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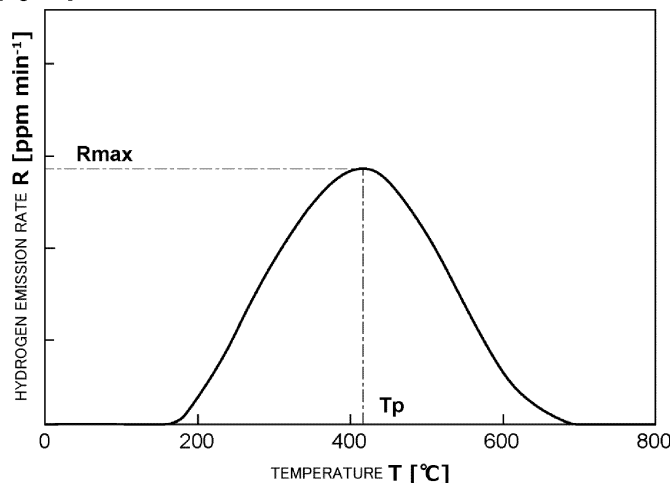
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(54) **AUSTENITIC STAINLESS STEEL**

(57) An austenitic stainless steel, wherein when an amount of hydrogen emitted at a heating rate of 100°C/h and in a temperature range from 25 to 800°C is measured by using thermal desorption analysis, an amount of emitted hydrogen [H] is less than 10.0 ppm, a peak temperature [Tp] of hydrogen emission is 350°C

or more,
a peak rate [Rmax] of hydrogen emission is 0.050 ppm/min or less, and
a number density of compounds having a diameter of 0.1 to 2 µm is 1.0 to 10.0/mm².

[Figure 1]



Description

TECHNICAL FIELD

5 **[0001]** The present disclosure relates to an austenitic stainless steel.

BACKGROUND ART

10 **[0002]** In recent years, hydrogen is drawing attention as a new energy source that replaces fossil fuels. Hydrogen is a clean energy source that does not emit CO₂. On the other hand, for example, hydrogen may cause hydrogen embrittlement that embrittles a material. Patent Document 1 then discloses an austenitic stainless steel with improved hydrogen gas embrittlement resistance.

LIST OF PRIOR ART DOCUMENTS

15

PATENT DOCUMENT

[0003] Patent Document 1: JP2015-196842A

20 SUMMARY OF INVENTION

TECHNICAL PROBLEM

25 **[0004]** However, the austenitic stainless steel disclosed in Patent Document 1 contains a large number of expensive alloying elements to improve hydrogen gas embrittlement resistance, which increases alloy costs. Accordingly, a problem with the austenitic stainless steel is that it is difficult to improve hydrogen embrittlement resistance without expensive alloying elements as essential elements.

30 **[0005]** Given the above-described situation, an objective of the present disclosure is to solve the above-described problem and to provide an austenitic stainless steel excellent in hydrogen embrittlement resistance without expensive alloying elements as essential elements.

SOLUTION TO PROBLEM

35 **[0006]** The gist of the present disclosure, which has been made to solve the above-described problem, is an austenitic stainless steel as described below.

(1) An austenitic stainless steel, wherein when an amount of hydrogen emitted at a heating rate of 100°C/h and in a temperature range from 25 to 800°C is measured by using thermal desorption analysis,

40 an amount of emitted hydrogen [H] is less than 10.0 ppm,
 a peak temperature [Tp] of hydrogen emission is 350°C or more,
 a peak rate [Rmax] of hydrogen emission is 0.050 ppm/min or less, and
 a number density of compounds having a diameter of 0.1 to 2 μm is 1.0 to 10.0/mm².

45 (2) The austenitic stainless steel according to the above (1), wherein a chemical composition of the austenitic stainless steel consists of, by mass%,

50 C: 0.080% or less,
 Si: 1.00% or less,
 Mn: 2.00% or less,
 P: 0.050% or less,
 S: 0.020% or less,
 Cr: 17.0 to 22.0%,
 Ni: 8.0 to 13.0%,
55 N: 0.250% or less,
 Nb: 0 to 0.20%,
 Ti: 0 to 0.20%,
 Mo: 0 to 1.0%,

Cu: 0 to 1.0%,
 Al: 0 to 0.30%,
 Co: 0 to 0.50%,
 V: 0 to 0.50%,
 W: 0 to 0.50%,
 B: 0 to 0.0050%,
 Ca: 0 to 0.010%,
 Mg: 0 to 0.010%,
 Zr: 0 to 0.50%,
 Ga: 0 to 0.05%,
 Hf: 0 to 0.10%,
 REM: 0 to 0.10%, and
 the balance: Fe and impurities.

(3) The austenitic stainless steel according to the above (1) or (2), wherein the austenitic stainless steel is used in a high-pressure hydrogen gas environment or a liquid hydrogen environment.

(4) The austenitic stainless steel according to the above (1) or (2), wherein the austenitic stainless steel is used for a component of a hydrogen gas production apparatus or a hydrogen gas supply apparatus.

ADVANTAGEOUS EFFECTS OF INVENTION

[0007] According to the present disclosure, it is possible to obtain an austenitic stainless steel excellent in hydrogen embrittlement resistance without expensive alloying elements as essential elements.

BRIEF DESCRIPTION OF DRAWINGS

[0008] [Figure 1] Figure 1 schematically shows a hydrogen emission curve (TDA curve).

DESCRIPTION OF EMBODIMENTS

[0009] The inventors have conducted various studies for increasing hydrogen embrittlement resistance of the austenitic stainless steel and for alloy saving and obtained the following findings (a) to (c).

(a) The hydrogen embrittlement of an austenitic stainless steel is likely to occur under a hydrogen environment at -100 to -40°C. The reason for this is considered that an austenite phase (hereinafter, also referred to as "γ phase") becomes unstable in this temperature region and a phase transformation into a brittle and weak α' phase occurs due to accumulation of strain or the like. Accordingly, it is conceivable to increase the contents of added elements such as Ni, Cu, and Mn to increase the stability of a γ phase. In the meantime, high alloying resulting from increased contents of such elements described above increases alloy costs.

(b) The inventors then paid attention to hydrogen contained in the steel to improve hydrogen embrittlement resistance without high alloying. In typical production processes of steel and under a hydrogen environment, minute hydrogen remains captured inside the steel due to penetration of hydrogen into the steel. When such hydrogen in the steel is built up at, for example, a site where strain accumulates, hydrogen embrittlement may be promoted. Accordingly, it is desirable that hydrogen in the steel is to be emitted. However, when hydrogen is present at a low temperature, which is typical for the use environment, it is difficult to emit the hydrogen to the outside of the steel.

(c) Accordingly, it is desirable to suppress aggregation of hydrogen present inside the steel to make the hydrogen less mobile. Specifically, it is effective to control the presence of hydrogen such that the hydrogen remains trapped inside the steel. When controlling the presence of hydrogen, it is preferable to perform warming processing in which heat treatment is performed for a long time in a temperature region from 50 to 400°C during production. It is also effective to provide sufficiently formed fine compounds including Ti and Nb, which contribute to trapping hydrogen.

[0010] An embodiment of the present disclosure has been made based on the above-described findings. The requirements of an austenitic stainless steel of the embodiment will now be described in detail.

1. Presence of hydrogen

[0011] In the austenitic stainless steel of the embodiment, the presence of hydrogen is controlled such that hydrogen remains less mobile, that is, remains trapped inside the steel. To grasp the presence of hydrogen quantitatively, the thermal

desorption analysis is used in the austenitic stainless steel of the embodiment.

[0012] The thermal desorption analysis is a method for detecting, by a gas chromatograph or a quadrupole mass spectrometer, hydrogen emitted when a stainless steel material is heated at a specific heating rate. Hydrogen is trapped in many defects including lattice defects such as vacancies, dislocations, and crystal grain boundaries, and interfaces of precipitates and inclusions. Then, the thermal desorption analysis can be used to quantitatively evaluate the amount of hydrogen trapped in these defects.

[0013] Here, a hydrogen emission curve (hereinafter, also referred to as "TDA curve") shown in Figure 1 obtained by the thermal desorption analysis will be described. In the TDA curve, the horizontal indicates a temperature, and the vertical indicates a hydrogen emission rate. The TDA curve of steel material is typically a curve that hits its peak in the hydrogen emission rate at a certain temperature. Then, a temperature at which the hydrogen emission rate reaches its peak in the TDA curve is referred to as a peak temperature [Tp] of hydrogen emission. In addition, the hydrogen emission rate at that time is referred to as a peak rate [Rmax] of hydrogen emission. Furthermore, it is also possible to calculate the amount of emitted hydrogen [H] by subjecting the curve to integration.

[0014] Each of the amount of emitted hydrogen [H], the peak temperature [Tp] of hydrogen emission, and the peak rate [Rmax] of hydrogen emission described above acts as an indicator for the presence of hydrogen in the steel.

[0015] Then, when the thermal desorption analysis is used to measure the amount of hydrogen emitted at the heating rate of 100°C/h within the temperature range from 25 to 800°C, the amount of emitted hydrogen [H] is less than 10.0 ppm, the peak temperature [Tp] of hydrogen emission is 350°C or more, and the peak rate [Rmax] of hydrogen emission is 0.050 ppm/min or less.

[0016] When the amount of emitted hydrogen [H] measured by the above-described method is 10.0 ppm or more, hydrogen is excessively captured in the steel, and it is difficult to improve hydrogen embrittlement resistance. Accordingly, the amount of emitted hydrogen [H] is less than 10.0 ppm. The amount of emitted hydrogen [H] is preferably 8.5 ppm or less and more preferably 6.5 ppm or less. Note that since hydrogen that cannot be emitted is trapped in the austenitic stainless steel of the embodiment, the amount of emitted hydrogen [H] is preferably more than 1.0 ppm and more preferably 1.5 ppm or more.

[0017] In addition, when the peak temperature [Tp] of hydrogen emission is less than 350°C, the level of trapping hydrogen decreases, so that hydrogen becomes easier to move in the steel. Accordingly, the peak temperature [Tp] of hydrogen emission is 350°C or more. The peak temperature [Tp] of hydrogen emission is preferably 370°C or more and more preferably 400°C or more. Here, although not particularly limited, the upper limit of the peak temperature [Tp] of hydrogen emission is, for example, 500°C or more.

[0018] Furthermore, when the peak rate [Rmax] of hydrogen emission is more than 0.050 ppm/min, the level of trapping hydrogen decreases, so that hydrogen becomes easier to move in the steel. Accordingly, the peak rate [Rmax] of hydrogen emission is 0.050 ppm/min or less. The peak rate [Rmax] is preferably 0.045 ppm/min or less and more preferably 0.035 ppm/min or less. Here, although not particularly limited, the lower limit of the peak rate [Rmax] of hydrogen emission is, for example, 0.005 ppm/min or more.

[0019] Note that the amount of emitted hydrogen [H], the peak temperature [Tp] of hydrogen emission, and the peak rate [Rmax] of hydrogen emission are measured by the thermal desorption analysis and specifically may be measured in the procedure described below.

[0020] After a test specimen of 10 mm (width direction) × 30 mm (rolling direction) is cut out from a steel sheet, the test specimen is subjected to degreasing and cleaning with organic solvent. Next, the thermal desorption analysis (TDA) is used to measure desorbed hydrogen by using the test specimen for evaluation. In TDA, heat is applied in an argon atmosphere within the temperature range from 25 (room temperature) to 800°C at the heating rate 100°C/h and hydrogen emitted (desorbed) from test specimen is measured by a chromatograph. The used chromatograph is capable of detecting hydrogen at an accuracy of 0.01 ppm. Based on measurement results in TDA, the TDA curve as in Figure 1 is created and the amount of emitted hydrogen [H], the peak temperature [Tp] of hydrogen emission, the peak rate [Rmax] of hydrogen emission are calculated. Note that it is possible to calculate the amount of emitted hydrogen by subjecting the curve to integration.

2. Number density of compounds

[0021] Furthermore, in the austenitic stainless steel of the embodiment, the number density of compounds is controlled to keep hydrogen solidly trapped in the steel. Specifically, the number density of compounds having a diameter of 0.1 to 2 μm is 1.0 to 10.0/mm².

[0022] Fine precipitates such as those including Nb and/or Ti, specifically, carbide and carbonitride have diameters of 0.1 to 2 μm and are effective for trapping hydrogen. Accordingly, the number density of compounds having a diameter of 0.1 to 2 μm is 1.0/mm² or more. The reason is that, when the number density of compounds having a diameter of 0.1 to 2 μm is less than 1.0/mm², hydrogen cannot be trapped sufficiently. The number density of compounds having a diameter of 0.1 to 2 μm is preferably 1.5/mm² or more.

[0023] In the meantime, the number density of compounds having a diameter of 0.1 to 2 μm is 10.0/mm² or less. When the number density of compounds having a diameter of 0.1 to 2 μm is more than 10.0/mm², hydrogen is excessively trapped, and what is worse, hydrogen embrittlement resistance degrades. The number density of compounds having a diameter of 0.1 to 2 μm is preferably 8.5/mm² or less and more preferably 6.5/mm² or less.

[0024] The number density of compounds may be measured in the procedure described below. Measurement samples are prepared in compliant with JIS Z 0555: 2003. The preparation is made such that the measurement area of a measurement sample is 72 mm². An automatic inclusion analyzer that comes with a SEM is used to verify compounds having a diameter of 0.1 to 2 μm and measure the number density. Here, the above-described diameter refers to an equivalent circle diameter, which can be calculated with the automatic inclusion analyzer. As the automatic inclusion analyzer, for example, the MQA (Metal Quality Analyzer: registered trademark) from ASPEx Corporation may be used and inclusions (compounds) defined in JIS Z 0555: 2003 may be measured and analyzed by using MQA analyzing software (Metals Cleanliness Rating: MQA-MCR (registered trademark)).

[0025] Generally, in fine compounds as described above include, for example, oxide that does not contribute to trapping hydrogen. Accordingly, it is desirable to measure precipitates such as carbide and carbonitride including Nb and/or Ti. However, it may be difficult to distinguish oxide from these precipitates by the automatic inclusion analyzer. In addition, oxide is typically coarse, and it is considered that oxide of 2 μm or less in size is minute. Accordingly, the number density of all compounds of 0.1 to 2 μm is measured without distinction.

3. Chemical composition

[0026] As described above, in the austenitic stainless steel of the embodiment, hydrogen embrittlement resistance is improved by controlling the presence of hydrogen. Accordingly, there is no need to particularly limit the chemical composition to the extent that the stainless steel is austenitic stainless steel. However, the effect of the steel sheet of the embodiment is produced particularly in a component design with reduced expensive alloying elements. Accordingly, the austenitic stainless steel of the embodiment preferably has the chemical composition presented below. The reason for limitation for each element is described below. Note that in the description below, "%" for a content refers to "by mass%".

C: 0.080% or less

[0027] C (carbon) is an element that is effective for stabilization of the austenite phase and produces an effect of improving strength. However, excessively contained C leads to degradation of toughness. Accordingly, the content of C is preferably 0.080% or less. The content of C is more preferably 0.070% or less, more preferably 0.065% or less, and more preferably 0.055% or less. On the other hand, to obtain the above-described effects, the content of C is preferably 0.010% or more and is preferably 0.020% or more.

Si: 1.00% or less

[0028] Si (silicon) is an element that is effective for deoxidation and improves hydrogen embrittlement resistance. However, excessively contained Si promotes generation of an intermetallic compound such as a σ phase and leads to degradation of toughness and the like. Accordingly, the content of Si is preferably 1.00% or less. The content of Si is more preferably 0.90% or less, more preferably 0.80% or less, more preferably 0.70% or less, and more preferably 0.60% or less. On the other hand, to obtain the above-described effects, the content of Si is preferably 0.10% or more, preferably 0.20% or more, and is preferably 0.30% or more.

Mn: 2.00% or less

[0029] Mn (manganese) is an element that is effective for stabilization of the austenite phase and is an element that is effective for improving hydrogen embrittlement resistance. However, excessively contained Mn increases alloy costs. Accordingly, the content of Mn is preferably 2.00% or less. The content of Mn is more preferably 1.90% or less, more preferably 1.80% or less, and more preferably 1.70% or less. On the other hand, to obtain the above-described effects, the content of Mn is preferably 0.50% or more, preferably 0.70% or more, and is preferably 0.90% or more.

P: 0.050% or less

[0030] P (phosphorus) is an impurity element contained in the steel and an element that leads to degradation of mechanical properties. Accordingly, the content of P is preferably 0.050% or less. The content of P is more preferably 0.040% or less and more preferably 0.030% or less. While it is preferable that the content of P is lowered as much as possible, excessively reducing P increases refining costs. Accordingly, the content of P is preferably 0.010% or more.

S: 0.020% or less

[0031] S (sulfur) is an impurity element contained in the steel and leads to degradation of mechanical properties. Accordingly, the content of S is preferably 0.020% or less. The content of S is more preferably 0.015% or less and more preferably 0.010% or less. While it is preferable that the content of S is lowered as much as possible, excessively reducing S increases refining costs. Accordingly, the content of S is preferably 0.0002% or more.

Cr: 17.0 to 22.0%

[0032] Cr (chromium) is an element necessary to maintain corrosion resistance of the stainless steel. Cr also produces an effect of improving strength. Accordingly, the content of Cr is preferably 17.0% or more. The content of Cr is more preferably 17.5% or more and is further preferably 18.0% or more. However, excessively contained Cr leads to degradation of toughness. Accordingly, the content of Cr is preferably 22.0% or less and is preferably 20.0% or less. The content of Cr is more preferably 19.5% or less.

Ni: 8.0 to 13.0%

[0033] Ni (nickel) produces an effect of improving hydrogen embrittlement resistance. An effect of improving strength also produced. Accordingly, the content of Ni is preferably 8.0% or more. The content of Ni is more preferably 8.5% or more, more preferably 9.0% or more, and more preferably 9.5% or more. However, since Ni is an expensive element, excessively contained Ni leads to an increase in alloy costs. Accordingly, the content of Ni is preferably 13.0% or less, more preferably 12.0% or less, and more preferably 11.0% or less.

N: 0.250% or less

[0034] N (nitrogen) is an element that is effective for improving hydrogen embrittlement resistance similarly to Mn and Ni. However, excessively contained N may causes inner defects such as blowholes while being melted to occur, so that a starting point of fracture is likely to occur. As a result, the impact resistance degrades. Accordingly, the content of N is preferably 0.250% or less. The content of N is more preferably 0.240% or less, more preferably 0.230% or less, more preferably 0.200% or less, and more preferably 0.170% or less. On the other hand, to obtain the above-described effects, the content of N is preferably 0.010% or more, preferably 0.040% or more, and is preferably 0.070% or more.

[0035] In addition to the above-described elements, one or more elements selected from Nb and Ti may further be contained to the extent indicated below. That is, the lower limits of these elements are preferably 0%. The reason for limitation for each element will be described.

Nb: 0 to 0.20%

[0036] Nb (niobium) forms fine precipitates and produces an effect of enhancing an action of trapping hydrogen. Accordingly, Nb may be contained as necessary. However, excessively contained Nb, which is an expensive element, leads to an increase in alloy costs. Furthermore, precipitates are excessively formed, which leads to degradation of toughness. Accordingly, the content of Nb is preferably 0.20% or less. The content of Nb is more preferably 0.18% or less and is further preferably 0.15% or less. On the other hand, to obtain the above-described effects, the content of Nb is more preferably 0.03% or more.

Ti: 0 to 0.20%

[0037] Ti (titanium) forms fine precipitates and produces an effect of enhancing an action of trapping hydrogen similarly to Nb. Accordingly, Ti may be contained as necessary. However, excessively contained Ti, which is an expensive element, leads to an increase in alloy costs. Accordingly, the content of Ti is preferably 0.20% or less. The content of Ti is more preferably 0.18% or less and further preferably 0.15% or less. On the other hand, to obtain the above-described effects, the content of Ti is more preferably 0.01% or more.

[0038] As described above, Ti and Nb form fine precipitates and enhance an action of trapping hydrogen, and therefore, the contents of Ti and Nb preferably satisfy Formula (i) described below:

$$0.01 \leq \text{Ti} + \text{Nb} \quad (\text{i})$$

where each element symbol in the formula represents a content (by mass%) of each element contained in the steel and is

zero when the element is not contained.

[0039] Specifically, when the total content of Ti and Nb, which is a left term value of Formula (i), is less than 0.01, the amounts of Ti and Nb in the steel are too low, which makes it difficult to form a sufficient number of compounds required to trap hydrogen. As a result, the number density of compounds whose diameters are 0.1 to 2 μm is likely to fall less than 1.0/mm². Consequently, it becomes difficult to improve hydrogen embrittlement resistance sufficiently. Accordingly, the left term value of Formula (i) is preferably 0.01 or more, preferably 0.03 or more, preferably 0.05 or more, and preferably 0.10 or more.

[0040] On the other hand, when the left term value of Formula (i) is more than 0.40, compounds are excessively formed, and the number density of compounds whose diameters are 0.1 to 2 μm is likely to be more than 10.0/mm². As a result, what is worse, the hydrogen embrittlement resistance is likely to degrade. Accordingly, the left term value of Formula (i) is preferably 0.40 or less, preferably 0.35 or less, and preferably 0.30 or less.

[0041] In addition to the above-described elements, one or more elements selected from Mo, Cu, Al, Co, V, W, B, Ca, Mg, Zr, Ga, Hf, and REM may further be contained to the extent indicated below. That is, the lower limit of these elements is preferably 0%. The reason for limitation for each element will be described.

Mo: 0 to 1.0%

[0042] Mo (molybdenum) produces an effect of improving strength and corrosion resistance. Accordingly, Mo may be contained as necessary. However, since Mo is an expensive element, excessively contained Mo leads to an increase in alloy costs. Accordingly, the content of Mo is preferably 1.0% or less. The content of Mo is more preferably 0.7% or less, more preferably 0.5% or less, and more preferably 0.4% or less. On the other hand, to obtain the above-described effects, the content of Mo is preferably 0.1% or more and preferably 0.2% or more.

Cu: 0 to 1.0%

[0043] Cu (copper) produces an effect of improving strength and corrosion resistance. Accordingly, Cu may be contained as necessary. However, since Cu is an expensive element, excessively contained Cu leads to an increase in alloy costs. Furthermore, the steel is excessively hardened, which leads to degradation of mechanical properties such as toughness. Accordingly, the content of Cu is preferably 1.0% or less. The content of Cu is more preferably 0.9% or less, more preferably 0.8% or less, and more preferably 0.6% or less. On the other hand, to obtain the above-described effects, the content of Cu is preferably 0.1% or more and more preferably 0.2% or more.

Al: 0 to 0.30%

[0044] Al (aluminum) is an element that produces a deoxidation effect. Accordingly, Al may be contained as necessary. However, excessively contained Al causes excessive formation of inclusions, leading to degradation of surface texture. The hot workability also degrades. Accordingly, the content of Al is preferably 0.30% or less. The content of Al is more preferably 0.25% or less, more preferably 0.20% or less, more preferably 0.15% or less, and more preferably 0.10% or less. On the other hand, to obtain the above-described effects, the content of Al is preferably 0.01% or more and preferably 0.02% or more.

Co: 0 to 0.50%

[0045] Co (cobalt) produces an effect of improving strength and corrosion resistance. Co also stabilizes the austenite phase, so that an effect of improving hydrogen embrittlement resistance is produced. Accordingly, Co may be contained as necessary. However, excessively contained Co, which is an expensive element, leads to an increase in alloy costs. The workability and the toughness also degrade. Accordingly, the content of Co is preferably 0.50% or less. The content of Co is more preferably 0.40% or less and more preferably 0.30% or less. On the other hand, to obtain the above-described effects, the content of Co is preferably 0.10% or more.

V: 0 to 0.50%

[0046] V (vanadium) precipitates as solid solution or carbonitride in the steel, and produces an effect of improving strength. Accordingly, V may be contained as necessary. However, excessively contained V causes excessive formation of carbonitride, leading to degradation of producibility during hot rolling. Accordingly, the content of V is preferably 0.50% or less. The content of V is more preferably 0.40% or less and more preferably 0.30% or less. On the other hand, to obtain the above-described effects, the content of V is preferably 0.05% or more.

W: 0 to 0.50%

[0047] W (tungsten) produces an effect of improving strength and corrosion resistance. Accordingly, W may be contained as necessary. However, excessively contained W leads to an increase in alloy costs. Accordingly, the content of W is preferably 0.50% or less. The content of W is more preferably 0.40% or less and more preferably 0.30% or less. On the other hand, to obtain the above-described effects, the content of W is preferably 0.05% or more.

B: 0 to 0.0050%

[0048] B (boron) produces an effect of enhancing grain boundaries and improving strength. Accordingly, B may be contained as necessary. However, excessively contained B leads to degradation of workability. Accordingly, the content of B is preferably 0.0050% or less. The content of B is more preferably 0.0030% or less and more preferably 0.0020% or less. On the other hand, to obtain the above-described effects, the content of B is preferably 0.0002% or more.

Ca: 0 to 0.010%

[0049] Ca (calcium) suppresses grain boundary segregation of low melting point elements and produces an effect of enhancing grain boundaries. Accordingly, Ca may be contained as necessary. However, when Ca is excessively contained, segregation is likely to occur, leading to degradation of toughness. Accordingly, the content of Ca is preferably 0.010% or less. The content of Ca is more preferably 0.007% or less and more preferably 0.005% or less. On the other hand, to obtain the above-described effects, the content of Ca is preferably 0.0002% or more.

Mg: 0 to 0.010%

[0050] Mg (magnesium) suppresses grain boundary segregation of low melting point elements and produces an effect of enhancing grain boundaries. Accordingly, Mg may be contained as necessary. However, excessively contained Mg causes formation of a large number of inclusions, which is likely to be a starting point of fracture, and as a result, the toughness may degrade. Accordingly, the content of Mg is preferably 0.010% or less. The content of Mg is more preferably 0.007% or less and more preferably 0.005% or less. On the other hand, to obtain the above-described effects, the content of Mg is preferably 0.0002% or more.

Zr: 0 to 0.50%

[0051] Zr (zirconium) produces a deoxidation effect. An effect of improving corrosion resistance is also produced. Accordingly, Zr may be contained as necessary. However, excessively contained Zr leads to degradation of toughness and workability. Accordingly, the content of Zr is preferably 0.50% or less. The content of Zr is more preferably 0.30% or less and more preferably 0.20% or less. On the other hand, to obtain the above-described effects, the content of Zr is preferably 0.01% or more.

Ga: 0 to 0.05%

[0052] Ga (gallium) produces an effect of improving hot workability. Accordingly, Ga may be contained as necessary. However, excessively contained Ga leads to degradation of producibility. Accordingly, the content of Ga is preferably 0.05% or less. The content of Ga is more preferably 0.04% or less and more preferably 0.02% or less. On the other hand, to obtain the above-described effects, the content of Ga is preferably 0.001% or more.

Hf: 0 to 0.10%

[0053] Hf (hafnium) produces an effect of improving strength and improving hydrogen embrittlement resistance. Accordingly, Hf may be contained as necessary. However, excessively contained Hf leads to degradation of workability. Accordingly, the content of Hf is preferably 0.10% or less. The content of Hf is preferably 0.07% or less and more preferably 0.05% or less. On the other hand, to obtain the above-described effects, the content of Hf is preferably 0.01% or more.

REM: 0 to 0.10%

[0054] REM produces an effect of improving hot workability. An effect of improving corrosion resistance is also produced. Accordingly, REM may be contained as necessary. However, excessively contained REM not only causes the effect to be saturated, but also causes degradation of hot workability. Accordingly, the content of REM is preferably

0.10% or less. The content of REM is preferably 0.07% or less and more preferably 0.05% or less. On the other hand, to obtain the above-described effects, the content of REM is preferably 0.01% or more.

[0055] REM refers to a total of 17 elements of Sc, Y, and lanthanoid, and the content of REM refers to a total content of the elements. Industrially, REM is often contained as misch metal.

[0056] In the chemical composition of the steel sheet of the embodiment, the balance is preferably Fe and impurities. Here, "impurities" refer to components that are introduced due to various factors in raw materials such as scrap and ore and production processes when the austenitic stainless steel is industrially produced and that are acceptable to the extent that they do not adversely affect the steel of the embodiment.

4. Application and shape

[0057] It is preferable that the austenitic stainless steel of the embodiment is used in a high-pressure hydrogen gas environment or a liquid hydrogen environment. For example, the austenitic stainless steel is preferably used for components of hydrogen gas production apparatuses or hydrogen gas supply apparatuses. Here, components of hydrogen gas production apparatuses or hydrogen gas supply apparatuses include, for example, a tank body, a mouthpiece, a liner, valve, a heat exchanger, and instrumentation such as a dispenser.

[0058] The shape of the steel is not particularly limited. For example, the steel may be a steel sheet. To use in the application described above, in the case of a steel sheet, it is assumed that the sheet thickness is 0.25 to 6.0 mm.

5. Production method

[0059] For example, the austenitic stainless steel of the present disclosure can stably be produced by production method as described below. Note that in the description below, the case of a steel sheet, which is taken as an example, will be mainly described.

5-1. Hot working process

[0060] The stainless steel is melted to produce a cast piece such as a slab. At this time, the chemical composition of the cast piece is preferably the chemical composition described above. Next, the cast piece is subjected to hot working by heating it to a predetermined temperature, so that a hot-worked material is obtained. When a steel sheet is to be produced, the material may be subjected to hot rolling. The heating temperature during hot working is preferably within the range of 1150 to 1250°C. Furthermore, to keep the number density of compounds in 1.0 to 10.0/mm², the working ratio in the temperature region of 900 to 1050°C during hot working is made 75 to 90%. For example, in the case of a steel sheet, the rolling reduction in the temperature region of 900 to 1050°C during hot rolling is within the range of 75 to 90%. When the rolling reduction in the above-described temperature region is less than 75%, the number density of compounds may fall to less than 1.0/mm². Note that, due to production constraints, the rolling reduction in the above-described temperature region is preferably 90% or less.

[0061] After hot working, annealing may be performed as needed to control microstructure. While annealing conditions are not particularly limited, for example, the annealing temperature is preferably within the range of 950 to 1150°C. In addition, the annealing time is preferably within the range of 0.5 to 15 minutes. Here, the annealing atmosphere may be an ambient air atmosphere. After hot working, or after annealing when the annealing has been performed after the hot rolling, pickling is performed to remove scale. Conditions for pickling are also not particularly limited. Pickling may be according to common procedures.

5-2. Cold working

[0062] The hot-worked material subjected to pickling and the like is subjected to cold working as needed, so that a cold-worked material is obtained. When a steel sheet is to be produced, the material may be subjected to cold rolling. While conditions during cold working, for example, the working ratio and the like during cold working are not particularly limited, in the case of a steel sheet, the rolling reduction is preferably 40% or more given such application.

[0063] Note that cold working may be performed several times. Heat treatment may also be performed between cold working and cold working. Conditions for heat treatment to be performed are preferably, but not particularly limited, for example, within the temperature range from 950 to 1150°C for 10 seconds to 10 minutes.

5-3. Annealing of cold-worked material

[0064] After cold working described above, the cold-worked material is subjected to annealing. The anneal temperature is preferably within the range of 950 to 1150°C. In addition, the annealing time is preferably within the range of 5 seconds to

3 minutes. The anneal temperature and the annealing time are in the range described above because recrystallization is promoted for homogeneous microstructures. While the annealing atmosphere is not particularly limited, for example, it may be an LNG combustion atmosphere or may be a reducing atmosphere including hydrogen gas. That is, both LNG annealing and BA annealing may be satisfactory. Note that BA annealing tends to provide a higher amount of emitted hydrogen [H]. Typical atmospheres including hydrogen gas include an atmosphere of 100% hydrogen gas or ammonia cracking gas (75% hydrogen gas+25% nitrogen gas). After annealing described above, pickling may be performed as needed. Conditions for pickling at this time are also not particularly limited. Pickling may be according to common procedures.

5-4. Warming processing

[0065] Subsequently, the cold-worked material subjected to annealing and pickling as needed is subjected to warming processing. The warming processing refers to a heat treatment in which the temperature region is held within 50 to 400°C for 0.5 to 360 hours. By performing heat treatment in this way at a low temperature for a long time, the presence of hydrogen can be controlled, and the amount of emitted hydrogen [H], the peak temperature [Tp] of hydrogen emission, and the peak rate [Rmax] of hydrogen emission can be kept in the range described above. After warming processing, cooling is performed as needed to adjust each process to bring about a desired austenitic stainless steel.

[0066] The atmosphere during warming processing is not particularly limited. An ambient air atmosphere or an atmosphere of N₂ gas or Ar gas under an atmospheric pressure may be satisfactory.

[0067] Embodiments of the austenitic stainless steel according to the present disclosure will now be described with reference to examples in detail. However, the present disclosure is not limited to the examples.

EXAMPLE

[0068] The stainless steels having chemical compositions shown in Table 1 are melted to obtain cast pieces. The obtained cast pieces were heated to 1230°C and subjected to hot rolling under the conditions shown in Table 2 for rolling reduction within the temperature region from 900 to 1050°C, so that hot-rolled sheets having a sheet thickness of 5.0 mm were obtained. Some examples were held at 1100°C for 3 minutes after hot rolling and subjected to hot-rolled sheet annealing. After hot rolling or hot-rolled sheet annealing, pickling was performed. Thereafter, the hot-rolled sheets were subjected to cold rolling, so that cold-rolled sheets having a sheet thickness of 1.2 mm were obtained. Here, as described in Table 2, cold rolling was performed once or twice. Subsequently, cold-rolled-sheet annealing was performed at 1080°C, and thereafter, pickling was performed. After pickling, warming processing was performed as shown in Table 2. Note that all the obtained steel sheets were austenitic stainless steels.

[0069] In Table 2, when cold rolling is performed once, it indicates that cold rolling at a cold rolling ratio of 76% was performed once to bring about a cold-rolled sheet having a sheet thickness of 1.2 mm. In Table 2, when cold rolling is performed twice, it indicates that cold rolling at a cold rolling ratio of 40% was performed once to bring about a steel sheet having a thickness of 3.0 mm, and thereafter, heat treatment of holding 1080°C for 1 minute was performed, followed again by cold rolling at a cold rolling ratio of 60% to bring about a cold-rolled sheet having a thickness of 1.2 mm. Furthermore, in Table 2, the entry "LNG" for cold-rolled-sheet annealing indicates that annealing was performed in an LNG combustion atmosphere for 1 minute or less, and "BA" indicates that annealing was performed in a 0.1 MPa H₂ atmosphere for 1 minute or less.

[0070] In Table 2, the entry "Yes 1" for warming processing indicates that warming processing of holding 80°C in an ambient air atmosphere for 7 days was performed, and "Yes 2" indicates that warming processing of holding 300°C under an atmospheric pressure in an atmosphere of N₂ gas or Ar gas for 4 hours.

[Table 1]

[0071]

Table 1

Steel Type	Chemical composition (mass%, Balance: Fe and impurities)														Nb+Ti
	C	Si	Mn	P	S	Cr	Ni	N	Nb	Ti	Mo	Cu	Al	Others	
A	0.055	0.40	1.00	0.035	0.005	17.1	11.2	0.040	0.03	0.01	1.0	0.25	0.01		0.04
B	0.072	0.55	0.90	0.030	0.002	21.1	8.6	0.170	0.05	0.03	0.2	0.2	0.02	Co: 0.10, V: 0.20, B: 0.0012	0.08
C	0.010	0.50	1.95	0.033	0.001	18.8	10.3	0.190	0.15	-	0.1	0.3	0.05	Ca: 0.0023, Mg: 0.0011, REM: 0.01	0.15
D	0.020	0.90	1.80	0.028	0.001	18.2	12.7	0.010	-	0.11	-	-	-		0.11
E	0.055	0.15	1.90	0.021	0.001	18.5	8.2	0.240	0.08	0.02	0.3	0.6	0.10	W: 0.10, B: 0.0021, Zr: 0.01, Ga: 0.02	0.10
F	0.065	0.40	1.60	0.025	0.001	18.8	8.9	0.085	0.05	0.05	0.4	0.9	0.02	REM(La:0.02, Y:0.02), Hf: 0.02	0.10
G	0.076	0.40	1.60	0.030	0.001	19.6	9.0	0.045	0.13	0.03	0.2	0.3	0.25		0.16
H	0.065	0.40	1.65	0.025	0.001	18.7	9.0	0.090	0.22	0.20	0.4	0.9	0.02		0.42
I	0.010	0.50	1.90	0.035	0.001	18.7	10.5	0.190	-	-	0.1	0.3	0.05	Ca: 0.0033, Mg: 0.0050	-

[0072] The obtained steel sheets were subjected to thermal desorption analysis from 25°C to 800°C at a heating rate of 100°C/h to examine the presence of hydrogen (amount of emitted hydrogen [H], peak temperature [Tp] of hydrogen emission, and peak rate [Rmax] of hydrogen emission). The number density of compounds having a diameter of 0.1 to 2 μm was also measured. Measurement for each value was performed according to the procedures described below.

(Presence of hydrogen)

[0073] After a test specimen of 10 mm (width direction) × 30 mm (rolling direction) was cut out from a steel sheet, the test specimen was subjected to degreasing and cleaning with organic solvent. Next, the thermal desorption analysis (TDA) was used to measure desorbed hydrogen by using the test specimen. In TDA, heat was applied in an argon atmosphere within the temperature range from 25 (room temperature) to 800°C at the heating rate 100°C/h and hydrogen emitted (desorbed) from test specimen was measured by a chromatograph. Based on measurement results in TDA, TDA curve was created and the amount of emitted hydrogen [H], the peak temperature [Tp] of hydrogen emission, and the peak rate [Rmax] of hydrogen emission were calculated.

(Number density of compounds)

[0074] Measurement samples were prepared in compliant with JIS Z 0555: 2003. The preparation was made such that the measurement area of a measurement sample was 72 mm². An automatic inclusion analyzer was used to verify compounds having a diameter of 0.1 to 2 μm and measure the number density. The automatic inclusion analyzer that was used was MQA (registered trademark) from ASPEX Corporation, and inclusions (compounds) defined in JIS Z 0555: 2003 were measured and analyzed by using analyzing software (MQA-MCR (registered trademark)).

[0075] As in the measurement of the values of physical properties described above, hydrogen embrittlement resistance was also evaluated. A slow strain rate tensile test (also referred to as "SSRT test") was conducted to evaluate hydrogen embrittlement resistance. The SSRT test was performed according to the procedures described below. First, tensile test specimens having a parallel portion of 4 mm width × 20 mm length were sampled. Subsequently, tensile tests were performed on the tensile test specimens at -40°C in hydrogen of 1 MPa at a strain rate of 3×10^{-5} /s. As a comparative test, a tensile test was performed at similar strain rate at -40°C in nitrogen under a 0.1 MPa atmospheric pressure. In some examples, similar tensile tests were also performed at -70°C.

[0076] When tensile strength and elongation after fracture did not degrade as compared to results of the comparative tests, "GOOD" was described in the table as the hydrogen embrittlement resistance being good. On the other hand, when at least one of tensile strength and elongation after fracture degraded as compared to results of the comparative tests, "NG" was described in the table as the hydrogen embrittlement resistance being poor.

[0077] The evaluations as those described above were conducted in the case of both -40°C and -70°C, and when tensile strength and elongation after fracture did not degrade at both temperature, "A" was described in the judgement of hydrogen embrittlement resistance. When tensile strength and elongation after fracture did not degrade at -40°C, but tensile strength and/or elongation after fracture degraded at -70°C, "B" was described in the judgement of hydrogen embrittlement resistance. When at least one of tensile strength and elongation after fracture degraded at -40°C, "C" was described in the judgement of hydrogen embrittlement resistance. The results are collectively described in Table 2 below.

[Table 2]

[0078]

Table 2

No	Steel Type	Manufacturing conditions					Presence of Hydrogen			Number Density of Compounds (/mm ²)	SSRT at 1 MPaH ₂		judgement of hydrogen embrittlement resistance	Note
		Working Ratio at 900 to 1050°C (%)	Hot-Rolled Sheet Annealing	Number of Cold Rolling	Cold-Rolled Sheet Annealing	Warming Processing	[H] (ppm)	[Tp] (°C)	[Rmax] (ppm/min)		-40°C	-70°C		
1	A	87	No	2	BA	Yes 1	6.5	420	0.040	1.7	GOOD	GOOD	A	Inventive Example
2		87	Yes	2	BA	Yes 1	6.7	400	0.040	1.4	GOOD	NG	B	
3		87	Yes	2	BA	No	8.5	<u>340</u>	<u>0.055</u>	1.3	NG	-	C	Comparative Example
4	B	87	No	1	BA	Yes 1	7.0	390	0.045	1.4	GOOD	NG	B	Inventive Example
5		82	No	1	LNG	Yes 2	2.3	380	0.020	1.6	GOOD	NG	B	Inventive Example
6		82	No	1	LNG	No	3.1	<u>330</u>	0.020	2.0	NG	-	C	Comparative Example
7	C	82	Yes	1	LNG	Yes 2	2.1	360	0.020	2.1	GOOD	NG	B	Inventive Example
8		82	Yes	1	LNG	No	2.9	<u>330</u>	0.025	2.1	NG	-	C	Comparative Example
9	D	83	No	1	LNG	Yes 1	0.9	360	0.015	5.2	GOOD	GOOD	A	Inventive Example
10		83	No	1	LNG	No	1.5	<u>320</u>	0.015	5.1	NG	-	C	Comparative Example
11	E	88	No	1	LNG	Yes 2	1.0	480	0.010	3.3	GOOD	GOOD	A	Inventive Example
12		88	No	1	LNG	No	1.2	<u>330</u>	0.015	3.4	NG	-	C	Comparative Example

(continued)

No	Steel Type	Manufacturing conditions					Presence of Hydrogen			Number Density of Compounds (/mm ²)	SSRT at 1 MPaH ₂		judgement of hydrogen embrittlement resistance	Note
		Working Ratio at 900 to 1050°C (%)	Hot-Rolled Sheet Annealing	Number of Cold Rolling	Cold-Rolled Sheet Annealing	Warming Processing	[H] (ppm)	[Tp] (°C)	[Rmax] (ppm/min)		-40°C	-70°C		
13	E	78	Yes	2	LNG	Yes 2	2.8	400	0.035	6.1	GOOD	NG	B	Inventive Example
14		78	Yes	2	LNG	No	3.8	370	0.055	6.2	NG	-	C	Comparative Example
15	F	86	Yes	1	BA	Yes 1	7.8	390	0.045	2.5	GOOD	NG	B	Inventive Example
16		86	Yes	1	BA	No	10.5	380	0.055	2.6	NG	-	C	Comparative Example
17	G	87	No	2	BA	Yes 1	5.5	370	0.040	8.2	GOOD	GOOD	A	Inventive Example
18		87	Yes	2	BA	Yes 1	5.8	370	0.045	6.2	GOOD	NG	B	Inventive Example
19		87	Yes	2	BA	No	7.0	370	0.055	5.3	NG	-	C	Comparative Example
20		87	Yes	1	BA	Yes 1	5.7	380	0.045	6.2	GOOD	NG	B	Inventive Example
21		87	Yes	1	BA	No	7.0	370	0.055	5.1	NG	-	C	Inventive Example
22		86	No	2	BA	Yes 1	10.2	400	0.055	10.2	NG	-	C	Comparative Example
23	I	83	No	2	BA	Yes 1	6.1	300	0.040	0.8	NG	-	C	Comparative Example
24	D	73	No	1	LNG	Yes 2	1.0	290	0.010	0.7	NG	-	C	Comparative Example
25	G	73	No	2	BA	Yes 1	50	310	0.035	0.8	NG	-	C	Comparative Example

Underlined: Deviates from the preferred manufacturing conditions of this embodiment, or deviates from the requirements of this embodiment. Number density of the compounds means the number density of the compounds having a diameter of 0.1 to 2 μm.

[0079] Nos. 1, 2, 4, 5, 7, 9, 11, 13, 15, 17, 18, and 20, which satisfied the requirements of the embodiment, had good hydrogen embrittlement resistance. On the other hand, Nos. 3, 6, 8, 10, 12, 14, 16, 19, and 21 to 25, which were not satisfy the requirements of the embodiment, were poor in hydrogen embrittlement resistance.

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Claims

1. An austenitic stainless steel, wherein when an amount of hydrogen emitted at a heating rate of 100°C/h and in a temperature range from 25 to 800°C is measured by using thermal desorption analysis,

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an amount of emitted hydrogen [H] is less than 10.0 ppm,
 a peak temperature [Tp] of hydrogen emission is 350°C or more,
 a peak rate [Rmax] of hydrogen emission is 0.050 ppm/min or less, and
 a number density of compounds having a diameter of 0.1 to 2 μm is 1.0 to 10.0/mm².

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2. The austenitic stainless steel according to claim 1, wherein a chemical composition of the austenitic stainless steel consists of, by mass%,

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C: 0.080% or less,

Si: 1.00% or less,

Mn: 2.00% or less,

P: 0.050% or less,

S: 0.020% or less,

Cr: 17.0 to 22.0%,

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Ni: 8.0 to 13.0%,

N: 0.250% or less,

Nb: 0 to 0.20%,

Ti: 0 to 0.20%,

Mo: 0 to 1.0%,

30

Cu: 0 to 1.0%,

Al: 0 to 0.30%,

Co: 0 to 0.50%,

V: 0 to 0.50%,

W: 0 to 0.50%,

35

B: 0 to 0.0050%,

Ca: 0 to 0.010%,

Mg: 0 to 0.010%,

Zr: 0 to 0.50%,

Ga: 0 to 0.05%,

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Hf: 0 to 0.10%,

REM: 0 to 0.10%, and

the balance: Fe and impurities.

3. The austenitic stainless steel according to claim 1 or 2, wherein the austenitic stainless steel is used in a high-pressure hydrogen gas environment or a liquid hydrogen environment.

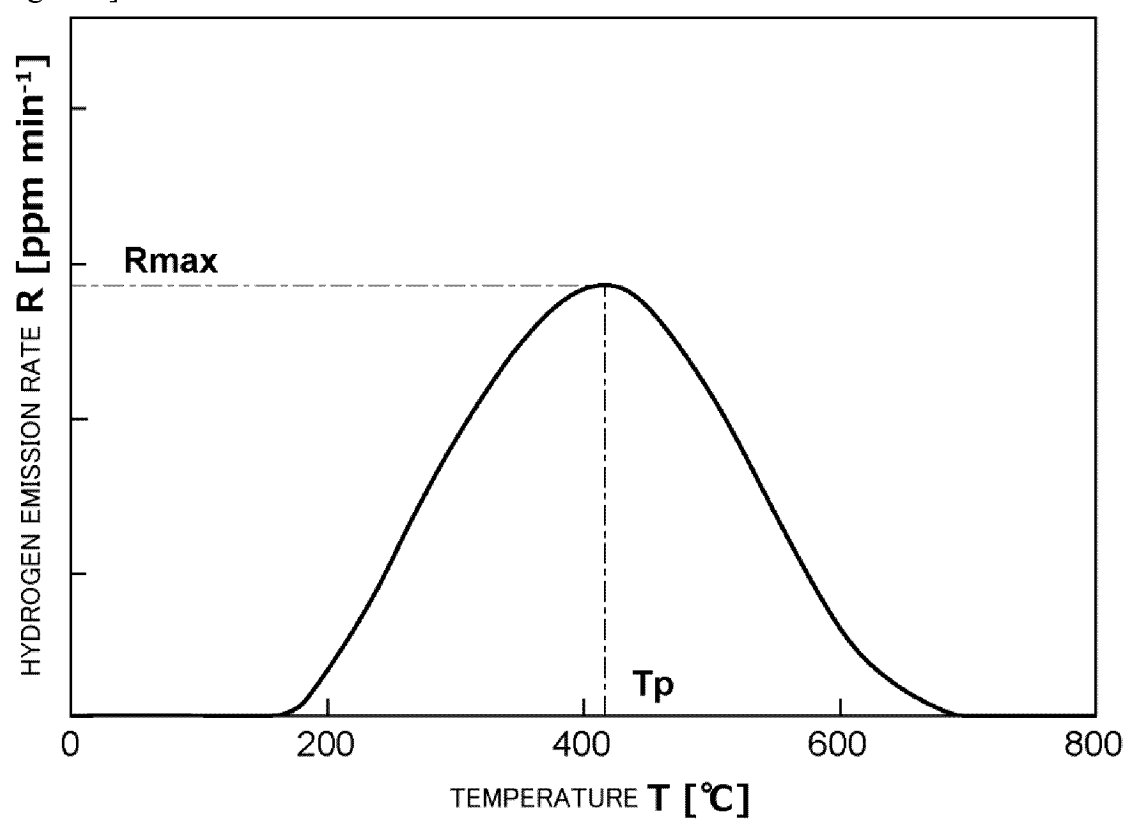
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4. The austenitic stainless steel according to claim 1 or 2, wherein the austenitic stainless steel is used for a component of a hydrogen gas production apparatus or a hydrogen gas supply apparatus.

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[Figure 1]



INTERNATIONAL SEARCH REPORT

International application No.

PCT/JP2023/028926

A. CLASSIFICATION OF SUBJECT MATTER

C21D 3/06(2006.01)i; *C21D 9/46*(2006.01)i; *C22C 38/00*(2006.01)i; *C22C 38/58*(2006.01)i
 FI: C22C38/00 302Z; C22C38/58; C21D3/06; C21D9/46 Q

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C21D3/06; C21D9/46; C22C38/00; C22C38/58

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

Published examined utility model applications of Japan 1922-1996
 Published unexamined utility model applications of Japan 1971-2023
 Registered utility model specifications of Japan 1996-2023
 Published registered utility model applications of Japan 1994-2023

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

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	WO 2009/107475 A1 (NATIONAL INSTITUTE OF ADVANCED INDUSTRIAL SCIENCE AND TECHNOLOGY) 03 September 2009 (2009-09-03)	1-4
A	WO 2007/058364 A1 (NAT INST FOR MATERIALS SCIENCE) 24 May 2007 (2007-05-24)	1-4
A	WO 2022/047714 A1 (BEKAERT(CHINA) TECHNOLOGY RESEARCH AND DEVELOPMENT CO., LTD.) 10 March 2022 (2022-03-10)	1-4

☐ Further documents are listed in the continuation of Box C. ☒ See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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"O" document referring to an oral disclosure, use, exhibition or other means	
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/JP2023/028926

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WO 2009/107475 A1	03 September 2009	US 2011/0005645 A1 EP 2248918 A1 CN 101960024 A KR 10-2010-0122910 A	
WO 2007/058364 A1	24 May 2007	US 2009/0277539 A1 EP 1956100 A1	
WO 2022/047714 A1	10 March 2022	(Family: none)	

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

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