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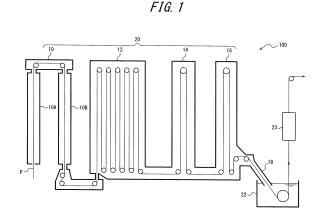
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## (54) METHOD FOR MANUFACTURING HOT-DIP GALVANIZED STEEL SHEET

A method of producing a hot-dip galvanized steel sheet whereby favorable coating appearance can be obtained with high coating adhesion even in the case of hot-dip galvanizing a steel strip whose Si content is 0.2 mass% or more is provided. The method includes: annealing a steel strip by conveying the steel strip through a heating zone, a soaking zone, and a cooling zone in this order inside an annealing furnace; and applying a hot-dip galvanized coating onto the steel strip discharged from the cooling zone. Reducing gas or non-oxidizing gas supplied into the soaking zone includes humidified gas and dry gas. While a width and a sheet passing speed of the steel strip passing through the soaking zone are constant, a variation of pressure in the annealing furnace is suppressed by adjusting a flow rate of the dry gas, and a variation range of an amount of moisture supplied into the soaking zone by the humidified gas is limited to 20 % or less.



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#### Description

#### **TECHNICAL FIELD**

**[0001]** The present disclosure relates to a method of producing a hot-dip galvanized steel sheet using a continuous hot-dip galvanizing apparatus that includes: an annealing furnace in which a heating zone, a soaking zone, and a cooling zone are arranged in this order; and a hot-dip galvanizing line adjacent to the cooling zone.

#### **BACKGROUND**

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**[0002]** In recent years, the demand for high tensile strength steel sheets which contribute to more lightweight structures and the like is increasing in the fields of automobiles, household appliances, building products, etc. As high tensile strength steel materials, for example, it is known that a steel sheet with favorable hole expandability can be produced by containing Si in steel, and a steel sheet with favorable ductility where retained austenite ( $\gamma$ ) forms easily can be produced by containing Si or Al in steel.

**[0003]** However, in the case of producing a galvannealed steel sheet using, as a base material, a high tensile strength steel sheet containing a large amount of Si (particularly, 0.2 mass% or more), the following problem arises. The galvannealed steel sheet is produced by, after heat-annealing the steel sheet as the base material at a temperature of about 600 °C to 900 °C in a reducing atmosphere or a non-oxidizing atmosphere, hot-dip galvanizing the steel sheet and further heat-alloying the galvanized coating.

[0004] Here, Si in the steel is an oxidizable element, and is selectively oxidized in a typically used reducing atmosphere or non-oxidizing atmosphere and concentrated in the surface of the steel sheet to form an oxide. This oxide decreases wettability with molten zinc in the galvanizing process, and causes non-coating. With an increase of the Si concentration in the steel, wettability decreases rapidly and non-coating occurs frequently. Even in the case where non-coating does not occur, there is still a problem of poor coating adhesion. Besides, if Si in the steel is selectively oxidized and concentrated in the surface of the steel sheet, a significant alloying delay arises in the alloying process after the hot-dip galvanizing, leading to considerably lower productivity.

**[0005]** In view of such problems, for example, JP 2010-202959 A (PTL 1) describes the following method. With use of a direct fired furnace (DFF), the surface of a steel sheet is oxidized and then the steel sheet is annealed in a reducing atmosphere to internally oxidize Si and prevent Si from being concentrated in the surface of the steel sheet, thus improving the wettability and adhesion of the hot-dip galvanized coating. PTL 1 describes that the reducing annealing after heating may be performed by a conventional method (dew point: -30 °C to -40 °C).

[0006] WO 2007/043273 A1 (PTL 2) describes the following technique. In a continuous annealing and hot-dip coating method that uses an annealing furnace having an upstream heating zone, a downstream heating zone, a soaking zone, and a cooling zone arranged in this order and a hot-dip molten bath, annealing is performed under the following conditions to internally oxidize Si and prevent Si from being concentrated in the surface of the steel sheet: heating or soaking the steel sheet at a steel sheet temperature in the range of at least 300 °C by indirect heating; setting the atmosphere inside the furnace in each zone to an atmosphere of 1 vol% to 10 vol% hydrogen with the balance being nitrogen and incidental impurities; setting the steel sheet end-point temperature during heating in the upstream heating zone to 550 °C or more and 750 °C or less and the dew point in the upstream heating zone to less than -25 °C; setting the dew point in the subsequent downstream heating zone and soaking zone to -30 °C or more and 0 °C or less; and setting the dew point in the cooling zone to less than -25 °C. PTL 2 also describes humidifying mixed gas of nitrogen and hydrogen and introducing it into the downstream heating zone and/or the soaking zone.

[0007] JP 2009-209397 A (PTL 3) describes the following technique. While measuring the dew point of furnace gas, the supply and discharge positions of furnace gas are changed depending on the measurement to control the dew point of the gas in the reducing furnace to be in the range of more than -30 °C and 0 °C or less, thus preventing Si from being concentrated in the surface of the steel sheet. PTL 3 describes that the heating furnace may be any of a direct fired furnace (DFF), a non-oxidizing furnace (NOF), and a radiant tube, but a radiant tube is preferable as it produces significantly advantageous effects.

CITATION LIST

Patent Literatures

55 [0008]

PTL 1: JP 2010-202959 A

PTL 2: WO 2007/043273 A1

PTL 3: JP 2009-209397 A

5 SUMMARY

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(Technical Problem)

**[0009]** However, with the method described in PTL 1, although the coating adhesion after the reduction is favorable, the amount of Si internally oxidized tends to be insufficient, and Si in the steel causes the alloying temperature to be higher than typical temperature by 30 °C to 50 °C, as a result of which the tensile strength of the steel sheet decreases. If the oxidation amount is increased to ensure a sufficient amount of Si internally oxidized, oxide scale attaches to rolls in the annealing furnace, inducing pressing flaws, i.e. pick-up defects, in the steel sheet. The means for simply increasing the oxidation amount is therefore not applicable.

**[0010]** With the method described in PTL 2, since the heating or soaking in the upstream heating zone, downstream heating zone, and soaking zone is performed by indirect heating, the oxidation of the surface of the steel sheet like that by direct firing in PTL 1 is unlikely to occur, and the internal oxidation of Si is insufficient as compared with PTL 1. The problem of an increase in alloying temperature is therefore more serious. Moreover, not only the amount of moisture brought into the furnace varies depending on the external air temperature change or the steel sheet type, but also the dew point of the mixed gas tends to vary depending on the external air temperature change, making it difficult to stably control the dew point in the optimal dew point range. Due to such large dew point variation, surface defects such as non-coating occur even within the aforementioned dew point ranges and temperature ranges. The production of stable products is therefore difficult.

**[0011]** With the method described in PTL 3, although the use of a DFF in the heating furnace may enable the oxidation of the surface of the steel sheet, stably controlling the dew point in a high dew point range of -20 °C to 0 °C in the aforementioned control range is difficult because humidified gas is not actively supplied to the annealing furnace. Besides, in the case where the dew point increases, the dew point in the upper part of the furnace tends to be high. For example, while a dew point meter in the lower part of the furnace indicates 0 °C, the atmosphere in the upper part of the furnace has a high dew point of +10 °C or more. Operating the furnace in such a state for a long time has been found to cause pick-up defects.

**[0012]** It could therefore be helpful to provide a method of producing a hot-dip galvanized steel sheet whereby favorable coating appearance can be obtained with high coating adhesion even in the case of hot-dip galvanizing a steel strip whose Si content is 0.2 mass% or more.

35 (Solution to Problem)

[0013] We provide the following:

[1] A method of producing a hot-dip galvanized steel sheet using a continuous hot-dip galvanizing apparatus that includes: an annealing furnace in which a heating zone, a soaking zone, and a cooling zone are arranged in the stated order; and a hot-dip galvanizing line adjacent to the cooling zone, the method comprising: annealing a steel strip by conveying the steel strip through the heating zone, the soaking zone, and the cooling zone in the stated order inside the annealing furnace; and applying a hot-dip galvanized coating onto the steel strip discharged from the cooling zone, using the hot-dip galvanizing line, wherein reducing gas or non-oxidizing gas is supplied into the soaking zone, the reducing gas or the non-oxidizing gas including: humidified gas humidified by a humidifying device; and dry gas not humidified by the humidifying device, and while a width and a sheet passing speed of the steel strip passing through the soaking zone are constant, a variation of pressure in the annealing furnace is suppressed by adjusting a flow rate of the dry gas, and a variation range of an amount of moisture supplied into the soaking zone by the humidified gas is limited to 20 % or less.

[2] The method of producing a hot-dip galvanized steel sheet according to [1], wherein a flow rate and a dew point of the humidified gas are set so that an amount of moisture M supplied into the soaking zone by the humidified gas and expressed in g/min satisfies the following Formula (1):

$$40 + Vf(W - 0.9)(S + 4)/90 < M < 60 + Vf(W - 0.9)(S + 4)/90 \dots (1)$$

where Vf is a volume of the soaking zone expressed in m³, W is the width of the steel strip passing through the

soaking zone and expressed in m, and S is the sheet passing speed of the steel strip expressed in m/s.

- [3] The method of producing a hot-dip galvanized steel sheet according to [2], wherein when at least one of the width and the sheet passing speed of the steel strip passing through the soaking zone varies, the flow rate and the dew point of the humidified gas are changed so that the amount of moisture M expressed in g/min satisfies the Formula (1).
- [4] The method of producing a hot-dip galvanized steel sheet according to any one of [1] to [3], wherein a dew point in the soaking zone measured at a dew point measurement port provided in the soaking zone is controlled to -25 °C or more and 0 °C or less, the dew point measurement port being provided in a region of upper 1/2 of the soaking zone in a height direction, and located 1 m or more away from a position of a supply port of the humidified gas provided in the soaking zone and 1 m or more away from an inner wall position of the soaking zone facing the supply port.
- [5] The method of producing a hot-dip galvanized steel sheet according to any one of [1] to [4], wherein the heating zone includes a direct fired furnace, the continuous hot-dip galvanizing apparatus includes an alloying line adjacent to the hot-dip galvanizing line, and the method further comprises heat-alloying the galvanized coating applied on the steel strip, using the alloying line.

#### (Advantageous Effect)

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**[0014]** With the disclosed method of producing a hot-dip galvanized steel sheet, it is possible to obtain favorable coating appearance with high coating adhesion even in the case of hot-dip galvanizing a steel strip whose Si content is 0.2 mass% or more.

#### BRIEF DESCRIPTION OF THE DRAWINGS

[0015] In the accompanying drawings:

- FIG. 1 is a sectional diagram illustrating the structure of a continuous hot-dip galvanizing apparatus 100 according to one of the disclosed embodiments; and
- FIG. 2 is a schematic diagram illustrating a system of supplying humidified gas and dry gas to a soaking zone 12 in FIG. 1.

## **DETAILED DESCRIPTION**

- **[0016]** The structure of a continuous hot-dip galvanizing apparatus 100 used in a method of producing a hot-dip galvanized steel sheet according to one of the disclosed embodiments is described first, with reference to FIG. 1. The continuous hot-dip galvanizing apparatus 100 includes: an annealing furnace 20 in which a heating zone 10, a soaking zone 12, and cooling zones 14 and 16 are arranged in this order; a hot-dip galvanizing bath 22 as a hot-dip galvanizing line adjacent to the cooling zone 16; and an alloying line 23 adjacent to the hot-dip galvanizing bath 22. In this embodiment, the heating zone 10 includes a first heating zone 10A (upstream heating zone) and a second heating zone 10B (downstream heating zone). The cooling zone includes a first cooling zone 14 (rapid cooling zone) and a second cooling zone 16 (slow cooling zone). A snout 18 connected to the second cooling zone 16 has its tip immersed in the hot-dip galvanizing bath 22, thus connecting the annealing furnace 20 and the hot-dip galvanizing bath 22.
- [0017] A steel strip P is introduced from a steel strip introduction port in the lower part of the first heating zone 10A into the first heating zone 10A. One or more hearth rolls are arranged in the upper and lower parts in each of the zones 10, 12, 14, and 16. In the case where the steel strip P is folded back by 180 degrees at one or more hearth rolls, the steel strip P is conveyed vertically a plurality of times inside the corresponding predetermined zone in the annealing furnace 20, forming a plurality of passes. While FIG. 1 illustrates an example of having 10 passes in the soaking zone 12, 2 passes in the first cooling zone 14, and 2 passes in the second cooling zone 16, the numbers of passes are not limited to such, and may be set as appropriate depending on the processing condition. At some hearth rolls, the steel strip P is not folded back but changed in direction at the right angle to move to the next zone. The steel strip P is thus annealed in the annealing furnace 20 by being conveyed through the heating zone 10, the soaking zone 12, and the cooling zones 14 and 16 in this order.
- **[0018]** Adjacent zones in the annealing furnace 20 communicate through a communication portion connecting the upper parts or lower parts of the respective zones. In this embodiment, the first heating zone 10A and the second heating zone 10B communicate through a throat (restriction portion) connecting the upper parts of the respective zones. The second heating zone 10B and the soaking zone 12 communicate through a throat connecting the lower parts of the respective zones. The soaking zone 12 and the first cooling zone 14 communicate through a throat connecting the lower parts of the respective zones. The first cooling zone 14 and the second cooling zone 16 communicate through a throat

connecting the lower parts of the respective zones. The height of each throat may be set as appropriate. To enhance the independence of the atmosphere in each zone, the height of each throat is preferably as low as possible. The gas in the annealing furnace 20 flows from downstream to upstream in the furnace, and is discharged from the steel strip introduction port in the lower part of the first heating zone 10A.

(Heating zone)

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[0019] In this embodiment, the second heating zone 10B is a direct fired furnace (DFF). The DFF may be a well-known DFF. A plurality of burners are distributed on the inner wall of the direct fired furnace in the second heating zone 10B so as to face the steel strip P, although not illustrated in FIG. 1. Preferably, the plurality of burners are divided into a plurality of groups, and the combustion rate and the air ratio in each group are independently controllable. Combustion exhaust gas in the second heating zone 10B is supplied into the first heating zone 10A, and the steel strip P is preheated by the heat of the gas.

**[0020]** The combustion rate is a value obtained by dividing the amount of fuel gas actually introduced into a burner by the amount of fuel gas of the burner under its maximum combustion load. The combustion rate at the time of combustion by the burner under its maximum combustion load is 100 %. When the combustion load is low, the burner cannot maintain a stable combustion state. Accordingly, the combustion rate is preferably adjusted to 30 % or more.

[0021] The air ratio is a value obtained by dividing the amount of air actually introduced into a burner by the amount of air necessary for complete combustion of fuel gas. In this embodiment, the heating burners in the second heating zone 10B are divided into four groups (#1 to #4), and the three groups (#1 to #3) upstream in the steel sheet traveling direction are made up of oxidizing burners, and the last group (#4) is made up of reducing burners. The air ratio of the oxidizing burners and the air ratio of the reducing burners are independently controllable. The air ratio of the oxidizing burners is preferably adjusted to 0.95 or more and 1.5 or less. The air ratio of the reducing burners is preferably adjusted to 0.5 or more and less than 0.95. The temperature in the second heating zone 10B is preferably adjusted to 800 °C to 1200 °C.

(Soaking zone)

[0022] In this embodiment, the soaking zone 12 is capable of indirectly heating the steel strip P using a radiant tube (RT) (not illustrated) as heating means. The average temperature Tr (°C) in the soaking zone 12 is measured by a thermocouple inserted into the soaking zone. The average temperature Tr (°C) in the soaking zone 12 is preferably adjusted to 700 °C to 900 °C.

**[0023]** Reducing gas or non-oxidizing gas is supplied into the soaking zone 12. As the reducing gas,  $H_2$ - $N_2$  mixed gas is typically used. An example is gas (dew point: about -60 °C) having a composition containing 1 vol% to 20 vol%  $H_2$  with the balance being  $N_2$  and incidental impurities. An example of the non-oxidizing gas is gas (dew point: about -60 °C) having a composition containing  $N_2$  and incidental impurities.

**[0024]** In this embodiment, the reducing gas or non-oxidizing gas supplied into the soaking zone 12 has two forms, namely, humidified gas and dry gas. Here, "dry gas" is reducing gas or non-oxidizing gas having a dew point of about -60 °C to -50 °C and not humidified by a humidifying device. Meanwhile, "humidified gas" is gas humidified by a humidifying device to a dew point of 0 °C to 30 °C.

**[0025]** For example, when producing a high tensile strength steel sheet having a chemical composition containing 0.2 mass% or more Si, it is preferable to supply the humidified gas to the soaking zone 12 in addition to the dry gas, in order to increase the dew point in the soaking zone. On the other hand, for example when producing an ordinary steel sheet (tensile strength of about 270 MPa), it is preferable to supply only the dry gas to the soaking zone 12 without supplying humidified gas.

[0026] FIG. 2 is a schematic diagram illustrating a system of supplying the humidified gas and the dry gas to the soaking zone 12. The humidified gas is supplied through three systems, namely, humidified gas supply ports 42A, 42B, and 42C, humidified gas supply ports 44A, 44B, and 44C, and humidified gas supply ports 46A, 46B, and 46C. In FIG. 2, a gas distribution device 24 feeds part of the reducing gas or non-oxidizing gas (dry gas) to a humidifying device 26. The remaining part passes through a dry gas pipe 32 as dry gas, and is supplied into the soaking zone 12 from dry gas supply ports 48A, 48B, 48C, and 48D.

**[0027]** The positions and number of dry gas supply ports are not limited, and may be determined as appropriate based on various conditions. Preferably, a plurality of dry gas supply ports are provided at the same height position. Moreover, the dry gas supply ports are preferably distributed evenly in the steel strip traveling direction.

**[0028]** The gas humidified in the humidifying device 26 is distributed into the three systems by a humidified gas distribution device 30, passes through respective humidified gas pipes 36, and is supplied into the soaking zone 12 from the humidified gas supply ports 42A, 42B, and 42C, the humidified gas supply ports 44A, 44B, and 44C, and the humidified gas supply ports 46A, 46B, and 46C.

[0029] The positions and number of humidified gas supply ports are not limited, and may be determined as appropriate based on various conditions. Preferably, a humidified gas supply port is provided at each of one or more locations in each of four sections obtained by dividing the soaking zone 12 into halves in the vertical direction and dividing the soaking zone 12 into halves in the entry-delivery direction. This enables uniform dew point control for the whole soaking zone 12. Reference sign 38 is a humidified gas flowmeter, and reference sign 40 is a humidified gas dew point meter. Since the dew point of the humidified gas can change due to slight dew condensation in the humidified gas pipes 34 and 36 or the like, the dew point meters 40 are desirably located immediately before the humidified gas supply ports 42, 44, and 46. [0030] The humidifying device 26 includes a humidifying module having a fluorine or polyimide hollow fiber membrane, flat membrane, or the like. Dry gas flows inside the membrane, whereas pure water adjusted to a predetermined temperature in a circulating constant-temperature water bath 28 circulates outside the membrane. The fluorine or polyimide hollow fiber membrane or flat membrane is a type of ion exchange membrane with affinity for water molecules. When moisture content differs between the inside and outside of the hollow fiber membrane, a force for equalizing the moisture content difference emerges and, with this force as a driving force, moisture transmits through the membrane and moves toward the part with lower moisture content. The temperature of dry gas varies with seasonal or daily air temperature change. In this humidifying device, however, heat exchange is possible by ensuring a sufficient contact area between gas and water through the vapor permeable membrane. Accordingly, regardless of whether the dry gas temperature is higher or lower than the circulating water temperature, the dry gas is humidified to the same dew point as the set water temperature, thus achieving highly accurate dew point control. The dew point of the humidified gas can be controlled to any value in the range of 5 °C to 50 °C. When the dew point of the humidified gas is higher than the pipe temperature, there is a possibility that dew condensation occurs in the pipe and dew condensation water enters directly into the furnace. The humidified gas pipe is therefore heated/heat-retained to be not less than the dew point of the humidified gas and not less than the external air temperature.

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**[0031]** Regardless of whether or not the humidified gas is supplied into the soaking zone 12, the pressure in the annealing furnace varies frequently depending on the combustion condition in the heating zone 10 and the cooling fan operating condition in the cooling zones 14 and 16. If the furnace pressure is excessively high, an excessive force acts on the furnace wall, which can damage the annealing furnace. If the furnace pressure is excessively low, oxygen outside the annealing furnace enters into the soaking zone 12 or the combustion gas in the heating zone 10 flows into the soaking zone 12, thus adversely affecting the steel sheet quality. Hence, such control that changes the flow rate of the gas supplied into the soaking zone 12 is typically performed so as to suppress the variation of the furnace pressure and preferably keep the furnace pressure constant. In the operation of supplying both the humidified gas and the dry gas to the soaking zone 12, the conventional control method changes not only the flow rate of the dry gas but also the flow rate of the humidified gas. Consequently, the amount of moisture supplied into the soaking zone by the humidified gas varies.

[0032] However, the soaking zone 12 needs to be constantly supplied with a necessary amount of moisture in terms of inducing internal oxidation of Si or Mn in the steel strip. If the flow rate of the humidified gas is decreased in order to suppress the variation of the furnace pressure, the amount of moisture supplied into the soaking zone 12 tends to become insufficient. This causes the dew point in the soaking zone 12 to fall below the lower limit of the appropriate range. As a result, partial non-coating occurs and degrades the coating appearance. Besides, in the operation that also involves alloying treatment, the alloying temperature tends to increase, making it impossible to obtain the desired tensile strength. If the flow rate of the humidified gas is increased in order to suppress the variation of the furnace pressure, the amount of moisture supplied into the soaking zone 12 tends to become excessive, as a result of which roll pick-up occurs. Such roll pick-up causes flaws on the steel strip surface, and degrades the coating appearance.

[0033] It is therefore important in this embodiment that, while the width and sheet passing speed of the steel strip passing through the soaking zone 12 are constant (hereafter also referred to as "under the same operation condition"), the variation of the pressure in the annealing furnace is suppressed by adjusting the flow rate of the dry gas, and the amount of moisture supplied into the soaking zone 12 by the humidified gas is kept as constant as possible, specifically, the variation range of the amount of moisture is limited to 20 % or less. This contributes to favorable coating appearance. Moreover, in the operation that also involves alloying treatment, a decrease in tensile strength can be prevented by decreasing the alloying temperature. Here, "the variation range of the amount of moisture" supplied into the soaking zone is defined as  $(M_{max} - M_{min})/M_{max}$ , where  $M_{max}$  is a maximum amount of moisture under the same operation condition and  $M_{min}$  is a minimum amount of moisture under the same operation condition. The amount of moisture can be calculated according to the below-mentioned Formula (2).

**[0034]** How the variation range of the amount of moisture is limited to 20 % or less is not limited. As an example, the dew point of the humidified gas is kept constant to control the variation range of its flow rate to 20 % or less. In the case where a plurality of humidified gas supply ports are provided as in this embodiment, it is preferable to keep both the flow rate of the humidified gas from each supply port and the total flow rate of the humidified gas as constant as possible (e.g. 20 % or less).

[0035] The amount of moisture M (g/min) introduced into the soaking zone 12 by the humidified gas needs to be

adjusted depending on the volume of the soaking zone and the width and sheet passing speed of the steel strip P passing through the soaking zone 12. As a result of keen examination, we discovered that setting the flow rate and dew point of the humidified gas so that the amount of moisture M (g/min) supplied into the soaking zone 12 by the humidified gas satisfies the following Formula (1) is effective in obtaining favorable coating appearance:

$$40 + Vf(W - 0.9)(S + 4)/90 < M < 60 + Vf(W - 0.9)(S + 4)/90 \dots (1)$$

where Vf is the volume (m³) of the soaking zone 12, W is the width (m) of the steel strip P passing through the soaking zone 12, and S is the sheet passing speed (m/s) of the steel strip P.

**[0036]** When at least one of the width W and sheet passing speed S of the steel strip P passing through the soaking zone 12 varies, it is effective to change the flow rate and dew point of the humidified gas so that the amount of moisture M (g/min) satisfies Formula (1).

[0037] The volume Vf of the soaking zone 12 is substantially a constant. In the case where the width W and sheet passing speed S of the steel strip P passing through the soaking zone 12 increase or in the case where one of the width W and the sheet passing speed S is constant and the other one of the width W and the sheet passing speed S increases, the area of the steel strip in contact with the gas in the soaking zone 12 per unit time increases. Accordingly, the amount of moisture by the humidified gas is increased based on Formula (1). In the case where the width W and sheet passing speed S of the steel strip P passing through the soaking zone 12 decrease or in the case where one of the width W and the sheet passing speed S decreases, on the other hand, the amount of moisture by the humidified gas needs to be decreased based on Formula (1). In the case where one of the width W and the sheet passing speed S decreases, too, the amount of moisture by the humidified gas is adjusted based on Formula (1). In any case, it is desirable to adjust the flow rate and dew point of the humidified gas so as to satisfy Formula (1), before the dew point in the soaking zone 12 changes as a result of a change in the operation condition.

[0038] The amount of moisture M (g/min) can be calculated from the dew point Tw (°C) and total flow rate Vm (Nm³/hr) of the humidified gas, according to Formula (2):

$$M = 0.08069 \times Vm \times 10^{7.5Tw/(Tw+237.3)}$$
 ... (2).

**[0039]** The flow rate Vm of the humidified gas supplied into the soaking zone 12 is not limited as long as the aforementioned control is performed, but is generally maintained in the range of 100 to 400 (Nm³/hr). The flow rate of the dry gas supplied into the soaking zone 12 is not limited, but is generally maintained in the range of 10 to 300 (Nm³/hr).

**[0040]** Water vapor has a lower specific gravity than nitrogen gas, and so tends to accumulate in the upper part in the soaking zone 12. Hence, a dew point measurement port 50 is located in a region of upper 1/2 of the soaking zone 12 in the height direction. The vicinity of each humidified gas supply port is a region where the dew point is locally high, and therefore is not suitable for dew point measurement. Accordingly, the dew point measurement port 50 is preferably located 1 m or more away from the position of each humidified gas supply port and 1 m or more away from the inner wall position of the soaking zone facing each of the supply ports. It is also preferable to control the flow rate of the humidified gas so that the dew point in the soaking zone 12 measured at the dew point measurement port 50 is maintained at -25 °C or more and 0 °C or less. This contributes to favorable coating appearance. Moreover, in the operation that also involves alloying treatment, a decrease in tensile strength can be prevented by decreasing the alloying temperature.

(Cooling zone)

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[0041] In this embodiment, the cooling zones 14 and 16 cool the steel strip P. The steel strip P is cooled to about 480 °C to 530 °C in the first cooling zone 14, and cooled to about 470 °C to 500 °C in the second cooling zone 16.

[0042] The cooling zones 14 and 16 are also supplied with the aforementioned reducing gas or non-oxidizing gas. Here, only the dry gas is supplied. The supply of the dry gas to the cooling zones 14 and 16 is not limited, but the dry gas is preferably supplied from introduction ports in two or more locations in the height direction and two or more locations in the longitudinal direction so that the dry gas is evenly introduced into the cooling zones. The total gas flow rate Qcd of the dry gas supplied into the cooling zones 14 and 16 is measured by a gas flowmeter (not illustrated) provided in the pipe. The total gas flow rate Qcd is not limited, but is set to about 200 to 1000 (Nm³/hr). The variation of the pressure in the annealing furnace may be suppressed by adjusting only the flow rate of the dry gas supplied into the cooling zones.

(Hot-dip galvanizing bath)

**[0043]** The hot-dip galvanizing bath 22 can be used to apply a hot-dip galvanized coating onto the steel strip P discharged from the second cooling zone 16. The hot-dip galvanizing may be performed according to a usual method.

(Alloying line)

**[0044]** The alloying line 23 can be used to heat-alloy the galvanized coating applied on the steel strip P. The alloying treatment may be performed according to a usual method. In this embodiment, the alloying temperature is kept from being high, thus preventing a decrease in tensile strength of the produced galvannealed steel sheet. Note that the alloying line 23 and the alloying treatment by the alloying line 23 are not essential in the present disclosure. The effect of obtaining favorable coating appearance can be achieved even in the case of not performing alloying treatment.

**[0045]** The steel strip P subjected to annealing and hot-dip galvanizing is not limited, but the advantageous effects according to the present disclosure can be effectively achieved in the case where the steel strip has a chemical composition containing 0.2 mass% or more Si, i.e. in the case of high tensile strength steel.

**EXAMPLES** 

(Experimental conditions)

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[0046] The continuous hot-dip galvanizing apparatus illustrated in FIGS. 1 and 2 was used to anneal each steel strip whose chemical composition is listed in Table 1 under each annealing condition listed in Table 2, and then hot-dip galvanize and alloy the steel strip. Steel sample ID A and steel sample ID B are both high tensile strength steels. In Table 2, "time" denotes the time elapsed from the operation start, where the type, sheet thickness, and sheet width of the passing steel strip and the operation condition of the continuous hot-dip galvanizing apparatus were changed with the passage of time as listed in Table 2.

**[0047]** A DFF was used as the second heating zone. The heating burners were divided into four groups (#1 to #4) where the three groups (#1 to #3) upstream in the steel sheet traveling direction were made up of oxidizing burners and the last group (#4) was made up of reducing burners, and the air ratios of the oxidizing burners and reducing burners were set to the values listed in Table 2. The length of each group in the steel sheet traveling direction was 4 m.

[0048] A RT furnace having a volume Vr of 700 m<sup>3</sup> was used as the soaking zone. The average temperature Tr in the soaking zone was set to the value listed in Table 2. As dry gas, gas (dew point: -50 °C) having a composition containing 15 vol $^{\circ}$  H $_2$  with the balance being N $_2$  and incidental impurities was used. Part of the dry gas was humidified by a humidifying device having 10 hollow fiber membrane-type humidifying modules, to prepare humidified gas. Dry gas of 500 L/min at the maximum and circulating water of 20 L/min at the maximum were flown in each module. A common circulating constant-temperature water bath capable of supplying pure water of 200 L/min in total was used for each module. The dry gas supply ports and the humidified gas supply ports were arranged at the positions illustrated in FIG. 2. [0049] Eight types of steel strips different from each other in any of steel sample ID, sheet thickness, and sheet width were passed as listed in Table 2. The first half (time 0:00 to 0:55) corresponds to Comparative Examples, and the latter half (time 0:55 to 1:50) corresponds to Examples. In detail, in the sheet passing in the first half, the flow rate of the dry gas supplied into the soaking zone, the flow rate of the humidified gas supplied into the soaking zone, and the flow rate of the dry gas supplied into the cooling zone were varied as listed in Table 2, to keep the furnace pressure constant. In the sheet passing in the latter half, while the type, width, and sheet passing speed of the steel strip passing through the soaking zone were constant, the dew point of the humidified gas was kept constant and the variation range of the flow rate of the humidified gas was limited to 20 % or less, as listed in Table 2. The furnace pressure was kept constant by adjusting the flow rate of the dry gas supplied into the soaking zone and the cooling zone.

**[0050]** In Table 2, the field "dew point" of the soaking zone indicates the dew point in the soaking zone measured at the position of the dew point measurement port 50 in FIG. 2. The field "dew point of vicinity of humidified gas supply port" indicates the dew point in the soaking zone measured at a position of 80 cm away from the humidified gas supply port 40B in FIG. 2. The field "dew point of humidified gas" indicates the dew point measured by the humidified gas dew point meter 40 in FIG. 2.

[0051] The dry gas (dew point: -50 °C) was supplied into the first and second cooling zones from their lowermost parts with the flow rate listed in Table 2.

[0052] The temperature of the molten bath was set to 460 °C, the A1 concentration in the molten bath was set to 0.130 %, and the coating weight was adjusted to  $50 \text{ g/m}^2$  per surface by gas wiping. The line speed was set to 1.0 m/s to 2.0 m/s, with the change of the sheet thickness. After the hot-dip galvanizing, alloying treatment was performed in an induction heating-type alloying furnace so that the coating alloying degree (Fe content) was 10 % to 13 %. The alloying temperature in the treatment is listed in Table 2.

#### (Evaluation methods)

[0053] The evaluation of the coating appearance was conducted through inspection by an optical surface defect meter (detection of non-coating defects or roll pick-up flaws of  $\phi$ 0.5 or more) and visual determination of alloying unevenness. Samples that passed in all criteria were rated as "Good", samples that had a low degree of alloying unevenness were rated as "Fair", and samples that failed in at least one of the criteria were rated as "Poor". The results are listed in Table 2. [0054] In addition, the tensile strength of a galvannealed steel sheet produced under each condition was measured. Steel sample ID A of high tensile strength steel was rated as "pass" when the tensile strength was 590 MPa or more, and steel sample ID B of high tensile strength steel was rated as "pass" when the tensile strength was 980 MPa or more. The results are listed in Table 2.

#### (Evaluation results)

[0055] In Comparative Examples, in the case where the dew point in the soaking zone was less than -25 °C, the coating appearance degraded due to partial non-coating, and also the tensile strength was rated as "fail" with an increase in alloying temperature. In the case where the dew point in the soaking zone was more than 0 °C, roll pick-up occurred, and caused flaws on the steel strip surface, resulting in degraded coating appearance. In time periods of 0:20, 0:35, and 0:45, the amount of moisture satisfied Formula (1), but the variation of the amount of moisture from the preceding or succeeding time period was significant and the dew point was outside the range of -25 °C to 0 °C, so that a low degree of alloying unevenness was seen.

**[0056]** In Examples, a predetermined amount of moisture was able to be stably supplied even when the total gas flow rate in the soaking zone changed, so that the surface appearance was favorable throughout the length and width of the coil, and desired tensile characteristics were obtained. In time periods of 1:20 to 2:00 in which the variation of the amount of moisture was limited to 20 % or less, Formula (1) was satisfied, and the dew point was controlled to -25 °C to 0 °C, especially high tensile strength and surface appearance were obtained stably.

Table 1

(mass%)												
Steel ID	С	Si	Mn	Р	S							
Α	0.08	0.25	1.5	0.03	0.001							
В	0.11	1.5	2.7	0.01	0.001							

		Category		Comparative Example	Example	Example									
5	Tensile strength (MPa)		575	594	1050	586	566	965	973	1002	096	866	1052	612	809
		Coating		Poor	Poor	Poor	Fair	Poor	Poor	Fair	Poor	Fair	Poor	Good	Good
10	Alloying treatment		570	530	495	530	512	555	542	512	558	\$10	492	505	507
	Cooling zone	Gas flow rate (Nm³/hr)	510	200	510	400	510	450	200	550	570	969	520	900	969
15	1)	Right side   Determination	Not satisfied	Not satisfied	Not satisfied	Not satisfied	Satisfied	Not satisfied	Not satisfied	Satisfied	Not satisfied	Satisfied	Not satisfied	Satisfied	Satisfied
	Formula (1)	Right side	64	64	09	09	09	72	72	72	88	88	88	88	<b>8</b> 8
20		Left side	44	44	40	40	40	52	52	52	89	89	89	89	89
		Variation range of amount of moisture (%)	0.001	2.0	46.0			21.2				36.9	6.2		7.0
25 30		Amount of moisture of humidified gas M (g/min)	0	34	72	39	55	42	49	54	57	82	96	72	89
		Flow rate of Dew point of moisture of humidified gas humidified gas humidified gas (Mni <sup>2</sup> /hr) (°C') M	,	10.0	15.0	10.0	10.0	10.0	10.0	10.0	10.0	14.0	19.0	15.0	15.0
	Soaking zone (RTF)		0	210	320	240	340	260	300	330	350	390	310	320	300
	Soakin	Flow rate of dry gas (Nm³/hr)	430	215	100	75	105	80	95	105	110	120	100	110	190
35		Average temperature Tr (°C)	801	805	825	830	829	831	829	826	833	830	825	808	802
		Dew point of vicinity of humidified gas supply port (°C)	-39.5	-20.3	6.1	-18.2	-12.7	-16.5	-12.1	-11.5	-10.3	-5.2	5.3	-7.2	-9.2
45		Dew point (°C)	-38.2	-28.3	2.2	-26.5	-18.2	-29.2	-26.5	-23.8	-29.2	-17.3	1.2	-16.2	-17.2
	6	Delivery temperature (°C)	681	682	730	725	723	735	721	722	732	723	719	829	683
	Heating zone (DFF)	Air ratio of Delivery reducing temperature burner (°C)	0.85	0.85	0.85	0.85	0.85	0.85	0.85	0.85	0.85	0.85	0.85	0.85	0.85
	H	Air ratio of oxidizing burner	0.95	0.95	1.10	1.10	1.10	1.10	1.10	1.10	1.10	1.10	1.10	0.95	0.95
		Sheet speed (m/s)		00.1	0.0	00.1	00.1	1.30	1.30	1.30	2.00	2.00	2.00	2.00	2.00
50		Sheet width (mm)		1000	006	006	006	1200	1200	1200	1500	1500	1500	1500	1500
		Sheet Sheet thickness width (mm) (mm)		8.	8.	8:	F.8	1.4	1.4	4:1	1.0	1.0	1.0	1.0	0.1
7	3 0		0:00 A	V	В	м	8	æ	m	В	В	m m	В	V	∢
Table 2		Time		0.02	0:10	0.15	0.20	0.25	0:30	0.35	0:40	0:45	0:20	0.55	1:00

					,		T				1		·	
	- Parketen	Category		Example	Example	Example	Example	Example	Example	Example	Example	Example	Example	Example
5		Tensile strength (MPa)		866	1001	1021	1032	1045	1030	1020	1055	1058	1020	1015
		Coating		Good	Good	Good	Good	Good	Good	Good	Good	Good	Good	Good
10	Alloying treatment	Alloying temperature (°C)	520	517	514	503	505	498	505	909	497	496	503	508
	Cooling zone	Gas flow rate (Nm³/hr)	550	200	510	520	490	450	550	520	480	400	200	350
15	a	Right side   Determination	Not satisfied	Not satisfied	Not satisfied	Satisfied								
	Formula (1)	Right side	88	88	88 88	88	88	88	72	72	72	09	09	09
20		Left side	89	89	89	89	89	89	52	52	32	40	40	40
		Variation range of amount of moisture (%)		17.2	J				5.7			12.5		
25	Wilder	How rate of Dew point of workure of range of humidified gas humidified gas amount of M moisture (g/min) (%)	54	19	65	72	11	81	57	55	54	52	47	45
	1100	Dew point of humidified gas (°C)	15.0	15.0	15.0	15.0	15.0	15.0	10.0	10.0	10.0	0.01	10.0	10.0
30	Soaking zone (RTF)		240	270	290	320	340	360	350	340	330	320	290	280
	Soakin	Flow rate of dry gas (Nm³/hr)	50	70	70	06	70	10	190	150	50	20	160	200
<ul><li>40</li><li>45</li></ul>		Average temperature Tr (°C)	820	825	830	827	833	830	828	830	831	829	830	833
		Dew point of vicinity of humidified gas supply port (°C)	-14.3	==	-10.7	-6.2	-5.2	4.7	-10.3	-7.5	-5.2	-12	-5.7	-8.3
		Dew point (°C)	-24.5	-21.3	-20.2	-15.2	-13.2	-12.3	-18.7	-16.5	-9.5	-6.3	-13.2	-19.2
	FF)	Delivery: temperature (°C)	722	720	723	720	723	721	717	725	721	716	725	722
	Heating zone (DFF)	Air ratio of reducing burner	0.85	0.85	0.85	0.85	0.85	0.85	0.85	0.85	0.85	0.85	0.85	0.85
	#	Air ratio of oxidizing burner	1.10	1.10	1.10	1.10	1.10	1.10	1.10	1.10	1.10	1.10	1.10	1.10
50		Sheet Sheet Sheet thickness width speed (mm) (mm) (m/s)		2.00	2.00	2.00	2.00	2.00	1.30	1.30	1.30	1.00	1.00	1.00
				1500	1500	1500	1500	1500	1200	1200	1200	900	006	006
<del>©</del>	£			1.0	1.0	1.0	1.0	1.0	1.4	1.4	1.4	1.8	1.8	8:1
55	Table 2 (cont'd)	Seed E		m	В	В	В	В	В	В	В	В	20	В
Iable 25		Time	1:05	1:10	1.15	1.20	125	130	135	1:40	1:45	1.50	155	2:00

#### INDUSTRIAL APPLICABILITY

**[0057]** With the disclosed method of producing a hot-dip galvanized steel sheet, favorable coating appearance with high coating adhesion can be obtained even in the case of hot-dip galvanizing a steel strip whose Si content is 0.2 mass% or more. Moreover, when further performing alloying treatment, the alloying temperature can be kept from becoming high, thus preventing a decrease in tensile strength of the produced galvannealed steel sheet.

#### REFERENCE SIGNS LIST

## 10 [0058]

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100 continuous hot-dip galvanizing apparatus

10 heating zone

10A first heating zone (upstream)

10B second heating zone (downstream, direct fired furnace)

12 soaking zone

14 first cooling zone (rapid cooling zone)

16 second cooling zone (slow cooling zone)

18 snout

20 20 annealing furnace

22 hot-dip galvanizing bath

23 alloying line

24 dry gas distribution device

26 humidifying device

25 28 circulating constant-temperature water bath

30 humidified gas distribution device

32 dry gas pipe

34, 36 humidified gas pipe

38 humidified gas flowmeter

30 40 humidified gas dew point meter

42A, 42B, 42C humidified gas supply port

44A, 44B, 44C humidified gas supply port

46A, 46B, 46C humidified gas supply port

48A, 48B, 48C, 48D dry gas supply port

50 dew point measurement port

52A upper hearth roll

52B lower hearth roll

P steel strip

Claims

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1. A method of producing a hot-dip galvanized steel sheet using a continuous hot-dip galvanizing apparatus that includes: an annealing furnace in which a heating zone, a soaking zone, and a cooling zone are arranged in the stated order; and a hot-dip galvanizing line adjacent to the cooling zone, the method comprising:

annealing a steel strip by conveying the steel strip through the heating zone, the soaking zone, and the cooling zone in the stated order inside the annealing furnace; and

applying a hot-dip galvanized coating onto the steel strip discharged from the cooling zone, using the hot-dip galvanizing line,

wherein reducing gas or non-oxidizing gas is supplied into the soaking zone, the reducing gas or the non-oxidizing gas including: humidified gas humidified by a humidifying device; and dry gas not humidified by the humidifying device, and

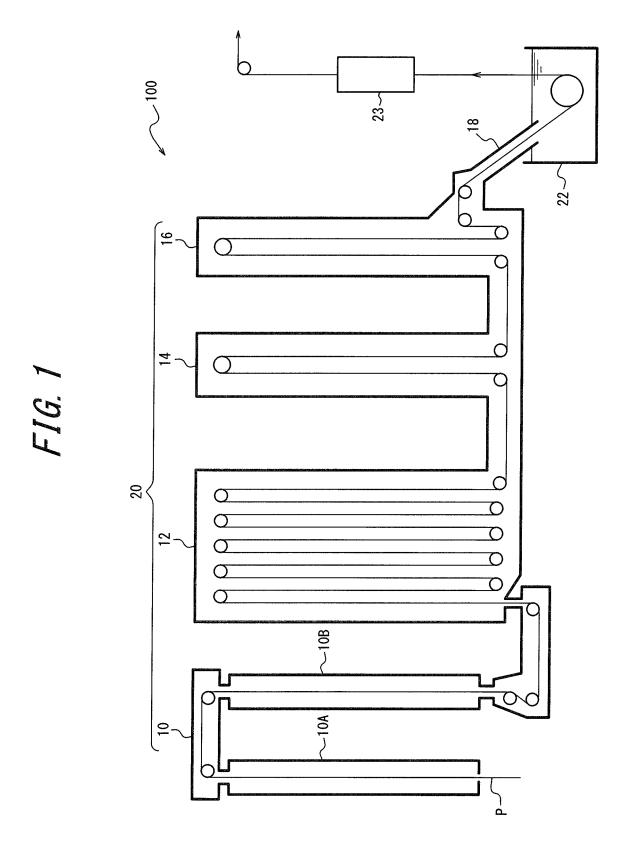
while a width and a sheet passing speed of the steel strip passing through the soaking zone are constant, a variation of pressure in the annealing furnace is suppressed by adjusting a flow rate of the dry gas, and a variation range of an amount of moisture supplied into the soaking zone by the humidified gas is limited to 20 % or less.

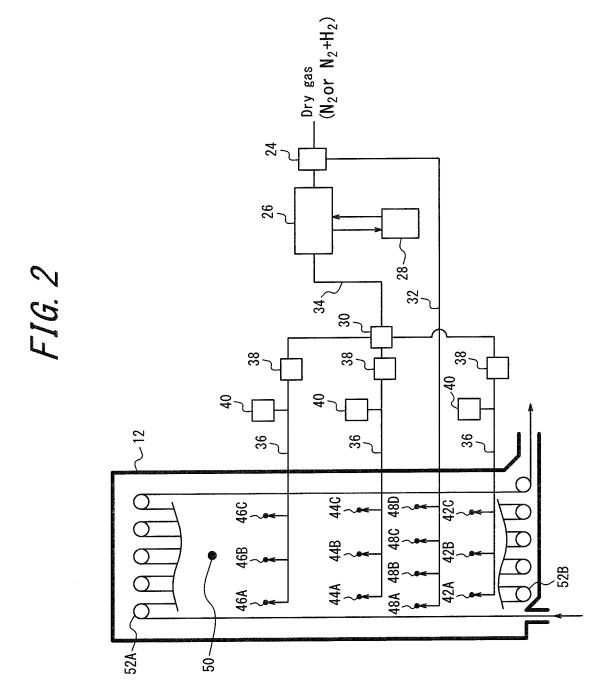
2. The method of producing a hot-dip galvanized steel sheet according to claim 1, wherein a flow rate and a dew point of the humidified gas are set so that an amount of moisture M supplied into the soaking zone by the humidified gas and expressed in g/min satisfies the following Formula (1):

$$40 + Vf(W - 0.9)(S + 4)/90 < M < 60 + Vf(W - 0.9)(S + 4)/90 \dots (1)$$

where Vf is a volume of the soaking zone expressed in m<sup>3</sup>, W is the width of the steel strip passing through the soaking zone and expressed in m, and S is the sheet passing speed of the steel strip expressed in m/s.

- 3. The method of producing a hot-dip galvanized steel sheet according to claim 2, wherein when at least one of the width and the sheet passing speed of the steel strip passing through the soaking zone varies, the flow rate and the dew point of the humidified gas are changed so that the amount of moisture M expressed in g/min satisfies the Formula (1).
- 4. The method of producing a hot-dip galvanized steel sheet according to any one of claims 1 to 3, wherein a dew point in the soaking zone measured at a dew point measurement port provided in the soaking zone is controlled to -25 °C or more and 0 °C or less, the dew point measurement port being provided in a region of upper 1/2 of the soaking zone in a height direction, and located 1 m or more away from a position of a supply port of the humidified gas provided in the soaking zone and 1 m or more away from an inner wall position of the soaking zone facing the supply port.
- 5. The method of producing a hot-dip galvanized steel sheet according to any one of claims 1 to 4, wherein the heating zone includes a direct fired furnace, the continuous hot-dip galvanizing apparatus includes an alloying line adjacent to the hot-dip galvanizing line, and the method further comprises heat-alloying the galvanized coating applied on the steel strip, using the alloying line.





#### INTERNATIONAL SEARCH REPORT International application No. PCT/JP2016/003631 A. CLASSIFICATION OF SUBJECT MATTER 5 C23C2/02(2006.01)i, C21D1/76(2006.01)i, C21D9/56(2006.01)i, C23C2/06 (2006.01)i, C23C2/28(2006.01)i, C23C2/40(2006.01)i, C22C38/00(2006.01)n, C22C38/04(2006.01)n According to International Patent Classification (IPC) or to both national classification and IPC FIELDS SEARCHED 10 Minimum documentation searched (classification system followed by classification symbols) C23C2/02, C21D1/76, C21D9/56, C23C2/06, C23C2/28, C23C2/40, C22C38/00, C22C38/04 Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched 1922-1996 Jitsuyo Shinan Koho Jitsuyo Shinan Toroku Koho 1996-2016 15 Kokai Jitsuyo Shinan Koho 1971-2016 Toroku Jitsuyo Shinan Koho 1994-2016 Electronic data base consulted during the international search (name of data base and, where practicable, search terms used) 20 DOCUMENTS CONSIDERED TO BE RELEVANT Category\* Citation of document, with indication, where appropriate, of the relevant passages Relevant to claim No. Y WO 2015/129202 A1 (JFE Steel Corp.), 1 - 503 September 2015 (03.09.2015), claims; paragraphs [0001] to [0016], [0027] to 25 [0031], [0035] to [0048]; fig. 1 to 3 & TW 201538743 A JP 02-310323 A (NKK Corp.), 1 - 526 December 1990 (26.12.1990), claims; page 2; examples; fig. 1 to 6 30 (Family: none) 35 Further documents are listed in the continuation of Box C. See patent family annex. 40 Special categories of cited documents later document published after the international filing date or priority date and not in conflict with the application but cited to understand "A" document defining the general state of the art which is not considered to the principle or theory underlying the invention earlier application or patent but published on or after the international filing 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 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) 45 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 "O" document referring to an oral disclosure, use, exhibition or other means document published prior to the international filing date but later than the document member of the same patent family Date of the actual completion of the international search Date of mailing of the international search report 50 06 September 2016 (06.09.16) 26 August 2016 (26.08.16) Name and mailing address of the ISA/ Authorized officer Japan Patent Office 3-4-3, Kasumigaseki, Chiyoda-ku, Tokyo 100-8915, Japan Telephone No. 55 Form PCT/ISA/210 (second sheet) (January 2015)

#### REFERENCES CITED IN THE DESCRIPTION

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## Patent documents cited in the description

- JP 2010202959 A [0005] [0008]
- WO 2007043273 A1 [0006] [0008]

• JP 2009209397 A [0007] [0008]