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- **SATO, Kazuhiro**  
Tokyo 100-8071 (JP)
- **NISHITANI, Norihisa**  
Tokyo 100-8071 (JP)
- **KAWANISHI, Yoshihiro**  
Tokyo 100-8071 (JP)
- **SHIMAYA, Keisuke**  
Tokyo 103-0027 (JP)
- **TESHIGAWARA, Tomoya**  
Tokyo 103-0027 (JP)
- **FUJIWAKI, Takeshi**  
Tokyo 103-0027 (JP)
- **TAGUCHI, Hiroki**  
Tokyo 103-0027 (JP)

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(71) Applicant: **Nippon Steel & Sumitomo Metal Corporation**  
**Tokyo 100-8071 (JP)**

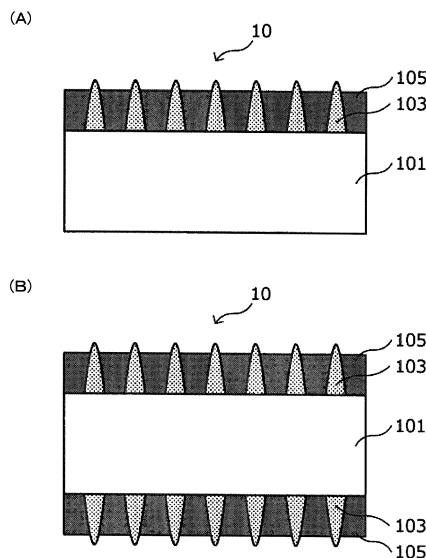
(72) Inventors:  
• **YASUI, Atsushi**  
**Tokyo 100-8071 (JP)**

(74) Representative: **Vossius & Partner**  
**Patentanwälte Rechtsanwälte mbB**  
**Siebertstrasse 3**  
**81675 München (DE)**

(54) **TREATED SURFACE STEEL BAND AND METHOD FOR MANUFACTURING TREATED SURFACE STEEL BAND**

(57) A surface treated steel strip according to the present invention includes: a base steel sheet; a zinc phosphate coating film layer made of needle zinc phosphate crystals which are formed in an island shape on a surface of the base steel sheet; and a lubricating coating film layer covering the surface of the base steel sheet and a part of the zinc phosphate coating film layer, and containing at least a lubricating component, in which an area ratio of the zinc phosphate crystals exposed from a surface of the lubricating coating film layer is 25% to 90%.

FIG.2



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## Description

[Technical Field]

5 **[0001]** The present invention relates to a surface treated steel strip and a manufacturing method of a surface treated steel strip.

[Background Art]

10 **[0002]** Among plastic workings of a steel sheet, in the working of an automobile transmission component or the like which has to be subjected to multistage press forming under a high contact pressure, there has been performed a phosphate soap treatment in which phosphate crystals mainly containing zinc phosphate are precipitated on a surface of a steel sheet to form a phosphate coating film, and then a reactive soap coating film containing sodium stearate (alkali soap) as a main component covers, as an upper layer, the phosphate coating film, in order to prevent seizing or die galling of the steel sheet with respect to a die. However, the phosphate soap treatment requires a long chemical reaction time for forming the phosphate coating film and the reactive soap coating film, so that a production cost is increased. Further, an unreacted soap component adheres to a die as a press scum when performing press forming, so that it is important to frequently clean the die.

15 **[0003]** Accordingly, in order to prevent seizing or die galling in a multistage plastic working such as multistage press forming under a high contact pressure, without performing the reactive soap treatment which requires a long time, there has been disclosed a technique of forming a lubricating coating film containing a lubricating component as an upper layer of a phosphate coating film (refer to the following Patent Document 1, for example).

[Prior Art Document]

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[Patent Document]

**[0004]** [Patent Document 1] Japanese Laid-open Patent Publication No. 2013-104125

30 [Disclosure of the Invention]

[Problems to Be Solved by the Invention]

35 **[0005]** Here, the treatment such as one disclosed in the aforementioned Patent Document 1 is often carried out in a state of a steel strip in a practical sense. When an object to be treated is a steel strip, in a slit process to be performed in a previous stage of a multistage plastic working, it is necessary to feed a steel sheet from the steel strip with the use of pinch rolls. Further, in order to secure die galling resistance when performing multistage press forming, it becomes important to lower a coefficient of static friction of the steel sheet fed from the steel strip, but, an excessive reduction in the coefficient of static friction increases a probability that the steel sheet slips at the pinch rolls to cause blanking. As described above, roll feedability (roll slip resistance) and the die galling resistance when performing the multistage press forming under the high contact pressure are properties which are in a mutual trade-off relation. In order to continuously perform the multistage plastic working while feeding the steel sheet from the steel strip, it is demanded to satisfy both of the roll slip resistance and the die galling resistance as described above.

40 **[0006]** Accordingly, the present invention has been made in view of the above-described problems, and an object of the present invention is to provide a surface treated steel strip and a manufacturing method of a surface treated steel strip capable of realizing both of roll slip resistance and die galling resistance being properties which are contrary to each other.

[Means for Solving the Problems]

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**[0007]** As a result of earnest studies for solving the above-described problems, the present inventor obtained the following findings.

55 (A) Before forming a lubricating coating film layer on a surface of a base steel sheet being a base material, zinc phosphate crystals are precipitated in an island shape on a surface of the base steel sheet. At this time, shapes of the zinc phosphate crystals are controlled to make a part of the zinc phosphate crystals to be exposed (protruded) from a surface of the lubricating coating film layer. Consequently, a surface treated steel strip in a form in which concave and convex made by the zinc phosphate crystals are exposed (protruded) from the surface of the lubricating

coating film layer, is finally obtained.

(B) The zinc phosphate crystals do not have a lubricating property, so that a coefficient of static friction of the surface treated steel strip in which the concave and the convex made by the zinc phosphate crystals are formed on the surface of the lubricating coating film layer, becomes larger than a coefficient of static friction of the lubricating coating film layer itself. The coefficient of static friction of the surface treated steel strip is correlative to an area ratio of the zinc phosphate crystals exposed from the surface of the lubricating coating film layer (sometimes referred to as an exposed area ratio, hereinafter). Specifically, by controlling the exposed area ratio of the zinc phosphate crystals, it becomes possible to control the coefficient of static friction of the surface treated steel strip.

(C) When the surface treated steel strip is conveyed in a state of being sandwiched between a pair of pinch rolls, a pressure (contact pressure) is applied to the surface treated steel strip by the pinch rolls, but, the pressure is not high enough to crush the zinc phosphate crystals exposed from the lubricating coating film layer. In other words, the concave and the convex at the surface of the lubricating coating film layer are not flattened by the pinch rolls. Accordingly, a value of the coefficient of static friction of the surface treated steel strip under the pinch rolls is maintained to be a large value. As a result of this, a slip of the surface treated steel strip under the pinch rolls is suppressed, which improves the roll slip resistance under the pinch rolls (under a low contact pressure).

(D) On the other hand, when multistage press forming is performed on the surface treated steel strip by a pressing device disposed on a downstream side of the pinch rolls, a very large pressure (contact pressure) is applied to the surface treated steel strip, so that the zinc phosphate crystals exposed from the lubricating coating film layer are crushed to flatten the concave and the convex at the surface of the lubricating coating film layer. Accordingly, the coefficient of static friction of the surface treated steel strip under the pressing device becomes a coefficient of static friction which the lubricating coating film layer originally has. As a result of this, since the original lubricating property (slidability) of the surface treated steel strip is exhibited under the pressing device, the die galling resistance under the pressing device (under a high contact pressure) improves.

**[0008]** The present invention has been completed based on the above-described findings, and the gist thereof is as follows.

**[0009]**

[1] A surface treated steel strip includes:

- a base steel sheet;
- a zinc phosphate coating film layer made of needle zinc phosphate crystals which are formed in an island shape on a surface of the base steel sheet; and
- a lubricating coating film layer covering the surface of the base steel sheet and a part of the zinc phosphate coating film layer, and containing at least a lubricating component, in which an area ratio of the zinc phosphate crystals exposed from a surface of the lubricating coating film layer is 25% to 90%.

[2] In the surface treated steel strip described in [1], the zinc phosphate crystals have an average grain diameter in a major axis direction of 25  $\mu\text{m}$  to 70  $\mu\text{m}$ , and have an average grain diameter in a minor axis direction of 3  $\mu\text{m}$  to 10  $\mu\text{m}$ .

[3] In the surface treated steel strip described in [1] or [2], a coating weight of the zinc phosphate coating film layer is 1.5 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup> per one side.

[4] In the surface treated steel strip described in any one of claims [1] to [3], a coating weight of the lubricating coating film layer is 1.0 g/m<sup>2</sup> to 12.0 g/m<sup>2</sup> per one side.

[5] In the surface treated steel strip described in any one of [1] to [4], the lubricating coating film layer contains an alkali metal silicate whose molar ratio represented by  $\text{SiO}_2/\text{M}_2\text{O}$  (M is alkali metal) is 2 to 5, and a polymeric wax made of at least either a polyethylene wax or a polypropylene wax having an average grain diameter of 0.1  $\mu\text{m}$  to 3.0  $\mu\text{m}$ , and a solid content of the alkali metal silicate and a solid content of the polymeric wax with respect to a total solid content mass of the lubricating coating film layer are 60 mass% to 90 mass% and 5 mass% to 40 mass%, respectively.

[6] A manufacturing method of a surface treated steel strip includes:

- a thermal refining step of performing thermal refining on a surface of a base steel sheet by utilizing a surface conditioner containing titanium colloid;
- a zinc phosphate coating film layer forming step of making needle crystals of zinc phosphate grow in an island shape on the surface of the base steel sheet after being subjected to the thermal refining of surface to form a zinc phosphate coating film layer on the surface of the base steel sheet; and
- a lubricating coating film layer forming step of forming a lubricating coating film layer by coating a lubricating

treatment agent containing at least a lubricating component to the surface of the base steel sheet and the zinc phosphate coating film layer so that a coating weight of the lubricating treatment agent becomes 1.0 g/m<sup>2</sup> to 12.0 g/m<sup>2</sup> per one side, and making an area ratio of the zinc phosphate crystals exposed from a surface of the lubricating coating film layer to be 25% to 90%.

[7] In the manufacturing method of the surface treated steel strip described in [6], the base steel sheet is heated in the zinc phosphate coating film layer forming step.

[8] In the manufacturing method of the surface treated steel strip described in [7], the base steel sheet is heated by applying steam to the base steel sheet.

#### [Effect of the Invention]

**[0010]** According to the present invention, it is possible to realize both of roll slip resistance under a low contact pressure and die galling resistance under a high contact pressure being properties which are contrary to each other.

#### [Brief Description of the Drawings]

#### **[0011]**

FIG. 1 is an explanatory diagram of a surface treated steel strip according to one embodiment of the present invention. FIG. 2(A) is an explanatory diagram schematically illustrating a configuration of the surface treated steel strip according to the same embodiment. FIG. 2(B) is an explanatory diagram schematically illustrating a configuration of the surface treated steel strip according to the same embodiment.

FIG. 3(A) is a front view illustrating one example of a zinc phosphate treatment bath with which a zinc phosphate coating film layer is formed on a surface of a steel strip. FIG. 3(B) is a plan view illustrating one example of the zinc phosphate treatment bath with which the zinc phosphate coating film layer is formed on the surface of the steel strip. FIG. 4 is a flow chart illustrating one example of a flow of a manufacturing method of the surface treated steel strip according to the same embodiment.

FIG. 5 is an explanatory diagram schematically illustrating a test method regarding die galling resistance.

FIG. 6 is an explanatory diagram schematically illustrating a test method regarding roll slip resistance.

FIG. 7 is an enlarged photograph of a surface treated steel strip of a comparative example.

FIG. 8 is an enlarged photograph of a surface treated steel strip of a comparative example.

FIG. 9 is an enlarged photograph of a surface treated steel strip of an example of the present invention.

FIG. 10 is an enlarged photograph of a surface treated steel strip of an example of the present invention.

#### [Best Mode for Carrying out the Invention]

**[0012]** Hereinafter, an example of embodiments of the present invention will be described while referring to the attached drawings. Note that in the present specification and the drawings, constituent elements having substantially the same functional configurations are denoted by the same reference numerals, and overlapped explanation thereof will be omitted.

(Regarding surface treated steel strip)

**[0013]** First, a surface treated steel strip according to one embodiment of the present invention will be described in detail while referring to FIG. 1 to FIG. 2B. FIG. 1 is an explanatory diagram for explaining a surface treated steel strip according to the present embodiment, and FIG. 2A and FIG. 2B are explanatory diagrams each schematically illustrating a configuration of the surface treated steel strip according to the present embodiment.

**[0014]** A surface treated steel strip 10 according to the present embodiment is obtained by performing a high-level lubricating treatment on a base steel sheet 101, as will be described below in detail. As schematically illustrated in FIG. 1, in a slit process to be performed in a previous stage of a multistage plastic working, such a surface treated steel strip 10 in a state of being wound in a coil shape is unwound by pinch rolls 1 and continuously passed in a predetermined sheet passage direction X. The passed surface treated steel strip 10 is subjected to a multistage presswork utilizing a die 2 in accordance with an intended product, to be worked into the intended product.

**[0015]** As already described above, in order to prevent seizing or die galling of the passed surface treated steel strip 10 (base steel sheet 101) with respect to the die 2, it is important to lower a coefficient of static friction of the surface treated steel strip 10, but, on the other hand, in order to stably unwind, by using the pinch rolls 1, the surface treated steel strip 10 in the state of being wound in the coil shape, it is important that the surface treated steel strip 10 has a

certain level of the coefficient of static friction. Accordingly, in the surface treated steel strip 10 according to the present embodiment, a surface treatment such as one to be described below in detail is performed on a surface of the base steel sheet 101 to be a base material to form a surface treated layer made of two layers, as illustrated in FIG. 2A and FIG. 2B.

**[0016]** As illustrated in FIG. 2A and FIG. 2B, the surface treated steel strip 10 according to the present embodiment has the base steel sheet 101 to be the base material, a zinc phosphate coating film layer 103 formed on the base steel sheet 101, and a lubricating coating film layer 105 formed on the zinc phosphate coating film layer 103. Note that the zinc phosphate coating film layer 103 and the lubricating coating film layer 105 may be formed only on one surface of the base steel sheet 101 as illustrated in FIG. 2A, and they may also be formed on two surfaces, which face each other, of the base steel sheet 101 as illustrated in FIG. 2B.

[Regarding base steel sheet 101]

**[0017]** The base steel sheet 101 is used as a base material of the surface treated steel strip 10. The base steel sheet 101 according to the present embodiment is not particularly limited, and it is possible to use a publicly-known steel sheet capable of realizing characteristics demanded with respect to an object manufactured through multistage plastic working processing at a subsequent stage. A manufacturing method and a material of such a publicly-known steel sheet are also not particularly limited, and it is possible to use a steel sheet manufactured by being appropriately subjected to publicly-known various processes of hot rolling, pickling, cold rolling, annealing, temper rolling, and so on after being subjected to a normal cast slab manufacturing process. Further, it is needless to say that such a base steel sheet 101 may be not only a carbon steel sheet but also a special steel sheet such as a stainless steel sheet or a high-alloy steel sheet.

**[0018]** As one example of such a base steel sheet 101, there can be cited a hot-rolled steel sheet containing chemical components as follows, for example.

**[0019]** A hot-rolled steel sheet being one example of the base steel sheet 101 contains, in mass%, C: 0.070% to 0.080%, Si: 0.030% to 0.080%, Mn: 1.15% to 1.30%, P: 0.015% to 0.028%, S: 0.000% to 0.040%, and the balance being composed of Fe and impurities.

**[0020]** By using such a hot-rolled steel sheet as the base steel sheet 101, it becomes possible to improve strength of an object to be manufactured.

[Regarding zinc phosphate coating film layer 103]

**[0021]** The zinc phosphate coating film layer 103 plays a role for increasing adhesiveness between the base steel sheet 101 and the lubricating coating film layer 105. This zinc phosphate coating film layer 103 is an aggregate of needle zinc phosphate crystals constituted of needle crystals of zinc phosphate precipitated through a chemical reaction on a surface of the base steel sheet 101. As schematically illustrated in FIG. 2A and FIG. 2B, the precipitated needle crystals of zinc phosphate do not completely cover the surface of the base steel sheet 101, and a part of the surface of the base steel sheet 101 remains in a state where it is not covered by the crystals of zinc phosphate. As a result of this, the needle crystals of zinc phosphate are distributed in an island shape on the surface of the base steel sheet 101, and the surface of the base steel sheet 101 where there is no needle crystal of zinc phosphate, exists as a flat portion. Note that the zinc phosphate coating film layer 103 appears in a state of being divided at a plurality of places on the surface of the base steel sheet 101 in a mutually independent manner. In the present invention, a state of the zinc phosphate coating film layer 103 which appears in a state of being divided at a plurality of places on the surface of the base steel sheet 101 in a mutually independent manner as described above, is referred to as an "island shape".

**[0022]** The zinc phosphate coating film layer 103 according to the present embodiment is formed on the base steel sheet 101 whose surface is subjected to thermal refining by using a specific surface conditioner as will be described later, and further, in a zinc phosphate coating film layer forming step of forming the zinc phosphate coating film layer 103, the base steel sheet 101 is heated, so that the zinc phosphate coating film layer 103 is constituted of needle crystals of zinc phosphate with larger ratio of grain diameters in a major axis direction and a minor axis direction. A coating weight of such a zinc phosphate coating film layer 103 is preferably set to 1.5 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup> per one side. The coating weight of the zinc phosphate coating film layer 103 is more preferably 3.0 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup> per one side. By making the coating weight of the zinc phosphate coating film layer 103 satisfy the range as described above, it becomes possible to make the lubricating coating film layer 105 to be brought into close contact with the base steel sheet 101 more securely, and even in a forming work under a high contact pressure, the zinc phosphate coating film layer 103 does not disappear, and the lubricating coating film layer 105 can be maintained to a final process of the forming work more securely.

**[0023]** Note that it is preferable that the needle crystals of zinc phosphate that constitute the zinc phosphate coating film layer 103 according to the present embodiment have an average grain diameter in a major axis direction of 25  $\mu$ m to 70  $\mu$ m, and have an average grain diameter in a minor axis direction of 3  $\mu$ m to 10  $\mu$ m. It is more preferable that the average grain diameter of the needle crystals of zinc phosphate is 25  $\mu$ m to 50  $\mu$ m in the major axis direction, and is 3

$\mu\text{m}$  to  $5\ \mu\text{m}$  in the minor axis direction. When the zinc phosphate crystals that constitute the zinc phosphate coating film layer 103 have such average grain diameters, it becomes possible to realize the adhesiveness as described above more securely.

**[0024]** Further, the needle crystals of zinc phosphate constituting the zinc phosphate coating film layer 103 according to the present embodiment are preferably needle crystals having a ratio of average grain diameters in the major axis direction and the minor axis direction of 2.5 or more. When the needle crystals of zinc phosphate having the average grain diameters as above are precipitated, it becomes possible to realize the adhesiveness between the base steel sheet 101 and the lubricating coating film layer 105 more securely.

**[0025]** This zinc phosphate coating film layer 103 can be formed by using a publicly-known treatment solution containing zinc phosphate and capable of precipitating needle crystals of zinc phosphate. Such a treatment solution is not particularly limited, and there can be cited, for example, a reactive zinc phosphate treatment solution for plastic working (more specifically, a reactive zinc phosphate treatment solution for plastic working in which a treatment time for complete covering is 20 seconds or more). When forming the zinc phosphate coating film layer 103, it is possible to make the zinc phosphate treatment solution as described above to be brought into contact with the base steel sheet 101 through a spray method or an immersion method, or it is possible to perform an electrolysis treatment by utilizing a reaction tank accommodating the zinc phosphate treatment solution as described above.

**[0026]** In order to make the zinc phosphate crystals precipitate in an island shape on the base steel sheet 101, it is only required to terminate the zinc phosphate treatment before the zinc phosphate crystals completely cover the entire surface of the base steel sheet 101. In order to achieve this, it is only required to regulate a contact time between the base steel sheet 101 and the zinc phosphate treatment solution or an electrolysis time to a short period of time. Specifically, it is only required that a commercially available zinc phosphate treatment solution for plastic working is used, and the treatment is terminated at a time shorter than an indicated treatment time. Regarding concrete treatment time and electrolysis condition, it is only required to specify the treatment time (electrolysis condition) capable of realizing the preferable coating weight as described above by previously examining a correspondence between the treatment time (or the electrolysis condition) and the coating weight. Further, it is also effective to heat the base steel sheet 101 in the zinc phosphate coating film layer forming step of forming the zinc phosphate coating film layer 103, in order to make the zinc phosphate crystals precipitate in an island shape on the base steel sheet 101.

**[0027]** It is possible to judge whether or not the zinc phosphate crystals precipitated through the treatment as described above are in an island shape, by performing a microscopic observation on the surface of the base steel sheet 101 after being subjected to the zinc phosphate treatment. Concretely, the surface of the base steel sheet 101 after being subjected to the zinc phosphate treatment is observed by a scanning electron microscope (SEM), and a case where an area of a flat portion determined through image processing is 30% or more may be judged that the zinc phosphate crystals are formed in an island shape. Such an observation of the zinc phosphate crystals may be performed before the formation of the lubricating coating film layer 105 to be described later, or it may be performed after the formation. However, when the observation is performed after the formation of the lubricating coating film layer 105, the zinc phosphate crystals are observed through the lubricating coating film layer 105, so that the SEM observation at a high acceleration voltage is performed. Concretely, by setting an acceleration voltage to 20 kV or more, it is possible to observe the crystals of zinc phosphate through the lubricating coating film layer 105, to thereby determine the area ratio of the flat portion. Note that in the observation before the formation of the lubricating coating film layer 105, it is possible to observe the zinc phosphate crystals even at a lower acceleration voltage.

**[0028]** Here, when forming the zinc phosphate coating film layer 103 on the base steel sheet 101, a thermal refining treatment of the surface of the base steel sheet 101 is performed by using a surface conditioner containing titanium colloid, previous to the zinc phosphate treatment as described above. The zinc phosphate crystals are precipitated from the surface of the base steel sheet 101 where a component of the surface conditioner is not adhered, and since the titanium colloid is a coarse colloidal particle, an area of the surface of the exposed base steel sheet 101 is suppressed. As a result of this, by performing the thermal refining treatment of surface by using the surface conditioner containing the titanium colloid, it becomes possible to make the needle crystals of zinc phosphate having the preferable average grain diameter ratio as described above to be precipitated more securely.

[Regarding lubricating coating film layer 105]

**[0029]** As schematically illustrated in FIG. 2A and FIG. 2B, the lubricating coating film layer 105 is a layer positioned on the surface of the base steel sheet 101, covering the surface of the base steel sheet 101 and at least a part of the zinc phosphate coating film layer 103, and having a surface from which at least a part of the zinc phosphate crystals is exposed. This lubricating coating film layer 105 is a layer containing at least a lubricating component, and preferably made of a binder component and a lubricating component.

**[0030]** In the surface treated steel strip 10 according to the present embodiment, since the zinc phosphate crystals in an island shape as described before are formed, the lubricating component and the binder component contained in the

lubricating coating film layer 105 are held among the zinc phosphate crystals in an island shape, as schematically illustrated in FIG. 2A and FIG. 2B. If a high contact pressure is applied to the surface of the surface treated steel strip 10 at a time of performing the multistage plastic working (at a time of performing the multistage presswork, for example), the lubricating component held among the zinc phosphate crystals in an island shape flows out between the base steel sheet 101 and a die. As a result of this, a coefficient of static friction of the lubricating coating film layer 105 is lowered to exhibit the lubricating property, and the die galling resistance is realized.

**[0031]** A coating weight of such a lubricating coating film layer 105 is 1.0 g/m<sup>2</sup> to 12.0 g/m<sup>2</sup> per one side, and an area ratio (an area ratio per unit area) of the zinc phosphate crystals exposed from the surface of the lubricating coating film layer 105 is 25% to 90%. By making the coating weight of the lubricating coating film layer 105 and the area ratio of the zinc phosphate crystals exposed from the surface of the lubricating coating film layer 105 fall within such ranges, it becomes possible to realize both of the die galling resistance and the roll slip resistance as described above.

**[0032]** A case where the coating weight of the lubricating coating film layer 105 becomes less than 1.0 g/m<sup>2</sup> is not preferable since an amount of the lubricating component held as the lubricating coating film layer 105 becomes insufficient, and sufficient die galling resistance cannot be realized. Further, a case where the coating weight of the lubricating coating film layer 105 exceeds 12.0 g/m<sup>2</sup> is not preferable since an amount of the lubricating component held as the lubricating coating film layer 105 becomes excessive, and the roll slip resistance cannot be realized. The coating weight per one side of the lubricating coating film layer 105 is more preferably 2.0 g/m<sup>2</sup> to 9.0 g/m<sup>2</sup>.

**[0033]** Note that the coating weight of the lubricating coating film layer 105 is also influenced by the coating weight of the zinc phosphate coating film layer 103. Specifically, when the coating weight of the zinc phosphate coating film layer 103 is small, the amount of the lubricating coating film layer 105 capable of being held also becomes small and, on the contrary, when the coating weight of the zinc phosphate coating film layer 103 is large, the amount of the lubricating coating film layer 105 capable of being held also becomes large. For example, when the coating weight of the zinc phosphate coating film layer 103 is 1.5 g/m<sup>2</sup> to 8.0 g/m<sup>2</sup>, the coating weight of the lubricating coating film layer 105 is preferably about 1.0 g/m<sup>2</sup> to 6.0 g/m<sup>2</sup>, and when the coating weight of the zinc phosphate coating film layer 103 is greater than 8.0 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup>, the coating weight of the lubricating coating film layer 105 is preferably about greater than 6.0 g/m<sup>2</sup> to 12.0 g/m<sup>2</sup>.

**[0034]** When the coating weight of the zinc phosphate coating film layer 103 is 1.5 g/m<sup>2</sup> to 8.0 g/m<sup>2</sup> per one side, it becomes possible to make the lubricating coating film layer 105 with the coating weight of about 1.0 g/m<sup>2</sup> to 6.0 g/m<sup>2</sup> to be favorably brought into close contact with the base steel sheet 101, resulting in that even in the forming work under a high contact pressure, the zinc phosphate coating film layer 103 does not disappear, and it becomes possible to maintain the lubricating coating film layer 105 to a final process of the forming work.

**[0035]** Meanwhile, among automotive parts such as a direct clutch, there exist parts each of which is subjected to multistage forming in a cylindrical shape, and then a periphery thereof is formed in a tooth shape. These parts are repeatedly subjected to sliding forming under a higher contact pressure, so that when the coating weight of the zinc phosphate coating film layer 103 is equal to or less than 8.0 g/m<sup>2</sup> per one side, there is a possibility that the zinc phosphate coating film layer 103 disappears due to sliding in a process in the middle of the forming, which causes reduction in dimensional accuracy of a finished product and a crack in the process in the middle of the forming. In such a case, it is preferable that the coating weight of the zinc phosphate coating film layer 103 is greater than 8.0 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup>, and the coating weight of the lubricating coating film layer 105 is about greater than 6.0 g/m<sup>2</sup> to 12.0 g/m<sup>2</sup>.

**[0036]** Here, it is preferable that the lubricating coating film layer 105 according to the present embodiment contains an alkali metal silicate whose molar ratio represented by SiO<sub>2</sub>/M<sub>2</sub>O (M is alkali metal selected from Li, Na, K, and the like) is 2 to 5 as the aforementioned binder component, and a polymeric wax made of at least either a polyethylene wax or a polypropylene wax having an average grain diameter of 0.1 μm to 3.0 μm as the aforementioned lubricating component.

**[0037]** By using the alkali metal silicate as described above as the binder component, it becomes possible to properly hold the lubricating component in the coating film, and in addition to that, it becomes possible to form a strong continuous coating film excellent in heat resistance on the surface of the steel strip. As a result of this, it becomes possible to exhibit a seizing resistance function for preventing a direct contact of metal between the surface treated steel strip according to the present embodiment and the die, a rust resistance function ascribable to a barrier property of a dense alkaline coating film, and the like. Here, a case where the aforementioned molar ratio is less than 2 is not preferable since a coating film strength cannot be sufficiently obtained and a plastic working performance deteriorates. Further, a case where the aforementioned molar ratio exceeds 5 is not preferable since the plastic working performance deteriorates, and in addition to that, stability of an alkali metal silicate aqueous solution used when forming the coating film deteriorates and practicality becomes poor. The molar ratio represented by SiO<sub>2</sub>/M<sub>2</sub>O in the alkali metal silicate is more preferably 3 to 4.

**[0038]** By using the polymeric wax as described above as the lubricating component, it is possible to suppress expansion of the lubricating coating film layer 105, and it is possible to further improve the plastic working performance of the lubricating coating film layer 105. Here, a case where the average grain diameter of the polymeric wax is less than

0.1  $\mu\text{m}$  is not preferable since oil is significantly dispersed from an interface of the polymeric wax into the coating film to deteriorate oil resistance of the coating film, and a case where the average grain diameter of the polymeric wax is greater than 3.0  $\mu\text{m}$  is not preferable since the polymeric wax in a chemical solution is not dispersed well, and it becomes difficult to form a uniform coating film. The average grain diameter of the polymeric wax is more preferably 0.5  $\mu\text{m}$  to 1.5  $\mu\text{m}$ . By using the polymeric wax having the average grain diameter as described above as the lubricating component, the lubricating component is easily filled in a concave portion of convex and concave of the zinc phosphate crystals, and as a result of this, the zinc phosphate crystals are easily exposed from the surface of the lubricating coating film layer 105.

**[0039]** A solid content of the aforementioned alkali metal silicate is preferably 60 mass% to 90 mass% with respect to a total solid content mass of the lubricating coating film layer 105, and a solid content of the aforementioned polymeric wax is preferably 5 mass% to 40 mass% with respect to the total solid content mass of the lubricating coating film layer 105.

**[0040]** A case where the solid content of the alkali metal silicate is less than 60 mass% is not preferable since continuity of a vitreous coating film formed of the alkali metal silicate becomes poor, and a possibility at which the coating film strength capable of enduring the plastic working can be obtained is lowered. Further, a case where the solid content of the alkali metal silicate exceeds 90 mass% is not preferable since the obtained coating film strength is saturated, which is disadvantageous in terms of cost. The solid content of the alkali metal silicate described above is more preferably 70 mass% to 80 mass% with respect to the total solid content mass of the lubricating coating film layer 105.

**[0041]** A case where the solid content of the polymeric wax is less than 5 mass% is not preferable since an amount of the lubricating component held by the lubricating coating film layer 105 becomes insufficient, which reduces a possibility that a sufficient lubricating property is exhibited. Further, a case where the solid content of the polymeric wax is greater than 40 mass% is not preferable since the amount of the lubricating component held by the lubricating coating film layer 105 becomes excessive, which reduces a possibility that sufficient roll slip resistance is exhibited. The solid content of the polymeric wax described above is more preferably 3 mass% to 10 mass% with respect to the total solid content mass of the lubricating coating film layer 105.

**[0042]** The lubricating coating film layer 105 according to the present embodiment can be formed by using a lubricating treatment agent obtained by mixing the lubricating component as described above in a solution or a dispersion liquid of the binder component as described above, and coating the lubricating treatment agent. Here, as a solvent to be used, any of water, an organic solvent, and a mixture of these may be employed, but, on a working environment, it is preferable to use a water-based solvent (water, or a mixed solvent between water and a water-miscible organic solvent such as alcohol). By adding, to such a solvent, the binder component (the alkali metal silicate, for example) of 60 mass% to 90 mass% with respect to the total solid content mass of the lubricating treatment agent and the lubricating component (the polymeric wax, for example) of 5 mass% to 40 mass% with respect to the total solid content mass of the lubricating treatment agent, and properly performing coating and drying, it is possible to form the lubricating coating film layer 105 having the solid content as described above.

**[0043]** Note that, in order to increase the dispersibility of the lubricating component, a publicly-known surface active agent may be added to the aforementioned lubricating treatment agent. Further, in order to modify viscosity of the aforementioned lubricating treatment agent, a viscosity modifier may be added in a range of exerting no influence on the coating film strength of the lubricating coating film layer 105. Such a viscosity modifier can employ a generally used one, and there can be cited, for example, an organic polymer thickener such as hydroxyethyl cellulose, carboxymethyl cellulose, polyacrylic amide, sodium polyacrylate, polyvinyl pyrrolidone, or polyvinyl alcohol. When such a viscosity modifier is used, a content thereof is preferably less than 10 mass% with respect to the total solid content mass of the lubricating coating film layer 105.

**[0044]** When coating the aforementioned lubricating treatment agent onto a base steel sheet 101 and the zinc phosphate coating film layer 103, it is possible to use a publicly-known method of an immersion treatment, a shower ringer treatment, a roll coating treatment, and the like. Further, the coating is only required to be performed to make the surface of the base steel sheet 101 and the zinc phosphate coating film layer 103 to be sufficiently covered by the aforementioned lubricating treatment agent, and a temperature and a coating time of the lubricating treatment agent are not particularly limited. Further, a drying temperature of the lubricating treatment agent is also not particularly limited, and it may be appropriately set in accordance with the component contained in the lubricating treatment agent.

[Regarding area ratio (25% to 90%) of zinc phosphate crystals exposed from surface of lubricating coating film layer 105]

**[0045]** In the lubricating coating film layer 105 according to the present embodiment, a part of the zinc phosphate crystals (zinc phosphate coating film layer 103) is exposed from a part of the surface of the lubricating coating film layer 105, as illustrated in FIG. 2A and FIG. 2B, so that the reduction in the coefficient of static friction of the lubricating coating film layer 105 is properly suppressed. As a result of this, it becomes possible to realize even the roll slip resistance when the steel sheet is unwound from the steel strip.

**[0046]** A case where the area ratio of the zinc phosphate crystals exposed from the surface of the lubricating coating



film layer 105 is less than 25% is not preferable since the degree of suppression of the reduction in the coefficient of static friction of the lubricating coating film layer 105 becomes insufficient, and it is not possible to realize sufficient roll slip resistance. Further, a case where the area ratio of the zinc phosphate crystals exposed from the surface of the lubricating coating film layer 105 is greater than 90% is not preferable since the degree of suppression of the reduction in the coefficient of static friction of the lubricating coating film layer 105 becomes excessive, and it is not possible to realize sufficient die galling resistance. The area ratio of the zinc phosphate crystals exposed from the surface of the lubricating coating film layer 105 is more preferably 30% to 60%.

**[0047]** When the coating weight of the lubricating coating film layer 105 and the area ratio of the zinc phosphate crystals exposed from the surface of the lubricating coating film layer 105 fall within the ranges as described above, the coefficient of static friction of the lubricating coating film layer 105 according to the present embodiment becomes 0.10 to 0.20. The coefficient of static friction of the lubricating coating film layer 105 is more preferably 0.12 to 0.15.

**[0048]** Note that by making the coating weight of the zinc phosphate coating film layer 103 fall within the preferable range as described above, it becomes possible to make the area ratio of the zinc phosphate crystals exposed from the lubricating coating film layer 105 fall within the aforementioned range more securely.

**[0049]** Further, for making the area ratio of the zinc phosphate crystals (zinc phosphate coating film layer 103) exposed from the surface of the lubricating coating film layer 105 to be 25% to 90%, it is necessary to make the zinc phosphate coating film layer 103 of 1.5 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup> per one side to be formed in an island shape, and in order to achieve this, there can be considered a method such that a temperature in the zinc phosphate treatment is increased or the treatment time is extended. However, regarding the temperature of the zinc phosphate treatment bath, it is practically difficult to increase the treatment temperature to 100°C or more since water is used as the solvent, and meanwhile, if the treatment time is extended, the crystals of zinc phosphate coating film to be precipitated are densely generated on the surface of the steel strip, resulting in that the zinc phosphate coating film layer 103 loses the island shape. Further, it is required to increase a temperature of the entire treatment bath for increasing the temperature of the zinc phosphate treatment bath, which causes a problem in terms of increase in energy cost. Furthermore, the extension of the treatment time also causes a problem in terms of reduction in productivity when the zinc phosphate treatment is performed in the zinc phosphate treatment bath with a certain length.

**[0050]** Accordingly, as one example of a method of forming the zinc phosphate coating film layer 103 of 1.5 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup> per one side in an island shape, there is proposed a method in which the temperature of the zinc phosphate treatment bath is partially increased by steam. With the use of such a method, a coating weight of the zinc phosphate crystals is increased, and in addition to that, by making the zinc phosphate crystals have more acicular shapes (sharper shapes), bulkiness of the zinc phosphate crystals is increased, which makes a lot of heads of the zinc phosphate crystals protrude from the lubricating coating film layer. The zinc phosphate crystal itself does not have slidability under a low contact pressure, so that the coefficient of static friction becomes large. On the other hand, under a high contact pressure, the zinc phosphate crystals are crushed to contribute to the slidability together with the lubricating coating film layer, so that the formability under the high contact pressure = the die galling resistance (simulated in L-shape press forming) is maintained to one equal to a conventional one.

**[0051]** Here, an example of an apparatus for forming the zinc phosphate coating film layer 103 of 1.5 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup> per one side in an island shape, is illustrated in FIG. 3A and FIG. 3B. The apparatus illustrated in FIG. 3A and FIG. 3B is configured such that steam generated by heaters 21 is applied to a surface of the base steel sheet 101 which is passed in a zinc phosphate treatment bath 20. In the zinc phosphate treatment bath 20, the steam generated through heating by the heaters 21 is agitated by agitators 22 to be applied to the surface of the base steel sheet 101. If partial heating is performed in the zinc phosphate treatment bath 20 and the steam is applied to the surface of the base steel sheet 101 to increase the treatment temperature at the time of forming the zinc phosphate coating film layer 103, it becomes possible to easily form the zinc phosphate coating film layer 103 in an island shape at a short treatment time with which the productivity is not impaired, without creating a state where the temperature of the entire zinc phosphate coating film treatment bath 20 is significantly increased. As a steam temperature increases, the formation of the zinc phosphate coating film layer 103 is accelerated more. For this reason, the steam temperature is preferably 100°C or more, and more preferably 120°C or more. When the temperature of steam becomes high, an energy cost increases and an effect of the steam temperature is saturated, so that the steam temperature is preferably set to 200°C or less.

**[0052]** The reason why the zinc phosphate coating film of 1.5 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup> per one side is formed in an island shape on the steel strip when the steam is applied to the surface of the base steel sheet 101 in the zinc phosphate treatment bath 20 as described above, is not clear. However, it is estimated that, on the surface of the base steel sheet 101, growth of the zinc phosphate crystals from a part of growing points of the zinc phosphate crystals is inhibited and the zinc phosphate coating film is formed in an island shape, and meanwhile, the zinc phosphate crystals formed in an island shape are heated to 100°C or more by the steam to be activated, and are brought into contact with the zinc phosphate treatment solution in the zinc phosphate treatment bath 20, which further accelerates the growth of the zinc phosphate crystals, resulting in that the zinc phosphate coating film of 1.5 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup> per one side can be formed in an island shape.

**[0053]** Note that the apparatus for forming the zinc phosphate coating film layer 103 of 1.5 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup> per one side in an island shape, is not particularly limited. An apparatus of a type in which the steam is directly applied to the base steel sheet 101 may be employed, or an apparatus of a type in which after mixture with the zinc phosphate treatment solution, the steam is directly applied to the base steel sheet 101 may be employed. Further, it is also possible to employ an apparatus of a type in which steam generated by the heaters 21 installed on wall surfaces of the zinc phosphate treatment bath 20 is agitated by the agitators 22, and the zinc phosphate treatment solution and the steam are simultaneously applied to the base steel sheet 101.

**[0054]** Note that by using the apparatus illustrated in FIG. 3A and FIG. 3B, it becomes possible to form the zinc phosphate coating film layer 103 in an island shape, and a maximum value of a coating weight of the zinc phosphate coating film layer 103 obtained by the blowing of steam is about 15.0 g/m<sup>2</sup>. By setting the coating weight of the zinc phosphate coating film layer 103 to 1.5 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup> per one side, it becomes possible to make the lubricating coating film layer 105 to be more securely brought into close contact with the base steel sheet 101, and even in the forming work under a high contact pressure, the zinc phosphate coating film layer 103 does not disappear, and it becomes possible to maintain the lubricating coating film layer 105 to a final process of the forming work more securely.

**[0055]** The surface treated steel strip 10 according to the present embodiment has been described above in detail while referring to FIG. 1 to FIGS. 3.

<Regarding measuring methods of various physical property values>

**[0056]** Subsequently, measuring methods of various physical property values realized by the surface treated steel strip 10 according to the present embodiment will be briefly described.

**[0057]** First, the average grain diameter of the zinc phosphate crystals which form the zinc phosphate coating film layer 103, and the average grain diameter of the polymeric wax contained in the lubricating coating film layer 105 can be measured through a publicly-known measuring method such that a field emission scanning electron microscope (FE-SEM) is used to observe the steel sheet surface at a low acceleration voltage.

**[0058]** Further, the coating weight per one side of each of the zinc phosphate coating film layer 103 and the lubricating coating film layer 105 can be measured through a publicly-known measuring method such as a gravimetric method.

**[0059]** Note that the thicknesses of the zinc phosphate coating film layer 103 and the lubricating coating film layer 105 can be measured by observing a cross section of the surface treated steel strip with the use of an electron microscope such as a SEM, but, the thickness of the lubricating coating film layer 105 can also be measured through a method to be described below. First, by using a glow discharge spectroscopy (GDS), emission spectrum intensities of a component (Si, for example) of the lubricating coating film layer 105 and the zinc phosphate crystal component (Zn, for example) are measured on a cross section of the surface treated steel strip 10 from a surface along a depth direction. By such measurement, it is possible to obtain two peaks corresponding to the respective components in the emission spectrum. Here, a thickness corresponding to a value between 50% values of respective peak intensities of the two components (a 50% value on the base steel sheet side regarding Si, and a 50% value on a surface layer side of the steel strip regarding Zn), can be set to the thickness of the lubricating coating film layer 105.

**[0060]** Further, the area ratio of the zinc phosphate crystals exposed from the surface of the lubricating coating film layer 105 can be obtained in a manner that the surface of the surface treated steel strip 10 is observed by an electron microscope such as a SEM, and an area of the zinc phosphate crystals detected within a field of view is specified by a publicly-known method. Here, regarding the specification of the area ratio, it is preferable to observe a plurality of places on the surface of the surface treated steel strip 10 and calculate an average of the specified area ratios.

**[0061]** Further, the coefficient of static friction of the lubricating coating film layer 105 can be measured through various test methods such as a round bead drawing test to be described below in detail.

**[0062]** The measuring methods of the various physical property values realized by the surface treated steel strip 10 according to the present embodiment have been briefly described above. Note that the above-described measuring methods are only examples, and it is also possible to specify the respective physical property values by using the other publicly-known measuring methods.

(Regarding manufacturing method of surface treated steel strip)

**[0063]** Subsequently, a manufacturing method of the surface treated steel strip 10 according to the present embodiment will be described while referring to FIG. 4. FIG. 4 is a flow chart illustrating one example of a flow of the manufacturing method of the surface treated steel strip 10 according to the present embodiment.

**[0064]** In the manufacturing method of the surface treated steel strip 10 according to the present embodiment, a pretreatment such as a degreasing treatment or a cleaning treatment is first performed, according to need, on a steel strip formed by winding a predetermined base steel sheet 101 (pretreatment step S101).

**[0065]** After that, a surface of the base steel sheet 101 is subjected to thermal refining by using a surface conditioner

containing titanium colloid (thermal refining step S103). Accordingly, the titanium colloid having a coarse grain diameter is adhered to the surface of the base steel sheet 101.

**[0066]** Subsequently, by the method as described before, needle crystals of zinc phosphate are precipitated on the surface of the base steel sheet 101 after being subjected to the thermal refining of surface (zinc phosphate coating film layer forming step S105). Accordingly, the needle crystals of zinc phosphate are precipitated in an island shape on the surface of the base steel sheet 101, resulting in that the zinc phosphate coating film layer 103 is formed. Note that as described above, by performing the precipitation of zinc phosphate in the apparatus illustrated in FIG. 3A and FIG. 3B, it becomes possible to favorably form the zinc phosphate coating film layer 103 in an island shape.

**[0067]** Next, a lubricating treatment agent is coated onto the base steel sheet 101 and the zinc phosphate coating film layer 103, and dried under a proper drying condition, to thereby form the lubricating coating film layer 105 (lubricating coating film layer forming step S107). Consequently, the surface treated steel strip 10 as illustrated in FIG. 2A and FIG. 2B is manufactured.

**[0068]** After that, it is also possible to perform a publicly-known post-treatment on the manufactured surface treated steel strip 10 according to need (post-treatment step S109).

**[0069]** The one example of the flow of the manufacturing method of the surface treated steel strip 10 according to the present embodiment has been briefly described above while referring to FIG. 4.

[Examples]

**[0070]** Hereinafter, the surface treated steel strip and the manufacturing method of the surface treated steel strip according to the present invention will be concretely described while showing Examples and Comparative Examples. Note that the Examples to be described below are only examples of the surface treated steel strip and the manufacturing method of the surface treated steel strip according to the present invention, and the surface treated steel strip and the manufacturing method of the surface treated steel strip according to the present invention are not limited to the examples to be described below.

**[0071]** Note that in the following Examples, the indication of "%" means "mass%" unless otherwise specified. Further, in the following Examples, each of coating weights means a coating weight per one side.

(1) Zinc phosphate treatment of steel sheet

**[0072]** On both surfaces of a cut sheet (300 mm × 300 mm) of SPH590 steel sheet (hot-rolled steel sheet having tensile strength of 590 MPa or more) with a sheet thickness of 3.2 mm, a zinc phosphate treatment was performed through an immersion treatment. At that time, in the Examples, by agitating steam generated by heaters installed on wall surfaces of an apparatus with agitators, the steam of 100°C to 120°C was applied for 5 seconds to the both surfaces of the steel sheet during the immersion treatment. An immersion time was changed from 5 seconds to 120 seconds to adjust a coating weight of zinc phosphate crystals, to thereby obtain zinc phosphate treated steel sheets shown in Table 1. The coating weight of the zinc phosphate crystals was determined from a mass change of the steel sheet before and after the zinc phosphate treatment.

(2) Formation of lubricating coating film

**[0073]** The following binder components, lubricating components, and extreme-pressure additive were added to pure water (deionized water), sufficiently agitated to be dispersed, and diluted by pure water to realize a solid content of 20%, to thereby adjust a chemical solution used for coating. This chemical solution was coated to surfaces of the above-described zinc phosphate treated steel sheet one side by one side with the use of a bar coater, dried at 60°C to form a lubricating coating film on each of the surfaces, to thereby form a lubricated steel sheet. A coating weight of the lubricating coating film was calculated from a mass change of the steel sheet before and after the formation of the lubricating coating film.

Chemical solution (2-1)

A) Binder components

**[0074]**

- Sodium metasilicate (molar ratio of  $\text{SiO}_2/\text{Na}_2\text{O}$ : 4)
- Acryl-based resin (monomer composition: 8% of acrylic acid, 52% of methacrylic acid, 40% of butyl acrylate)

B) Lubricating components

**[0075]**

- 5
- Organic polymeric compound: polyethylene wax (molecular weight of 20000, average grain diameter of 3  $\mu\text{m}$ )
  - Solid lubricant: molybdenum disulfide (average grain diameter of 2  $\mu\text{m}$ )
  - Soap: sodium stearate

C) Extreme-pressure additive

10

**[0076]**

- Phosphorous acid ester

15

D) Solid content

**[0077]**

- 20
- Solid content of sodium metasilicate with respect to total solid content of lubricating coating film layer: 88%
  - Solid content of polyethylene wax with respect to total solid content of lubricating coating film layer: 5%

Chemical solution (2-2)

A) Binder components

25

**[0078]**

- Sodium metasilicate (molar ratio of  $\text{SiO}_2/\text{Na}_2\text{O}$ : 5)
- Acryl-based resin (monomer composition: 8% of acrylic acid, 52% of methacrylic acid, 40% of butyl acrylate)

30

B) Lubricating components

**[0079]**

- 35
- Organic polymeric compound: polyethylene wax (molecular weight of 2000, average grain diameter of 0.5  $\mu\text{m}$ )
  - Solid lubricant: molybdenum disulfide (average grain diameter of 2  $\mu\text{m}$ )
  - Soap: sodium stearate

C) Extreme-pressure additive

40

**[0080]**

- Phosphorous acid ester

45

D) Solid content

**[0081]**

- 50
- Solid content of sodium metasilicate with respect to total solid content of lubricating coating film layer: 80%
  - Solid content of polyethylene wax with respect to total solid content of lubricating coating film layer: 5%

Chemical solution (2-3)

A) Binder components

55

**[0082]**

- Sodium metasilicate (molar ratio of  $\text{SiO}_2/\text{Na}_2\text{O}$ : 2)

- Acryl-based resin (monomer composition: 8% of acrylic acid, 52% of methacrylic acid, 40% of butyl acrylate)

B) Lubricating components

**[0083]**

- Organic polymeric compound: polyethylene wax (molecular weight of 10000, average grain diameter of 1.0  $\mu\text{m}$ )
- Solid lubricant: molybdenum disulfide (average grain diameter of 2  $\mu\text{m}$ )
- Soap: sodium stearate

C) Extreme-pressure additive

**[0084]**

- Phosphorous acid ester

D) Solid content

**[0085]**

- Solid content of sodium metasilicate with respect to total solid content of lubricating coating film layer: 60%
- Solid content of polyethylene wax with respect to total solid content of lubricating coating film layer: 30%

(3) Size of crystal

**[0086]** The steel sheet surface after being subjected to the zinc phosphate treatment was observed by a SEM of 500 magnifications at an acceleration voltage of 5 kV. An area ratio of zinc phosphate crystals exposed from the surface was calculated based on an area of crystal portions by performing binarization of an image. Further, four or more crystal exposed from the surface were selected in an arbitrary manner, lengths of a major axis and a minor axis were measured to calculate an average crystal size, and the size was set to a crystal size of zinc phosphate.

(4) Evaluation method

a-1) Die galling resistance

**[0087]** In order to simulate a multistage plastic working under a high contact pressure, an L-shape press forming equipment was used to perform multistage ironing on a side surface of an L-bent sample of the lubricated steel sheet through the following procedures, as in FIG. 5 which illustrates an outline, and a lubricating property of the steel sheet was evaluated. At this time, if the galling occurs on the steel sheet surface when the number of times of forming is two or less, the multistage forming is not realized, so that a steel sheet on which the galling does not occur when the number of times of forming is three or more was evaluated as acceptable. Note that a steel sheet on which the galling occurred when the number of times of forming was two or less was represented as  $\times$ , and a steel sheet on which the galling did not occur when the number of times of forming was three or more was represented as o.

a-2) Procedures

**[0088]**

Procedure (1) A general rust preventive oil is coated to both surfaces of a sample so that a coating weight becomes 1.5 g/m<sup>2</sup>.

Procedure (2) A clearance between a punch and a die is set to satisfy a sheet thickness - 0.25 (sheet thickness reduction width = 0.15 mm), and first ironing (L-shape forming) is performed on the sample.

Procedure (3) On the L-shaped sample after being subjected to the ironing in the procedure (2), the ironing is repeatedly performed while further reducing the clearance at a pitch of -0.25 until the die galling occurs on the steel sheet surface, and the number of times of ironing at which the die galling occurs is determined.

b-1) Roll slip resistance

**[0089]** In order to simulate roll feed resistance under a high contact pressure, front and rear surfaces of a steel sheet were sandwiched by round beads, and a coefficient of static friction was calculated from a load at a time of drawing the steel sheet, as in FIG. 6 which illustrates an outline. It is confirmed that when the coefficient of static friction is 0.09 or less, the force for feeding the steel sheet cannot be applied and it is difficult to pass the sheet in an actual line, and when the coefficient of static friction is greater than 0.20, the force required for feeding the steel sheet becomes too large and it is difficult to pass the sheet in an actual line. Therefore, a steel sheet having the coefficient of static friction of not less than 0.10 nor more than 0.20 was set as acceptable. Note that a steel sheet whose coefficient of static friction is out of the range was represented as ×, and a steel sheet whose coefficient of static friction is within the range was represented as o.

b-2) Procedures

**[0090]**

Procedure (1) A general rust preventive oil is coated to both surfaces of a sample so that a coating weight becomes 1.5 g/m<sup>2</sup>.

Procedure (2) Round beads (R5) are pressed against the both surfaces of the steel sheet to sandwich the steel sheet at a load of 1 [kN], the steel sheet is drawn, and a coefficient of static friction when drawing the steel sheet is calculated.

[Table 1]

TYPE	No.	ZINC PHOSPHATE COATING FILM LAYER			LUBRICATING COATING FILM LAYER		ZINC PHOSPHATE EXPOSED AREA RATIO [%]	COEFFICIENT OF STATIC FRICTION [-]	PROPERTY		PRESENCE / ABSENCE OF STEAM
		COATING WEIGHT [g/m <sup>2</sup> ]	MAJOR AXIS [μm]	MINOR AXIS [μm]	CHEMICAL SOLUTION	COATING WEIGHT [g/m <sup>2</sup> ]			DIE GALLING RESISTANCE	ROLL SLIP RESISTANCE	
COMPARATIVE EXAMPLE	1	1	33	2	2-1	3	5	0.07	×	×	ABSENCE
EXAMPLE	2	1.5	32	4	2-1	1.5	50	0.12	○	○	PRESENCE
EXAMPLE	3	1.5	30	4	2-1	3.7	40	0.11	○	○	PRESENCE
EXAMPLE	4	1.5	29	3	2-1	5.9	25	0.10	○	○	PRESENCE
COMPARATIVE EXAMPLE	5	3.3	29	5	2-1	1	95	0.14	×	○	ABSENCE
EXAMPLE	6	3.3	38	5	2-1	1.5	90	0.14	○	○	PRESENCE
EXAMPLE	7	3.3	31	4	2-1	3.7	60	0.12	○	○	PRESENCE
EXAMPLE	8	3.3	30	4	2-1	4	60	0.12	○	○	PRESENCE
EXAMPLE	9	3.3	30	4	2-1	5	40	0.11	○	○	PRESENCE
EXAMPLE	10	3.3	27	3	2-1	5.9	30	0.11	○	○	PRESENCE
COMPARATIVE EXAMPLE	11	3.3	24	3	2-1	8	10	0.09	○	×	ABSENCE
EXAMPLE	12	5	58	6	2-1	3.7	70	0.13	○	○	PRESENCE
EXAMPLE	13	8	66	7	2-1	1.5	90	0.14	○	○	PRESENCE
EXAMPLE	14	8	64	7	2-1	3.7	80	0.13	○	○	PRESENCE
EXAMPLE	15	8	62	7	2-1	5.9	40	0.11	○	○	PRESENCE
COMPARATIVE EXAMPLE	16	8	61	6	2-1	7	23	0.09	○	×	ABSENCE
EXAMPLE	17	6.5	62	7	2-2	3	70	0.11	○	○	PRESENCE
EXAMPLE	18	5.5	60	6	2-2	5	50	0.13	○	○	PRESENCE
COMPARATIVE EXAMPLE	19	0	-	-	2-2	5	0	0.06	×	×	ABSENCE
EXAMPLE	20	2	35	4	2-3	2	44	0.13	○	○	PRESENCE
EXAMPLE	21	12	60	8	2-1	6	70	0.13	○	○	PRESENCE
EXAMPLE	22	12	60	8	2-1	8	60	0.12	○	○	PRESENCE
EXAMPLE	23	12	60	8	2-1	12	45	0.11	○	○	PRESENCE
COMPARATIVE EXAMPLE	24	12	50	25	2-1	8	10	0.07	○	×	ABSENCE
EXAMPLE	25	15	70	10	2-1	2	90	0.20	○	○	PRESENCE
EXAMPLE	26	15	70	10	2-1	6	80	0.14	○	○	PRESENCE
EXAMPLE	27	15	70	10	2-1	8	75	0.13	○	○	PRESENCE
EXAMPLE	28	15	70	10	2-1	12	55	0.12	○	○	PRESENCE
COMPARATIVE EXAMPLE	29	15	85	40	2-1	12	20	0.08	○	×	ABSENCE
COMPARATIVE EXAMPLE	30	1.9	100	20	2-1	3.3	10	0.08	○	×	ABSENCE
COMPARATIVE EXAMPLE	31	4.5	200	50	2-1	3.3	24	0.07	○	×	ABSENCE

## (5) Evaluation results

**[0091]** The above Table 1 shows evaluation results when forming lubricating coating films on various zinc phosphate treated steel sheets with different coating weights of zinc phosphate crystals.

**[0092]** As is apparent from Table 1, in Nos. 2 to 4, 6 to 10, 12 to 15, 17, 18, 20 having preferable zinc phosphate coating film layers and lubricating coating film layers and satisfying the exposed area ratios of zinc phosphate crystals, sufficient properties realizing both of the die galling resistance and the roll slip resistance were exhibited.

**[0093]** On the other hand, in Nos. 1, 19 in which the coating weight of the zinc phosphate coating film layer was 0 or insufficient, the coefficient of static friction became low, resulting in that the die galling resistance and the roll slip

resistance deteriorated. In each of Nos. 11, 24, 29, 30, 31 in which the average grain diameter in the major axis direction or the average grain diameter in the minor axis direction of the zinc phosphate crystals was out of the range of the present invention although the coating weight of the zinc phosphate coating film layer and the lubricating coating film of the upper layer satisfied the preferable conditions, the coefficient of static friction became low, resulting in that the roll slip resistance deteriorated. In No. 5 in which a lot of zinc phosphate crystals were exposed, an amount of the lubricating coating film itself was small, resulting in that the die galling resistance deteriorated. In No. 16, the exposed area ratio of the zinc phosphate crystals was small, and the coefficient of static friction did not become 0.10 or more.

**[0094]** FIG. 7 to FIG. 10 illustrate enlarged photographs of surface treated steel strips of comparative examples and examples of the present invention. A comparative example (No. 30) illustrated in FIG. 7 has a coating weight of an island-shaped zinc phosphate coating film layer of 1.9 g/m<sup>2</sup>, a major axis of 100 μm, a minor axis of 20 μm, a coating weight of the lubricating coating film layer (chemical solution 2-1) of 3.3 g/m<sup>2</sup>, and a zinc phosphate exposed area ratio of 10%, and a comparative example (No. 31) illustrated in FIG. 8 has a coating weight of an island-shaped zinc phosphate coating film layer of 4.5 g/m<sup>2</sup>, a major axis of 200 μm, a minor axis of 50 μm, a coating weight of the lubricating coating film layer (chemical solution 2-1) of 3.3 g/m<sup>2</sup>, and a zinc phosphate exposed area ratio of 24%. An example of the present invention (No. 14) illustrated in FIG. 9 has a coating weight of an island-shaped zinc phosphate coating film layer of 8.0 g/m<sup>2</sup>, a major axis of 64 μm, a minor axis of 7 μm, a coating weight of the lubricating coating film layer (chemical solution 2-1) of 3.7 g/m<sup>2</sup>, and a zinc phosphate exposed area ratio of 80%, and an example of the present invention illustrated in FIG. 10 has a coating weight of an island-shaped zinc phosphate coating film layer of 8.0 g/m<sup>2</sup>, a major axis of 31 μm, a minor axis of 4 μm, a coating weight of the lubricating coating film layer (chemical solution 2-1) of 3.7 g/m<sup>2</sup>, and a zinc phosphate exposed area ratio of 60%.

**[0095]** In the examples of the present invention, the temperature of the zinc phosphate treatment bath was partially increased by the steam to increase the coating weight of the zinc phosphate crystals, and in addition to that, by making the zinc phosphate crystals have more acicular shapes (sharper shapes), bulkiness of the zinc phosphate crystals was increased, which made a lot of heads of the zinc phosphate crystals protrude from the lubricating coating film layer. The zinc phosphate crystal itself does not have slidability under a low contact pressure, so that the coefficient of static friction became large. On the other hand, under a high contact pressure, the zinc phosphate crystals were crushed to contribute to the slidability together with the lubricating coating film layer, so that the formability under the high contact pressure = the die galling resistance (simulated in L-shape press forming) was maintained to one equal to a conventional one. As described above, it became possible to realize both of the roll slip resistance and the die galling resistance being properties which are contrary to each other.

**[0096]** In the foregoing, the preferred embodiments of the present invention have been described in detail while referring to the attached drawings, but, the present invention is not limited to such examples. It is apparent that a person ordinary skilled in the art to which the present invention pertains is able to devise various variation or modification examples within the scope of the technical spirit described in the claims, and it should be understood that such examples belong to the technical scope of the present invention as a matter of course.

[Explanation of Codes]

#### **[0097]**

- 1 pinch rolls
- 2 die
- 10 surface treated steel strip
- 20 zinc phosphate treatment bath
- 21 heater
- 22 agitator
- 101 base steel sheet
- 103 zinc phosphate coating film layer
- 105 lubricating coating film layer

#### **Claims**

1. A surface treated steel strip, comprising:

- a base steel sheet;
- a zinc phosphate coating film layer made of needle zinc phosphate crystals which are formed in an island shape on a surface of the base steel sheet; and



a lubricating coating film layer covering the surface of the base steel sheet and a part of the zinc phosphate coating film layer, and containing at least a lubricating component, wherein an area ratio of the zinc phosphate crystals exposed from a surface of the lubricating coating film layer is 25% to 90%.

2. The surface treated steel strip according to claim 1, wherein the zinc phosphate crystals have an average grain diameter in a major axis direction of 25  $\mu\text{m}$  to 70  $\mu\text{m}$ , and have an average grain diameter in a minor axis direction of 3  $\mu\text{m}$  to 10  $\mu\text{m}$ .

3. The surface treated steel strip according to claim 1 or 2, wherein a coating weight of the zinc phosphate coating film layer is 1.5 g/m<sup>2</sup> to 15.0 g/m<sup>2</sup> per one side.

4. The surface treated steel strip according to any one of claims 1 to 3, wherein a coating weight of the lubricating coating film layer is 1.0 g/m<sup>2</sup> to 12.0 g/m<sup>2</sup> per one side.

5. The surface treated steel strip according to any one of claims 1 to 4, wherein:

the lubricating coating film layer contains an alkali metal silicate whose molar ratio represented by  $\text{SiO}_2/\text{M}_2\text{O}$  (M is alkali metal) is 2 to 5, and a polymeric wax made of at least either a polyethylene wax or a polypropylene wax having an average grain diameter of 0.1  $\mu\text{m}$  to 3.0  $\mu\text{m}$ ; and a solid content of the alkali metal silicate and a solid content of the polymeric wax with respect to a total solid content mass of the lubricating coating film layer are 60 mass% to 90 mass% and 5 mass% to 40 mass%, respectively.

6. A manufacturing method of a surface treated steel strip, comprising:

a thermal refining step of performing thermal refining on a surface of a base steel sheet by utilizing a surface conditioner containing titanium colloid;

a zinc phosphate coating film layer forming step of making needle crystals of zinc phosphate grow in an island shape on the surface of the base steel sheet after being subjected to the thermal refining of surface to form a zinc phosphate coating film layer on the surface of the base steel sheet; and

a lubricating coating film layer forming step of forming a lubricating coating film layer by coating a lubricating treatment agent containing at least a lubricating component to the surface of the base steel sheet and the zinc phosphate coating film layer so that a coating weight of the lubricating treatment agent becomes 1.0 g/m<sup>2</sup> to 12.0 g/m<sup>2</sup> per one side, and making an area ratio of the zinc phosphate crystals exposed from a surface of the lubricating coating film layer to be 25% to 90%.

7. The manufacturing method of the surface treated steel strip according to claim 6, wherein the base steel sheet is heated in the zinc phosphate coating film layer forming step.

8. The manufacturing method of the surface treated steel strip according to claim 7, wherein the base steel sheet is heated by applying steam to the base steel sheet.

FIG.1

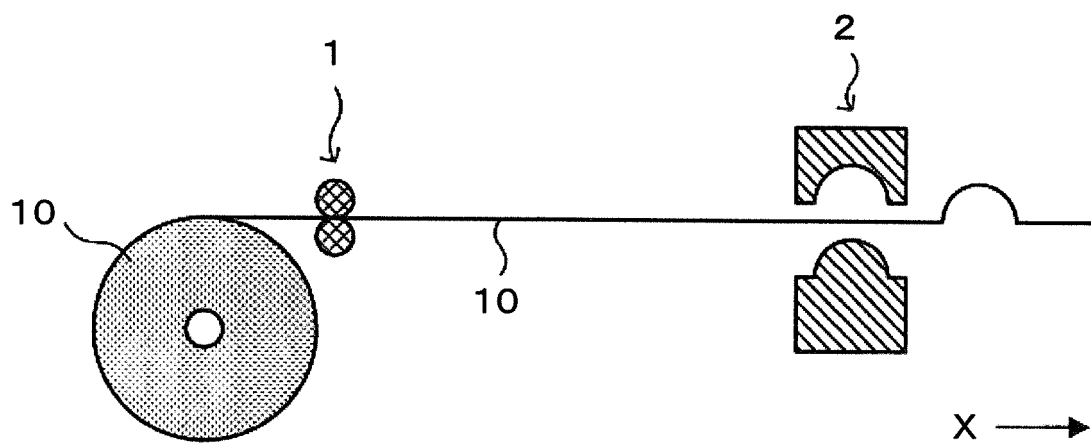
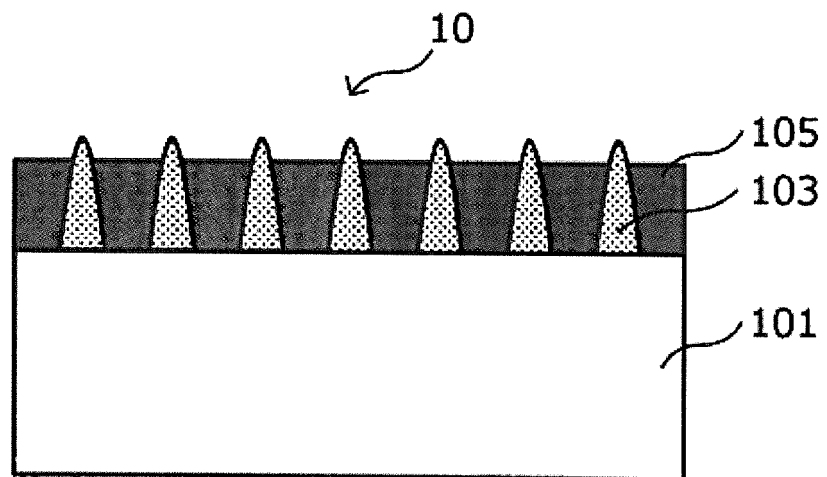


FIG.2

(A)



(B)

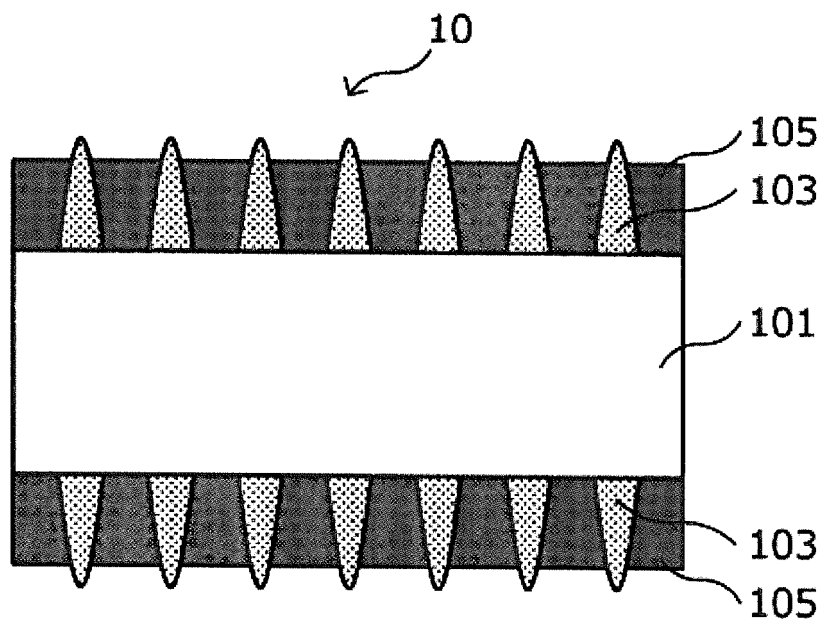


FIG.3

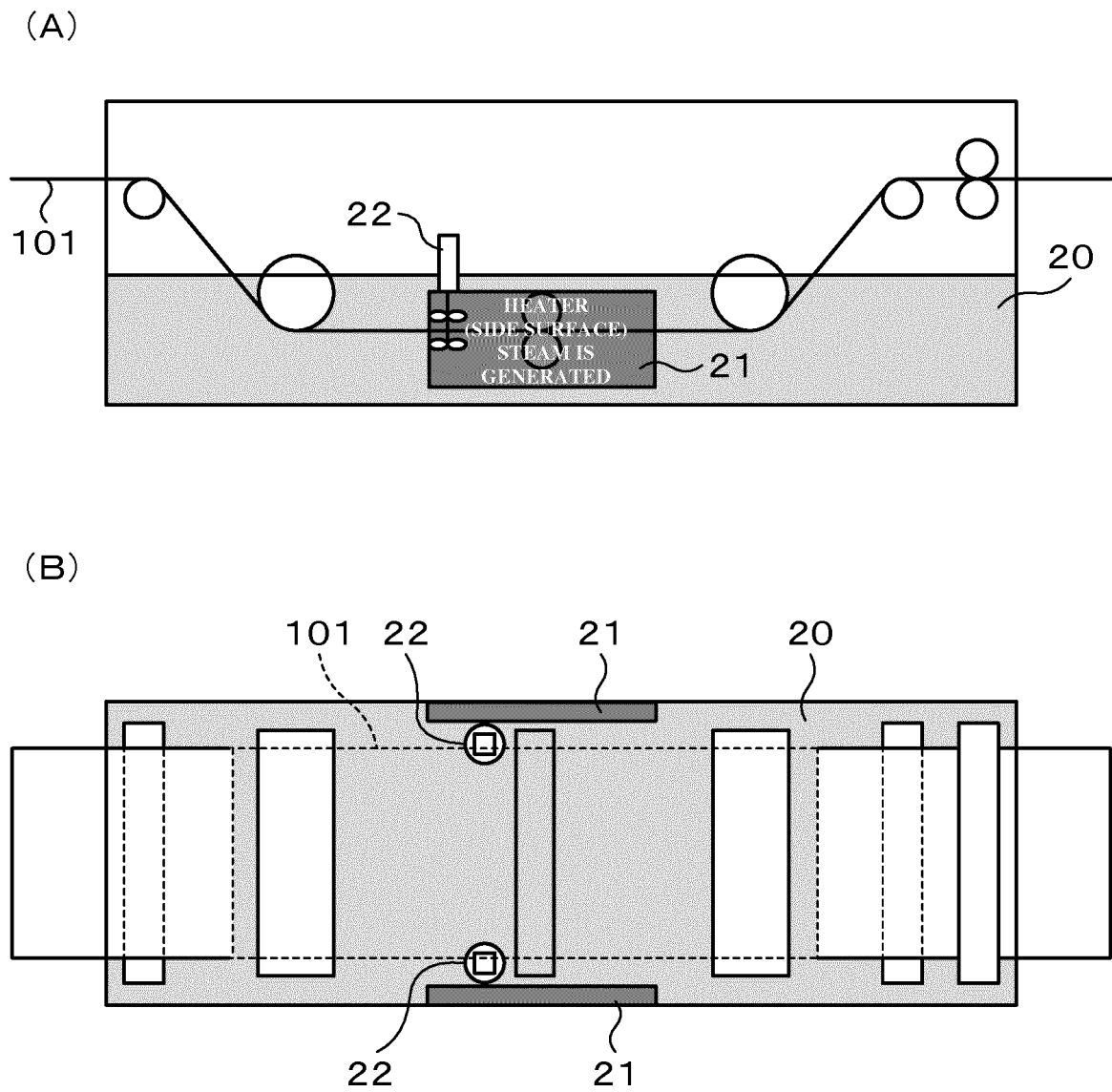


FIG.4

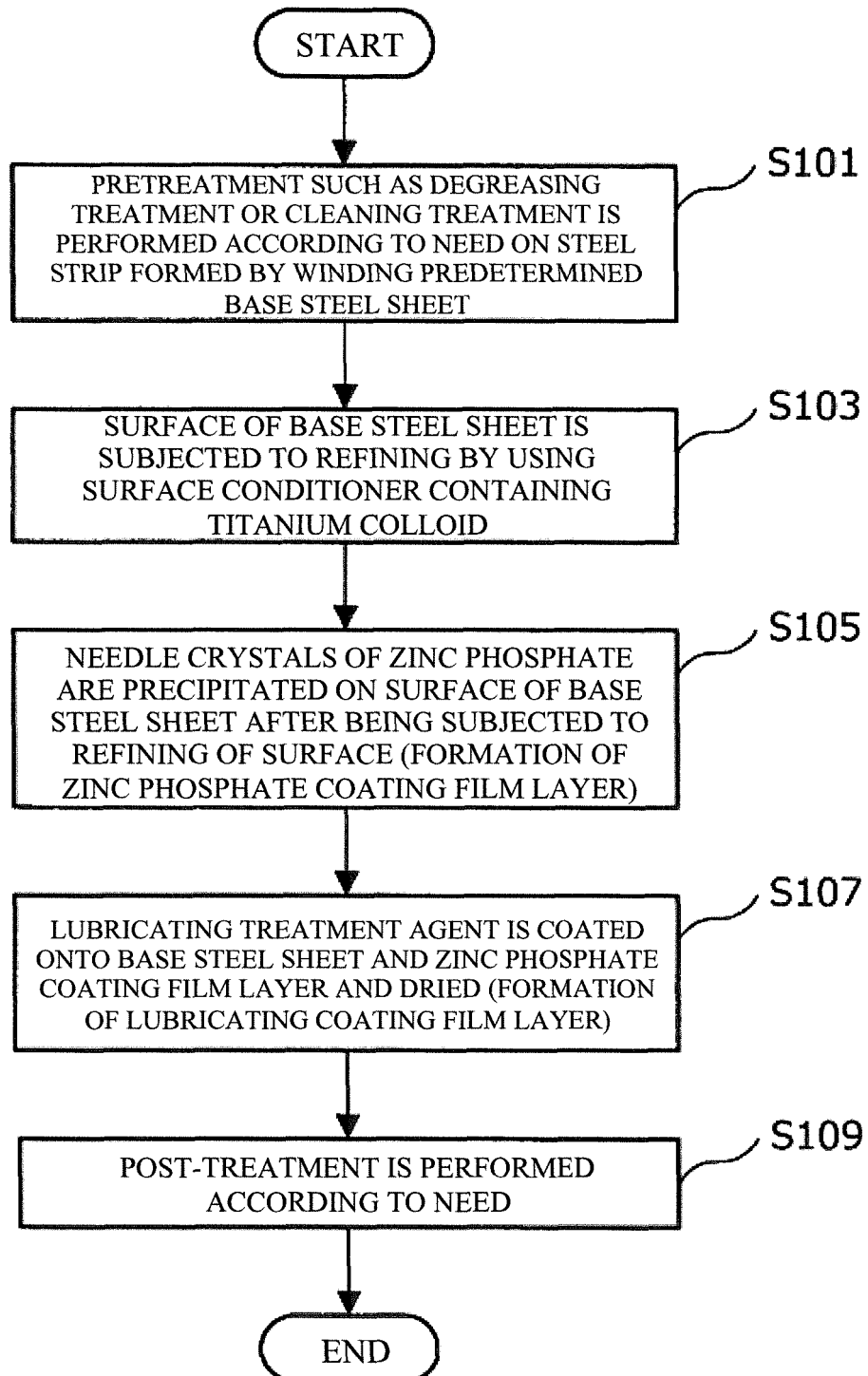


FIG.5

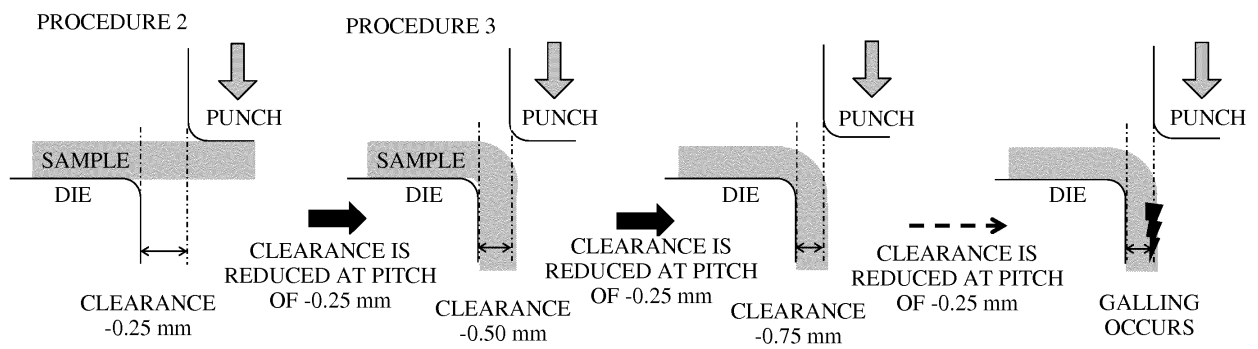


FIG.6

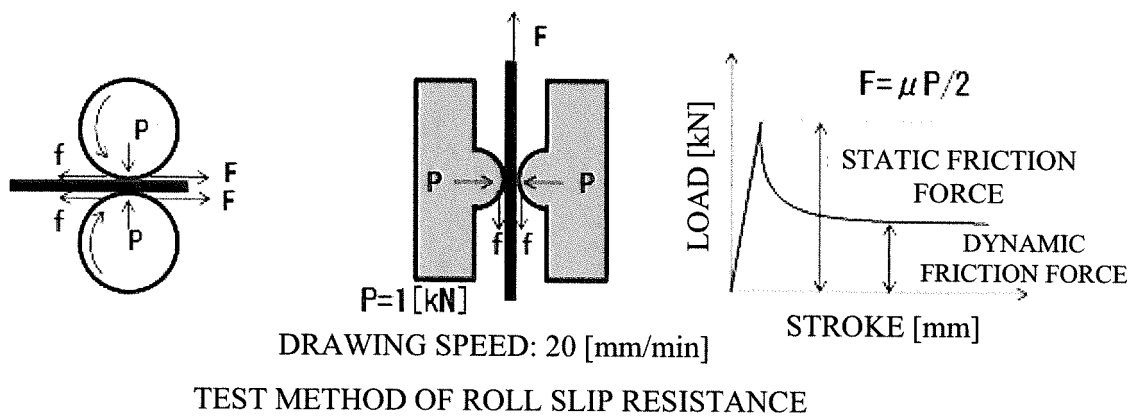


FIG.7

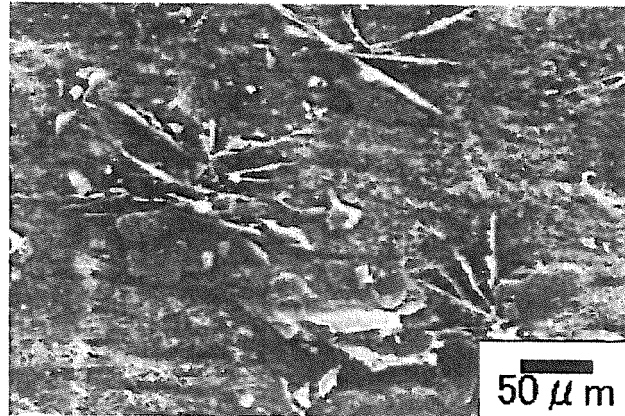


FIG.8

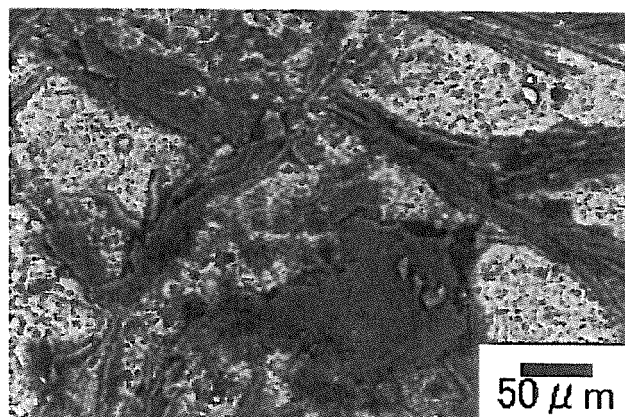




FIG.9

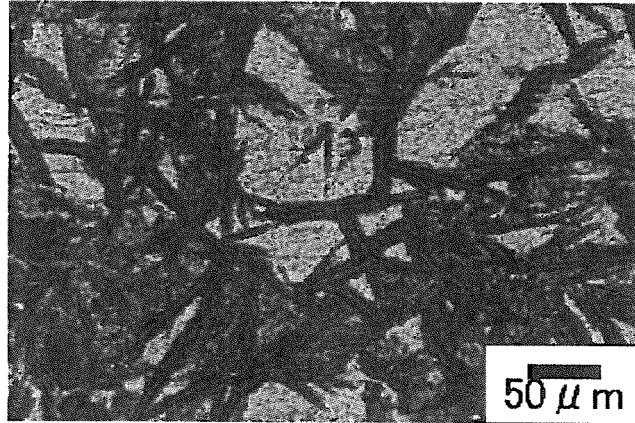
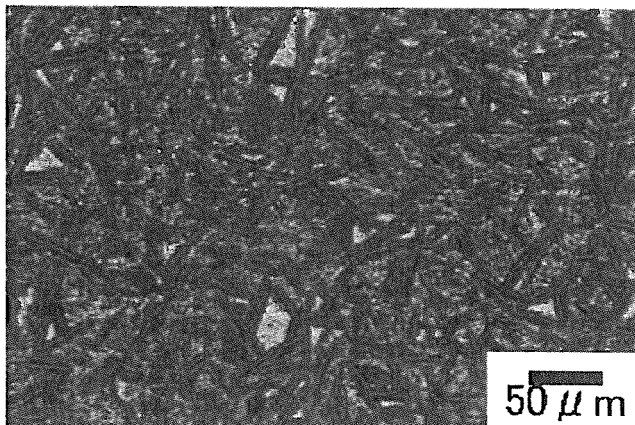


FIG.10



## INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2017/015160

## A. CLASSIFICATION OF SUBJECT MATTER

C23C28/00(2006.01)i, C10M103/06(2006.01)i, C10M125/26(2006.01)i,  
C10M143/04(2006.01)i, C23C22/12(2006.01)i

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)

C23C28/00, C10M103/06, C10M125/26, C10M143/04, C23C22/12

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

Jitsuyo Shinan Koho 1922-1996 Jitsuyo Shinan Toroku Koho 1996-2017

Kokai Jitsuyo Shinan Koho 1971-2017 Toroku Jitsuyo Shinan Koho 1994-2017

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.
A	JP 2013-104125 A (Nippon Steel & Sumitomo Metal Corp.), 30 May 2013 (30.05.2013), (Family: none)	1-8
A	JP 2005-290552 A (Nisshin Steel Co., Ltd.), 20 October 2005 (20.10.2005), (Family: none)	1-8

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

☐ See patent family annex.

\* Special categories of cited documents:

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"P" document published prior to the international filing date but later than the priority date claimed

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"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

"&" document member of the same patent family

Date of the actual completion of the international search  
26 April 2017 (26.04.17)

Date of mailing of the international search report  
16 May 2017 (16.05.17)

Name and mailing address of the ISA/  
Japan Patent Office  
3-4-3, Kasumigaseki, Chiyoda-ku,  
Tokyo 100-8915, Japan

Authorized officer

Telephone No.

**REFERENCES CITED IN THE DESCRIPTION**

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**Patent documents cited in the description**

- JP 2013104125 A [0004]