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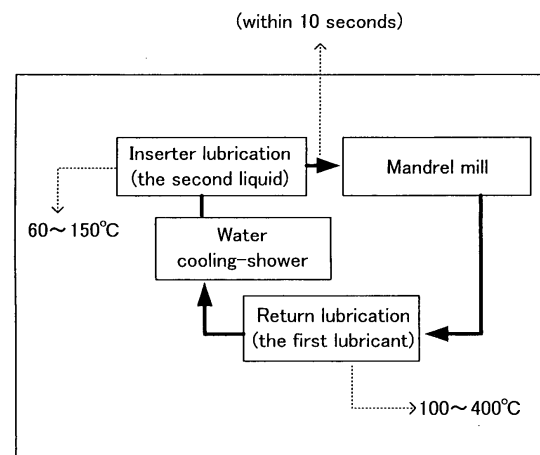
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## (54) **PROCESS FOR PRODUCING SEAMLESS TUBE**

(57) The present invention provide a method of manufacturing seamless pipes comprising the steps of: the first coating step for forming a water-resistant coating by applying a first liquid onto the surface of a hot mandrel bar right after the previous elongation-rolling process; a water-cooling step for water-cooling the mandrel bar treated by the first coating step; a second coating step for applying a second liquid containing an inorganic lubricant which is excellent in high-temperature fluidity on the surface of the mandrel bar treated by the water-cooling step, then making the second liquid permeate into the water-resistant coating to give it high-temperature adhesion, and an elongation-rolling step.

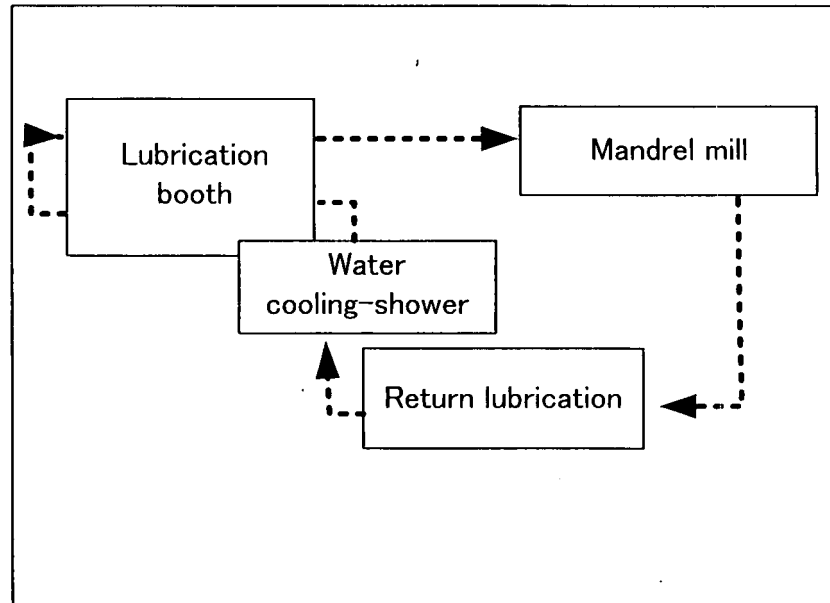
This elongation-rolling step is characterizing in that the elongation-rolling of a hollow shell shall be commenced in 10 seconds after the second coating step by using the mandrel bar.

FIG. 1 (A)



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FIG. 1 (B)



## Description

## Technical Field

**[0001]** The present invention relates to a method of manufacturing seamless pipes or tubes (hereinafter, refer to "pipes" as "pipes or tubes".) according to Mannesmann mandrel-mill process. Specifically, the present invention relates to a method of manufacturing seamless pipes which enables to effectively inhibit seizing between a hollow-shell and a mandrel bar as a tool for forming inner surface of pipes and to effectively inhibit occurrence of flaws on inner surface of the pipes when a hollow shell of which material is high-alloy steel such as stainless-steel or 13 Cr steel is in elongation-rolling process.

## Background Art

**[0002]** Seamless pipes are used in many industrial fields such as energy, automobile, chemistry, industrial machinery, and construction. Seamless pipes are heavily used especially for oil well tubular and for transporting crude oil and gas. Thus, such pipes play an important role in the field related to the world's energy-resources development.

**[0003]** The manufacturing method of seamless pipes in accordance with the Mannesmann mandrel-mill process is, for example, carried out based on the process shown in FIG. 3. Firstly, a billet 1 as a raw material of a seamless pipe is inserted into a rotary-hearth type heating furnace 2 and heated. The billet 1 heated in the rotary-hearth type heating furnace 2 is taken out from the furnace, after that, it is pierced and elongated by piercer (piercing-rolling apparatus) 3 to become a hollow shell 4. Then, from the back-end side of the hollow shell 4, a mandrel bar 5a is inserted into a through hole of the hollow shell 4, and the hollow shell 4 is elongated and rolled to become an element pipe 4a. Later, the mandrel bar 5a is withdrawn from the element pipe 4a.

**[0004]** In the manufacturing of seamless pipes in accordance with the above Mannesmann mandrel-mill process, at a time of elongation-rolling of the hollow shell 4 by mandrel-mill, the mandrel bar 5a is normally inserted into a hollow shell 4 of a temperature of 1100~1200°C and is exposed in a state that the bar 5a tends to seize. Therefore, lubricants are coated onto the outer surface of the mandrel bar 5a. The lubricants can work as a protecting coating which inhibits seizing between the mandrel bar 5a and the hollow shell 4. As such lubricants coated onto the surface of the mandrel bar 5a, used are lubricants for hot-rolling which are excellent in lubrication performance in hot condition. From the viewpoint of lower cost and excellent lubrication performance, for instance, an aqueous graphite-type lubricant mainly contains graphite is heavily used. This lubricant is usually supplied from a storage tank to production lines. The lubricant is showery injected onto the surface of the mandrel bar 5a to go through inside of the hollow shell 4 from a ring-shaped nozzle disposed at a position just before the mandrel bar 5a being inserted into the hollow shell 4. In this way, empirically-determined certain amount of lubricants is applied onto the surface of the mandrel bar 5a.

**[0005]** Shape of outer diameter and wall thickness of the element pipe elongated and rolled by a mandrel-mill are influenced by number of revolution of each stand and shape of rolls' holes. They are also influenced by changes of friction coefficient between a mandrel bar 5a and a hollow shell 4. In other words, as a lubricant to be coated to the mandrel bar 5a, by using the lubricant which is capable to reduce friction coefficient between the mandrel bar 5a and the hollow shell 4, the hollow shell 4 is uniformly deformed in the circumferential direction and in the longitudinal direction during the elongation-rolling. Therefore it is possible to obtain element pipes having stable shape of outer diameter and stable local variations in the wall thickness.

**[0006]** After the process of elongation-rolling, the mandrel bar 5a is withdrawn from an element pipe 4a with use of bar stripper. However, if lubricity of the lubricant coated onto the outer surface of the mandrel bar 5a is insufficient, the element pipe 4a (hollow shell 4) and the mandrel bar 5a would seize each other, thereby flaws occur on the inner surface of the element pipe 4a. Or, the seizing prevents the mandrel bar 5a from its withdrawal.

**[0007]** In general, in hot metal working, oxidized scale is produced on the surface of both tool and the inner surface of the material to be processed. An iron oxide coating consists of this oxidized scale, particularly a coating consists of dense and relatively low hardness of material including FeO and Fe<sub>3</sub>O<sub>4</sub> gives a favorable influence for lubricity during the pipe manufacturing. Nevertheless, different from a normal steel like carbon steel, a stainless-steel such as SUS 304 series, SUS 316 series, SUS 347 series, SUS 410 series, and SUS 430 series, or an alloy steel such as STBA 25 and STBA 26, respectively defined in the Japanese Industrial Standard (JIS), does not easily produce such oxidized scale. In addition, as these steels have mechanical properties such as high-strength and high-toughness, the tool corresponding to the above steel needs to be made of heat-resistant low alloy such as Co-Mo series alloy and Cr-Mo-V series alloy, e. g. JIS-SKD 61. Moreover, the tool made of these materials does not easily give oxidized scale. Thus, rolling load and friction coefficient during the process becomes higher, the mandrel bar 5a and the hollow shell 4 tend to seize each other. These factors often produce flaws on the inner surface of the element pipe. Highly accurate surface texture is required for pipe production of stainless steel or alloy steel. When such surface flaws are produced, even though they are mild damages, the obtained pipe itself cannot be shipped as a final product. Therefore, after treatment such as

surface polishing is needed. On the other hand, when a deep flaw is made on the pipe surface, the pipe is defective. Accordingly, in a manufacturing of seamless pipes in accordance with the Mannesmann mandrel-mill process, performance of lubricants coated onto the surface of the mandrel bar 5a gives large impact not only on the product quality but also on its productivity.

**[0008]** Normally, a mandrel bar is inserted into a hollow shell of a temperature of 1100~1200°C, and the hollow shell having the mandrel bar therein is treated by elongation-rolling process. So, temperature of the mandrel bar right after elongation-rolling is high in the range of 100~400°C. Such a mandrel bar is cooled and lubricants are coated thereon again before the use for elongation-rolling with the following hollow shell.

**[0009]** About lubricants to be applied on the mandrel bar, Patent document 1 discloses an art which inhibits seizing between a hollow shell and a mandrel bar by the following method: chemically reacting two-liquid containing the first liquid containing graphite powder and aqueous resin having reaction-curing property and the second liquid containing boric acid and so on; then, putting the hardened lubricant coating to intervene between the hollow shell and the mandrel bar.

**[0010]** Patent document 2 discloses a one-component type lubricant of the first liquid of the above Patent document 1 of which aqueous resin is altered by a copolymer and further including mica.

**[0011]** Further, Patent document 3 discloses an art which inhibits seizing by applying a particular lubricant for more than once to secure the predetermined thickness of lubricant coating. Patent Document 1: Japanese Patent Application Laid-Open (JP-A) No. 63-230797

Patent Document 2: JP-A No. 8-165489

Patent Document 3: JP-A No. 2004-34072

## Disclosure of the Invention

### Problems to be solved by the Invention

**[0012]** A mandrel bar withdrawn from an element pipe is reused (the so-called "cyclic usage") for the elongation-rolling of the following hollow shell after being transported to the bar-cooling zone to be cooled down to the certain temperature. In the bar-cooling zone, practically adoptable cooling methods in the field are air-cooling and water-cooling. If sufficient space cannot be secured for putting many mandrel bars for air-cooling, water-cooling is adopted because of a short period of cooling time. In such a case, the mandrel bar is provided for the elongation-rolling of the following hollow shell soon after the water-cooling.

**[0013]** If a mandrel bar is water-cooled, presumably, in general, there may be two timings of lubricants coating onto the surface of the bar, that is: right after the finish of elongation-rolling of the previous hollow shell (pre-water-cooling); and a period between after complete evaporation of water on the surface of a water-cooled bar and right before the elongation-rolling of the following hollow shell. When a lubricant is coated right after the elongation-rolling, as the mandrel bar to be coated by the lubricant is a high temperature of 100~400°C and water-cooled as the following step, such lubricants are required to have heat resistance and water resistance as well as seizure resistance. While, when lubricant is coated onto the surface of the bar after its water-cooling and right before the elongation-rolling of the following hollow shell, in order to raise the manufacturing efficiency, it is necessary to form a lubricant coating having a sufficient seizure resistance within a short period after the lubricant coating. In other words, the lubricant is required to have quick-drying property.

**[0014]** The lubricant disclosed in Patent document 1 is not designed to have water resistance and heat resistance, thereby it is necessary to apply lubricants after water-cooling and before elongation-rolling. However, it is not possible to form a dry-coating over the entire thickness of the lubricant coating within a short period so as to have favorable seizure resistance. On the other hand, it takes a long time to obtain a lubricant coating having favorable seizure resistance by sufficient drying. Accordingly, there is a problem that the operating time for elongation becomes longer so that the manufacturing efficiency declines.

**[0015]** While, the lubricant disclosed in Patent document 2 is designed to be used basically at room temperature and water resistance is not concerned; it is necessary to use it after water-cooling. However, it is not concerned about quick-drying property, thus the lubricant has a similar problem to the one disclosed in Patent document 1.

**[0016]** Further, in Patent document 3 disclosing an art, there is no description about heat resistance and water resistance required to the lubricant to be applied before water-cooling and about quick-drying property after water-cooling. So, there is no suggestion how such a lubricant should be used in detail when the mandrel bar is water-cooled.

**[0017]** Accordingly, an object of the present invention is to provide a method of manufacturing seamless pipes in case of having a manufacturing process including water-cooling of mandrel bars, wherein sufficient productivity is maintained, and it is possible to effectively inhibit occurrence of flaws on the surface of the pipes even when the materials to be worked are stainless-steel or high-alloy steel, by forming a lubricant coating having excellent seizure resistance on the surface of the mandrel bars.

## Means for Solving the Problems

**[0018]** The present inventors have been carried out serious studies. As the result, the present inventors have acquired following ideas for solving the above problems.

(1) By coating a lubricant onto a hot mandrel bar right after elongation-rolling and before water-cooling, it is capable to obtain a coating having sufficient thickness and to sufficiently dry the coating within a short period of time.

(2) The lubricant to be coated before water-cooling (hereinafter, refer to "the first liquid".) is required to have heat resistance to the temperature up to 400°C and water resistance not to be washed away by the following water-cooling step.

(3) However, even though the first liquid has the above heat resistance, the coating to be formed tends to be porous due to high temperature, so its adhesion to the surface of the mandrel bar is not sufficient. Therefore, without any after-treatment, seizure resistance of the mandrel bar is not sufficient. Such insufficiency can be remarkably observed particularly on working materials like stainless steel and high-alloy steel. Moreover, the coating may be partly peeled in contact with peripheral members before the next elongation-rolling process.

(4) In order to cover the shortcomings, by coating a lubricant (hereinafter, refer to "the second liquid".) having relatively low-viscosity over the first liquid after the water-cooling, the second liquid permeate into the porous coating, thereby it is capable to make the lubricant coating dense. In addition, by adding a component giving seizure resistance with the second liquid, it is capable to reinforce the seizure resistance of the coating. As seen above, it is capable to improve adhesion between the lubricant coating and the mandrel bar, and it is also capable to enhance seizure resistance of the coating. It is also possible to reinforce the lubricity of an area where lubricant coating is partly peeled.

(5) After the coating of the second liquid, there is no water-cooling step. Therefore, the second liquid is not required to have water resistance.

(6) However, as an elongation-rolling step is carried out soon after the coating, the second liquid needs to have quick-drying property. If a second liquid without having quick-drying property, non-dried coating is formed by the time of elongation-rolling; such a coating gives insufficient adhesion. While, if waited until the coating is sufficiently dried up, the delay negatively impacts on the pipes' manufacturing productivity.

**[0019]** The present invention is completed based on the above ideas, the summary of the invention is as follows.

**[0020]** The first aspect of the invention is a method of manufacturing seamless pipes comprising the steps of: the first coating step for forming a water-resistant coating by applying a first liquid onto a surface of a mandrel bar right after the previous elongation-rolling process; the water-cooling step for water-cooling the mandrel bar treated by the first coating step; the second coating step for applying a second liquid containing an inorganic lubricant which is excellent in high-temperature fluidity onto the surface of the mandrel bar treated by the water-cooling step, then making the second liquid permeate into the water-resistant coating to give it high-temperature adhesion, and an elongation-rolling step, wherein, the elongation-rolling step is characterizing in that the elongation-rolling of a hollow shell shall be started within 10 seconds after the second coating step by using the mandrel bar.

**[0021]** The wording "a (hot) mandrel bar right after the previous elongation-rolling process" means a mandrel bar inserted into a hollow shell of a temperature of 1100~1200°C and withdrawn from the newly-made element pipe (originally hollow shell) after elongation-rolling. The temperature of the bar is usually 100~400°C. Also, the wording "water resistance" means that normally even if a lubricant on the surface of the mandrel bar is water-sprayed, more than half (50 mass %) of the lubricant can be remained without being washed away. The evaluation method in detail will be described later. Further, the wording "high-temperature adhesion" means that a lubricant coating adhered on the surface of the mandrel bar cannot easily come off by the contact of a hollow shell, even though the bar is inserted into the hollow shell of a temperature of 1100~1200°C. The evaluation in detail is made based on whether or not seizing between a mandrel bar and the inner surface of a hollow shell occurs.

**[0022]** The second aspect of the invention is a method of manufacturing seamless pipes according to the first aspect of the invention, wherein the first liquid is an aqueous solution or an aqueous dispersion containing 20~40 mass % of a solid lubricant and 10~30 mass % of a water dispersible resin, and the second liquid is an aqueous solution or an aqueous dispersion containing 5~30 mass % of one or more selected from a group consisting of amine borate, potassium borate, potassium (or sodium) molybdate, and potassium (or sodium) carbonate, together with 0~30 mass % of a solid lubricant.

**[0023]** The third aspect of the invention is a method of manufacturing seamless pipes according to the second aspect of the invention, wherein the solid lubricant is graphite, mica, or a mixture thereof.

**[0024]** The fourth aspect of the invention is a method of manufacturing seamless pipes according to any one of the first to the third aspects of the invention, wherein the first coating step is carried out with a mandrel bar of 100~400°C, and the second coating step is carried out with a mandrel bar of 60~150°C.

**[0025]** The fifth aspect of the invention is a method of manufacturing seamless pipes according to according to any

one of the first to the fourth aspects of the invention, wherein material of the hollow shell is an alloy steel containing 5 mass % or more of Cr or a stainless steel.

#### Effects of the Invention

**[0026]** According to the present invention, it is capable to provide a method for manufacturing seamless pipes in case of having a manufacturing process including water-cooling of mandrel bars, wherein sufficient productivity is maintained, and it is possible to effectively inhibit occurrence of flaws on the surface of the pipes even when the materials to be machined are stainless steel or high-alloy steel, by forming a lubricant coating having excellent seizure resistance on the surface of the mandrel bars.

**[0027]** Such effects and advantages of the invention will be made apparent from the best mode for carrying out the invention, which will be described as follows.

#### Brief Description of the Drawings

**[0028]**

FIG. 1 (A) is a schematic view showing an operation cycle of a mandrel bar of the present invention;  
FIG. 1 (B) is a schematic view showing an operation cycle of a mandrel bar of the prior art for reference;  
FIG. 2 is a diagrammatic view of testing machine for measuring seizure resistance; and  
FIG. 3 is a schematic view showing manufacturing steps of a seamless pipe.

#### Description of the reference numerals

**[0029]**

- 1 billet
- 2 rotary-hearth type heating furnace
- 3 piercing-rolling apparatus
- 4 hollow shell
- 4a element pipe
- 5 mandrel mill
- 5a mandrel bar
- 6 sizing mill
- 7 cooling floor
- 40 rolling tester
- 41 flat-plate type tool
- 42 mill roll
- 43 test piece

#### Best Mode for Carrying Out the Invention

**[0030]** FIG. 1 (A) is a schematic view showing an operation cycle of a mandrel bar in the method for manufacturing seamless pipes of the first embodiment of the present invention. As a reference for comparison, FIG. 1 (B) is a schematic view showing a conventional operation cycle of a mandrel bar.

**[0031]** In FIG. 1 (A), a mandrel bar provided for elongation-rolling of a pipe in a mandrel mill is withdrawn from an element pipe, the first liquid is coated onto the mandrel bar at a temperature of 100~400°C (so-called "return lubrication"). The coated mandrel bar is cooled down to 60~150°C by water shower, then the second liquid is coated on the surface of the cooled mandrel bar (so-called "insertion lubrication"). Later, the mandrel bar coated by the second liquid is again provided for elongation-rolling of a pipe in a mandrel mill. The period of time is within 10 seconds from coating of the second liquid to beginning of the next elongation-rolling.

**[0032]** On the other hand, in a conventional operation cycle shown in FIG. 1 (B), the solution coated in the step of the first liquid coating has been used for the purpose of cooling the mandrel bar uniformly in the following cooling shower. The solution has been largely washed away. So, a lubricant to give a function of lubrication has applied during the step of second liquid coating. However, it takes time for drying the coating after the coating on the cooled mandrel bar, an area the so-called "lubrication booth" was needed for retention of mandrel bars for the certain period of time.

**[0033]** In the operation cycle of a mandrel bar in the method for manufacturing seamless pipes of the first embodiment of the invention, the lubrication booth as shown in FIG. 1 (B) is not necessary, and it is capable to begin the following

elongation-rolling right after inserter lubrication; thereby it is possible to enhance the manufacturing efficiency.

**[0034]** Here, the first liquid and the second liquid for the use of a method for manufacturing seamless pipes of the present embodiment will be explained as below.

(The first liquid)

**[0035]** In the first liquid, the base substance to be used is a solid lubricant including graphite, mica, or a mixture thereof. The upper limit of the content of the solid lubricant to the total amount of lubricant is 40 mass %, preferably 35 mass %. When the content of the solid lubricant is excessive, spray coating becomes difficult. In addition, excessive solid lubricant makes supply of the lubricant from a storage tank to production lines difficult. In the end, showery injection from a nozzle to the surface of a mandrel bar becomes impossible. On the other hand, the lower limit of the content of the solid lubricant to the total amount of lubricant is 20 mass %, preferably 25 mass %. When the content of the solid lubricant is too small, seizure resistance becomes lower. There is a fear of having flaws on the pipes when elongated and rolled ones are hollow shells made particularly of stainless steel and high-alloy steel.

**[0036]** Graphite may be either natural graphite or synthetic graphite. The purity of graphite is preferably 81% or more, and the average particle diameter is preferably 40 $\mu$ m or less. If the purity of graphite is less than 81%, the lubricity may be blocked by impurity mainly including inorganic substance such as alumina (Al<sub>2</sub>O<sub>3</sub>) and silica (SiO<sub>2</sub>) as misplaced materials. Also, if the average particle diameter is 40 $\mu$ m or more, such graphite is not properly dispersed in the first liquid. Therefore, the graphite cannot be supplied to the surface of a mandrel bar and of a hollow shell when used, and it cannot be stably stored under a condition of dispersion.

**[0037]** Mica may be either natural mica or synthetic mica. As natural mica, the examples include sericite, muscovite, and phlogopite. Further, the examples of synthetic mica include tetrasilicic potassium mica, tetrasilicic sodium mica, and fluor-phlogopite mica. Mica shall be included in order to improve lubricity of the lubricant under a high temperature around 1000°C. Similar to the effect of graphite, mica enhances the lubrication effect between a mandrel bar and a hollow shell. Graphite lowers friction coefficient; while, mica intervenes between a mandrel bar and a hollow shell and inhibits their fusion-bonding each other. Thus mica gives a function to inhibit occurrence of flaws on the inner surface of the hollow shell (element pipe). The average particle diameter of mica is preferably 40 $\mu$ m or less, and the purity of the same is preferably 81% or more. Because, if the average particle diameter becomes over 40 $\mu$ m, such particles cannot be properly dispersed, thereby smooth supply thereof to the lubrication surface becomes difficult. Also, if the purity of mica is less than 81%, the lubricity may be blocked by impurity mainly including inorganic substance such as alumina (Al<sub>2</sub>O<sub>3</sub>) and silica (SiO<sub>2</sub>) as misplaced materials.

**[0038]** A water dispersible resin to be mixed in the first liquid can make the solid lubricant as a base substance stably dispersed in water. Thus, the first liquid can be easily supplied when used. Therefore, the resin is included so as to make the first liquid uniformly spread on the surface of a mandrel bar and a hollow shell, and to give supplemental lubricity to the first liquid.

**[0039]** As a water dispersible resin to be mixed in the first liquid, for example, there may be a vinyl acetate copolymer, an acrylic acid ester copolymer, a methacrylic acid ester copolymer, copolymer of two or more thereof, and an ethylene-vinyl acetate copolymer. The average particle diameter of such water dispersible resins is preferably 40 $\mu$ m or less. If the average particle diameter becomes over 40 $\mu$ m, such particles cannot be properly dispersed, thereby smooth supply thereof to the lubrication surface becomes difficult. The upper limit of the content of the water dispersible resin to the total amount of lubricants is 30 mass %, preferably 25 mass %. If the content of water dispersible resin is excessive, viscosity of the lubricant becomes too high, therefore spray coating becomes difficult. On the other hand, the lower limit of the same is 10 mass %, preferably 15 mass %. If the content of the water dispersible resin is too small, the adhesiveness and water-resistance of the first liquid is not sufficient, thereby lubrication coating may be peeled.

**[0040]** Moreover, in order to enhance the high-temperature adhesiveness of the first liquid to the mandrel bar and to adequately disperse and settle out the solid lubricant such as graphite and mica, a water soluble polymer may be mixed in a range which water resistance is not damaged, for example 3 mass % or less. Examples of the water soluble polymer include methyl cellulose and carboxyl methyl cellulose, or polysaccharide such as alginate.

**[0041]** Water is contained so as to uniformly apply the lubricant on the surface of the mandrel bar. When the content becomes less than 30 mass %, viscosity of the lubricant becomes too high to deal with. On the other hand, when the content becomes over 70 mass %, bumping phenomenon is intensified, so uniform coating cannot be obtained. Moreover, adhesiveness of the first liquid is deteriorated, thereby it is impossible to obtain the desired amount of adhesion. Thus, content of water is 30~70 mass %, preferably 40~60 mass %.

**[0042]** In the above lubricant, if necessary, 0.01~2.0 mass % of the following respective materials can be added: a generally marketed silicone-series defoamant such as dimethyl siloxane; and an antiseptic agent including a thiazoline series compound such as 1,2-benzisothiazoline-3-on, a triazine series compound such as Hexahydro-1,3,5-tris (2-hydroxyethyl)-S-triazine, and a pyridine series compound such as sodium 2-pyridine thiol-1-oxide. When a defoamant is added, it inhibits foaming of the lubricant; when an antiseptic agent is added, it inhibits decay of the same.

**[0043]** In the amount of the first liquid to be adhered on the surface of the mandrel bar, the amount of the solid lubricant therein is 50~150g/m<sup>2</sup>, preferably 80~120g/m<sup>2</sup>. When the amount of the first liquid is excessive, coating having more than necessary thickness for seizure resistance is formed, it is economically disadvantageous. While, when the amount of the adhered first liquid is too small, sufficient seizure resistance performance cannot be obtained.

(The second liquid)

**[0044]** The second liquid to be applied onto the surface of the mandrel bar after water-cooling is an aqueous solution or an aqueous dispersion containing: 5~30 mass % of one or more selected from a group consisting of amine borate, potassium borate, potassium molybdate or sodium molybdate, and potassium carbonate or sodium carbonate; and 0~30 mass % of a solid lubricant.

**[0045]** Available amine borate may be any amines which can form water soluble salt, for instance, there may be monoethanol amine, monoisopropanol amine, diethanol amine, and triethanol amine.

**[0046]** To the second liquid, a solid lubricant may be added, if necessary. As available solid lubricants, similar to the first liquid, graphite, mica, or a mixture thereof may be used. In addition to these, or alone, it is possible to use layered silicate.

**[0047]** The upper limit of the content of the solid lubricant to be mixed in the second liquid, it is 40 mass % to the total amount of the second liquid, preferably 30 mass %. When the amount of the above solid lubricant is excessive, the second liquid has a difficulty to permeate into the porous coating made of the first liquid. Therefore, adhesiveness of the lubrication coating cannot be sufficiently improved. On the other hand, when no solid lubricant is used in the second liquid, the lubrication coating to be formed may lack the seizure resistance.

Examples

**[0048]** The first liquid and the second liquid described above were coated on the surface of test pieces, then the following evaluations thereof were carried out.

(1) Water resistance

**[0049]** Eight kinds of the first liquids whose composition are shown in Table 1 (30 mass % of graphite, 5~40 mass % of water dispersible resin) were respectively spray-coated on metal test pieces heated at 300°C so as the amount of adhesion to be about 100g/m<sup>2</sup> and left them for 5 seconds. Then, the individual test pieces were exposed to a cooling-shower continuously until the water did not come to a boil (about 100°C). The condition of the cooling-shower is as follows.

Nozzle: shower nozzle  
Water pressure: about 0.2MPa  
Water flow: 10L/min  
Water temperature: 20~25°C

Graphite used was scaly graphite (purity: 81% or more); water dispersible resin was a mixture of a vinyl acetate series resin and a vinyl acetate-acryl series resin at a ratio of 1:1. The peeling conditions of the coating under the above circumstances were evaluated in accordance with the following criteria.

(Criteria of Water resistance evaluation)

**[0050]**

○: Little coating was peeled (the peeled portion was less than 15% of the total);  
△: 15% or more and less than 80% of the coating was peeled; and  
×: almost all coating was peeled (the peeled portion was 80% or more of the total).

The results are shown in Table 1. The results of spray coatability observed in the above test are also shown in Table 1. The spray coatability was evaluated in accordance with the following criteria.

(Criteria of Spray coatability evaluation)

**[0051]**

- : It was possible to uniformly spray on the entire surface for coating;  
 Δ: the coating became slightly uneven over the entire surface for coating; and  
 × : the coating became uneven over the entire surface for coating.

[0052]

| (Table 1) |                         | (mass %)          |                  |
|-----------|-------------------------|-------------------|------------------|
| Graphite  | Water dispersible resin | Spray coatability | Water resistance |
| 30.0      | 40.0                    | ×                 | -                |
|           | 35.0                    | ×                 | -                |
|           | 30.0                    | Δ                 | ○                |
|           | 25.0                    | ○                 | ○                |
|           | 20.0                    | ○                 | ○                |
|           | 15.0                    | ○                 | ○                |
|           | 10.0                    | ○                 | Δ                |
|           | 5.0                     | ○                 | ×                |

## (2) Evaluation tests for drying property and seizure resistance

**[0053]** Drying property and seizure resistance were evaluated by using a rolling tester 40 diagrammatically shown in FIG. 2. This rolling tester 40 is to demonstrate a condition of elongation-rolling on a single stand of a mandrel mill. A heated test piece 43 was rolled between a roll 42 and a flat-plate type tool 41 movably disposed in the rolling direction (the right-and-left direction in FIG. 2) and lubricant had been coated on the surface. The damage situation of the flat-plate type tool 41 was observed after the rolling.

**[0054]** For a test piece 43, used was a 18 Cr-8 Ni stainless steel plate (SUS 304) of 20mm in width, 10mm in thickness, and 250mm in length. Onto the flat-plate type tool 41, five kinds of the first liquids of which compositions are shown in Table 2 (three kinds of Examples, and two kinds of Comparative examples) were spray-coated so as the coating amount to be about 35g/m<sup>2</sup>, and it was cooled down to 80°C by water. Later, the second liquid of eight kinds of Examples (A~H) of which compositions are shown in Table 3 and three kinds of Comparative examples (I~K) of which compositions are shown in Table 4 were spray-coated over the coating of the first liquid so as the coating amount to be about 20g/m<sup>2</sup>. Finally, in order to coincide with a pitch of operation cycle of the actual mandrel mill, drying property was checked by finger touch after 10 seconds from the lubricant's coating, and downward rolling by the roll 42 was carried out.

**[0055]** The elongation condition was as follows.

Heating temperature: 1000°C  
 Circumferential velocity of the roll: 392.5mm/sec  
 Velocity of the tool movement: 15mm/sec  
 Rolling reduction: 30%.

[0056]

(Table 2)

| The first liquid        |         | (mass %) |      |                     |      |
|-------------------------|---------|----------|------|---------------------|------|
|                         | Example |          |      | Comparative example |      |
|                         | 1       | 2        | 3    | 4                   | 5    |
| Graphite                | 30.0    | -        | 15.0 | 45.0                | 15.0 |
| Mica                    | -       | 30.0     | 15.0 | -                   | -    |
| Water dispersible resin | 25.0    | 25.0     | 25.0 | 25.0                | 25.0 |
| Dispersant              | 2.0     | 2.0      | 2.0  | 2.0                 | 2.0  |

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(continued)

|       | Example |      |      | Comparative example |      |
|-------|---------|------|------|---------------------|------|
| Water | 43.0    | 43.0 | 43.0 | 28.0                | 58.0 |

[0057]

(Table 3)

| The second liquid (Examples) (mass %) |         |      |      |      |      |      |      |      |
|---------------------------------------|---------|------|------|------|------|------|------|------|
|                                       | Example |      |      |      |      |      |      |      |
|                                       | A       | B    | C    | D    | E    | F    | G    | H    |
| Graphite                              | 20.0    | -    | 15.0 | 20.0 | -    | 20.0 | 20.0 | 20.0 |
| Mica                                  | -       | 20.0 | 15.0 | -    | -    | -    | -    | -    |
| Amine borate                          | -       | -    | -    | -    | 30.0 | -    | -    | -    |
| Potassium borate                      | -       | -    | -    | 5.0  | -    | -    | -    | -    |
| Sodium molybdate                      | 15.0    | 15.0 | 15.0 | -    | -    | -    | -    | -    |
| Potassium molybdate                   | -       | -    | -    | -    | -    | 15.0 | -    | -    |
| Sodium carbonate                      | -       | -    | -    | -    | -    | -    | 15.0 | -    |
| Potassium carbonate                   | -       | -    | -    | -    | -    | -    | -    | 15.0 |
| Dispersant                            | 2.0     | 2.0  | 2.0  | 2.0  | 2.0  | 2.0  | 2.0  | 2.0  |
| Water                                 | 63.0    | 63.0 | 53.0 | 73.0 | 68.0 | 63.0 | 63.0 | 63.0 |

[0058]

(Table 4)

| The second liquid (Comparative examples) (mass %) |                     |      |      |
|---------------------------------------------------|---------------------|------|------|
|                                                   | Comparative example |      |      |
|                                                   | I                   | J    | K    |
| Graphite                                          | 35.0                | 20.0 | 20.0 |
| Mica                                              | -                   | -    | -    |
| Amine borate                                      | -                   | 3.0  | 35.0 |
| Potassium borate                                  | -                   | -    | -    |
| Sodium molybdate                                  | 15.0                | -    | -    |
| Potassium molybdate                               | -                   | -    | -    |
| Sodium carbonate                                  | -                   | -    | -    |
| Potassium carbonate                               | -                   | -    | -    |
| Dispersant                                        | 2.0                 | 2.0  | 2.0  |
| Water                                             | 48.0                | 75.0 | 43.0 |

[0059] Graphite used was scaly graphite (purity: 81% or more); water dispersible resin was a mixture of a vinyl acetate series resin and a vinyl acetate-acryl series resin at a ratio of 1:1.

[0060] The drying property of the lubricant and seizing situation of the flat-plate type tool 41 after rolling were recorded in accordance with the following criteria.

(Criteria of drying property)

[0061]

- 5 Dried: ○  
Drying was slightly uncompleted: △  
Drying was not done: ×

(Criteria of seizing)

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[0062]

- 15 No seizing was observed: ○  
A little seizing was observed: △  
Seizing was largely observed: ×

[0063] The results of the drying property evaluation and seizure resistance evaluation are shown in Tables 5 and 6.

[0064]

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(Table 5)

Drying property

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| The 2nd liquid | A | B | C | D | E | F | G | H | I | J | K |
|----------------|---|---|---|---|---|---|---|---|---|---|---|
| The 1st liquid |   |   |   |   |   |   |   |   |   |   |   |
| 1              | ○ | ○ | ○ | △ | △ | ○ | ○ | ○ | ○ | △ | × |
| 2              | ○ | ○ | ○ | △ | △ | ○ | ○ | ○ | ○ | △ | × |
| 3              | ○ | ○ | ○ | △ | △ | ○ | ○ | ○ | ○ | △ | × |
| 4              | — | — | — | — | — | — | — | — | — | — | — |
| 5              | ○ | ○ | ○ | △ | △ | ○ | ○ | ○ | ○ | △ | × |

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[0065] The first liquid 4 could not be tested due to the unavailability of spray-coating. The second liquids D and J were not favorable in drying property as these contained a lot of water. Further, the second liquids E and K were not favorable in their drying property as these contained a lot of amine borate.

[0066]

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(Table 6)

Seizure resistance

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| The 2nd liquid | A | B | C | D | E | F | G | H | I | J | K |
|----------------|---|---|---|---|---|---|---|---|---|---|---|
| The 1st liquid |   |   |   |   |   |   |   |   |   |   |   |
| 1              | ○ | ○ | ○ | △ | △ | ○ | ○ | ○ | × | × | × |
| 2              | ○ | ○ | ○ | △ | △ | ○ | ○ | ○ | × | × | × |
| 3              | ○ | ○ | ○ | △ | △ | ○ | ○ | ○ | × | × | × |
| 4              | — | — | — | — | — | — | — | — | — | — | — |
| 5              | × | × | × | × | × | × | × | × | × | × | × |

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[0067] The first liquid 4 could not be tested due to the unavailability of spray-coating. The first liquid 5 had an insufficient seizure resistance because of the small amount of solid lubricant. The second liquids D and E had an insufficient seizure

resistance because of the decline of drying property. The second liquid I was not good at permeability due to the large amount of solid lubricant, thereby the seizure resistance was insufficient. The second liquid K was not good at permeability due to the large amount of inorganic lubricant, thereby the seizure resistance was insufficient. The second liquid J had an insufficient seizure resistance because of the small amount of inorganic lubricant.

[0068] The above has described the present invention associated with the most practical and preferred embodiments thereof. However, the invention is not limited to the embodiments disclosed in the specification. Thus, the invention can be appropriately varied as long as the variation is not contrary to the subject substance and conception of the invention which can be read out from the claims and the whole contents of the specification. It should be understood that a method of manufacturing seamless pipes with such an alternation are included in the technical scope of the invention.

## Claims

1. A method of manufacturing seamless pipes comprising the steps of:

a first coating step for forming a water-resistant coating by applying a first liquid onto a surface of a mandrel bar right after a previous elongation-rolling process;  
a water-cooling step for water-cooling said mandrel bar treated by said first coating step;  
a second coating step for applying a second liquid containing an inorganic lubricant which is excellent in high-temperature fluidity onto the surface of said mandrel bar treated by said water-cooling step, then making said second liquid permeate into said water-resistant coating to give it high-temperature adhesion, and an elongation-rolling step,

wherein, said elongation-rolling step is **characterizing in that** the elongation-rolling of a hollow shell shall be started within 10 seconds after said second coating step by using said mandrel bar.

2. A method of manufacturing seamless pipes according to claim 1, wherein said first liquid is an aqueous solution or an aqueous dispersion containing 20~40 mass % of a solid lubricant and 10~30 mass % of a water dispersible resin, and said second liquid is an aqueous solution or an aqueous dispersion containing 5~30 mass % of one or more selected from a group consisting of amine borate, potassium borate, potassium (or sodium) molybdate, and potassium (or sodium) carbonate, together with 0~30 mass % of a solid lubricant.

3. A method of manufacturing seamless pipes according to claim 2, wherein said solid lubricant is graphite, mica, or a mixture thereof.

4. A method of manufacturing seamless pipes according to any one of claims 1~3, wherein said first coating step is carried out with a mandrel bar of 100~400°C, and said second coating step is carried out with a mandrel bar of 60~150°C.

5. A method of manufacturing seamless pipes according to any one of claims 1~4, wherein material of said hollow shell is an alloy steel containing 5 mass % or more of Cr or a stainless steel.

FIG. 1 (A)

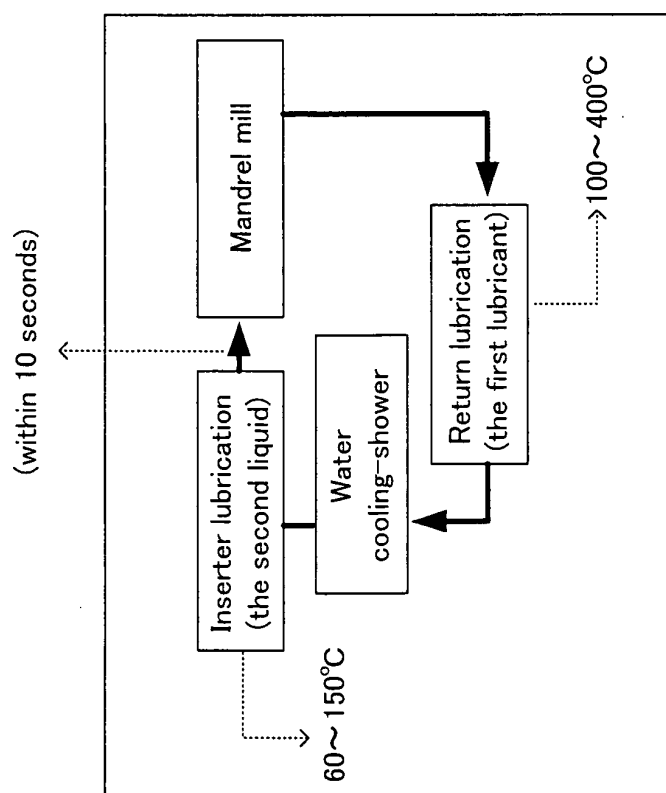


FIG. 1 (B)

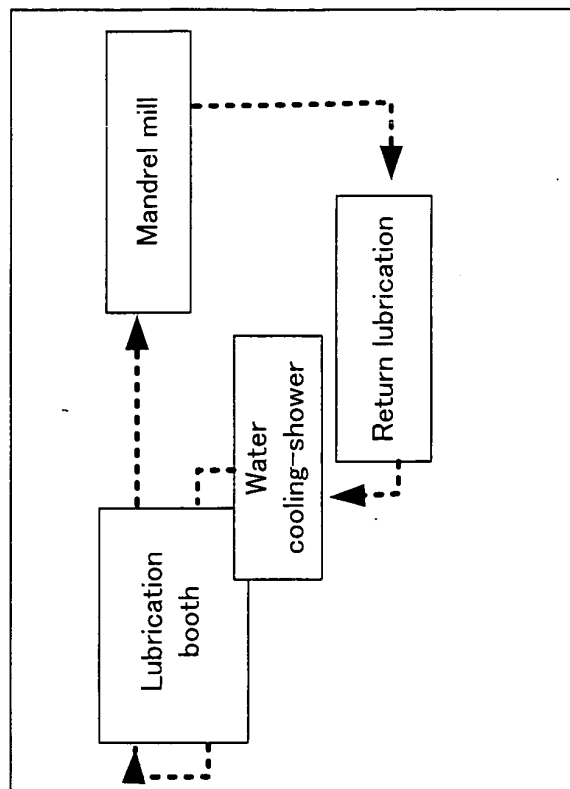


FIG. 2

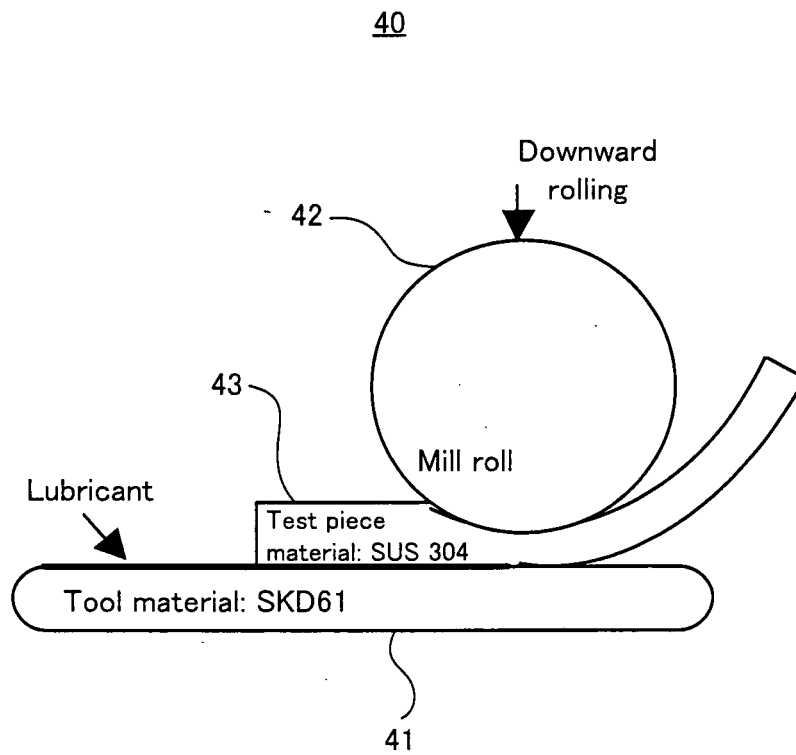
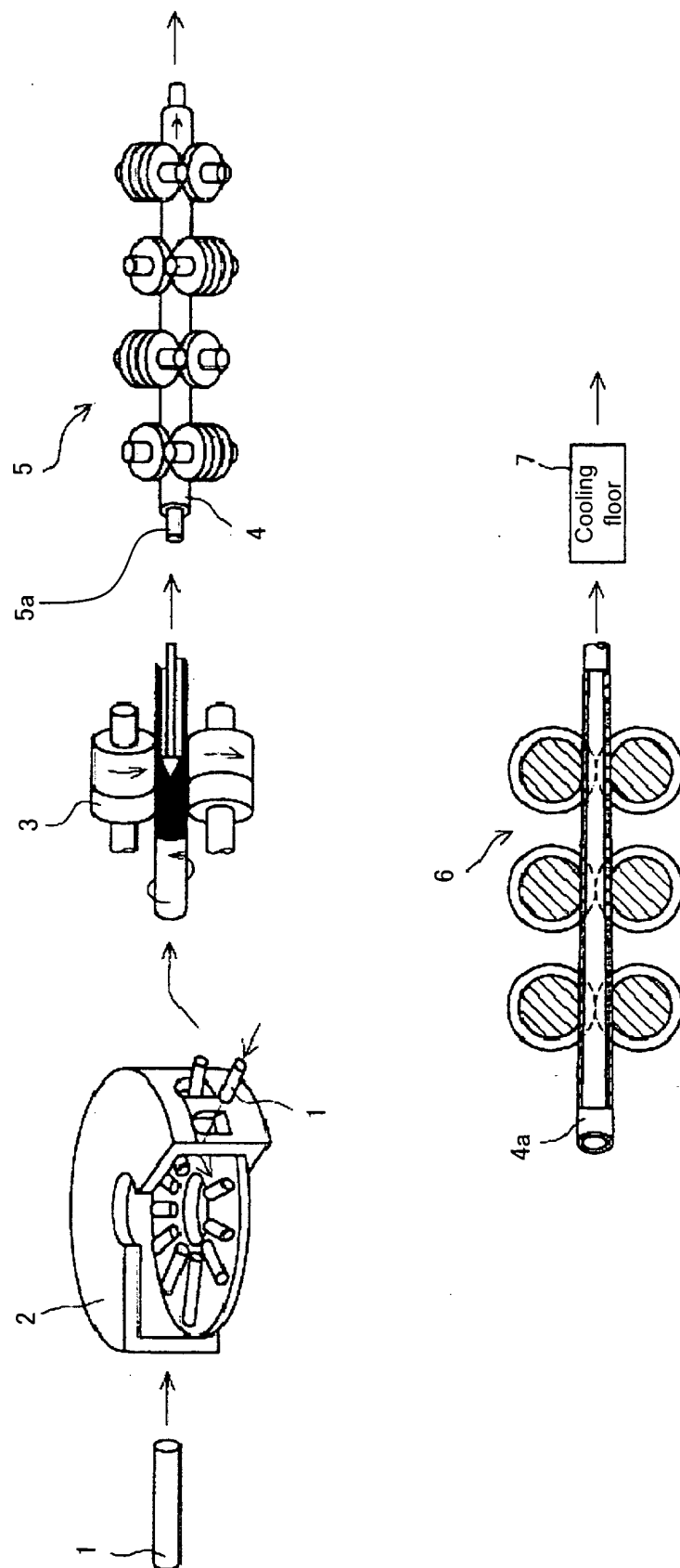


FIG. 3



## INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2006/306928

| <b>A. CLASSIFICATION OF SUBJECT MATTER</b><br><b>B21B25/04</b> (2006.01) , <b>B21B17/04</b> (2006.01)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |                                                                                                                                                                                                     |                                                                                |
|--------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------------|--------------------------------------------------------------------------------|
| According to International Patent Classification (IPC) or to both national classification and IPC                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                                                                                                                                                                                                     |                                                                                |
| <b>B. FIELDS SEARCHED</b><br>Minimum documentation searched (classification system followed by classification symbols)<br><b>B21B25/04</b> (2006.01) , <b>B21B17/04</b> (2006.01)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  |                                                                                                                                                                                                     |                                                                                |
| Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched<br>Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2006<br>Kokai Jitsuyo Shinan Koho 1971-2006 Toroku Jitsuyo Shinan Koho 1994-2006                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                    |                                                                                                                                                                                                     |                                                                                |
| Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                       |                                                                                                                                                                                                     |                                                                                |
| <b>C. DOCUMENTS CONSIDERED TO BE RELEVANT</b>                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                                                                                                                                                                                     |                                                                                |
| Category*                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                          | Citation of document, with indication, where appropriate, of the relevant passages                                                                                                                  | Relevant to claim No.                                                          |
| A                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | JP 61-252293 A (Hoechst Gosei Kabushiki Kaisha),<br>10 November, 1986 (10.11.86),<br>Claims; page 2, lower left column, lines 9 to 11; example 5<br>(Family: none)                                  | 1-5                                                                            |
| A                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                  | JP 63-230797 A (Sumitomo Metal Industries, Ltd.),<br>27 September, 1988 (27.09.88),<br>Claims; page 2, lower left column, line 1 to page 3, upper right column, line 10; examples<br>(Family: none) | 1-5                                                                            |
| <input checked="" type="checkbox"/> Further documents are listed in the continuation of Box C. <input type="checkbox"/> See patent family annex.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                   |                                                                                                                                                                                                     |                                                                                |
| * Special categories of cited documents:<br>"A" document defining the general state of the art which is not considered to be of particular relevance<br>"E" earlier application or patent but published on or after the international filing date<br>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)<br>"O" document referring to an oral disclosure, use, exhibition or other means<br>"P" document published prior to the international filing date but later than the priority date claimed<br>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention<br>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone<br>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art<br>"&" document member of the same patent family |                                                                                                                                                                                                     |                                                                                |
| Date of the actual completion of the international search<br>01 June, 2006 (01.06.06)                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                              |                                                                                                                                                                                                     | Date of mailing of the international search report<br>13 June, 2006 (13.06.06) |
| Name and mailing address of the ISA/<br>Japanese Patent Office                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                     |                                                                                                                                                                                                     | Authorized officer                                                             |
| Facsimile No.                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                                      |                                                                                                                                                                                                     | Telephone No.                                                                  |

Form PCT/ISA/210 (second sheet) (April 2005)

## INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2006/306928

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

| Category* | Citation of document, with indication, where appropriate, of the relevant passages                                                                             | Relevant to claim No. |
|-----------|----------------------------------------------------------------------------------------------------------------------------------------------------------------|-----------------------|
| A         | JP 8-165489 A (Sumitomo Metal Industries, Ltd.),<br>25 June, 1996 (25.06.96),<br>Claims; column 4, line 40 to column 6, line 45;<br>examples<br>(Family: none) | 1-5                   |

Form PCT/ISA/210 (continuation of second sheet) (April 2005)

**REFERENCES CITED IN THE DESCRIPTION**

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**Patent documents cited in the description**

- JP 63230797 A [0011]
- JP 8165489 A [0011]
- JP 2004034072 A [0011]