverable shear value of at least 2 recoverable shear units.

(2) It must have a mending time less than about 30 seconds.

(3) It must have a positive elastic drag coefficient of at least 1.2 dynes per square centimeter per unit of shear as measured over a distance of at least 7 shear units with the apparatus shown in FIGURE 1 of the drawing or with equivalent apparatus at a shear rate in the range between about 0.5 and about 50 reciprocal seconds.

The recoverable shear of a liquid is a term indicating in non-dimensional shear units the extent to which the liquid will "recoil" when a deforming force acting on it is instantaneously removed and the potential energy stored in the liquid in response to the force is released as kinetic energy. Several methods for measuring the recoverable shear of viscoelastic liquids have been developed in recent years. One such method is based upon use of the Weissenberg Rheogonimeter to measure the normal force acting on the liquid and calculation of the recoverable shear from the relationship $S = \tau \cdot s$, where S is the normal stress, τ is the applied shear stress, and s is recoverable shear. Other methods are based upon flow birefringence measurements, capillary entrance effect determinations, direct recoil measurements, and determination of the extent to which the viscoelastic jet effect occurs. These methods have all been described in the rheological literature and will therefore be familiar to those skilled in the art. Tests have shown that liquids useful for purposes of the invention must have recoverable shear values in excess of 2 units. Those having lower values do not give the required flow behavior and are not considered to be superelastic liquids.

In addition to having recoverable shear values in excess of 2 shear units, superelastic liquids are characterized by mending times less than 30 seconds at the temperatures at which they are employed. The mending time is the period during which 2 samples of the material must be held in intimate contact with one another before they will rupture along a new surface when pulled apart. It is an accepted rheological term.

The third requirement for a superelastic liquid suitable for purposes of the invention is that it have a positive elastic drag coefficient in excess of about 1.2 dynes per square centimeter per unit of shear when measured over a distance equivalent to at least 7 shear units in apparatus of the type shown in FIGURE 1 of the drawing or in equivalent apparatus. The elastic drag of a liquid may be defined as the drag force in excess of viscous drag which is exerted by the liquid on a body moved within it at constant velocity. The elastic drag coefficient is an indication of the rate of change in the elastic drag force as the body is moved and can be calculated by dividing the difference between the drag force exerted on the body at two different points within the liquid by the distance between the points. Most viscometers do not permit the measurement of elastic drag. An instrument which has been developed specifically for testing superelastic liquids and determining their elastic drag is shown in FIGURE 1 of the drawing.

The ball test device depicted in FIGURE 1 includes an elongated cylinder 11 of glass or similar material provided with an outer jacket 12 having inlet 13 and an outlet 14 through which water or other fluid may be circulated

from a thermostatically controlled vessel in order to maintain a liquid within the cylinder at an essentially constant temperature. A steel ball 15 is suspended within the cylinder at the end of a line 16. The line passes over pulley 17, supported at the outer end of the cantilever arm 18 on stanchion 19, and is wound on drum 20. The pulley is preferably mounted on a ball bearing in order to minimize friction. An electric motor 21 which can be operated at constant speed and is connected to the drum shaft through suitable gears not shown permits rotation of the drum. The ball is pulled upwardly through the column of liquid in the cylinder as the drum rotates. A strain gauge 22 is mounted on the cantilever arm to detect deflection of the arm and provide an indication of the force required to raise the ball through the liquid. The strain gauge signal, obtained from a standard strain gauge measuring circuit 23, is fed through leads 24 to recorder 25. The recorder chart moves at a rate proportional to the drum speed and hence the recorded information represents a plot of the force required to raise the ball through the liquid versus the distance through which the ball travels within the liquid.

FIGURES 2 and 3 in the drawing are plots of the results obtained with a typical nonelastic liquid and a typical superelastic liquid in apparatus of the type described above, utilizing a half-inch diameter ball and a 20 inch cylinder having an inside diameter of 37 millimeters. For this particular apparatus it can be shown that shear rate in reciprocal seconds is 0.0424 times the ball velocity in centimeters per minute. The drag is measured in grams. The nonelastic liquid used to obtain the curve shown in FIGURE 2 of the drawing was an aqueous solution containing bentonite clay in a concentration of 44 pounds per barrel. The superelastic liquid on which the curve of FIGURE 3 is based was an organic liquid prepared by dissolving a commercial aluminum soap in kerosene in a concentration of 14 pounds per barrel and allowing the aluminum soap molecules to form soap micelles in solution. The tests were carried out at a temperature of 77° F. The ball was raised through the liquid in the apparatus at a rate of 122 centimeters per minute in each test.

The curve shown in FIGURE 2 demonstrates that, except for effects due to the inertia of the ball, the gel strength of the bentonite solution at the beginning of the run, and emergence of the ball at the end of the run, the force required to move the ball through the bentonite solution decreased continuously. The solution therefore had a negative elastic drag coefficient. Such behavior is typical of nonelastic liquids. The drag coefficient of such liquids is always either zero or negative, never positive. Where a negative coefficient is obtained, the magnitude will depend upon the viscosity of the liquid, the gel strength of the liquid, the ball velocity, and the ball and tube diameters in the test apparatus.

It will be noted from FIGURE 3 of the drawing that the force required to raise the ball through the superelastic liquid was quite different from that required in the earlier test. The elasticity and tensile strength of the modified kerosene resulted in a marked increase in the force necessary to move the ball after the effect of inertia had been overcome. This increase continued until a "rupture" point was reached. The initial portion of the curve therefore shows a positive elastic drag coefficient which cannot be attributed to ball inertia or liquid gel strength. Rupture occurred at the point where the drag on the ball exceeded the tensile strength of the superelastic liquid. Rupture of the bonds within the liquid eliminated the drag component due to elasticity and tensile strength and hence the force necessary for ball movement decreased following rupture. Under the test conditions employed, mending of the bonds occurred rapidly as the ball continued to move. The resulting elastic drag again caused an increase in rate in the force required for ball movement. Again the curve shows a positive drag coefficient. If a line is drawn through the

low points on the curve, where the ball is affected only by viscous drag, such a line will show a zero or negative drag coefficient similar to that obtained with a non-elastic liquid. This repeated increase in the force required to move the ball through the liquid is typical of superelastic liquids and provides a convenient means for readily identifying such liquids.

As was pointed out earlier, the elastic drag coefficient of viscoelastic liquids tested in apparatus such as that of FIGURE 1 of the drawing represents the slope of the force-distance curve. Tests have shown that superelastic liquids suitable for purposes of the invention must have positive drag coefficients in excess of about 1.2 dynes per square centimeter per unit of shear when measured in apparatus of the type shown in FIGURE 1 over a distance equivalent to at least 7 shear units. In the particular apparatus employed in obtaining the curves in FIGURES 2 and 3 this value corresponds to a drag coefficient of at least 0.04 gram per centimeter. The modified kerosene employed in obtaining the curve shown in FIGURE 3 gave a positive coefficient of about 0.67 gram per centimeter at a shear rate of 5.2 reciprocal seconds. The bentonite solution with which the curve in FIGURE 2 was obtained, on the other hand, gave a negative drag coefficient and was therefore not a superelastic liquid.

The results obtained when superelastic liquids are tested in apparatus of the type shown in FIGURE 1 of the drawing depend to some extent upon the velocity with which the ball is moved through the liquid and the dimensions of the ball and cylinder employed. If the ball velocity is too low, the shear rate may be insufficient to show the elastic properties of the liquid. An increase in the width of the gap between the ball and cylinder has the same effect as the reduction of ball velocity. If the ball velocity is too high or the gap between the ball and cylinder is too small, the elastic properties of the liquid may not be readily discernible. In like manner, the use of a very short cylinder may not permit sufficient ball movement for rupture to occur. In all cases, however, a superelastic liquid will give a positive drag coefficient in excess of about 1.2 dynes per square centimeter per unit shear at some shear rate between about 0.5 and about 50 reciprocal seconds when tested in such equipment over a distance equivalent to at least 7 shear units.

Measurements made with apparatus of the type shown in FIGURE 1 of the drawing can in first approximation be converted to absolute rheological units compatible with measurements made in capillary or rotating coaxial cylinder viscometers by means of Stokes law corrected for finite cylinder diameter valid for Newtonian liquids. This relationship is expressed by the following equation:

$$\mu = 3120 \cdot \frac{F}{a} \cdot \frac{F}{V} = \frac{\tau}{D}$$

where μ is the viscosity of the liquid in poises; F is the drag of the ball in grams; a is the ball radius in centimeters; τ is the shear stress in dynes per square centimeter; D is the shear rate in reciprocal seconds;

$$f = 1 - 2.104 \frac{a}{b} + 2.09 \left(\frac{a}{b} \right)^3 - 0.95 \left(\frac{a}{b} \right)^5 - \frac{1}{16} R_e$$

b is the radius of the cylinder in centimeters;

$$R_e = \frac{2a \cdot d \cdot V}{60\mu}$$

and d is the liquid density in grams per cubic centimeter.

The value of F to be used in the above equation should be derived from a plot of force versus ball travel obtained with apparatus of the type shown in FIGURE 1 by extrapolating the curve to the point where the ball emerges from the liquid in order to nullify end effects. It has been shown that these equations are accurate within 1 percent up to an a/b ratio of 0.32 and hold for apparatus such as that depicted in FIGURE 1 up to an a/b ratio of 0.5.

It has also been shown experimentally that the shear rate in first approximation in apparatus of the type shown above is given by the equation:

$$D = \frac{\pi \cdot V}{60(b-a)}$$

where D is the shear rate in reciprocal seconds, V is the ball velocity in centimeters per minute, b is the cylinder radius in centimeters and a is the ball radius in centimeters. This expression holds for values of a between 0.635 and 1.805 centimeters and for value of $(b-a)$ between 0.33 and 1.87 centimeters with an average error of 3 percent. Substitution of this equation into the expression given earlier permits the derivation of equation for the shear stress, τ , in dynes per square centimeter.

In like manner it can be shown that the number of shear units in a system such as that shown in FIGURE 1 is given by the expression

$$S = \frac{\pi \cdot x}{(b-a)}$$

where S is shear units, x is ball travel in centimeters, b is the radius of the cylinder in centimeters, and a is the radius of the ball in centimeters. The value of S obtained from the equation is dimensionless.

Application of the equations above will readily permit the testing of any viscoelastic liquid in apparatus of the type shown in FIGURE 1 in order to determine whether the liquid has superelastic properties and will simplify interpretation of the results of the tests. The equations also permit comparison of the results obtained in the ball test with those obtained in other types of rheological equipment.

The high elasticity and tensile strength of superelastic liquids which permit their use for purposes of the invention are apparently due to the presence of bonds between the particles which are able to resist considerable deformation but can be ruptured under shear stresses and will rapidly be reformed when the stresses are alleviated. Such liquids resemble gels in some respects but can readily be distinguished from true gels by the fact that they have no yield point, have no angle of repose, and will mend rapidly, generally within a few seconds. Superelastic liquids can readily be prepared by dissolving aluminum soaps and related compounds in hydrocarbon solvents to produce the required state or by dissolving high molecular weight polymers in organic solvents. Examples of liquids which have the required superelastic properties under certain conditions include those prepared by the solution of hydroxy aluminum soaps in gasoline, kerosene, and similar hydrocarbons, those obtained by adding viscous nitrocellulose or similar material to butyl acetate or a related solvent, and those produced by dissolving natural rubber in toluene or the like.

A preferred class of superelastic liquids particularly effective for purposes of the invention are those derived by the solution of hydroxy aluminum soaps of organic acids in naphtha, gasoline, kerosene, gas-oil, crude-oil and the like. Such liquids are generally prepared with mixed hydroxy aluminum soaps of fatty and naphthenic acids. Fatty acid soaps which may be employed include mono- and dihydroxy aluminum soaps of fatty acids containing from about 12 to about 24 carbon atoms per molecule. Typical of such soaps are those derived from lauric acid, myristic acid, palmitic acid, oleic acid, linoleic acid, stearic acid, arachidic acid, behenic acid, and mixed acids derived from naturally-occurring materials such as coconut oil, tallow fat, cottonseed oil, soybean oil and the like. Naphthenic acid soaps which may be utilized include aluminum hydroxy and dihydroxy soaps of hexahydrobenzoic acid and substituted hexahydrobenzoic acid. The use of mixed soaps containing both a fatty acid derivative and a naphthenic acid derivative generally results in more stable superelastic liquids and hence such soaps are preferred for purposes of the invention. The use of aluminum naphthenate and aluminum coconut oil acid soaps

is particularly effective. In lieu of employing mixed aluminum soaps, it is sometimes advantageous to utilize an aluminum naphthenate or similar soap and a free fatty acid.

The aluminum soaps or related compounds can be used with naphtha, gasoline, kerosene, gas-oil or similar liquid hydrocarbons by first adding an aluminum naphthenate, oleate or similar soap to the hydrocarbon and agitating it until all of the soap has been dissolved. The soap will normally be added in a concentration of about 1 to about 10 percent by weight, the exact amount utilized depending upon the particular liquid and soap employed. From about 1 to about 10 weight percent of an aluminum coconut acid soap or a mixture of free fatty acids can then be added with agitation in order to effect formation of the superelastic liquid. The concentration required will again depend upon the material selected. It is generally necessary to age the liquid containing the soap for a period ranging from a few minutes to 24 hours or longer in order to develop the requisite superelastic properties. In lieu of this procedure, the soaps can be dissolved in separate portions of kerosene, or a similar hydrocarbon liquid, which can later be combined to effect the formation of a superelastic liquid. Still another procedure is to mill the soaps together to produce a mixture which can be incorporated in hydrocarbons to produce superelasticity. Regardless of the procedure utilized, it is usually necessary to age the materials for a considerable period in order to produce a liquid having the superelastic properties desired.

A variety of materials containing aluminum and similar metallic soaps are marketed commercially for use in thickening liquids hydrocarbons in the manufacture of greases and lubricants and are suitable for the preparation of superelastic liquids in accordance with the invention. One such material is an aluminum stearate manufactured by the Witco Chemical Company of New York and marketed under the trade name "Witco 171S." Similar products are listed in the sales literature of other manufacturers and will therefore be familiar to those skilled in the art. The use of such commercial materials generally facilitates the preparation of superelastic liquids in the field and is therefore preferred.

It will be understood that all liquid hydrocarbons thickened with aluminum soaps and related materials do not possess superelastic properties. The development of such properties depends in part upon the amount of soap or similar material employed in the liquid. If insufficient material is used, a stringy material which is not superelastic will be obtained. If excessive material is used, a gel or precipitate which also lacks superelastic properties will result. The amount required may vary widely depending upon the particular materials employed and the concentrations in which they are used. Tests of the type described earlier should therefore be carried out in equipment such as that shown in FIGURE 1 of the drawing or in equivalent apparatus to insure that the material and concentrations selected for field use will result in a liquid having superelastic properties. Once the concentrations and mixing procedure required for a particular system have been established, that system can then be used in the field without further tests.

A superelastic liquid is utilized for overcoming lost circulation problems in accordance with the invention by entraining solid particles in the liquid and pumping the result mixture into the borehole. The particles utilized preferably range between about 100 mesh on the Tyler screen scale and about $\frac{1}{8}$ of an inch in size. The unique flow properties of superelastic liquids result in the deposition of the entrained solids on the upstream side of flow restrictions as the liquids flow through such restrictions. This deposition continues until the flow channel has been completely closed and no further flow takes place. Openings many times the diameter of the largest particles used can thus be plugged. A variety of fibrous, flaky or gran-

ular material may be employed. Suitable materials include sand, sawdust, walnut hulls, shredded cellophane and the like. Particles of irregular shape which will interlock with one another as they accumulate on the walls of the channel through which the liquid flows are preferred. The interlocking improves bridging of the particles by reducing disturbance of the accumulated material as the pressure differential across a partially-formed bridge increases. The use of relatively small particles also reduces disturbance, since such particles project a lesser distance into the flow stream than do larger ones. Coarse sawdust has been found to be a particularly effective solid for use in the superelastic liquid, either alone or in combination with sand or a similar material. The low weight of shredded cellophane per unit of volume and the resultant ease of transporting the quantities required for lost circulation control to remote well sites constitutes a particular advantage for this material. From about 0.5 to about 4.0 pounds of cellophane per barrel of superelastic liquid is usually effective. Sawdust and similar solids may be employed in quantities up to about 20 pounds per barrel. Sand is generally used in amounts up to about 50 pounds per barrel. Mixtures of these and similar materials may be employed in intermediate quantities.

When a lost circulation problem is encountered during the drilling, completion or workover of a well or similar borehole, the superelastic liquid and entrained solids may be pumped into the borehole through the drilling rig pump or by means of an auxiliary pump. Experience has shown that passage of the liquids and solids through the pump does not adversely affect the superelastic properties. Sufficient pump pressure to force the liquid and entrained solids into the vugs and fissures in the strata surrounding the borehole should be used but care should be taken to avoid pressures sufficient to fracture the strata. As pointed out earlier, fracturing creates additional fissures and thus aggravates lost circulation problems. Fracturing pressures generally range from about 0.6 to about 0.9 pound per square inch per foot of borehole depth. The superelastic liquid utilized can be mixed in batches of from 10 to 20 barrels in a small tank or cementing truck and pumped directly through the drill pipe into the lost circulation zone. In some instances it may be desirable to prepare the liquid in advance and store it until needed. Tests have shown that the liquids are generally stable over long periods and can be stored without difficulty. The desirability of preparing the liquid in advance will depend largely upon the superelastic liquid utilized, the accessibility of the borehole site to a ready supply of the material required and the probability that lost circulation problems will be encountered.

The placement of a single plug of about 200 barrels of superelastic liquid containing sawdust, shredded cellophane or similar solids is generally sufficient to overcome most circulation problems. In severe cases, however, it may be necessary to utilize several plugs in succession. Following the recovery of circulation, conventional drilling muds may be pumped into the well or borehole in order to form a filter cake over the plugged lost circulation zone and permit the resumption of normal operations. It is frequently desirable, however, to set a cement plug after circulation has been regained and thus assure a permanent seal over the lost circulation zone. Conventional cementing equipment and techniques may be employed. The initial use of a superelastic liquid containing entrained solids will permit the installation of a cement plug in lost circulation zones where a series of plugs might otherwise have to be injected and allowed to set if the superelastic liquid were not used.

The effectiveness of organic liquids containing soaps and similar agents in sufficient quantities to provide superelastic properties for plugging lost circulation zones with entrained solids can readily be seen by considering the results of laboratory tests carried out in equipment

designed to simulate a well bore surrounded by a vugular formation. The equipment utilized included a vertical Lucite column having an inside diameter of $5\frac{1}{2}$ inches which served as the well bore. A 20 foot length of Lucite tubing representing drill pipe extended through a well head at the top of the column to a point just above the bottom of the column. A conventional mud pump, injection line, and hose were used to circulate fluid into the upper end of the tubing. Vugs were provided at intermediate points in the column by drilling holes and installing horizontal lengths of Lucite tubing. The vugs had a $\frac{3}{4}$ inch inside diameter adjacent the column and tapered at 60° to a $\frac{1}{2}$ inch inside diameter. Each tube representing a vug was connected to a discharge tank through a meter so that the amount of fluid which escaped through the vug could be measured. Examinations of vugular limestones and similar formations indicate that the vugs used in the test apparatus are probably more difficult to plug than the tortuous channels normally found in such formations. A discharge line at the top of the Lucite column was provided in order to permit the circulation of fluid into the well bore through the drill pipe, past the vugs, and out of the annulus. The vugs were lighted to permit close observation of the plugging action of fluids so circulated.

Tests were first carried out with a conventional drilling mud provided by adding bentonite to water in a concentration of 44 pounds per barrel. Measurements of the force required to move a ball through the liquid were carried out in apparatus of the type shown in FIGURE 1 of the drawing. It was established that the bentonite mud did not possess superelastic properties. Solids were then entrained in the mud by stirring in 20 pounds of crushed walnut hulls and 2 pounds of shredded cellophane per barrel. The resulting mixture was then circulated through the apparatus. It was found that the mud containing the entrained solids readily flowed into the vugs and was thus lost from the well bore. Continued circulation evidenced no tendency for the entrained solids to be deposited within the vugs. No plugging occurred even though circulation was continued for several hours. Similar tests were made at other flow rates ranging from about 1 barrel per hour to about 7 barrels per hour, using mud containing solids in concentrations up to about 50 pounds per barrel. No plugging took place in any of these tests.

Following the tests with bentonite mud, a superelastic liquid was prepared with kerosene and a commercial aluminum stearate marketed as a thickening agent for hydrocarbons. The thickening agent was stirred into the kerosene in a concentration of 14 pounds per barrel at a temperature of 80° F. and the solution was allowed to stand. Tests of the liquid carried out at intervals in the ball test apparatus shown in FIGURE 1 of the drawing indicated that the material did not initially possess superelastic properties but that such properties had developed after about 24 hours. The elastic drag coefficient as measured in the ball test apparatus was about 0.67 gram per centimeter at a shear rate of 5.2 reciprocal seconds. Walnut hulls and shredded cellophane were entrained in the liquid in concentrations of 20 pounds per barrel and 2 pounds per barrel respectively. The liquid and entrained solids were then circulated in the Lucite well bore under the same conditions previously used in testing the bentonite mud.

Visual observation of the behavior of the superelastic liquid and solids in the well bore vugs showed that the solids were deposited by the liquid just upstream from the point where the vug diameter decreased from $\frac{3}{4}$ of an inch to $\frac{1}{2}$ of an inch. FIGURE 4 of the drawing is a schematic representation of a vug illustrating this deposition of solids by the liquid. The deposition continued very rapidly until the vugs had been completely sealed off and no further flow took place. Tests were

made with circulation rates in the vugs ranging from about 0.4 to about 24 barrels per hour and in each case complete plugging occurred. The volume of liquid which flowed through each vug prior to plugging ranged from about 0.01 to about 0.1 barrel. No difficulties due to plugging in the circulation system or drill string were encountered.

The results of the above tests demonstrate the unique flow properties of superelastic liquids and the ability of such liquids to rapidly plug vugs and fissures with entrained solid particles. Complete plugging of $\frac{1}{2}$ inch vugs with entrained particles less than $\frac{1}{8}$ an inch in diameter was secured with the loss of from only 0.01 to about 0.1 barrel of liquid per vug. More than twenty barrels of conventional drilling mud containing identical solid particles escaped through each vug and produced no plugging whatsoever. It is thus apparent that use of the superelastic liquids with entrained solids greatly reduces the quantity of material required to plug lost circulation zones and permits the plugging of such zones much more efficiently than has been possible heretofore.

It will be apparent that the process of this invention is not limited to the specific kerosene-aluminum soap solution utilized in the test described above and that any organic liquid containing a soap, polymer or similar material in a concentration sufficient to give the required superelastic properties may be employed. The equipment shown in FIGURE 1 of the drawing or equivalent apparatus permits the testing of viscoelastic liquids to determine whether they possess superelastic properties which will permit their use for purposes of the invention. The rheological relationships set forth facilitate such testing and will permit the preparation of a variety of liquids suitable for purposes of the invention by those skilled in the art.

What is claimed is:

1. A process for overcoming lost circulation in a borehole penetrating subsurface strata which comprises dissolving a high molecular weight polymer in an organic solvent in a concentration sufficient to produce, at the temperature conditions prevailing within the lost circulation zone, a viscous liquid having a recoverable shear rate of at least two recoverable shear units, a mending time less than about 30 seconds, and a positive elastic drag coefficient in the apparatus of FIGURE 1 of the drawing of at least 1.2 dynes per centimeter per unit of shear over at least 7 shear units at a shear rate between about 0.5 and about 50 reciprocal seconds, entraining solid particles in said viscous liquid, and thereafter injecting said viscous liquid containing said solid particles into said borehole and the surrounding lost circulation zone at a pressure below the formation fracturing pressure.

2. A process as defined by claim 1 wherein said additive agent is an aluminum soap.

3. A process as defined by claim 1 wherein said organic liquid is kerosene.

4. A process as defined by claim 1 wherein said solid particles include particles ranging between about 100 mesh and $\frac{1}{8}$ inch in size.

5. A process for overcoming lost circulation in a borehole penetrating subsurface strata which comprises dissolving an aluminum soap in a hydrocarbon liquid in a concentration sufficient to produce, at the temperature conditions prevailing within the lost circulation zone, a viscous liquid having a recoverable shear value of at least two recoverable shear units, a mending time less than about 30 seconds, and a positive elastic drag coefficient in the apparatus of FIGURE 1 of the drawing of at least 1.2 dynes per centimeter per unit of shear over at least 7 shear units at a shear rate between about 0.5 and about 50 reciprocal seconds, entraining solid particles in said viscous liquid, and thereafter injecting said viscous liquid containing said solid particles into said

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borehole and the surrounding lost circulation zone at a pressure below the formation fracturing pressure.

6. A process as defined by claim 5 wherein said aluminum soap is an aluminum stearate.

7. A process as defined by claim 5 wherein said aluminum soap is dissolved in said petroleum fraction in the presence of a free fatty acid.

8. A process as defined by claim 5 wherein said solid particles comprise sawdust.

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