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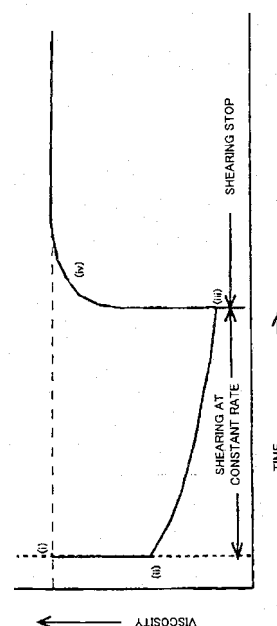
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(54) **LUBRICANT COMPOSITION FOR SEAMLESS STEEL PIPE WORKING**

(57) Provided is a lubricant composition for seamless steel pipes or tubes working which totally satisfies various properties, such as storage stability, transferability inside an apparatus tube, sprayability to lubricated sites, and property of uniform adhesion to a high-temperature mandrel bar without losing non-carburizability or excellent lubricity to hard-to-work materials such as 13 chromium-steel and stainless steel. Prepared is a lubricant composition which has a viscosity property represented by the following approximate expression: $Y = a \cdot X^b$ wherein Y: viscosity (mPa·s), x: shear rate (s^{-1}); and a: 4000 to 40000 and b: -1.0 to -0.3 while the lubricant composition is stored to be kept still, and a: 1000 to 20000, and b: -1.0 to -0.15 after 90 seconds from the time when shearing a lubricant composition is finished.

FIG. 1



Description

Technical Field

5 **[0001]** The present invention relates to a lubricant for hot rolling, which is mainly used for the production of a seamless steel pipe or tube by the Mannesmann process.

Background Art

10 **[0002]** As is well known, in the production of a seamless metal pipe or tube by the Mannesmann process, a billet or bloom heated is made into a hollow blank with piercing mill, and then the hollow blank is finished into a product by means of a continuance mill (mandrel mill) and a sizing mill. On the step of elongation in Mannesmann process, inside face of a hollow blank is forcibly lubricated with a lubricant in order to prevent seizing. The lubricant is a graphite-based lubricant made mainly of granular graphite, scaly graphite, soil-form graphite or the like. It is used in a solid state or in the state
15 of a liquid wherein a graphite is mixed with a binder.

[0003] In lubricity a graphite-based lubricant is suitable for hot rolling of a hard-to-work material such as stainless steel or high-alloy steel. However, the corrosion resistance of the material is hindered by carburization. Specifically, when a graphite-based lubricant is used for hot rolling of a hard-to-work material such as stainless steel or high-alloy, the inside face of a mother pipe or tube is carburized; therefore, grain boundaries due to grain-boundary - precipitation and the vicinity thereof are selectively corroded, so that the corrosion resistance thereof lowers, thereby losing the performance of product.

[0004] In order to improve this problem, Patent Document 1 suggests, instead of a conventional graphite-based lubricant, a lubricant composition based on a combination of an oxide-based layered compound, boric acid, and an alkali metal borate. Patent Document 2 suggests a lubricant composition' based on a combination of artificial mica, vermiculite
25 or bentonite, Li, Na or K borate, a metaborate or pyroborate, and a hydrate thereof. These lubricants, wherein an oxide-based layered compound is combined with boric acid, are effective as non-carburizable lubricant that is excellent in lubricity.

[0005] On the other hand, non-carburizability is not required for ordinary graphite-based lubricant. Accordingly, these graphite-based lubricants can give the above-mentioned various properties when an ordinary organic thickener (for example, a water-soluble acrylic resin, or a water-soluble cellulose such as sodium carboxymethylcellulose) is sufficiently added to the graphite-based lubricants. For example, Patent Document 3 discloses a lubricant wherein both of a water-soluble polymer and a water-dispersible polymer are blended in large amounts, whereby the above-mentioned various properties are attained.

[0006] Patent Document 1: Japanese Patent Application Laid-Open (JP-A) No. 64-16894

35 **[0007]** Patent Document 2: JP-A No. 5-171165

[0008] Patent Document 3: JP-A No. 2-51592

Disclosure of the Invention

40 Problems to be Solved by the Invention

[0009] The lubricants disclosed in the Patent Documents 1 and 2 are supplied in a solid state onto a face of a tool (for example mandrel bar), or are applied to a tool in a state that they are dispersed into water. Accordingly, in order to use the lubricants in a continuance mill (mandrel mill), it becomes necessary that they have various properties totally, such as storage stability before use, transferability inside an apparatus tube, sprayability to a lubricated site, and property of uniform adhesion to a high - temperature mandrel bar. However, it is difficult to attain these properties totally.

[0010] In elongating a hollow blank of stainless steel or high-alloy steel by a continuance mill (mandrel mill), non-carburizability is persistently required in order to keep corrosion resistance thereof. Thus, the above-mentioned polymers cannot be blended in large amounts.

50 **[0011]** Accordingly, an object of the present invention is to provide a lubricant composition which totally has various properties, such as storage stability, transferability inside an apparatus tube, sprayability to a lubricated site, and property of uniform adhesion to a high-temperature mandrel bar without losing non-carburizability or excellent lubricity in elongating hard-to-work materials (for example, such as 13 chromium steel and stainless steel) by a continuance mill (mandrel mill).

55 Means for Solving the Problems

[0012] The present invention is a lubricant composition for seamless steel pipes or tubes working which has a viscosity property represented by the following approximate expression:

$$Y = a \cdot X^b$$

wherein Y: viscosity (mPa·s) X: shear rate (s⁻¹) ; and

a: 4000 to 40000 and b: -1.0 to -3.0 while a lubricant composition is stored still, and a: 1000 to 20000, and b: -1.0 to -0.15 after 90 seconds from the time when shearing a lubricant composition is finished.

[0013] The wording "90 seconds from the time when shearing a lubricant composition is finished" means the time of 60 seconds after the following measurement start: a lubricant composition is stirred; and at 30 seconds from the end of the stirring, the viscosity of lubricant composition is measured at a given shear rate. Consequently, the wording "90 seconds" corresponds to the total value of the above-mentioned 30 seconds and 60 seconds. The wording "the time when shearing a lubricant composition is finished" means the time when the above-mentioned stirring operation is finished.

[0014] It is preferred that the lubricant composition for seamless steel pipes or tubes working comprises 10 to 40% by mass of an oxide-based layered compound, 5 to 30% by mass of one or more alkali metal salts or amine salts of boric acid, 0.11 to 3.0% by mass of one or more water-soluble polymers which are soluble in an aqueous solution of the alkali metal salt (s) or amine salt (s) of boric acid, and water as the balance.

[0015] It is preferred that the lubricant composition for seamless steel pipes or tubes working comprises, as water-soluble polymer(s), a pseudo plastic fluidity water-soluble polymer, or a pseudo plastic fluidity water-soluble polymer and a thixotropic fluidity water-soluble polymer.

[0016] Alternatively, it is preferred that the lubricant composition for seamless steel pipes or tubes working comprises, as water-soluble polymer(s), a pseudo plastic fluidity water-soluble polymer in an amount of 0.01 to 1.0% by mass of the whole of the lubricant composition, and a thixotropic fluidity water-soluble polymer in an amount of 0.1 to 2.0% by mass of the whole of the lubricant composition.

Effects of the Invention

[0017] According to the invention, it is possible to provide a lubricant composition for seamless steel pipes or tubes working which totally has various properties, such as storage stability, transferability inside an apparatus tube, sprayability to lubricated sites, and property of uniform adhesion to a high-temperature mandrel bar without losing non-carburizability or excellent lubricity to hard-to-work materials such as 13 chromium steel and stainless steel.

Best Mode for Carrying Out the Invention

[0018] An oxide-based layered compound used as main agent in a lubricant composition for seamless steel pipes or tubes working of the invention is, for example, any natural mica or artificial mica. Examples of the mica include:

Potassium tetrasilicone mica {KMg_{2.5}(Si₄O₁₀)F₂},
Sodium tetrasilicone mica {NaMg_{2.5}(Si₄O₁₀) F₂}, and
Natural golden mica {KMg₃(AlSi₂O₁₀) (OH)₂}.

In the lubricant composition for seamless steel pipes or tubes working of the invention, one or more therefrom can be used. Instead of the mica, or together with the mica, vermiculite, bentonite or the like can be used. Sodium tetrasilicone mica is the most preferable for the lubricant composition for seamless steel pipes or tubes working of the invention.

[0019] The average particle diameter of the oxide-based layered compound is from 1 to 40 μm, preferably from 5 to 30 μm. If the particle diameter is too small, the effect of interlayer slip becomes small. On the other hand, if the particle diameter is too large, problems such that a nozzle is blocked when the lubricant composition is sprayed are caused. The adding amount of the oxide-based layered compound is from 10 to 40% by mass of the lubricant composition of the lubricant of the invention for seamless steel pipes or tubes working, preferably from 20 to 30% by mass thereof. If the adding amount of the oxide-based layered compound is too small, the seizing resistance becomes low so that a problem is caused about the lubricity thereof. On the other hand, if the adding amount of the oxide-based layered compound is too large, the viscosity of the lubricant composition becomes too high so that a problem is caused about the workability.

[0020] Alkali metal salt(s) or amine salt(s) of boric acid in a lubricant composition help (s) the oxide-based layered compound, which is a main agent, spreading on a high-temperature mandrel bar, or the salt (s) itself/themselves work (s) as a single or plural lubricating auxiliaries. Examples of the alkali metal salt(s) of boric acid include lithium borate, sodium borate, and potassium borate. Ametaborate, a pyroborate and/or a hydrate, such as borax (Na₂B₄O₇·10H₂O) can be used.

[0021] The adding amount of alkali metal salt (s) or amine salt (s) of boric acid into the lubricant composition is from 5 to 30% by mass, preferably from 10 to 20% by mass. If the adding amount of the salt (s) is too large, the salt (s) hinder (s) the lubricity of the main agent. If the adding amount of the salt(s) is too small, it hinders a main object, that is, the property of uniform adhesion to a mandrel bar caused by the main agent. Moreover, the lubricity is insufficient, so that shortage of lubricity is induced.

[0022] As water-soluble polymer(s) used in a lubricant composition for seamless steel pipes or tubes working of the invention, the following can be used: a single or a combination of two or more selected from natural, semi-natural and synthetic water-soluble polymers having the viscosity conditions prescribed in the present invention. These water-soluble polymers will be specifically described later.

[0023] Next, an ideal viscosity which has been found by the present inventors is shown in FIG. 1. FIG. 1, wherein viscosity is represented by its vertical axis and time is represented by its horizontal axis, is a graph of the viscosity variation of a lubricant composition changed with time under the following conditions:

(i): a lubricant composition is kept still,

(ii) to (iii): a lubricant composition is sheared at a constant rate, and

(iii) to (iv): the shearing of a lubricant composition is finished.

[0024] The inventors have found out that a lubricant composition of which viscosity property is as the following (1) to (4), has excellent lubricity in the production of a seamless metal pipe or tube according to the Mannesmann process:

(1) In order to store a lubricant in a state that solid particles of the oxide-based layered compound is even and stable, the viscosity at the time (i) when a lubricant composition is kept still has to be high. If the viscosity is low, the solid particles settle down.

(2) In order to keep the transferability inside an apparatus tube and the sprayability, the viscosity at the time (ii) to (iii) when a lubricant composition is sheared needs to be low.

(3) For the property of fine and even adhesion to a high-temperature bar, which gives a great influence on lubricity, the viscosity at the time (ii) has to be high. From a microscopic viewpoint, a lubricant is continuously sprayed and adheres onto a tool surface. On a coat of lubricant adhered formerly, a lubricant sprayed later arrives. That is, a coat of lubricant adhered formerly is sheared. Accordingly, if the viscosity at the time (ii) is low, the lubricant is unfavorably peeled down or scattered by the pressure of spraying lubricant.

(4) In order to keep uniform and stable retention property of the solid particles on a tool surface, the viscosity needs to rise immediately after the finish of the shearing at the times of (iii) to (iv). If it takes much time that the viscosity recovers or the viscosity does not recover, the lubricant unfavorably flows down from the tool surface.

[0025] In order to realize that a lubricant has the viscosity showed as FIG. 1, and attains various properties, such as storage stability, transferability inside an apparatus tube, sprayability to lubricated sites, and property of uniform adhesion to a high-temperature mandrel bar without adding a large amount of a polymer, the inventors have found out that the lubricant viscosity has to fulfill the expression described below. In other words, the inventors have measured the viscosity property of the present invention lubricant having the above-mentioned various properties by a viscosity-measuring method described in Examples which is described later, so as to find that the parameters a and b which are counted from the measured viscosity curve has to be within the range described in the following approximate expression:

$$Y = a \cdot X^b$$

wherein Y: viscosity (mPa·s) X: shear rate (s⁻¹); and

a: 4000 to 40000 and b: -1.0 to -0.3 while a lubricant composition is stored still; and

a: 1000 to 20000, and b: -1.0 to -0.7.5 after 90 seconds from the time when shearing a lubricant composition is finished.

[0026] In the approximate expression:

$$Y = a \cdot X^b,$$

a and b are set into the range of 4000 to 40000 and that of -1.0 to -0.3, respectively, while a lubricant composition is stored to be kept still. If a is less than 4000, the viscosity of a lubricant composition is low while a lubricant composition is kept still. Consequently, the oxide-based layered compound settles down while a lubricant composition is stored. If a is more than 40000, the fluidity of a lubricant composition is substantially lost, so as to cause a problem about the transfer

thereof.

[0027] Further, if b is more than -0.3 , the difference between the viscosity of a lubricant composition while it is kept still and that while a lubricant composition is sheared (i.e., transferred or sprayed) is small, so as to cause a problem about the transfer or spray thereof. If b is less than -1.0 , the viscosity of a lubricant composition becomes too low when it is sprayed. Consequently, at the time of applying a lubricant composition onto a tool, the lubricant adhered formerly is peeled down or scattered by the pressure of spraying lubricant itself. More preferably a and b are as follows:

a : 7000 to 30000, and
 b : -0.5 to -0.8 .

[0028] The approximate expression: $Y = a \cdot X^b$ is described herein with reference to FIG. 1. As the value a is larger and the value b is smaller while a lubricant composition is stored still (that is, when the value X is limitlessly near 0), the viscosity at the time (i) in FIG. 1 is higher so that the stability is better. For example, in the case of $X = 0.01$, $a = 100000$ and $b = -1$, $Y = 10,000,000$ mPa·s.

[0029] In the approximate expression:

$$Y = a \cdot X^b,$$

a and b are set into the range of 1000 to 20000 and that of -1.0 to -0.15 , respectively, after 90 seconds from the time when shearing a lubricant composition is finished. If a is less than 1000 or b is less than -1.0 , the water-soluble polymer (s) is /are sheared and thus it takes much time that the viscosity recovers. Alternatively, a lubricant composition is sheared and its viscosity does not recover, and at this time the lubricant flows down. If a is more than 20000 or b is more than -0.15 , a problem is caused when a lubricant composition is transferred or sprayed. More preferably, a and b are as follows:

a : 3000 to 20000, and
 b : -0.3 to -0.8 .

[0030] As the value a is larger and the value b is smaller while a lubricant composition is sheared (that is, when the value X is larger immediately after a lubricant composition is stirred), the viscosity at the time (ii) and (iii) in FIG. 1 is higher. If a is too large and b is too small, the viscosity does not become low and that hinders the transferability and sprayability. For example, in the case of $X = 10$, $a = 100000$ and $b = -1$, $Y = 10,000$ mPa·s.

[0031] The time after a lubricant composition is sheared (that is, when the value X just after the stirring is small) is (iv) in FIG. 1. In the case that numerical values of a and b are similar to the numerical values when a lubricant composition is stored still, the viscosity rapidly recovers. In this case, the lubricant that adheres to a tool does not easily flow down. Accordingly, such an appropriate viscosity property as in the invention described above is crucial.

[0032] The inventors have made eager investigations on conditions for attaining the above-mentioned viscosity property, so as to find out the following: the use of only a pseudo plastic fluidity water-soluble polymer, as the water-soluble polymer(s) used in a lubricant composition of the invention, may cause the above mentioned property to be attained; however, the addition of both of a pseudo plastic fluidity water-soluble polymer and a thixotropic fluidity water-soluble polymer easily causes the above mentioned property to be attained.

[0033] Typical examples of the pseudo plastic fluidity water-soluble polymer include bio-gums such as xanthan gum, welan gum and ramsan gum. The chemical structure of xanthan gum is a water-soluble polymer polysaccharide wherein linking blocks each having two glucoses, two mannoses and one glucuronic acid as constituents are recurred. A model of the chemical structure is illustrated in FIG. 2.

[0034] Typical examples of the thixotropic fluidity water-soluble polymer include carboxymethylcellulose salts (Na, K, and amine salts). As an example thereof, a chemical structure model of a long-chain polymer wherein glucoses are subjected to glucoside bonding is illustrated in FIG. 3.

[0035] Pseudo plastic fluidity and/or thixotropic fluidity can also be attained by incorporating, into a lubricant composition, one or more selected from the following materials which are different from the above and cannot be easily distinguished from each other dependently on the molecular weight, another component or other components (for example, a metal (such as Ca) ion), or pH: bio-gums such as gellan gum, and succinoglucon; natural polysaccharides such as tamarind, tara gum, locust bean gum, and carrageenan; cellulose derivatives such as methylcellulose, ethylcellulose, hydroxyethylcellulose, hydroxypropylcellulose, hydroxyethylmethylcellulose, and hydroxypropylmethylcellulose; polyacrylic acid salts (Na, K, and amine salts), and alginic acid salts (Na, K, and amine salts).

[0036] Next, the viscosity property of a pseudo plastic fluidity water-soluble polymer in static and dynamic states is

shown in FIG. 4, and that of a thixotropic fluidity water-soluble polymer is shown in FIG. 5. According to only the pseudo plastic fluidity water-soluble polymer, a change from the static state to the dynamic state is like a straight line. Thus, the change comes under the scope of the invention, but satisfaction is not sufficiently given. On the other hand, the viscosity change of the thixotropic fluidity water-soluble polymer, wherein the viscosity difference from the static state to the dynamic state is small, is gentler than the viscosity change of the pseudo plastic fluidity water-soluble polymer. About the thixotropic fluidity water-soluble polymer, some amount of time (yield value) is necessary from the start of the dynamic state to a drop in the viscosity. About the thixotropic fluidity water-soluble polymer, time is also necessary for a rise in the viscosity in the case that the dynamic state is shifted to the static state. In the case that time is required for such a viscosity change, the lubricant sprayed onto a mandrel bar cannot undergo viscosity change for ideal film-formation in a film-forming step from the spray to drying. As a result, the lubricant does not adhere evenly onto the mandrel bar at ease. Accordingly, the object of the invention cannot be sufficiently realized by only the thixotropic fluidity water-soluble polymer.

[0037] In order to realize the ideal viscosity form shown in FIG. 1, it is desired to add the above-mentioned two polymers together. Specifically, the addition percentage of the pseudo plastic fluidity water-soluble polymer is from 0.01 to 1.0% by mass of the whole of the lubricant composition and the addition percentage of the thixotropic fluidity water-soluble polymer is from 0.1 to 2.0% by mass of the whole of the lubricant composition. More preferably, the addition percentage of the pseudo plastic fluidity water-soluble polymer is from 0.05 to 0.5% by mass of the whole of the lubricant composition and in connection therewith the addition percentage of the thixotropic fluidity water-soluble polymer is from 0.5 to 1.5% by mass of the whole of the lubricant composition. If the addition percentage of the pseudo plastic fluidity water-soluble polymer gets larger than that of the thixotropic fluidity water-soluble polymer, the viscosity change by shear becomes too large so that a lubricant composition does not adhere evenly onto a mandrel bar at ease. If the percentage of the thixotropic fluidity water-soluble polymer becomes too large, the dispersion stability of the oxide-based layered compound and the spreading property to a high-temperature mandrel bar are deteriorated so that the property of the uniform adhesion gets worse.

[0038] A lubricant which includes each fluidity water-soluble polymer of the above-mentioned two has a mixed viscosity property of that shown in FIG. 4 and FIG. 5. A qualitative viscosity property thereof is as follows: in FIG. 1, at time(i) when a lubricant composition is kept still, the viscosity is high because of the nature of the pseudo plastic fluidity water-soluble polymer; when shear is applied thereto thereafter, a drop in the viscosity at (i) to (iii), having a synthetic form of pseudo plastic fluidity and thixotropic fluidity states, is exhibited; and at last the viscosity property is converted into a rise in the viscosity shown at (iv) immediately after shearing is finished.

[0039] If the total percentage of the two polymers is more than 3.0% by mass of the total of a lubricant composition, a problem of carburization is unfavorably caused. If the percentage of the two polymers is less than 0.11% by mass of the total of a lubricant composition, the dispersion stability of the oxide-based layered compound is deteriorated so that the resultant is not suitable for a lubricant composition of the invention.

[0040] The addition of an ordinary antifoaming agent or dispersing agent causes a risk of carburization if the agent is an organic compound; therefore, the addition of a small amount (0.5% or less by mass of the total of the lubricant composition) thereof is permissible. Inorganic agents, which do not contain carbon, are allowed to be added as long as the agents do not give a significant influence onto basic performances of the invention.

Examples

(1) Preparations of samples for evaluation

[0041] Eighteen working examples shown in Tables 1 to 2 and 10 comparative examples shown in Table 3, that is, 28 samples in total were prepared. Tables 1 to 3 also show peculiar numeric constants "a" and "b" in the approximate expressions of these samples while they were stored still, and those after 90 seconds from the time when shearing thereto was finished.

(2) Performance-evaluating tests

1) Viscosity

(i) Measuring conditions

[0042] Measuring device: B-model rotary viscometer was used.
Measuring temperature: It was set to 25°C.

Shearing conditions:

[0043] a 500-mL of the samples were respectively put into a 500-mL beaker, and then stirred at 3000 rpm for 1 minute by means of a propeller of 50 mm diameter.

Rotation number:

[0044] Low rotation (1.5 rpm) [shear rate: 0.323 to 0.366 S⁻¹]

[0045] High rotation (60 rpm) [shear rate: 12.9 to 14.6 s⁻¹]

[0046] The reason why the shear rate of the samples had a numerical range was that in the rotary viscometer, wherein viscosity is measured while a cone is rotated on its plate, cones actually used for the samples were different from each sample.

(ii) Measuring method

[0047] Stationary viscosity: After each of the samples was stirred and kept still for 24 hours, it started to measure the viscosity at low shear rate. After 60 seconds from the start of the measurement, data of the viscometer was read out and was multiplied by a coefficient. Subsequently, it started to measure the viscosity at high shear rate. After 60 seconds from the start of the measurement, data of the viscometer was read out and was multiplied by a coefficient. The result was recorded as stationary viscosity.

[0048] Shear viscosity: Each of samples was stirred to shear thereto with the propeller. After 30 seconds from the finish of shearing, it started to measure the viscosity at low shear rate. After 60 seconds from the start of the measurement, data of the viscometer was read out and was multiplied by a coefficient. The sample was again stirred, and after 30 seconds therefrom it started to measure the viscosity at high shear rate. After 60 seconds from the start of the measurement, data of the viscometer was read and was multiplied by a coefficient. The result was recorded as shear viscosity.

[0049] In the measurements, the time "90 seconds from the time when shearing was finished (or after 90 seconds from the finish of the shearing)" means the time of 60 seconds after the following measurement start: an operation of shearing a sample was performed by stirring the lubricant composition; and after 30 seconds from the finish of the operation, it started to measure the viscosity at a given shear rate. Thus, the time "90 seconds" corresponds to the total value of the above-mentioned 30 seconds and 60 seconds. The "time when shearing was finished (or the time of the finish of the shearing)" means the time when rotating the propeller for the above-mentioned stirring is stopped.

(iii) Decision of the peculiar numeric constants "a" and "b"

[0050] Logarithms of both sides of the approximate expression $Y = a \cdot X^b$ are given as follows: $\log(Y) = b \log(X) + \log(a)$

[0051] According to X and Y obtained in the above-mentioned measurements, $\log(X)$ and $\log(Y)$ are calculated. These are plotted in a graph corresponding to its vertical axis and its horizontal axis, respectively. As a result, there is a substantially linear relationship (relationship of a linear function) between them. From the graph, "b", which is the gradient of the linear function and "a", which is the vertical axis intercept, can be obtained by the least-squares method.

2) Storage stability

(i) Test method

[0052] The samples were respectively stored in a 500-mL glass container, and kept still for 7 days. Thereafter, a state of separation thereof was observed.

(ii) Evaluating method: The samples were evaluated inaccordance with the following evaluating criterion:

[0053]

⊙: No supernatant was generated, and no sedimentation was caused on the bottom.

○: Supernatant was generated in an amount of less than 5%, and no sedimentation was caused on the bottom.

Δ: Supernatant was generated in an amount of 5% or more, and no sedimentation was caused on the bottom.

x: Sedimentation was caused on the bottom regardless of the generation of supernatant.

[0054] The word "supernatant" means a substantially transparent liquid portion containing no solid. By observing a beaker of sample from a side-direction, the height of a supernatant in the beaker is measured. The height of a supernatant

in the beaker was evaluated as the percentage thereof into the height of the liquid in the beaker.

[0055] The wording "sedimentation on the bottom" means a state that a solid lubricant settles down on the bottom and thus a hard layer having no fluidity can be perceived.

3) Carburization

(i) Test method

[0056] Four examples 4, 11 and 13 and Comparative Examples 6 were respectively sprayed and applied onto a mandrel bar made of SKD61 and having an outer diameter of 140.5 mm and an effective portion length of 18 m, and then dried and solidified to form a substantially uniform lubricant coat having a film thickness of 100 μm on the surface of the mandrel bar. The mandrel bar, onto which the lubricant was applied, was inserted into an hollow shell. Continuance mill (mandrel mill) composed of 7 stands was used to roll a hollow blank into an mother pipe or tube.

[0057] Specification of a hollow blank before elongation:

Material: Austenite stainless steel (SUS304L);

Working: piercing by piercing mill with inclined-roll;

Shape: outer diameter: 181.0 mm, thickness: 16.0 mm, and length: 7000 mm.

Shape of a mother pipe or tube after elongation:

Outer diameter: 151.0 mm, thickness: 5.0 mm, and length: 25300 mm.

[0058] After the hollow blanks were respectively rolled with continuance mill (mandrel mill), the mother pipe or tube was rolled to determined outer diameter by sizing mill. A product had an outer diameter of 63.5 mm, thickness of 7.0 mm and a length of 40000 mm. An arc-form test piece having a thickness of 5 mm, a width of 25 mm and a length of 50 mm was made from a product. This test piece was used to make a sulfuric acid-copper sulfate corrosion test prescribed in JIS G0575, and then the state of a grain boundary corrosive crack generated in the inside surface was observed.

(ii) Evaluating method: An evaluation was made in accordance with the following criterion:

[0059]

⊙: No crack.

x: A crack was generated.

4) Sprayability

(i) Test conditions

[0060] Spray manner: an airless spray.

[0061] Spraying pressure: It was set to 3.0 MPa.

[0062] Nozzle: 1/4MVVP5010 (manufactured by Kabushiki Kaisha Ikeuchi (transliteration)).

[0063] Spray pattern: Fan-shaped

[0064] Spray angle:

[0065] 50 degrees (angle at which water was spread into a fan shape when water was sprayed)

[0066] Sample temperature: It was set to 25°C to make an evaluation.

(ii) Evaluation: The spreading property was evaluated by measuring the spray angle. The result of the evaluation was recorded in accordance with the following evaluating criterion:

[0067]

⊙: The spray angle was a almost predetermined angle (50 degrees).

O: The spray angle was somewhat narrower than the predetermined angle (40 to 45 degrees).

△: The spray angle was far narrower than the predetermined angle (20 to 39 degrees).

x: The spray angle was hardly spread (less than 20 degrees), or sprayed particles were large.

5) Adhesion property (adhesion amount)

i) Test conditions

- 5 [0068] Spray manner: an airless spray.
 [0069] Spraying pressure: It was set to 3.0 MPa.
 [0070] Nozzle: 1/4MVVP5010 (manufactured by Kabushiki Kaisha Ikeuchi (transliteration)).
 [0071] Spray angle: 50 degrees
 [0072] Test piece temperatures: The temperatures were set to 60, 80, 100, and 120°C.
 10 [0073] Nozzle/test-piece distance: It was set to 250 mm to make the test.
 [0074] Test speed: It was set to 2 m/second.

(ii) Evaluating method: Each sample was sprayed onto a test piece (a 65 mm x 120 mm x 30 mm steel plate) under the test conditions. After spraying, the test piece was heated up to 120°C to dry the lubricant. Thereafter, the test piece naturally cooled down to 20-40°C. The lubricant coat was shaved off with a knife and its weight was measured. The weight of the coat was divided by the adhesion area (0.0078 m²) and the result was defined as the adhesion amount. It was evaluated in accordance with the following evaluating criterion:

[0075]

- 20 ◎: The coat adhered evenly, and the adhesion amount was 50 g/m² or more.
 0: The coat adhered substantially evenly, and the adhesion amount was 50 g/m² or more.
 △: The lubricant somewhat flowed down, and the adhesion amount was 40 g/m² or more.
 x: The lubricant flowed down largely, and the adhesion amount was 30 g/m² or less.
 25 xx: The adhesion property was bad because of repellency, and the adhesion amount was 30 g/m² or less.

(3) Test results

30 [0076] The results of the tests 1) to 5) are shown in Tables 1 to 3.

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[Table 1]

		Example 1	Example2	Example 3	Example 4	Example 5	Example 6	Example 7	Example 8	Example 9
Sodium tetrasilicone mica		10.00	10.00	20.00	15.00	20.00	20.00	20.00	20.00	30.00
Natural golden mica										
Bentonite										
Potassium borate			10.00	13.00	8.00	10.00	10.00	15.00	15.00	12.00
Amine borate		5.00	5.00		8.00		5.00	5.00	5.00	3.00
Xanthan gum		0.50	0.40	0.70	0.10		0.40	0.50	0.30	0.30
Welan gum						0.05				
CMC (A)		0.50						0.50		
CMC (B)			0.50							0.30
CMC (C)					1.00	1.00			0.50	
CMC (D)										
Na polyacrylate (A)										
Na polyacrylate (B)							0.20			
Water (Calculated amount)		84.00	74.10	66.30	67.90	68.95	64.40	59.00	59.20	54.40
Liquid state										
Stationary viscosity	(low shear rate)	18850	15930	53500	14180	15400	15800	22080	26000	30900
	(high shear rate)	1080	1390	1750	1780	1870	1040	1720	1700	1860
After shearing	(low shear rate)	19520	17400	48420	6325	2250	18900	25000	18000	24130
	(high shear rate)	1090	1410	1690	1410	1170	1060	1630	1600	1710
At a time of stationary storage	(coefficient a)	8154	7795	19638	7719	8302	7119	10451	11691	13562
	(coefficient b)	-0.777	-0.663	-0.929	-0.564	-0.573	-0.739	-0.693	-0.741	-0.763
Immediately after the shearing	(coefficient a)	8380	8332	18115	4074	1858	8125	11232	8856	11110
	(coefficient b)	-0.784	-0.683	-0.911	-0.408	-0.178.	-0.783	-0.742	-0.657	-0.719

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Table continued

	Example 1	Example2	Example 3	Example 4	Example 5	Example 6	Example 7	Example 8	Example 9
Storage stability	⊙	⊙	⊙	⊙	⊙	⊙	⊙	⊙	⊙
Carburization	⊙	⊙	⊙	⊙	⊙	⊙	⊙	⊙	⊙
Sprayability	(25°C)	⊙	⊙	○	⊙	⊙	○	⊙	⊙
Adhesive property	(60°C)	⊙	⊙	⊙	○	○	Δ	⊙	⊙
	(80°C)	⊙	⊙	⊙	⊙	○	Δ	⊙	⊙
	(100°C)	⊙	⊙	⊙	⊙	⊙	○	⊙	⊙
	(120°C)	⊙	⊙	○	⊙	⊙	○	⊙	⊙

[Table 2]										
		Example 10	Example 11	EXample 12	Example 13	Example 14	Example 15	Example 16	Example 17	Example 18
Sodium tetrasilicone mica		40.00			10.00		5.00	5.00		20.00
Natural golden mica			20.00		10.00	10.00	5.00	5.00	10.00	
Bentonite				20.00		10.00	5.00	5.00		
Potassium borate		12.00	12.00	10.00	10.00	20.00	5.00	5.00	30.00	30.00
Amine borate		3.00	3.00	10.00	5.00		10.00	10.00		
Xanthan gum			0.60					0.10	0.20	
Welan gum		0.05		0.15	0.20	0.15	0.10			0.02
CMC (A)					1.50					
CMC (B)										0.50
CMC (C)							1.00	1.00	1.00	
CMC (D)		0.50								
Na polyacrylate (A)			2.00			1.00				
Na polyacrylate (B)				0.30						
Water (Calculated amount)		44.45	62.40	59.55	63.30	58.85	68.90	68.90	58.80	49.48
Liquid state										
Stationary viscosity	(low shear rate)	12070	42400	32000	45100	17800	50000	9500	18000	9225
	(high shear-rate)	1740	2480	1450	4230	1650	3050	1800	2150	1680
After shearing	(low shear rate)	8920	32100	17500	9830	7200	8500	5800	133300	1975
	(high shear rate)	1580	2500	1100	2230	900	2100	1560	1980	835
At a time of stationary storage	(coefficient a)	6843	18453	12923	22542	8866	22031	5835	9657	5600

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		Example 10	Example 11	EXample 12	Example 13	Example 14	Example 15	Example 16	Example 17	Example 18
	(coefficient b)	-0.526	-0.771	-0.841	-0.643	-0.646	-0.76	-0.452	-0.577	-0.463
Immediately after the shearing	(coefficient a)	5371	15193	7779	6364	3915	5643	3947	7611	1534
	(coefficient b)	-0.47	-0.693	-0.752	-0.403	-0.565	-0.38	-0.357	-0.517	-0.234
Storage stability		⊙	⊙	⊙	⊙	○	⊙	⊙	⊙	○
Carburization		⊙	⊙	⊙	⊙	⊙	⊙	⊙	⊙	⊙
Sprayability	(25°C)	⊙	⊙	○	○	⊙	⊙	⊙	⊙	⊙
Adhesive property	(60°C)	⊙	⊙	Δ	⊙	○	⊙	⊙	⊙	Δ
	(80°C)	⊙	⊙	Δ	⊙	○	⊙	⊙	⊙	Δ
	(100°C)	⊙	○	○	⊙	⊙	⊙	⊙	⊙	○
	(120°C)	⊙ Δ		○	⊙	○	⊙	⊙	⊙	Δ

[Table 3]

		Comparati ve Example 1	Comparati ve Example 2	Comparati ve Example 3	Comparati ve Example 4	Comparati ve Example 5	Comparati ve Example 6	Comparati ve Example 7	Comparati ve Example 8	Comparati ve Example 9	Comparativ e Example 10
Sodium tetrasilicone mica		20.00	30.00			20.00	10.00	20.00	7.00	42.00	
Natural golden mica				15.00	50.00		10.00				20.00
Bentonite						10.00					10.00
Potassium borate		10.00		5.00	30.00	10.00	15.00	20.00	2.00	30.00	10.00
Amine borate			10.00	5.00		5.00	5.00		2.00	5.00	5.00
Xanthan gum		3.00					0.50	0.30	0.30		0.005
Welan gum					0.30	2.00				0.005	
CMC (A)			1.00						1.00		0.05
CMC (B)								5.00			
CMC (E)							5.00				
Na polyacrylate (A)											
Na polyacrylate (B)				1.00						0.15	
Water (Calculated amount)		67.00	59.00	74.00	19.70	53.00	54.50	54.70	87.70	22.845	54.945
Liquid state		Unable to be prepared			Unable to be prepared	Unable to be prepared		Unable to be prepared			
Stationar y viscosity	(low shear rate)		400	2100			26800		4400	87600	800
	(high shear rate)		340	570			3870		520	100000	130
After shearing	(low shear rate)		350	700			11000		1000	100000	1200
	(high shear rate)		314	500			2300		468	8700	140

Table continued

		Comparative Example 1	Comparative Example 2	Comparative Example 3	Comparative Example 4	Comparative Example 5	Comparative Example 6	Comparative Example 7	Comparative Example 8	Comparative Example 9	Comparative Example 10
At a time of stationary storage	(coefficient a)		381	1433			15201		2353	91065	469
	(coefficient b)		-0.044	-0.354			-0.526		-0.58	-0.038	-0.494
Immediately after the shearing	(coefficient a)		339	63.4			6954		800	48892	639
	(coefficient b)		-0.03	-0.091			-0.425		-0.206	-0.663	-0.584
Storage stability			X	X			⊙		⊙	⊙	X
Carburization			⊙	⊙			X		⊙	⊙	⊙
Sprayability	(25°C)		⊙	○			○		⊙	X	⊙
Adhesive property	(60°C)		X	X			○		X	X	X
	(80°C)		X	X			○		X	X	X
	(100°C)		X	X			○		X	X	X
	(120°C)		XX	XX			○		X	X	XX
CMC*(A): molecular weight (100,000), viscosity (800/2%**) CMC*(B): molecular weight (250,000), viscosity (1600/1%**) CMC*(C): molecular weight (175,000), viscosity (2500/2%) CMC*(D): molecular weight (195,000), viscosity (3500/2%) CMC*(E): molecular weight (30,000), viscosity (15/2%) Na polyacrylate (A): molecular weight (500,000), viscosity (75/1%)											

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Table continued

Na polyacrylate (B): molecular weight (1,650,000), viscosity (300/0.2%)
*CMC = Na salt of carboxymethylcellulose
**: It means that the viscosity of a 2% solution thereof in water at 25°C was 800 mPa·s.

(4) Conclusion

[0077] From the above-mentioned test results, it was proved that a lubricant composition of the invention has performances good in all of storage stability, sprayability, and adhesion property. On the other hand, about the group of a lubricant compositions of Comparative Examples, satisfactory performances therefor were unable to be obtained.

[0078] The above has described the invention in connection with embodiments which appear to be most practical and preferable at the present time. However, the invention is not limited to the embodiments disclosed in the specification of the present application, and can be appropriately varied within a scope not contrary to the subject matter and the conception of the invention read out from the claims and the whole of the specification. It should be understood that any lubricant composition for seamless steel pipes or tubes working with such a variation is also included in the technical scope of the invention.

Industrial Applicability

[0079] A lubricant composition for seamless steel pipes or tubes working of the invention is useful for working in the Mannesmann process for producing a seamless steel pipe.

Brief Description of the Drawings

[0080]

FIG. 1 is a chart showing an ideal viscosity form as a lubricant composition for seamless steel pipes or tubes working.

FIG. 2 is a view of a chemical structure model of an example of the pseudo plastic fluidity water-soluble polymer.

FIG. 3 is a view showing a chemical structure model of a long-chain polymer wherein glucoses are subjected to glucoside bonding, the polymer being an example of the thixotropic fluidity water-soluble polymer.

FIG. 4 is a chart showing viscosity properties of static and dynamic states of a pseudo plastic fluidity water-soluble polymer.

FIG. 5 is a chart showing viscosity properties of static and dynamic states of a thixotropic fluidity water-soluble polymer.

Claims

1. A lubricant composition for seamless steel pipes or tubes working, which has a viscosity property represented by the following approximate expression: $Y = a \cdot X^b$
 wherein Y: viscosity (mPa·s), X: shear rate (s^{-1}); and
 a: 4000 to 40000 and b: -1.0 to -0.3 while a lubricant composition is stored still, and
 a: 1000 to 20000, and b: -1.0 to -0.15 after 90 seconds from the time when shearing a lubricant composition is finished.
2. The lubricant composition for seamless steel pipes or tubes working according to claim 1, which comprises 10 to 40% by mass of an oxide-based layered compound, 5 to 30% by mass of one or more alkali metal salts or amine salts of boric acid, 0.11 to 3.0% by mass of one or more water-soluble polymers which are soluble in an aqueous solution of the alkali metal salt(s) or amine salt(s) of boric acid, and water as the balance.
3. The lubricant composition for seamless steel pipes or tubes working according to claim 2, which comprises, as the water-soluble polymer(s), a pseudo plastic fluidity water-soluble polymer, or a mixture of a pseudo plastic fluidity water-soluble polymer and a thixotropic fluidity water-soluble polymer.
4. The lubricant composition for seamless steel pipes or tubes working according to claim 2, which comprises, as the water-soluble polymer(s), a pseudo plastic fluidity water-soluble polymer in an amount of 0.01 to 1.0% by mass of the whole of the lubricant composition, and a thixotropic fluidity water-soluble polymer in an amount of 0.1 to 2.0% by mass of the whole of the lubricant composition.

FIG. 1

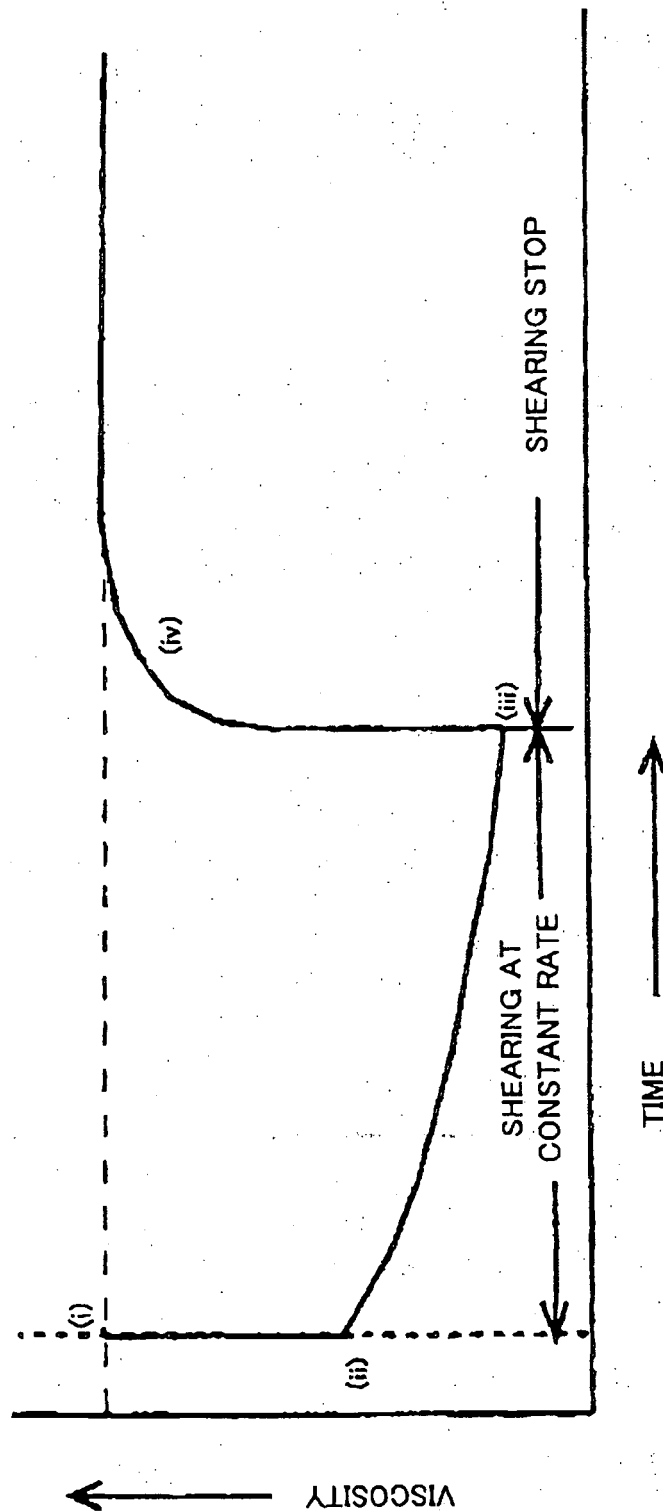
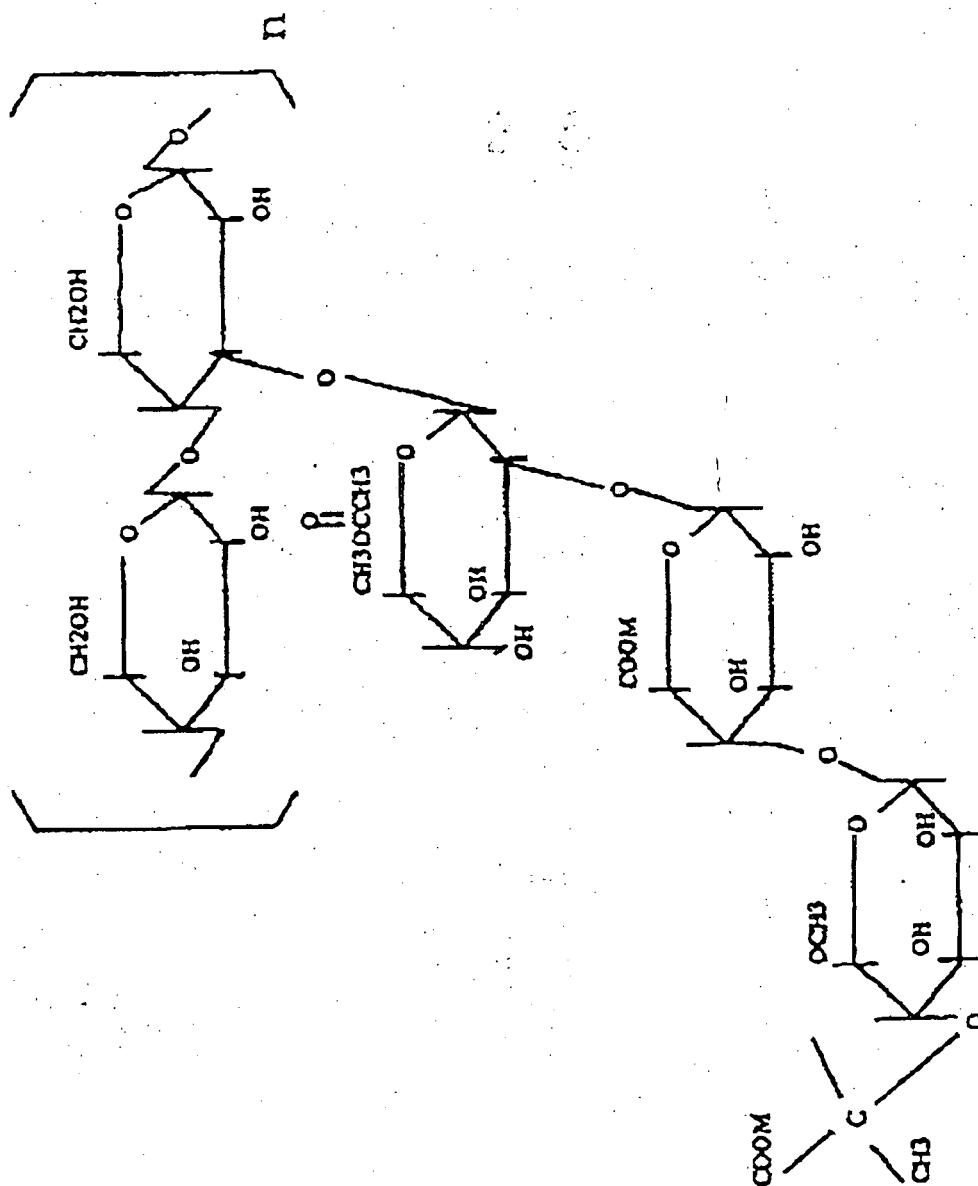
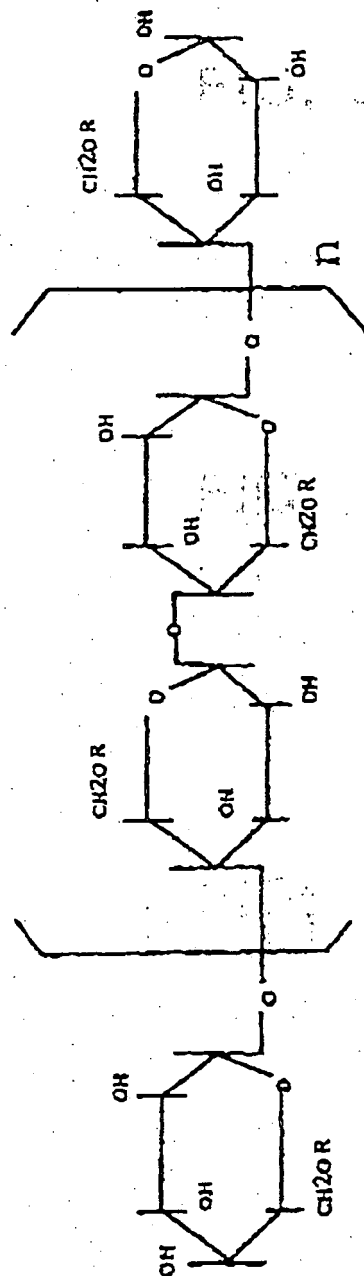


FIG. 2



M is Na, K or $1/2\text{Ca}$

FIG. 3



R is H, CH₃, C₂H₅, CH₂COONa, CH₂CH₂OH, CH₂CH(OH)CH₃ or the like.

FIG. 4

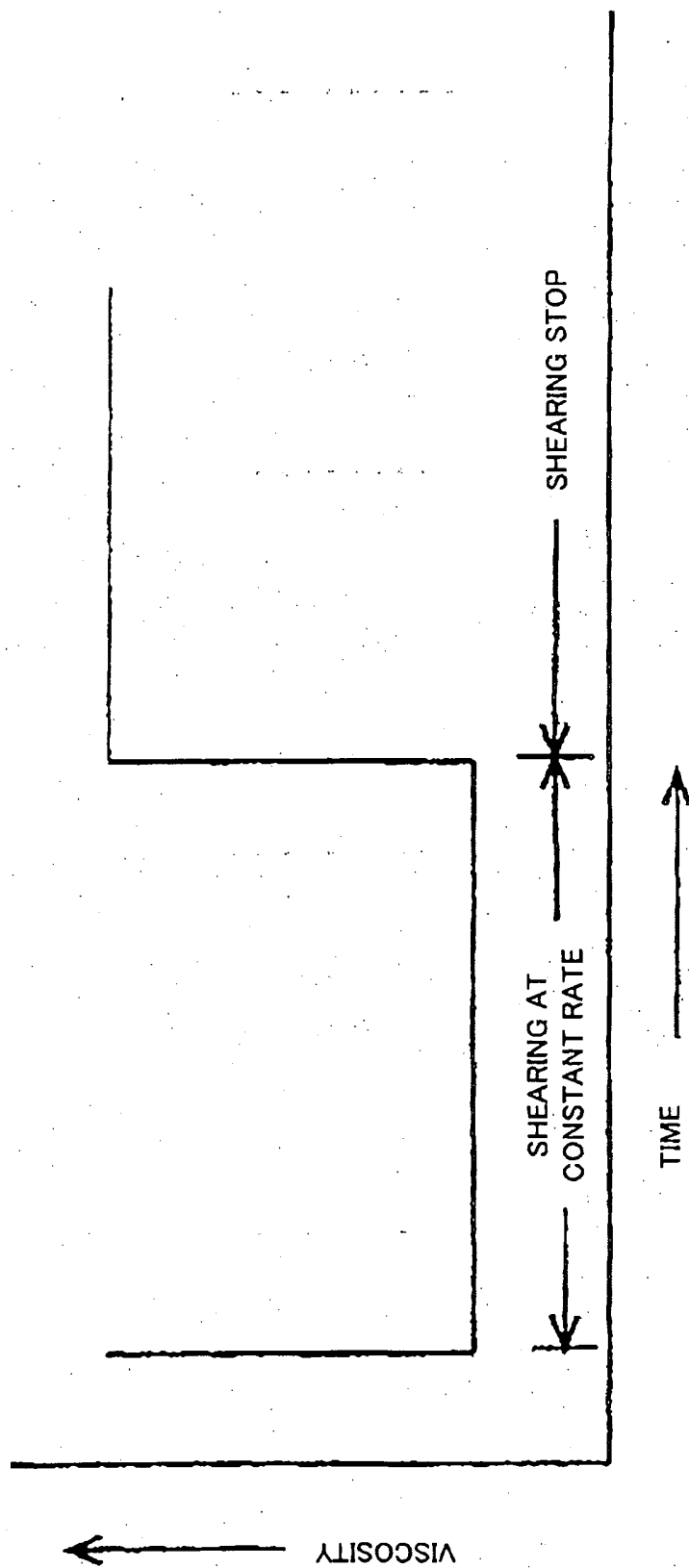
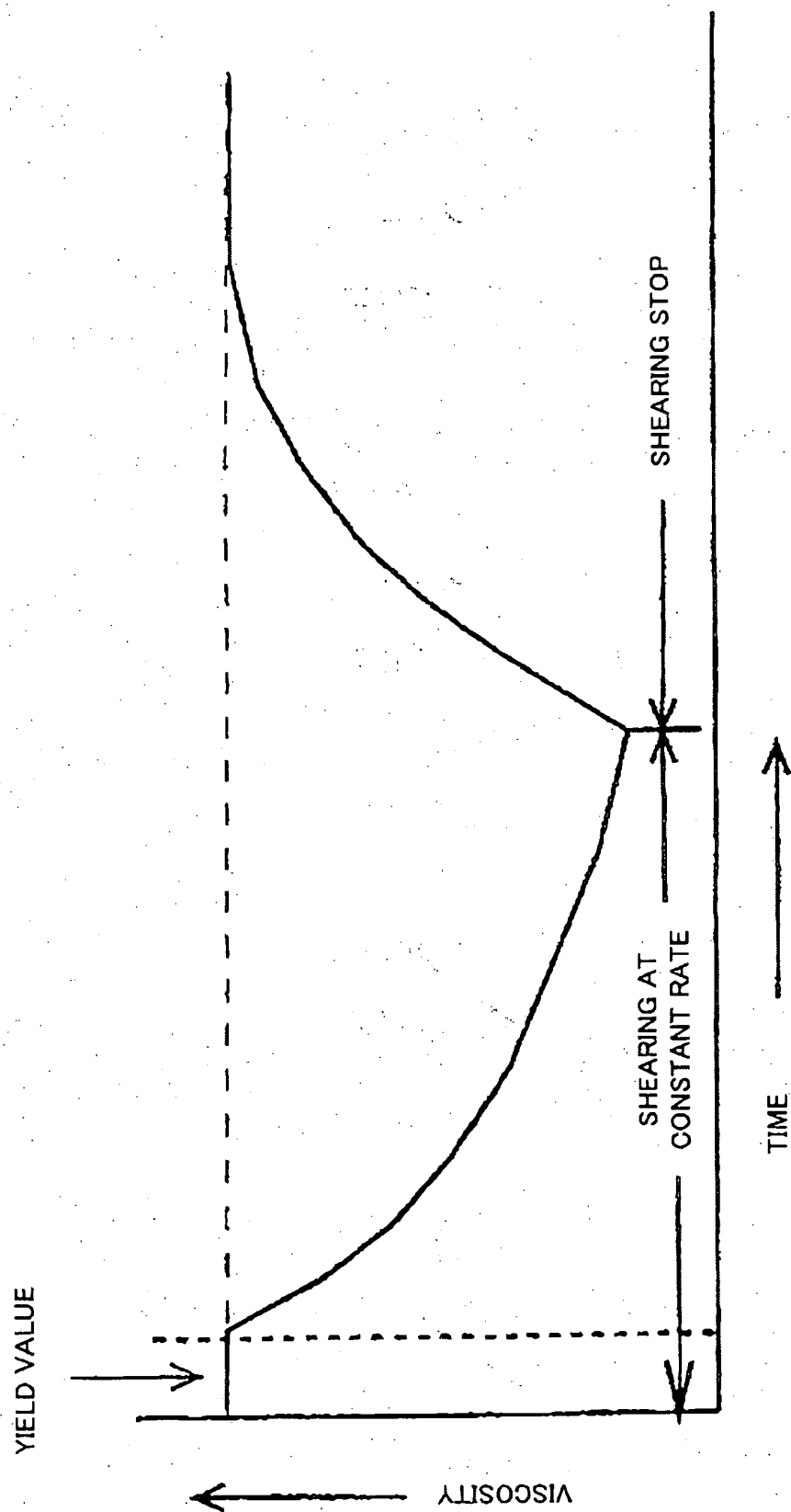


FIG. 5



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2004/012833

A. CLASSIFICATION OF SUBJECT MATTER

Int.Cl.⁷ C10M171/02, 173/02//B21B25/04 (C10M173/02, 103:00, 103:06, 145:40) C10N10:02, 20:02, 30:00, 30:06, 40:24, 50:02

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)

Int.Cl.⁷ C10M171/02, 103/00, 103/06, 125/26-125/30, 145/40, 173/00-173/02, C10N10:02, 20:02, 30:00, 30:06, 40:20-40:24, 50:02, B21B25/04

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Jitsuyo Shinan Koho 1922-1996 Toroku Jitsuyo Shinan Koho 1994-2004
Kokai Jitsuyo Shinan Koho 1971-2004 Jitsuyo Shinan Toroku Koho 1996-2004

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2001-234189 A (Sumitomo Metal Industries, Ltd.), 28 August, 2001 (28.08.01), (Family: none)	1-4
X	JP 3-12498 A (Sumitomo Metal Industries, Ltd.), 21 January, 1991 (21.01.91), (Family: none)	1-4
X	JP 2002-28705 A (Sumitomo Metal Industries, Ltd.), 29 January, 2002 (29.01.02), (Family: none)	1-4

☐ Further documents are listed in the continuation of Box C.

☐ See patent family annex.

* Special categories of cited documents:

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Date of the actual completion of the international search
22 November, 2004 (22.11.04)

Date of mailing of the international search report
14 December, 2004 (14.12.04)

Name and mailing address of the ISA/
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