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(54) **HIGH-STRENGTH STEEL PLATE FOR SOUR-RESISTANT LINE PIPES AND HIGH-STRENGTH STEEL PIPE USING SAME**

HOCHFESTES STAHLBLECH FÜR SAUER GASRESISTENTE LEITUNGSROHRE UND HOCHFESTES STAHLROHR DAMIT

TÔLE D'ACIER À HAUTE RÉ S I S T A N C E P O U R T U Y A U D E C A N A L I S A T I O N R É S I S T A N T À L'ACIDITÉ ET TUYAU EN ACIER À HAUTE RÉ S I S T A N C E L'UTILISANT

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(73) Proprietor: **JFE Steel Corporation**

**Tokyo 100-0011 (JP)**

(72) Inventors:

- **SHIMAMURA Junji**  
**Tokyo 100-0011 (JP)**

- **YOKOTA Tomoyuki**

**Tokyo 100-0011 (JP)**

- **IZUMIKAWA Shinichi**

**Tokyo 100-0011 (JP)**

(74) Representative: **Haseltine Lake Kempner LLP**

**Bürkleinstrasse 10  
80538 München (DE)**

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**Description**

## TECHNICAL FIELD

5 **[0001]** This disclosure relates to a high strength steel plate for sour-resistant line pipes that is used in the field of sour-resistant line pipes and that is excellent in the uniformity of material homogeneity, in particular HIC characteristics, in the steel plate, and to a high strength steel pipe using the same.

## BACKGROUND

10 **[0002]** In general, a line pipe is manufactured by forming a steel plate manufactured by a plate mill or a hot rolling mill into a steel pipe by UOE forming, press bend forming, roll forming, or the like.

15 **[0003]** The line pipe used to transport crude oil and natural gas containing hydrogen sulfide is required to have so-called sour resistance such as resistance to hydrogen-induced cracking (HIC resistance) and resistance to sulfide stress corrosion cracking (SSCC resistance), in addition to strength, toughness, weldability, and so on. Above all, in HIC, hydrogen ions caused by corrosion reaction adsorb on the steel surface, penetrate into the steel as atomic hydrogen, diffuse and accumulate around non-metallic inclusions such as MnS in the steel and the hard second phase structure, and become molecular hydrogen, thereby causing cracking due to its internal pressure.

20 **[0004]** Several methods have been proposed to prevent such HIC. JPH5-271766A (PTL 1) and JPH7-173536A (PTL 2) propose methods for suppressing central segregation in a high strength steel plate by keeping the C and Mn contents low, while performing morphological control of sulfide inclusions by keeping the S content low and adding Ca, and supplementing the decrease in strength associated therewith by adding Cr, Mo, Ni, and the like and performing accelerated cooling.

25 **[0005]** On the other hand, the demand for a steel plate with higher strength and higher toughness is increasing from the viewpoint of increasing the size of steel structures and reducing costs. For the purposes of property improvement and alloying element reduction of steel plates and elimination of heat treatment, high strength steel plates are usually manufactured by applying a so-called TMCP (Thermo-Mechanical Control Process) technique combining controlled rolling and controlled cooling.

30 **[0006]** In order to increase the strength of the steel material by using TMCP technique, it is effective to increase the cooling rate at the time of controlled cooling. However, when the control cooling is performed at a high cooling rate, the surface layer of the steel plate is rapidly cooled, and the hardness of the surface layer becomes higher than that of the inside of the steel plate, and the hardness distribution in the plate thickness direction becomes uneven. Therefore, it is a problem in terms of ensuring the material homogeneity in the steel plate.

35 **[0007]** To solve the above problems, for example, JP2002-327212A (PTL 3) and JP3711896 B (PTL 4) describe methods for producing a steel plate for line pipes in which a steel plate surface after subjection to accelerated cooling is heated to a higher temperature than the interior using a high-frequency induction heating device such that the hardness is reduced at the surface layer.

40 **[0008]** On the other hand, when the scale thickness on the steel plate surface is uneven, the cooling rate is also uneven at the underlying steel plate during cooling, causing a problem of local variation in the cooling stop temperature in the steel plate. As a result, unevenness in scale thickness causes variation in the steel plate material property in the plate width direction. On the other hand, JPH9-57327A (PTL 5) and JP3796133B (PTL 6) describe methods for improving the shape of a steel plate by performing descaling immediately before cooling to suppress cooling unevenness caused by scale thickness unevenness. JP2013124398A describes a steel sheet or steel slab for high strength sour resistant line pipe, for which a concentration mapping analysis in a region is carried out.

## 45 CITATION LIST

## Patent Literature

50 **[0009]**

PTL 1: JPH5-271766A  
 PTL 2: JPH7-173536A  
 PTL 3: JP2002-327212A  
 55 PTL 4: JP3711896 B  
 PTL 5: JPH9-57327A  
 PTL 6: JP3796133B

SUMMARY

(Technical Problem)

5 **[0010]** However, although the techniques of PTLs 1 to 4 focus on central segregation area, none of these documents consider the uniformity of the HIC resistance in the plate width direction. Variation in central segregation in the plate width direction in a slab result in variation in the HIC resistance in the plate width direction of the rolled steel plate.

**[0011]** Further, according to the present inventors' study, it turned out that there is still a room for improvement in high strength steel plates obtained by the methods described in PTLs 5 and 6 in terms of uniformity of the HIC resistance in the plate width direction. The reason can be considered as follows. The methods of PTLs 5 and 6 apply descaling to reduce the surface characteristics defects due to the scale indentation during hot leveling and to reduce the variation in the cooling stop temperature of the steel plate to improve the steel plate shape. However, no consideration is given to the cooling conditions for obtaining a uniform material property.

10 **[0012]** Thus, conventionally, when combining low-cost chemical compositions and controlled cooling at a high cooling rate, it was impossible to manufacture a high strength steel plate that has both of proper material homogeneity in the steel plate and proper HIC resistance.

**[0013]** It would thus be helpful to provide a high strength steel plate for sour-resistant line pipes that is excellent in HIC resistance in which variation in the HIC resistance in the plate width direction is suppressed, and a high strength steel pipe using the same.

20 (Solution to Problem)

**[0014]** To solve the above problems, the present inventors made intensive studies on the chemical compositions and microstructures of steel materials and the manufacturing methods of a high strength steel plate having a strength of X65 grade in accordance with the API standard in order to prevent HIC generation from the central segregation area, suppress the variation in the HIC resistance in the plate width direction, and improve the material homogeneity in the steel plate. As a result, it was discovered that it is possible to suppress the variation in central segregation in the plate width direction of a steel plate by a combined use of secondary cooling of a cast steel (slab) under particular conditions and controlled cooling after hot rolling under particular conditions, and the present disclosure was completed based on this discovery.

25 **[0015]** We thus provide a high strength steel plate as defined in claim 1 and a high strength steel pipe using the high strength steel plate as defined in claim 2.

(Advantageous Effect of Invention)

30 **[0016]** The high strength steel plate for sour-resistant line pipes disclosed herein has excellent HIC resistance in which variation in the HIC resistance in the plate width direction is suppressed. Accordingly, the high strength steel pipe disclosed herein using this high strength steel plate has excellent HIC resistance in which variation in the HIC resistance in the pipe circumferential direction is suppressed.

40 BRIEF DESCRIPTION OF THE DRAWINGS

**[0017]** In the accompanying drawings:

45 FIG. 1 is a schematic view illustrating a C cross-section of a steel plate for explanation of the positions of EPMA analyzing regions in an example;

FIG. 2 is a schematic view illustrating a C cross-section of a steel plate for explanation of the portions for HIC test piece sampling in an example; and

50 FIGS. 3A and 3B are diagrams for explaining secondary cooling of cast steel in continuous casting for producing a high strength steel plate according to the present embodiment, where FIG. 3A is a schematic diagram illustrating an injection range and a water flux density distribution of coolant when the coolant is injected from one two-fluid spray nozzle, and FIG. 3B is a schematic diagram illustrating an injection range and a water flux density distribution of coolant and a lap margin of the injection range when the coolant is injected from two two-fluid spray nozzles.

DETAILED DESCRIPTION

55 **[0018]** Hereinafter, the high strength steel plate for sour-resistant line pipes according to the present disclosure will be described in detail.

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[Chemical composition]

**[0019]** First, the chemical composition of the high strength steel plate disclosed herein and the reasons for limitation thereof will be described. Hereinbelow, all units shown by % are mass%.

C: 0.02 % to 0.08 %

**[0020]** C contributes effectively to the improvement of strength. However, when the content is less than 0.02 %, sufficient strength can not be secured. On the other hand, when the content is more than 0.08 %, the hardness of the surface layer is increased during accelerated cooling, and the HIC resistance deteriorates. The toughness also decreases. Therefore, the C content is set in a range of 0.02 % to 0.08 %.

Si: 0.01 % to 0.50 %

**[0021]** Si is added for deoxidation. However, when the content is less than 0.01 %, the deoxidizing effect is insufficient. On the other hand, when the content is more than 0.50 %, the toughness and weldability deteriorate. Therefore, the Si content is set in a range of 0.01 % to 0.50 %.

Mn :0.50 % to 1.80 %

**[0022]** Mn contributes effectively to the improvement of strength and toughness. However, if the content is less than 0.50 %, the addition effect is poor, while if it exceeds 1.80 %, the hardness of the surface layer is increased during accelerated cooling, and the HIC resistance deteriorates. The weldability also decreases. Therefore, the Mn content is set in a range of 0.50 % to 1.80 %.

P: 0.001 % to 0.015 %

**[0023]** P is an inevitable impurity element that degrades the weldability and increases the hardness of the central segregation area, causing deterioration in HIC resistance. This tendency becomes more pronounced when the content exceeds 0.015 %. Therefore, the upper limit is set at 0.015 %. The content is preferably 0.008 % or less. Although a lower P content is preferable, the P content is set to 0.001 % or more from the viewpoint of refining cost.

S: 0.0002 % to 0.0015 %

**[0024]** S is an inevitable impurity element that forms MnS inclusions in the steel and degrades the HIC resistance, and hence a lower S content is preferable. However, up to 0.0015 % is acceptable. Although a lower S content is preferable, the S content is set to 0.0002 % or more from the viewpoint of refining cost.

Al: 0.01 % to 0.08 %

**[0025]** Al is added as a deoxidizing agent. However, an Al content below 0.01 % provides no addition effect, while an Al content beyond 0.08 % lowers the cleanliness of the steel and deteriorates the toughness. Therefore, the Al content is in a range of 0.01 % to 0.08 %.

Ca: 0.0005 % to 0.005 %

**[0026]** Ca is an element effective for improving HIC resistance through morphological control of sulfide inclusions. If the content is less than 0.0005 %, however, the addition effect is not sufficient. On the other hand, if the content exceeds 0.005 %, not only the addition effect saturates, but also the HIC resistance is deteriorated due to the reduction in the cleanliness of the steel. Therefore, the Ca content is in a range of 0.0005 % to 0.005 %.

**[0027]** The basic components according to the present disclosure have been described above. Optionally, however, the chemical composition according to the present disclosure may also contain at least one selected from the group consisting of Cu, Ni, Cr, and Mo in the following ranges to further improve the strength and toughness of the steel plate.

Cu: 0.50 % or less

**[0028]** Cu is an element effective for improving the toughness and increasing the strength. To obtain this effect, the Cu content is preferably 0.05 % or more, yet if the content is too large, the weldability deteriorates. Therefore, when Cu is

added, the Cu content is up to 0.50 %.

Ni: 0.50 % or less

5 **[0029]** Ni is an element effective for improving the toughness and increasing the strength. To obtain this effect, the Ni content is preferably 0.05 % or more, yet excessive addition of Ni is not only economically disadvantageous but also deteriorates the toughness of the heat-affected zone. Therefore, when Ni is added, the Ni content is up to 0.50 %.

Cr: 0.50 % or less

10 **[0030]** Cr, like Mn, is an effective element for obtaining a sufficient strength even at low C content. To obtain this effect, the Cr content is preferably 0.05 % or more, yet if the content is too large, the weldability deteriorates. Therefore, when Cr is added, the Cr content is up to 0.50 %.

15 Mo: 0.50 % or less

**[0031]** Mo is an element effective for improving the toughness and increasing the strength. To obtain this effect, the Mo content is preferably 0.05 % or more, yet if the content is too large, the weldability deteriorates. Therefore, when Mo is added, the Mo content is up to 0.50 %.

20 **[0032]** The chemical composition according to the present disclosure may further optionally contain one or more selected from the group consisting of Nb, V, and Ti in the following ranges.

**[0033]** At least one selected from the group consisting of Nb: 0.005 % to 0.1 %, V: 0.005 % to 0.1 %, Ti: 0.005 % to 0.1 % All of Nb, V, and Ti are elements that can be optionally added to enhance the strength and toughness of the steel plate. If the content of each added element is less than 0.005 %, the addition effect is poor, while if it exceeds 0.1 %, the toughness of the welded portion deteriorates. Therefore, the content of each added element is preferably in a range of 0.005 % to 0.1 %.

25 **[0034]** The balance other than the above-described elements is Fe and inevitable impurities. However, there is no intention in this expression of precluding the inclusion of other trace elements, without impairing the action or effect of the present disclosure.

30 [Mn-concentrated spots]

**[0035]** In the high strength steel plate for sour-resistant line pipes disclosed herein, it is important that in a cross-section perpendicular to a rolling direction (plate length direction) of the steel plate, the number of Mn-concentrated spots that are approximated to an elliptical shape having a major axis length of more than 1.5 mm, in a measuring region located  $\pm 5$  mm from a plate thickness center toward a plate thickness direction, is 3 or less per 100 mm in length in a plate width direction.

35 **[0036]** As used herein, a "Mn-concentrated spot" refers to a site in which the Mn concentration is higher than the addition amount of Mn (the Mn content in the steel plate) due to segregation. This site is specifically identified as a site in which the Mn concentration is 1.60 % or more when the Mn content in the steel plate is 1.50 % or less, and as a site in which the Mn concentration is at least 0.10 % higher than the Mn content in the steel plate when the Mn content in the steel plate is more than 1.50 % and 1.80 % or less.

40 **[0037]** According to the present inventors' studies, it was revealed that HIC cracking is likely to occur from the positions of those Mn-concentrated spots having a major axis length of more than 1.5 mm among the Mn-concentrated spots specified as described above, and that HIC cracking occurs when the number of Mn-concentrated spots having a major axis length of more than 1.5 mm exceeds 3 per 100 mm in length in the plate width direction. Therefore, in the present disclosure, the number of Mn-concentrated spots having a major axis length of more than 1.5 mm is 3 or less per 100 mm in length in the plate width direction.

45 **[0038]** In the present disclosure, "the number of Mn-concentrated spots having a major axis length of more than 1.5 mm per 100 mm in length in the plate width direction" is measured as follows. First, a sample for analysis is cut out from a steel plate and polished for preparation. This setup is carried out such that the surface of the sample becomes a cross-section perpendicular to the plate length direction of the steel plate (a C cross-section). Then, as illustrated in FIG. 1, in the C cross section, Mn concentration mapping is performed using an electron probe microanalyzer (EPMA) for three regions centering on each of the three points at the plate thickness center ( $t/2$  position;  $t$  is the plate thickness) of the steel plate and ranging  $\pm 5$  mm in the plate thickness direction (i.e., 10 mm thick) and  $\pm 200$  mm in the plate width direction (i.e., 400 mm wide), at a  $W/4$  position, a  $W/2$  position, and a  $3W/4$  position from one end in the plate width direction of the steel plate, where  $W$  denotes the plate width (hereinafter, simply referred to as " $W/4$  position", " $W/2$  position", and " $3W/4$  position", respectively). Note that the three regions may be one overlapping area depending on the plate width of the steel plate. The mapping is performed using an electronic probe with an accelerating voltage of 25 kV and a diameter of 0.15 mm. In each EPMA analyzing region (10 mm thick and 400 mm wide), the number of Mn-concentrated spots having a major axis length

of more than 1.5 mm is counted and converted to the number per 100 mm in length in the plate width direction.

**[0039]** It is preferable that the steel microstructure of the high strength steel plate for sour-resistant line pipes disclosed herein is bainite microstructure in order to have a tensile strength as high as 520 MPa or more. In this case, the bainite microstructure includes a microstructure called bainitic ferrite or granular ferrite which contributes to transformation strengthening. These microstructures appear through transformation during or after accelerated cooling. If different microstructures such as ferrite, martensite, pearlite, martensite austenite constituent, retained austenite, and the like are mixed in the bainite microstructure, a decrease in strength, a deterioration in toughness, a rise in surface hardness, and the like occur. Therefore, it is preferable that microstructures other than the bainite phase have smaller proportions. However, when the volume fraction of such microstructures other than the bainitic phase is sufficiently low, their effects are negligible, and up to a certain amount is acceptable. Specifically, in the present disclosure, if the total of the steel microstructures other than bainite (such as ferrite, martensite, pearlite, martensite austenite constituent, and retained austenite) is less than 5 % by volume fraction, there is no adverse effect, and this is acceptable.

[Uniformity of the HIC resistance in the plate width direction]

**[0040]** In the high strength steel plate for sour-resistant line pipes disclosed herein, it is important that the HIC resistance at a W/4 position, a W/2 position, and a 3W/4 position is 10 % or less in terms of CAR, and that the variation in the HIC resistance in the plate width direction in terms of  $3\sigma$  is 5% or less when  $\sigma$  denotes a standard deviation of CARs. This means that the high strength steel plate has excellent HIC resistance in which variation in the HIC resistance in the plate width direction is suppressed. The HIC resistance at a W/4 position, a W/2 position, and a 3W/4 position is preferably 5 % or less in terms of CAR.

**[0041]** In this disclosure, the "HIC resistance at a W/4 position, a W/2 position, and a 3W/4 position" is evaluated as follows. As illustrated in FIG. 2, in a C-section of the steel plate, centering on the plate thickness center at a W/4 position, a W/2 position, and a 3W/4 position (total of three points) in the plate width direction, test pieces of 20 mm thick and 20 mm wide are collected. From each of the three test pieces thus obtained, three samples are collected, and a total of nine samples are subjected to hydrogen-induced cracking (HIC) resistance examination. This examination is conducted in the Method A environment according to NACE TM0284, and the crack area ratio (CAR) is determined as a hydrogen-induced cracking criterion. In the high strength steel plate for sour-resistant line pipes disclosed herein, all nine CARs thus obtained are 10 % or less, and preferably 5 % or less.

**[0042]** Further, in this disclosure, the "variation in the HIC resistance in the plate width direction" is evaluated in terms of  $3\sigma$  when the standard deviation of nine CARs described above is calculated as  $\sigma$ .

[Tensile strength]

**[0043]** The high strength steel plate disclosed herein is a steel plate for steel pipes having a strength of X60 grade or higher in API 5L, and thus has a tensile strength of 520 MPa or more.

[Manufacturing method]

**[0044]** Hereinafter, the method and conditions for manufacturing the above-described high strength steel plate for sour-resistant line pipes will be described concretely. The manufacturing method disclosed herein comprises: subjecting steel having the above chemical composition to continuous casting to prepare a cast steel (slab); heating the slab; then hot rolling the slab to obtain a steel plate; and then subjecting the steel plate to controlled cooling. At this time, by performing secondary cooling in the continuous casting under particular conditions, and by performing the slab heating and controlled cooling under particular conditions, it is possible to manufacture a high strength steel plate for sour-resistant line pipes that has excellent HIC resistance in which variation in the HIC resistance in the plate width direction is suppressed.

[Secondary cooling of a slab during continuous casting]

**[0045]** As illustrated in FIGS. 3A and 3B, the following secondary cooling method is used. Coolant is sprayed on a cast steel 20 in a mist form from a plurality of two-fluid spray nozzles 10A and 10B to cool the cast steel 20 while feeding the cast steel 20 in its longitudinal direction. The plurality of two-fluid spray nozzles 10A and 10B are arranged at predetermined intervals in the width direction of the cast steel 20. Regarding the two-fluid spray nozzles 10 (10A and 10B), the positions on the cast steel at which a water flux density is 50 % of the water flux density immediately below the two-fluid spray nozzles 10 are located away by a distance S (mm) from both ends of each spraying range of the coolant in the width direction of the cast steel 20. The overlapping margin between the spraying ranges of the coolant sprayed from the two-fluid spray nozzles 10A and 10B adjacent to each other is set in a range of 1.6 S to 2.4 S.

**[0046]** FIGS. 3A and 3B schematically illustrate the injection ranges and the water flux density distributions of the coolant

injected from two-fluid spray nozzle(s). FIG. 3A illustrates a distance S from both ends of the injection range at which a ratio of a water flux density at that position to a water flux density immediately below the two-fluid spray nozzle 10 is 50 %, and FIG. 3B illustrates the overlapping margin between the injection ranges of the coolant injected from the two two-fluid spray nozzles 10A and 10B.

**[0047]** The distance S from both ends of the injection range of the coolant injected from the two-fluid spray nozzle 10 can be obtained as follows. First, a water flux density distribution in the width direction of the cast steel of the coolant injected from the two-fluid spray nozzle 10 is measured. The water flux density distribution can be measured by placing the two-fluid spray nozzle 10 above a group of measures finely divided in the width direction of the cast steel 1 and weighing the coolant injected from the two-fluid spray nozzle 10 for each measuring apparatus.

**[0048]** The reason for setting the overlapping margin in a range of 1.6 S to 2.4 S is as follows. That is, in the case of the cast steel being subjected to secondary cooling with a plurality of two-fluid spray nozzles, even if the two-fluid spray nozzles are arranged such that the water flux density of the coolant injected from each two-fluid spray nozzle is uniform in the width direction of the cast steel, the collision pressure is low at both ends of each injection range of the coolant, resulting in low ability of cooling cast steel. Thus, it is impossible to cool the cast steel uniformly in the width direction. However, if the overlapping margin is adjusted in the range of 1.6 S to 2.4 S, it is possible to uniformly cool the cast steel in the width direction, considering the collision pressure distribution in addition to the water flux density distribution in the width direction of the cast steel. That is, according to this method, it is possible to cool the cast steel without lowering the cooling ability in a region over which the injection ranges of the coolant from the adjacent two-fluid spray nozzles 10A and 10B overlap, and to suppress the surface temperature deviation in the width direction of the cast steel, enabling substantially uniform cooling. Accordingly, it is possible to prepare a slab with suppressed variation in the central segregation in the width direction.

**[0049]** Although FIG. 3B illustrates an example using two two-fluid spray nozzles 10A and 10B, in the case of performing secondary cooling of the cast steel with three or more two-fluid spray nozzles, the overlapping margin of the injection ranges of the coolant may be set as described above for those adjacent to each other among three or more two-fluid spray nozzles.

**[0050]** Further, the two-fluid spray nozzle include, but is not limited to, for example, a mist nozzle provided with a feed pipe for coolant and air, a mixing pipe, and a nozzle tip.

[Slab heating temperature]

Slab heating temperature: 1000 °C to 1300 °C

**[0051]** If the slab heating temperature is lower than 1000 °C, carbides do not solute sufficiently and the necessary strength can not be obtained. On the other hand, if the slab heating temperature exceeds 1300 °C, the toughness is deteriorated. Therefore, the slab heating temperature is set to 1000 °C to 1300 °C. This temperature is the temperature in the heating furnace, and the slab is heated to this temperature to the center.

[Rolling finish temperature]

**[0052]** In a hot rolling step, in order to obtain high toughness for base metal, a lower rolling finish temperature is preferable, yet on the other hand, the rolling efficiency is lowered. Thus, the rolling finish temperature in terms of a temperature of the surface of the steel plate needs to be set in consideration of the required toughness for base metal and rolling efficiency. From the viewpoint of improving the strength and the HIC resistance, it is preferable to set the rolling finish temperature at or above the Ar<sub>3</sub> transformation temperature in terms of a temperature of the surface of the steel plate. As used herein, the Ar<sub>3</sub> transformation temperature means the ferrite transformation start temperature during cooling, and can be determined, for example, from the components of steel according to the following equation. Further, in order to obtain high toughness for base metal, it is desirable to set the rolling reduction ratio in a temperature range of 950 °C or lower, which corresponds to the austenite non-recrystallization temperature range, to 60 % or more. The temperature of the surface of the steel plate can be measured by a radiation thermometer or the like.

$$Ar_3(^{\circ}C) = 910 - 310[\%C] - 80[\%Mn] - 20[\%Cu] - 15[\%Cr] - 55[\%Ni] - 80[\%Mo],$$

where [%X] indicates the content by mass% of the element X in steel.

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[Cooling start temperature in the controlled cooling]

Cooling start temperature: ( $A_{r3} - 10^{\circ}\text{C}$ ) or higher in terms of a temperature of the surface of the steel plate

5 **[0053]** If the temperature of the surface of the steel plate is low at the start of cooling, ferrite forms in a large amount before controlled cooling, in particular, when the temperature drop from the  $A_{r3}$  transformation temperature exceeds  $10^{\circ}\text{C}$ , ferrite forms in a volume fraction of more than 5 %, causing a significant reduction in the strength and a deterioration in the HIC resistance. Therefore, the temperature of the surface of the steel plate at the start of cooling is set to ( $A_{r3} - 10^{\circ}\text{C}$ ) or higher.

10

[Cooling rate of the controlled cooling]

Average cooling rate in a temperature range from  $750^{\circ}\text{C}$  to  $550^{\circ}\text{C}$  in terms of an average temperature of the steel plate:  $15^{\circ}\text{C/s}$  or higher

15

**[0054]** If the average cooling rate in a temperature range from  $750^{\circ}\text{C}$  to  $550^{\circ}\text{C}$  in terms of an average temperature of the steel plate is lower than  $15^{\circ}\text{C/s}$ , a bainite microstructure can not be obtained, causing deterioration in the strength and HIC resistance. Therefore, the cooling rate in terms of an average temperature of the steel plate is set to  $15^{\circ}\text{C/s}$  or higher. From the viewpoint of variation in the strength and hardness of the steel plate, the steel plate average cooling rate is preferably  $20^{\circ}\text{C/s}$  or higher. The upper limit of the average cooling rate is not particularly limited, yet is preferably  $80^{\circ}\text{C/s}$  or lower such that excessive low-temperature transformation products will not be generated.

20

[Cooling stop temperature]

25 Cooling stop temperature:  $250^{\circ}\text{C}$  to  $550^{\circ}\text{C}$  in terms of an average temperature of the steel plate

**[0055]** After the completion of rolling, a bainite phase is generated by performing controlled cooling to quench the steel plate to a temperature range of  $250^{\circ}\text{C}$  to  $550^{\circ}\text{C}$  which is the temperature range of bainite transformation. When the cooling stop temperature exceeds  $550^{\circ}\text{C}$ , bainite transformation is incomplete and sufficient strength can not be obtained. In addition, if the cooling stop temperature is lower than  $250^{\circ}\text{C}$ , the hardness markedly increases in the surface layer. The cooling stop temperature is preferably  $350^{\circ}\text{C}$  to  $500^{\circ}\text{C}$ .

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**[0056]** Although an average temperature of the steel plate can not be directly measured physically, a temperature distribution in a cross section in the plate thickness direction can be determined in real time, for example, by difference calculation using a process computer on the basis of the surface temperature at the start of cooling measured with a radiation thermometer and the target surface temperature at the end of cooling. The average value of temperatures in the plate thickness direction in the temperature distribution is referred to as the "average temperature of the steel plate" in this description.

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[High strength steel pipe]

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**[0057]** By forming the high strength steel plate disclosed herein into a tubular shape by press bend forming, roll forming, UOE forming, or the like, and then welding the butting portions, a high strength steel pipe for sour-resistant line pipes (such as a UOE steel pipe, an electric-resistance welded steel pipe, and a spiral steel pipe) that has excellent material homogeneity in the steel plate and that is suitable for transporting crude oil and natural gas can be manufactured.

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**[0058]** For example, an UOE steel pipe is manufactured by milling and beveling the edges of a steel plate, forming the steel plate into a steel pipe shape by C press, U-ing press, and O-ing press, then seam welding the butting portions by inner surface welding and outer surface welding, and optionally subjecting it to an expansion process. Any welding method may be applied as long as sufficient joint strength and joint toughness are guaranteed, yet it is preferable to use submerged arc welding from the viewpoint of excellent weld quality and manufacturing efficiency.

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EXAMPLES

**[0059]** Steels having the chemical compositions listed in Table 1 (Steel Sample IDs A to M) were prepared and subjected to continuous casting to obtain slabs with a slab width of 1600 mm. Secondary cooling was performed with the overlapping margin of the injection ranges of the coolant injected in a mist form from the three two-fluid spray nozzles arranged at predetermined intervals in the width direction being set as listed in Table 2. Note that a distance S from both ends of the injection range of the coolant in the width direction of the cast steel 20 to the position where the ratio of a water flux density at that position to a water flux density immediately below the two-fluid spray nozzles is 50 % was fixed to 70 mm.

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**[0060]** Each slab thus obtained was heated to the temperature as listed in Table 2, and then hot rolled with the rolling finish temperature and the rolling reduction ratio as listed in the table, to thereby obtain a steel plate with the plate thickness as listed in the table. Then, each steel plate was subjected to controlled cooling using a water-cooling type controlled-cooling device under the conditions listed in Table 2.

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Table 1

Steel sample ID	Chemical composition (mass%)											Ar <sub>3</sub> Temperature (°C)			
	C	Si	Mn	P	S	Al	Ca	Cu	Ni	Cr	Mo		Nb	V	Ti
A	0.064	0.32	1.41	0.004	0.0004	0.024	0.0022					0.010			777
B	0.075	0.20	1.52	0.003	0.0005	0.032	0.0030								765
C	0.044	0.21	1.28	0.005	0.0005	0.020	0.0017	0.23	0.11	0.19	0.025				768
D	0.049	0.14	1.33	0.004	0.0004	0.019	0.0021			0.22	0.10	0.035			777
E	0.046	0.24	1.23	0.006	0.0006	0.022	0.0015			0.20	0.14	0.021		0.015	783
F	0.053	0.25	1.27	0.004	0.0008	0.025	0.0023			0.35		0.035	0.035		787
G	0.041	0.28	1.32	0.003	0.0005	0.021	0.0011			0.25	0.11	0.029		0.011	779
H	0.048	0.30	1.25	0.003	0.0004	0.020	0.0014	0.15	0.16	0.30	0.18	0.031		0.008	764
I	<u>0.086</u>	0.17	1.33	0.005	0.0006	0.021	0.0021		0.26		0.15	0.022		0.010	751
J	0.034	0.22	1.88	0.006	0.0008	0.025	0.0014	0.12	0.25						733
K	0.046	0.20	1.28	<u>0.018</u>	0.0004	0.023	0.0020			0.23	0.12	0.015			780
L	0.054	0.15	1.31	0.006	<u>0.0026</u>	0.022	0.0016		0.22	0.11	0.10	0.027	0.044	0.010	767
M	0.055	0.19	1.44	<u>0.017</u>	0.0005	0.022	0.0026								778

Note 1: Underlined if outside the scope of the disclosure.

Table 2

No.	Steel sample ID	Plate thickness (mm)	Overlapping margin for secondary cooling of slab	Heating temp. (°C)	Rolling finish temperature (°C)	Rolling reduction ratio (%)	Cooling start temp. (°C)	Cooling start temp. - A <sub>r3</sub> (°C)	Cooling rate (steel plate average) (°C/s)	Cooling stop temp. (°C)	Category
1	A	34	2.0S	1150	880	70	820	43	37	480	Exampe
2	B	16	2.0S	1130	910	75	830	65	54	520	
3	C	25	2.1S	1080	880	75	810	42	33	510	
4	D	15	1.9S	1080	870	75	810	33	45	440	
5	E	20	2.2S	1080	850	80	790	7	31	450	
6	F	34	2.4S	1080	840	75	810	23	29	500	
7	G	34	2.0S	1080	870	70	830	51	43	390	
8	G	34	2.0S	1110	830	75	770	-9	23	460	
9	G	20	2.0S	1110	800	75	780	1	34	470	
10	G	20	2.0S	1100	850	75	810	31	31	440	
11	H	34	1.8S	1080	860	75	810	46	37	350	
12	H	25	1.6S	1150	850	75	820	56	47	500	
13	H	38	2.3S	1080	850	75	800	36	32	480	

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No.	Steel sample ID	Plate thickness (mm)	Overlapping margin for secondary cooling of slab	Heating temp. (°C)	Rolling finish temperature (°C)	Rolling reduction ratio (%)	Cooling start temp. (°C)	Cooling start temp. - Ar <sub>3</sub> (°C)	Cooling rate (steel plate average) (°C/s)	Cooling stop temp. (°C)	Category
14	G	34	2.2S	<u>970</u>	850	75	810	31	31	480	Comparative example
15	G	34	2.0S	1080	780	75	<u>740</u>	<u>-39</u>	25	420	
16	G	34	1.9S	1080	830	75	810	31	5	500	
17	G	34	2.3S	1080	850	75	800	21	37	<u>180</u>	
18	G	20	2.0S	1080	850	75	830	51	<u>13</u>	450	
19	G	20	<u>1.5S</u>	1100	850	75	820	41	35	430	
20	G	34	<u>2.5S</u>	1130	850	75	810	31	32	450	
21	H	34	<u>2.6S</u>	1080	850	75	810	46	38	460	
22	B	16	<u>1.4S</u>	1140	910	75	830	65	52	500	
23	<u>I</u>	34	2.0S	1080	840	75	800	49	37	430	
24	<u>J</u>	34	2.0S	1080	820	75	780	47	27	520	
25	<u>K</u>	34	2.0S	1080	860	75	820	40	33	470	
26	<u>L</u>	34	2.0S	1080	850	75	810	43	32	450	
27	<u>M</u>	16	2.0S	1130	890	75	840	62	42	520	

Note 1: Underlined if outside the scope of the disclosure.

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[Identification of microstructure]

5 **[0061]** The microstructure of each obtained steel plate was observed with an optical microscope and a scanning electron microscope. The microstructures at the plate thickness center (i.e.,  $t/2$  position) of the steel plate are listed in Table 3.

[Evaluation of tensile property]

10 **[0062]** From each obtained steel plate, a full-thickness test piece (as prescribed in API-5L specification) in the transverse direction (direction orthogonal to the rolling direction) was taken and subjected to tensile test as a tensile test piece to measure the yield stress (0.5% proof stress) and the tensile strength. The target ranges were a yield stress of 450 MPa or more and a tensile strength of 520 MPa or more. The results are listed in Table 3.

[Evaluation of variation in the HIC resistance in the plate width direction]

15 **[0063]** Three samples were respectively collected from a  $W/4$  position, a  $W/2$  position, and a  $3W/4$  position in the manner described above, and CARs were measured. The maximum value of the nine measured values thus obtained is presented in the column of "HIC resistance" in Table 3. Table 3 also lists  $3\sigma$  when the standard deviation of nine CARs is calculated as  $\sigma$ . The target range was 10 % or less for the maximum value and 5 % or less for  $3\sigma$ .

20 [Measurement of Mn-concentrated spots]

25 **[0064]** The number of Mn-concentrated spots having a major axis length of more than 1.5 mm was counted per 100 mm in length in the plate width direction in the manner described above. The target range was 3 or less. The results are listed in Table 3.

[DWTT test]

30 **[0065]** From each obtained steel plate, a DWTT test piece conforming to the API-5L was taken and tested at test temperatures of 0 °C to -80 °C to determine a transition temperature at which the SA value (Shear Area: percent ductile fracture) was 85 %. The target range for transition temperature was -50 °C or lower. The results are listed in Table 3.

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Table 3

No.	Steel sample ID	Plate thickness (mm)	Microstructure (t/2 position)	Yield strength (MPa)	Tensile strength (MPa)	HIC resistance (%)	Variation in HIC resistance, $3\sigma$ (%)	Mn-concentrated spots (pcs.)	DWTT 85% SATT ( $^{\circ}$ C)	Category
1	A	34	B	460	575	1	0.3	1	-50	Example
2	B	16	B	477	589	2	1.2	2	-50	
3	C	25	B	480	624	0	0	1	-60	
4	D	15	B	485	622	0	0	0	-65	
5	E	20	B	462	581	0	0	0	-55	
6	F	34	B	458	578	3	0.7	2	-50	
7	G	34	B	504	637	2	0.4	1	-50	
8	G	34	B	466	574	0	0	2	-55	
9	G	20	B	506	621	0	0	0	-60	
10	G	20	B	511	628	0	0	0	-65	
11	H	34	B	498	629	1	0.2	2	-65	
12	H	25	B	522	639	2	0.5	2	-60	
13	H	38	B	531	634	4	1	3	-60	

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No.	Steel sample ID	Plate thickness (mm)	Microstructure (t/2 position)	Yield strength (MPa)	Tensile strength (MPa)	HIC resistance (%)	Variation in HIC resistance, 3σ (%)	Mn-concentrated spots (pcs.)	DWTT 85% SATT (°C)	Category
14	G	34	B	415	<u>505</u>	4	2	2	-60	Comparative example
15	G	34	F+B	420	510	12	8	5	-65	
16	G	34	F+P	410	<u>513</u>	<u>15</u>	<u>7</u>	<u>6</u>	-70	
17	G	34	B+M	541	654	<u>14</u>	<u>6</u>	<u>5</u>	-30	
18	G	20	F+P	430	<u>511</u>	12	6	4	-50	
19	G	20	B	482	632	<u>22</u>	<u>7</u>	<u>8</u>	-60	
20	G	34	B	476	639	<u>18</u>	<u>8</u>	<u>8</u>	-60	
21	H	34	B	489	644	<u>22</u>	<u>8</u>	<u>12</u>	-35	
22	B	16	B	468	597	8	6	4	-55	
23	<u>I</u>	34	B	510	647	<u>22</u>	<u>8</u>	<u>6</u>	-30	
24	<u>J</u>	34	B	516	650	<u>16</u>	<u>6</u>	<u>6</u>	-65	
25	<u>K</u>	34	B	501	614	<u>14</u>	<u>7</u>	<u>5</u>	-60	
26	<u>L</u>	34	B	483	620	<u>12</u>	<u>8</u>	<u>6</u>	-60	
27	<u>M</u>	16	B	503	616	<u>13</u>	<u>7</u>	<u>5</u>	-60	

Note 1: Underlined if outside the scope of the disclosure.

Note 2: For microstructure, B denotes bainite, F denotes ferrite, M denotes martensite, and P denotes pearlite.

**[0066]** For Nos. 1 to 13, which are our examples, the chemical compositions were within the scope of the present disclosure and the manufacturing conditions were within the range suitable for obtaining steel plates according to the present disclosure. All of our samples had a yield stress of 450 MPa or more, a tensile strength of 520 MPa or more, a 85 % SATT of -50 °C or lower in the DWTT test, and small variation in the HIC resistance in the plate width direction, any of which properties were considered good.

**[0067]** In contrast, for Nos. 14 to 22, which are comparative examples, although the chemical compositions were within the scope of the present disclosure, the manufacturing conditions were outside the scope of the preferred conditions for obtaining steel plates according to the present disclosure. For No. 14, the slab heating temperature was low, the homogenization of the microstructure and the solid solution state of carbides were insufficient, and the strength was low. For No. 15, since ferrite generated excessively due to the low cooling start temperature, the strength was low and the HIC resistance was inferior.

For Nos. 16 and 18, since pearlite excessively generated as a microstructure in the mid-thickness part due to the controlled cooling condition outside the suitable range, the strength was low and the HIC resistance was inferior.

For No. 17, since hard phases such as martensite and martensite austenite constituent (MA) were formed due to the low cooling stop temperature, the DWTT property and the HIC resistance were inferior.

For Nos. 19 to 22, since the secondary cooling conditions of the slabs were outside the suitable range, Mn concentration in the central segregation area was high, variation in the HIC resistance in the plate width direction was large, and the HIC resistance was inferior.

For Nos. 23 to 27, since the chemical compositions were outside the scope of the present disclosure, Mn concentration in the central segregation area was high, variation in the HIC resistance in the plate width direction was large, and the HIC resistance was inferior.

#### INDUSTRIAL APPLICABILITY

**[0068]** The high strength steel plates for sour-resistant line pipes according to the present disclosure have excellent HIC resistance in which variation in the HIC resistance in the plate width direction is suppressed. Therefore, steel pipes (such as electric-resistance welded steel pipes, spiral steel pipes, and UOE steel pipes) manufactured by cold-forming the disclosed steel plate can be suitably used for transportation of crude oil and natural gas that contain hydrogen sulfides where sour resistance is required.

#### REFERENCE SIGNS LIST

#### **[0069]**

10, 10A, 10B two-fluid spray nozzle  
20 cast steel

#### Claims

**1.** A high strength steel plate for sour-resistant line pipes comprising: a chemical composition containing, by mass%, C: 0.02 % to 0.08 %, Si: 0.01 % to 0.50 %, Mn: 0.50 % to 1.80 %, P: 0.001 % to 0.015 %, S: 0.0002 % to 0.0015 %, Al: 0.01 % to 0.08 %, and Ca: 0.0005 % to 0.005 %, optionally at least one selected from the group consisting of Cu: 0.50 % or less, Ni: 0.50 % or less, Cr: 0.50 % or less, and Mo: 0.50 % or less, Nb: 0.005 % to 0.1 %, V: 0.005 % to 0.1 %, and Ti: 0.005 % to 0.1 %, with the balance being Fe and inevitable impurities, wherein

in a cross-section perpendicular to a rolling direction of the steel plate, the number of Mn-concentrated spots that are approximated to an elliptical shape having a major axis length of more than 1.5 mm is 3 or less per 100 mm in length in a plate width direction, in three regions centering on each of the three points at the plate thickness center of the steel plate and ranging  $\pm 5$  mm in the plate thickness direction and  $\pm 200$  mm in the plate width direction at a W/4 position, a W/2 position and a 3W/4 position from one end in the plate width direction of the steel plate, where W denotes the plate width, and where the Mn-concentrated spot is defined as a site in which the Mn concentration is 1.60% or more when the Mn content in the steel plate is 1.50% or less, and as a site in which the Mn concentration is at least 0.10 % higher than the Mn content in the steel plate when the Mn content in the steel plate is more than 1.50 % and 1.80% or less,

where, in the cross-section perpendicular to the rolling direction of the steel plate, centering on the plate thickness center at the W/4 position, the W/2 position, and the 3W/4 position (total of three points) in the plate width direction, test pieces of 20 mm thick and 20 mm wide are collected, and from each of the three test pieces thus obtained, three samples are collected, and a total of nine samples are subjected to hydrogen-induced cracking (HIC)

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resistance examination, HIC resistance of all the nine samples are 10 % or less in terms of CAR and where the CAR is measured according to NACE TM0284, variation in the HIC resistance in the plate width direction in terms of  $3\sigma$  is 5 % or less when  $\sigma$  denotes a standard deviation of CARs of the nine samples, and a tensile strength is 520 MPa or more, where the tensile strength is measured according to API-5L.

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2. A high strength steel pipe using the high strength steel plate for sour-resistant line pipes as recited in claim 1.

### Patentansprüche

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1. Hochfeste Stahlplatte für sauergasresistente Leitungsrohre, umfassend: eine chemische Zusammensetzung, enthaltend, in Masse-%, C: 0,02 % bis 0,08 %, Si: 0,01 % bis 0,50 %, Mn: 0,50 % bis 1,80 %, P: 0,001 % bis 0,015 %, S: 0,0002 % bis 0,0015 %, Al: 0,01 % bis 0,08 % und Ca: 0,0005 % bis 0,005 %, optional mindestens eine ausgewählt aus der Gruppe, bestehend aus Cu: 0,50 % oder weniger, Ni: 0,50 % oder weniger, Cr: 0,50 % oder weniger und Mo: 0,50 % oder weniger, Nb: 0,005 % bis 0,1 %, V: 0,005 % bis 0,1 % und Ti: 0,005 % bis 0,1 %, wobei der Rest Fe und unvermeidliche Verunreinigungen sind, wobei

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in einem Querschnitt senkrecht zu einer Walzrichtung der Stahlplatte die Anzahl der Mn-konzentrierten Punkte, die sich einer elliptischen Form annähern, die eine Hauptachsenlänge von mehr als 1,5 mm aufweist, 3 oder weniger pro 100 mm Länge in Plattenbreitenrichtung beträgt, in drei Regionen, die sich in jedem der drei Punkte an dem Plattendickenmittelpunkt der Stahlplatte zentrieren und in einem Bereich von  $\pm 5$  mm in der Plattendickenrichtung und  $\pm 200$  mm in der Plattenbreitenrichtung an einer W/4-Position, einer W/2-Position und einer 3W/4-Position von einem Ende in der Plattenbreitenrichtung der Stahlplatte liegen, wo W die Plattenbreite bezeichnet und wo der Mn-konzentrierte Punkt definiert als eine Stelle ist, in der die Mn-Konzentration **1,60** % oder mehr beträgt, wenn der Mn-Gehalt in der Stahlplatte 1,50 % oder weniger beträgt, und als eine Stelle, in der die Mn-Konzentration mindestens 0,10 % höher als der Mn-Gehalt in der Stahlplatte ist, wenn der Mn-Gehalt in der Stahlplatte mehr als 1,50 % und 1,80 % oder weniger beträgt, wo in dem Querschnitt senkrecht zu der Walzrichtung der Stahlplatte, zentriert auf dem Plattendickenmittelpunkt an der W/4-Position, der W/2-Position und der 3W/4-Position (insgesamt drei Punkte) in Plattenbreitenrichtung, 20 mm dicke und 20 mm breite Probestücke gesammelt werden und von jedem der drei so erlangten Probestücke drei Proben genommen werden, und insgesamt neun Proben einer Prüfung von wasserstoffinduzierter Rissbildungs-Widerstand (hydrogen-induced cracking, HIC) unterzogen werden, der HIC-Widerstand aller neun Proben 10 % oder weniger in Bezug auf CAR beträgt und wo der CAR gemäß NACE TM0284 gemessen wird, die Variation des HIC-Widerstands in Plattenbreitenrichtung in Bezug auf  $3\sigma$  5 % oder weniger beträgt, wenn  $\sigma$  eine Standardabweichung der CAR der neun Proben bezeichnet, und eine Zugfestigkeit 520 MPa oder mehr beträgt, wobei die Zugfestigkeit gemäß API-5L gemessen wird.

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2. Hochfestes Stahlrohr unter Verwendung der hochfesten Stahlplatte für sauergasresistente Leitungsrohre nach Anspruch 1.

### Revendications

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1. Plaque d'acier à haute résistance pour tuyaux de canalisation résistantes aux acides, comprenant : une composition chimique contenant, en % en masse, C : 0,02 % à 0,08 %, Si : 0,01 % à 0,50 %, Mn : 0,50 % à 1,80 %, P : 0,001 % à 0,015 %, S : 0,0002 % à 0,0015 %, Al : 0,01 % à 0,08 %, et Ca : 0,0005 % à 0,005 %, éventuellement au moins un élément choisi dans le groupe constitué de Cu : 0,50 % ou moins, Ni : 0,50 % ou moins, Cr : 0,50 % ou moins, et Mo : 0,50 % ou moins, Nb : 0,005 % à 0,1 %, V : 0,005 % à 0,1 %, et Ti : 0,005 % à 0,1 %, le reste étant du Fe et des impuretés inévitables, dans lequel

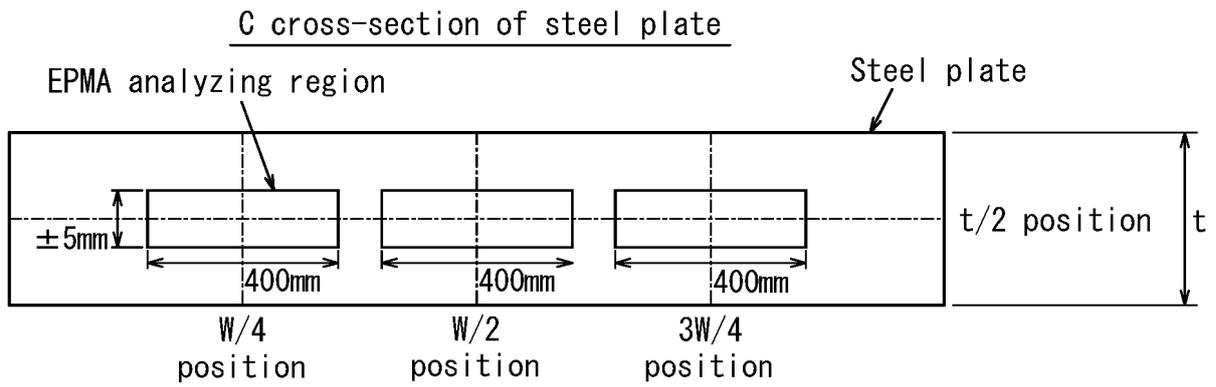
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dans une section transversale perpendiculaire à une direction de laminage de la plaque d'acier, le nombre de taches concentrées en Mn qui sont approximées par une forme elliptique ayant une longueur d'axe majeur de plus de 1,5 mm est de 3 ou moins par 100 mm de longueur dans une direction de la largeur de la plaque, dans trois régions centrées sur chacun des trois points au centre de l'épaisseur de la plaque d'acier et allant de  $\pm 5$  mm dans la direction de l'épaisseur de la plaque et de  $\pm 200$  mm dans la direction de la largeur de la plaque à une position W/4, une position W/2 et une position 3W/4 à partir d'une extrémité dans la direction de la largeur de la plaque d'acier, où W désigne la largeur de la plaque, et où la tache concentrée en Mn est définie comme un site dans lequel la concentration en Mn est de 1,60 % ou plus lorsque la teneur en Mn dans la plaque d'acier est de 1,50 %

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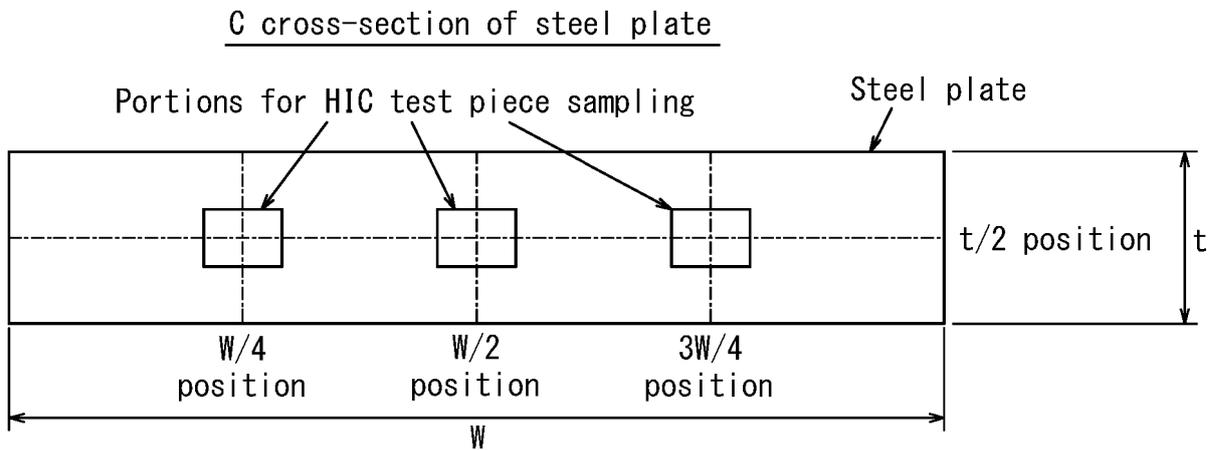
ou moins, et comme un site dans lequel la concentration en Mn est au moins 0,10 % supérieure à la teneur en Mn dans la plaque d'acier lorsque la teneur en Mn dans la plaque d'acier contient plus de 1,50 % et 1,80 % ou moins, où, dans la section transversale perpendiculaire à la direction de laminage de la plaque d'acier, centrée sur le centre d'épaisseur de la plaque à la position W/4, la position W/2 et la position 3W/4 (total de trois points) dans la direction de la largeur de la plaque, des éprouvettes de 20 mm d'épaisseur et de 20 mm de largeur sont prélevées, et à partir de chacune des trois éprouvettes ainsi obtenues, trois échantillons sont prélevés, et un total de neuf échantillons sont soumis à un examen de résistance à la fissuration induite par l'hydrogène (HIC), la résistance HIC de tous les neuf échantillons est de 10 % ou moins en termes de CAR et lorsque le CAR est mesuré selon la norme NACE TM0284, la variation de la résistance HIC dans la direction de la largeur de la plaque en termes de  $3\sigma$  est de 5 % ou moins lorsque  $\sigma$  désigne un écart type des CAR des neuf échantillons, et une résistance à la traction est de 520 MPa ou plus, où la résistance à la traction est mesurée selon la norme API-5L.

2. Un tuyau en acier à haute résistance utilisant la plaque d'acier à haute résistance pour tuyaux de canalisation résistants aux acides telle que mentionnée dans la revendication 1.

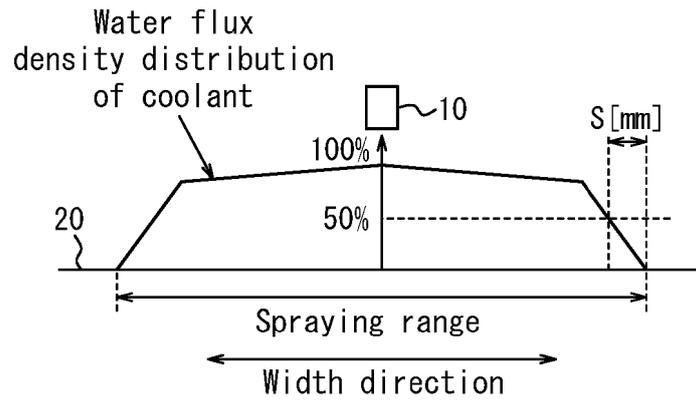
**FIG. 1**



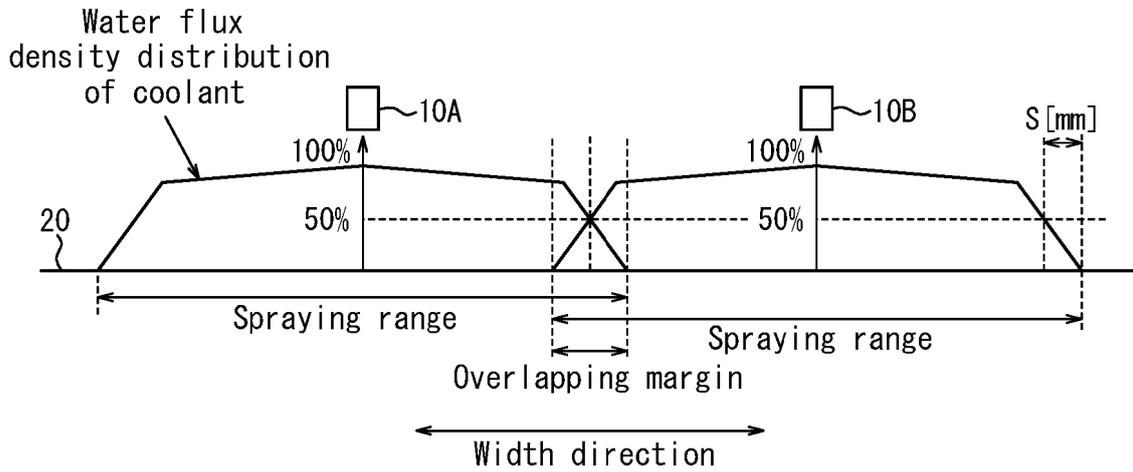
**FIG. 2**



**FIG. 3A**



**FIG. 3B**



**REFERENCES CITED IN THE DESCRIPTION**

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- JP 3796133 B [0008] [0009]
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