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(54) **490-MPA-GRADE THICK STEEL PLATE WITH HIGH CORE FATIGUE STRENGTH AND MANUFACTURING METHOD THEREFOR**

(57) The present disclosure provides a 490-MPa-grade thick steel plate with high core fatigue strength and manufacturing method therefor. The steel plate comprises the following chemical components in mass percentage: C: 0.045 to 0.076%, Si: 0.19 to 0.31%, Mn: 0.95 to 1.13%, P: ≤ 0.008%, S: ≤ 0.002%, Als: 0.010 to 0.040%, Nb: 0.014 to 0.038%, V: 0.025 to 0.041%, Ti:

0.011 to 0.022%, Ni: 1.35 to 1.55%, Ce: 0.020 to 0.040%, and Fe and inevitable impurities. The steel plate exhibits an excellent core fatigue performance, and can be used for supporting parts and components which have certain requirements for the core fatigue performance of the steel plate in the buildings, engineering machinery, ocean engineering, etc.

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Description**TECHNICAL FIELD**

- 5 **[0001]** The present disclosure belongs to the technical field of material, and particularly relates to a 490-MPa-grade thick steel plate with excellent core fatigue strength and manufacturing method therefor.

BACKGROUND

- 10 **[0002]** With the development of the current economy and the continuous progress of equipment design and manufacturing capabilities, there is an increasing demand for the high-strength steel for large components, and the materials for the main structures are constantly developing towards high strength, high toughness and large thickness. Thick steel plates are important structural materials of large structures, equipment and facilities for high-rise buildings, ocean development, crude oil tanks, oil and gas pipelines, ships and warships, etc.
- 15 **[0003]** At present, thick steel plates are usually rolled from thick casting slabs, but the most continuous casting slabs are limited by their solidification characteristics and existing smelting equipment and processes. The uneven distribution of chemical composition in the cross-section, internal segregation, porosity, shrinkage and other defects in the casting slab are serious, which can have a significant impact on the strength, toughness, fatigue performance and other indicators of the steel plate. Especially the center segregation of the casting slab, it can be "inherited" to the steel plate during rolling
- 20 process, resulting in the abnormal microstructure and unqualified flaws detected at the center part in steel plate thickness, and affecting the safety of steel structure components due to the occurrence of brittle zones and premature damage in the core during the next processing or in use. In addition, the current inspection standards often focus on the performance at 1/4 thickness of the steel plate, and have less constraint on the core performance. Therefore, it is necessary to improve the comprehensive mechanical properties at the central part of thick steel plates, especially in harsh service environments
- 25 where higher requirements for toughness, fatigue performance, etc. are needed.
- [0004]** Patent Document 1 discloses a 460-MPa-grade hot-rolled steel plate for automotive structures with good fatigue performance and manufacturing method therefor. The steel plate comprises the following chemical elements in mass percentage: C: 0.03-0.06%, Mn: 1.0-1.2%, Nb: 0.025-0.035%, Ti: 0.025-0.035%, Si: <0.10%, S: ≤0.005%, P: ≤0.015%, N: ≤40ppm, Als: 0.025-0.050%, the balance being Fe and inevitable impurities. Nb-Ti microalloying technology is used in a
- 30 short process to fix S and N (S ≤ 0.005%, N ≤ 40ppm) with a trace amount of Ti in steel, reducing the inclusions of MnS in the steel, fully utilizing the fine grain effect of Nb and Ti, obtaining fine F+P, and thus improving the fatigue performance of the steel grade while achieving high strength. Processes of rolling, laminar flow and coiling are adopted for production in this disclosure. The steel has a thickness of 1.0 to 3.0 mm, a yield strength of 460 to 560 MPa and a tensile strength of 500 to 640 MPa.
- 35 **[0005]** Patent Document 2 discloses a high-strength steel for automotive beam with good fatigue performance and formability and manufacturing method therefor. The steel comprises the following chemical components in mass percentage: C: 0.04-0.07%, Si: 0.05-0.15%, Mn: 1.3-1.6%, P: ≤ 0.013%, S: ≤ 0.004%, Nb: 0.02-0.04%, Ti: ≤ 0.002%, N: ≤ 0.004%, Al: 0.010-0.030%, O: ≤ 0.002%, and Ti/O < 2, the balance being Fe and inevitable impurities. The steel for automobile beam with a thickness of 2.0 to 7.0 mm produced by this disclosure has the following mechanical properties:
- 40 ReL ≥ 480 MPa, Rm ≥ 600 MPa, A ≥ 20% and a weight reduction of 17% for the parts. The steel grade has a thickness of 2.0 to 7.0 mm and the following mechanical properties: ReL ≥ 480 MPa, Rm ≥ 600 MPa and A ≥ 20%. The automotive beam steel not only has a good fatigue performance, but also has an excellent formability.
- [0006]** Patent Document 3 discloses a quenched and tempered FO460 steel plate for shipping with a large thickness and manufacturing method therefor. The steel plate comprises the following chemical elements: C: 0.06-0.10%; Si: 0.05-0.14% ; Mn: 1.40-1.80%; S: ≤ 0.002%; P: ≤ 0.008%; Als: 0.015%-0.045%; N: 0.003%-0.015%; Nb: 0.01-0.04%; Cu: 0.16-0.35% ; Ni: 0.30-0.60% ; Cr: 0.15-0.30%; Ti: 0.008-0.014%; the balance being Fe and inevitable impurities. The steel plate of this disclosure has a thickness of 60 to 100 mm, a yield strength of ≥ 460 MPa and a tensile strength of ≥ 570 MPa, which can meet the technical requirements for marine steel plate in harsh and demanding environments of ocean.
- 45 **[0007]** Patent Document 4 discloses a quenched and tempered steel plate with a yield strength of 420-MPa grade for building structures and production method therefor. The composition design of Nb and Ti microalloying treatment is carried out on the basis of Fe-Mn-C system. The percentages of the chemical components of the steel plate are respectively: C: 0.13-0.18%, Si: 0.20-0.50%, Mn: 1.40-1.70%, P: ≤ 0.015%, S: ≤ 0.005%, Cr: ≤ 0.30%, Mo: ≤ 0.30%, Ni: ≤ 0.30%, Cu: ≤ 0.30%, Al: 0.020-0.050%, V: ≤ 0.015%, Nb: 0.025-0.050%, Ti: 0.010-0.020%, N: ≤ 0.006%, the balance being Fe and inevitable impurity elements. The production process is as follows. smelting raw materials are sequentially subjected to
- 50 converter smelting, external refining and RH furnace refining to obtain high-purity molten steel. The molten steel is poured into steel slabs ranging from 370 mm to 450 mm. Then reasonable technologies of slab heating, rolling, online direct quenching and tempering heat treatment are utilized. Thus, a steel plate material with a yield strength of 420 MPa grade is obtained, which is suitable for large-scale steel structure construction projects such as high-rise buildings, large-span

sports venues, airports, exhibition centers and industrial plants, etc. The steel grade of this disclosure has a thickness of 50 to 100 mm, a yield strength ReL of 410 to 540 MPa and a tensile strength Rm of 530 to 680 MPa.

Prior Art Documents

Patent Documents

[0008]

Patent Document 1: CN107641760B

Patent Document 2: CN109161795A

Patent Document 3: CN113174535A

Patent Document 4: CN112981235A

SUMMARY

The technical problems to be solved by the disclosure

[0009] Although the above-mentioned patent documents 1 and 2 both disclose automotive steels with good fatigue performance, the thickness of the steel is only 1 to 7 mm, and the technical methods are not applicable to the manufacturing of thick steel plates. Although patent documents 3 and 4 disclose two methods for manufacturing steel plates with a large thickness, both are silent on the fatigue performance of the steel plates.

[0010] In view of the above-mentioned existing technologies, the objective of the present disclosure is to provide a 490-MPa-grade thick steel plate with excellent core fatigue strength and manufacturing method therefor. The steel plate of the present disclosure has a thickness of 60 to 100 mm, a yield strength of ≥ 490 MPa, a tensile strength of ≥ 600 MPa, and a fatigue strength of ≥ 340 MPa at the center in plate thickness. The present steel plate exhibits an excellent core fatigue performance, and the strength, toughness and fatigue performance of the steel plate are further improved. Especially, the present steel plate has an excellent comprehensive mechanical properties at the center parts. Thus, the present disclosure can solve the problems of uneven distribution of cross-sectional chemical composition, internal segregation and early onset of damage caused by brittle zones in the core of high-strength steel for large components, and is particularly suitable for application fields with high requirements for toughness, fatigue performance, etc.

The means to solve the technical problems

[0011] In order to achieve the above objectives, the present disclosure provides a 490-MPa-grade thick steel plate with high core fatigue strength, comprising the following chemical components in mass percentage:

C: 0.045-0.076%, Si: 0.19-0.31%, Mn: 0.95-1.13%, P: $\leq 0.008\%$, S: $\leq 0.002\%$, Als: 0.010-0.040%, Nb: 0.014-0.038%, V: 0.025-0.041%, Ti: 0.011-0.022%, Ni: 1.35-1.55%, Ce: 0.020-0.040%, and Fe and inevitable impurities.

[0012] One or two of B: 0.0005-0.0009% and Mo: 0.15-0.25% can be added into the steel plate.

[0013] Preferably, the steel plate with excellent core fatigue performance of the present disclosure has a microstructure of quasi-polygonal ferrite (QF) + lath bainite (BF) + pearlite (P), wherein the proportion of QF phase is 30% to 60%, the proportion of BF phase is 40% to 70%, and the proportion of P phase is 0.1% to 3% in percentage by area, which can further ensure that the steel has a good strength and toughness performance.

[0014] Preferably, the steel plate with excellent core fatigue performance of the present disclosure has an average grain size of 8 to 12 μm , which can further effectively improve the strength, toughness and fatigue performance of the steel plate.

[0015] Preferably, in the steel plate with excellent core fatigue performance of the present disclosure, the oxide inclusions are mainly $\text{Ce}_2\text{O}_3+\text{Al}_2\text{O}_3$, Ce_2O_3 , Al_2O_3 and composite inclusions with $\text{Ce}_2\text{O}_3+\text{Al}_2\text{O}_3$, Ce_2O_3 and Al_2O_3 as cores respectively, with $\text{Ce}_2\text{O}_3+\text{Al}_2\text{O}_3$ and the composite inclusion with $\text{Ce}_2\text{O}_3+\text{Al}_2\text{O}_3$ as the core accounting for 90% or more in number, Ce_2O_3 and the composite inclusion with Ce_2O_3 as the core accounting for 1% to 10% in number, and Al_2O_3 and the composite inclusion with Al_2O_3 as the core accounting for 1% or less in number.

[0016] Preferably, the steel plate with excellent core fatigue performance of the present disclosure has an inclusion density of 100 to 500 per mm^2 , wherein inclusions with a size of 0.2 to 2 μm account for 95% or more in number, inclusions with a size of >2 to 5 μm account for 5% or less in number, inclusions with a size of >5 to 10 μm account for 0.01% or less in number, and there are no inclusions with a size of greater than 10 μm .

[0017] The present disclosure further provides a manufacturing method for a 490-MPa-grade thick steel plate with high

core fatigue strength, preferably comprising the following steps:

- 1) desulfurizing molten steel, wherein the molten steel is controlled to satisfy $S \leq 0.002\%$;
- 2) vacuuming for a time period of ≥ 21 minutes, and continuous casting at a casting speed of 0.5 to 1.0 m/min; wherein a two-stage electromagnetic stirring is used, with current parameters of 420A and 455A respectively; and for dynamic soft reduction, a solid fraction is 0.35 to 0.70, and a reduction amount is 6 to 10 mm;
- 3) conventional continuous casting into a slab and heating the casted slab, wherein a heating temperature is controlled to be 1201 to 1245 °C, and a tapping temperature is not lower than 1180 °C;
- 4) performing a two-stage rolling, wherein for the first stage, an initial rolling temperature is not lower than 1063 °C, a reduction amount per pass is ≥ 20 mm, and a reduction amount for the last two passes is ≥ 40 mm; and wherein an initial rolling temperature for the second stage is not higher than 943 °C, a reduction rate for the first two passes is greater than 15%, a reduction rate for the remaining rolling passes is controlled to be 8% to 10%, and a finishing rolling temperature is 821 to 843 °C. Rapid cooling is performed after finishing rolling, wherein a cooling rate is 0.5 to 5 °C/s, and a self-tempering temperature is controlled to be not higher than 430 °C;
- 5) performing tempering heat treatment in an industrial furnace, wherein a tempering temperature is 611 to 631 °C, an in-furnace time is: (product thickness in mm $\times$ 1.5) min, and a continuous holding time is not less than (product thickness in mm $\times$ 0.9) min after reaching the temperature. After the tempering is finished, air cooling is performed to room temperature.

Effect of the disclosure

[0018] The steel plate of the present disclosure has a thickness of 60 to 100 mm, a yield strength of ≥ 490 MPa, a tensile strength of ≥ 600 MPa and a fatigue strength of ≥ 340 MPa at the center part in plate thickness. The steel plate exhibits excellent core fatigue performance, and can be used for supporting parts and components which have certain requirements for the core fatigue performance of steel plates in buildings, engineering machinery, ocean engineering, etc. The present disclosure has the advantages of simple manufacturing process and can be implemented in various metallurgical enterprises.

DETAILED DESCRIPTION

[0019] The specific embodiments and examples will be combined in the following to elaborate the present disclosure, but those skilled in the art should understand that these specific embodiments and examples are used to illustrate the present disclosure, not to limit the present disclosure.

[0020] Throughout the entire description, unless otherwise specified, the terms used in this disclosure should be understood as having meanings commonly used in the field. Therefore, unless otherwise defined, all technical and scientific terms used in this disclosure have the same meaning as the general understanding of the technical personnel in the field to which the disclosure belongs. If there is a contradiction, this description is preferred.

[0021] The present disclosure provides the following technical solutions to obtain the above-mentioned effects: According to a typical embodiment of the present disclosure, a 490-MPa-grade thick steel plate with high core fatigue strength and manufacturing method therefore are provided. The steel plate comprises the following chemical components in mass percentage: C: 0.045 to 0.076%, Si: 0.19 to 0.31%, Mn: 0.95 to 1.13%, P: $\leq 0.008\%$, S: $\leq 0.002\%$, Als: 0.010 to 0.040%, Nb: 0.014 to 0.038%, V: 0.025 to 0.041%, Ti: 0.011 to 0.022%, Ni: 1.35 to 1.55%, Ce: 0.020 to 0.040%, and Fe and inevitable impurities.

[0022] One or two of B: 0.0005-0.0009% and Mo: 0.15-0.25% can be added into the steel plate.

[0023] Each chemical component is controlled in the present disclosure based on the principles as follows:

C and Mn are very effective elements for improving the strength of a steel. On one hand, an increase in carbon content leads to the increase in the tensile strength and yield strength of steel, but the elongation and impact toughness will be decreased; moreover, a phenomenon of hardening may occur in the welding heat affected region of the steel, resulting in the generation of the welding cold cracks. With an increase of Mn content, the strength of the steel significantly increases, while the impact transition temperature remains almost unchanged. Mn is also an element that expands the austenite region. The increase of Mn content can improve stability of austenite, reduce critical cooling rate, strengthen ferrite, significantly improve hardenability, and slow down the rate of microstructure decomposition and transformation during tempering after quenching, thereby improving the stability of the tempered microstructure. On the other hand, C and Mn elements are also elements that are highly prone to segregation at the core of the casted slab and cause central segregation. Therefore, the present disclosure controls C and Mn at low levels (C: 0.045-0.076%, Mn: 0.95-1.13%), adopts other alloy elements to balance the strength and toughness of the steel plate, and reduces the adverse effects of central segregation on the performances of the core.

[0024] Si element can enhance the hardness and strength of the solid solution in steel, not only increasing the

hardenability of the steel, but also enhancing the resistance to tempering of the quenched steel, such that the steel can be tempered at higher temperatures, thereby improving the toughness and resistance to delayed fracture of the steel. Si can significantly improve the elastic limit, yield strength and yield-to-tensile ratio of the steel. Excessive Si content can deteriorate the thermal conductivity of steel, and the surfaces of steel ingots and slabs are prone to cracking or crack defects. The Si content of the steel is designed to be 0.19% to 0.31% in the present disclosure.

[0025] P and S are impurity elements in steel and also elements that are prone to segregation, which can form serious segregation and inclusions in local areas of the steel, reducing plasticity and toughness. The present disclosure strictly controls the content levels of sulfur and phosphorus in steel in terms of metallurgical quality, i.e., $P \leq 0.008\%$ and $S \leq 0.002\%$.

[0026] Al is the main deoxidizing element in steel. In addition, Al has a high melting point. During production, Al in the steel can form AlN with N. However, AlN can hinder the growth of high-temperature austenite and plays a role in grain refinement. The Al content of the steel of the present disclosure is controlled to be 0.010% to 0.040%.

[0027] Nb and Ti are two strong elements that form carbides and nitrides, which have an extremely strong affinity with nitrogen and carbon and can form extremely stable carbides and nitrides with nitrogen and carbon. The dispersed distribution of the second phase particles of carbonitride of Nb distributed along the austenite grain boundaries can greatly increase the coarsening temperature of the original austenite grains. In the recrystallization temperature region of austenite during rolling, carbonitride precipitates of Nb can serve as the nucleation core of the austenite grains; while in the non-recrystallization temperature range, dispersedly distributed carbonitride precipitates of Nb can effectively pin the austenite grain boundaries, prevent further growth of the austenite grains, refine ferrite grains and thereby achieve the goal of improving strength and impact toughness. The nitrides of Ti can effectively pin the austenite grain boundaries, contribute to control the growth of austenite grains, and greatly improve the low-temperature toughness of the welding heat affected region. Therefore, through the fine grain strengthening and precipitation strengthening effects of Nb and Ti microalloying elements, steel plates can achieve excellent strength and toughness. The Nb content of the steel of the present disclosure is designed to be 0.014 to 0.038%, and the Ti content is designed to be 0.011 to 0.022%.

[0028] V is a relatively strong carbide-forming element, which can enhance the strength of the steel by fine grain strengthening, precipitation strengthening and solid solution strengthening. In addition, when the mass percentage of V in the steel is less than 0.1%, the ductile brittle transition temperature of the steel is decreased with an increase of V content. When the mass percentage of V exceeds 0.1%, the ductile brittle transition temperature is increased with an increase of V content. In the steel containing Si and Mn, addition of a small amount of V can significantly alleviate the effects of these two elements on growth of grain and elevation of the ductile brittle transition temperature. The composite addition of V and Nb can enhance the strength of the steel and improve the toughness of the steel. The content of V element of the present disclosure is 0.025 to 0.041%.

[0029] Ni can strengthen the ferrite matrix in the steel, inhibit coarse pre-eutectoid ferrite, significantly enhance the toughness of the steel, reduce the ductile brittle transition temperature of the steel, and enhance the low-temperature impact toughness of the steel. The Ni content is designed to be 1.35 to 1.55% in the present disclosure.

[0030] Ce is a rare earth element that has a strong affinity for oxygen and sulfur, and has effects of purification and significant modification in the steel. Solid solution can be enriched at grain boundaries by the diffusion mechanisms in the steel, reducing the segregation of inclusion elements at grain boundaries, resulting in strengthening of the grain boundaries and improvement in properties related to the grain boundaries, such as low-temperature brittleness and toughness, etc. The added amount of Ce is 0.020 to 0.040% in the present disclosure.

[0031] The main function of Mo element is solid solution strengthening in the steel. A small amount of Mo can form refractory carbides, hinder the growth of austenite grains during heating, refine the microstructure of product, and enhance strength, hardness and wear resistance. Mo can improve hardenability, alleviate or eliminate tempering brittleness caused by other alloying elements, greatly benefit for the toughness of the steel, improve tempering stability, and effectively eliminate or reduce the residual stress in the steel. However, excessive Mo is prone to coarse martensite during the rapid cooling and the welding cooling processes, reducing the low-temperature toughness of the substrate and deteriorating welding performance. Therefore, the present disclosure preferably controls the Mo content to be 0.15 to 0.25%.

[0032] B is an element that strongly enhances hardenability. The addition of B can effectively inhibit the nucleation and growth of pre-eutectoid ferrite. Due to the non-equilibrium segregation of B at the austenite grain boundaries, the γ - α phase transformation is strongly suppressed, which promotes the austenite to form small low-carbon martensite during quenching, thereby improving the yield strength and the tensile strength of the steel. The B content of the present disclosure is preferably 0.0005 to 0.0009%.

[0033] In the steel plate with excellent core fatigue performance according to the present disclosure, the oxide inclusions are mainly $Ce_2O_3+Al_2O_3$, Ce_2O_3 , Al_2O_3 and composite inclusions with $Ce_2O_3+Al_2O_3$, Ce_2O_3 and Al_2O_3 as cores respectively, with $Ce_2O_3+Al_2O_3$ and the composite inclusion with $Ce_2O_3+Al_2O_3$ as the core accounting for 90% or more in number, Ce_2O_3 and the composite inclusion with Ce_2O_3 as the core accounting for 1% to 10% in number, and Al_2O_3 and the composite inclusion with Al_2O_3 as the core accounting for 1% or less in number.

[0034] Preferably, in the steel plate with excellent core fatigue performance according to the present disclosure, a

density of the inclusions is 100 to 500 per mm^2 , wherein inclusions with a size of 0.2 to 2 μm account for 95% or more in number, inclusions with a size of >2 μm to 5 μm account for 5% or less in number, inclusions with a size of >5 μm to 10 μm account for 0.01% or less in number, and there are no inclusions with a size of greater than 10 μm .

[0035] In the above-mentioned technical schemes, the control of inclusion types has the effects of refining grain size and promoting bainite transformation. The micro control of the inclusion size has the effects of refining grain size, promoting bainite transformation and improving the strength, toughness and fatigue strength of the steel plate. Specifically, fine inclusions with a high density can serve as heterogeneous nucleation points for ferrite during the cooling process of the steel plate, promoting the formation of the ferrite, therefore refining grain size and promoting bainite transformation.

[0036] According to another embodiment of the present disclosure, a manufacturing method for a 490-MPa-grade thick steel plate with high core fatigue strength is provided, which preferably comprises the following steps:

- 1) desulfurizing molten steel, wherein the molten steel is controlled to satisfy $S \leq 0.002\%$;
- 2) vacuuming for a time period of ≥ 21 minutes, and continuous casting at a casting speed of 0.5 to 1.0 m/min; wherein a two-stage electromagnetic stirring is used, with current parameters of 420A and 455A respectively; and for dynamic soft reduction, a solid fraction is 0.35 to 0.70, and a reduction amount is 6 to 10 mm;
- 3) conventional continuous casting into a slab and heating the casted slab, wherein a heating temperature is controlled to be 1201 to 1245 $^{\circ}\text{C}$, and a tapping temperature is not lower than 1180 $^{\circ}\text{C}$;
- 4) performing a two-stage rolling, wherein for the first stage, an initial rolling temperature is not lower than 1063 $^{\circ}\text{C}$, a reduction amount per pass is ≥ 20 mm, and a reduction amount for the last two passes is ≥ 40 mm; and wherein an initial rolling temperature for the second stage is not higher than 943 $^{\circ}\text{C}$, a reduction rate for the first two passes is greater than 15%, a reduction rate for the remaining rolling passes is controlled to be 8% to 10%, and a finishing rolling temperature is 821 to 843 $^{\circ}\text{C}$. Rapid cooling is performed after finishing rolling, wherein a cooling rate is 0.5 to 5 $^{\circ}\text{C/s}$, and a self-tempering temperature is controlled to be not higher than 430 $^{\circ}\text{C}$;
- 5) performing tempering heat treatment in an industrial furnace, wherein a tempering temperature is 611 to 631 $^{\circ}\text{C}$, an in-furnace time is: (product thickness in mm $\times$ 1.5) min, and a continuous holding time is not less than (product thickness in mm $\times$ 0.9) min after reaching the temperature. After the tempering is finished, air cooling is performed to room temperature.

[0037] The design points and reasons for the manufacturing method of the present disclosure are as follows:

Defects such as center segregation in the casted slab can be greatly improved by controlling the casting speed of continuous casting, adopting two-stage electromagnetic stirring, and controlling the dynamic light reduction and total reduction within the scope of the present disclosure as described above.

[0038] By controlling the heating temperature and tapping temperature of the casted slab within the scope of the present disclosure as described above, sufficient austenization in the steel of the present disclosure can be further ensured.

[0039] In the present disclosure, a two-stage rolling process is adopted, in which the reduction amount of passes is changed, instead of using a simple rolling process with large reduction amount and few passes, thereby superimposedly refining the recrystallization grains and ferrite nucleation grains of the two stages. Preferably, by setting and controlling the initial rolling temperature of the first stage to not be lower than 1063 $^{\circ}\text{C}$, in combination with the reduction amount per pass, the rolling pressure can be effectively transmitted to the center part of the casted slab, fully refining the austenite grains. By controlling the initial rolling temperature of the second stage to be not more than 943 $^{\circ}\text{C}$, the reduction rate of the first two passes to be greater than 15%, the reduction rate of the remaining rolling passes to be 8% to 10%, and the finishing rolling temperature to be 821 to 843 $^{\circ}\text{C}$, the recrystallized grains and the ferrite nucleated grains can be further superimposedly refined, thereby further improving the toughness of the steel plate, and indirectly providing sufficient time to reduce center segregation and center looseness.

[0040] Cooling is performed after rolling, wherein the cooling rate is controlled and the self-tempering temperature is controlled to be not higher than 430 $^{\circ}\text{C}$, which can further ensure that a microstructure of quasi-polygonal ferrite + lath bainite + pearlite is obtained in the steel plate, and the microstructure with desired proportions is obtained, and thus excellent basic performance and fatigue performance are achieved.

[0041] The tempering temperature is controlled to be 611 to 631 $^{\circ}\text{C}$. A too low tempering heating temperature can cause insufficient precipitation of some elements during tempering of the steel according to the present disclosure, resulting in insufficient strength and poor toughness of the steel plate. The temperature being too high can lead to a decrease in the strength of the steel of the present disclosure. Meanwhile, when the steel plate is sent into the industrial furnace for tempering, it will cause a decrease in furnace temperature. Therefore, it is necessary to control a sufficient continuous holding time to be not less than (product thickness in mm $\times$ 0.9) min after reaching the temperature, so that the elements are precipitated and diffused sufficiently during tempering of the steel plate, and the internal stress is eliminated sufficiently, thereby obtaining excellent comprehensive performances.

[0042] By using the chemical composition and manufacturing method of the present disclosure for smelting, rolling, cooling and tempering process with specific parameters, the steel plates that meet the requirements of the present

disclosure can be manufactured.

[0043] The present disclosure will be further described in detail by combining following examples, comparative examples and experimental data.

5 EXAMPLES

[0044] The steel compositions with different steel components shown in Table 1 were processed according to the process shown in Table 2 to obtain steel plates.

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Table 1: The chemical components and contents in mass percentage in Examples and Comparative Examples of the present disclosure

Element	Example 1	Example 2	Example 3	Example 4	Example 5	Example 6	Example 7	Example 8	Comparative Example 1	Comparative Example 2	Comparative Example 3
C	0.045	0.071	0.067	0.058	0.061	0.076	0.051	0.048	0.059	0.07	0.06
Si	0.23	0.31	0.27	0.28	0.24	0.19	0.25	0.29	0.08	0.14	0.27
Mn	1.13	1.02	1.11	0.98	1.08	0.95	1.10	1.05	1.45	1.40	1.00
P	0.006	0.007	0.005	0.008	0.006	0.007	0.007	0.005	0.007	0.004	0.004
S	0.001	0.002	0.002	0.001	0.002	0.002	0.002	0.001	0.0022	0.001	0.001
Al	0.015	0.010	0.025	0.040	0.037	0.021	0.031	0.028	0.028	0.045	0.040
Nb	0.038	0.017	0.019	0.031	0.014	0.025	0.028	0.022	0.028	0.035	0.035
V	0.041	0.028	0.032	0.025	0.037	0.030	0.040	0.039	0.025	0.025	0.025
Ti	0.022	0.013	0.015	0.020	0.014	0.017	0.019	0.011	0.0013	0.010	0.021
Cu	-	-	-	-	-	-	-	-	-	0.30	-
Cr	-	-	-	-	-	-	-	-	-	0.15	-
Ni	1.35	1.51	1.39	1.45	1.41	1.55	1.47	1.38	0.80	1.45	1.45
Ce	0.020	0.022	0.025	0.027	0.030	0.033	0.037	0.040	-	-	0.01
B	0.0006	-	0.0007	0.0005	-	-	0.0009	0.0007	-	0.0015	0.0005
Mo	0.15	0.25	-	-	0.21	0.15	-	0.18	-	-	-
N	-	-	-	-	-	-	-	-	0.0034	0.0030	-
O	-	-	-	-	-	-	-	-	0.0015	-	-

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[0045] The specific process parameters for Examples 1-8 and Comparative Examples are shown in Table 2:

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Table 2: Main process parameters for each example and comparative example of the present disclosure

Example	1	2	3	4	5	6	7	8	Comparative Example 1	Comparative Example 2	Comparative Example 3
Casting speed [m/min]	1.0	0.95	0.85	0.90	0.95	1.0	0.95	0.9	1.2	1.0	0.95
Electromagnetic stirring		two-stage, with current parameters of 420A and 455A, respectively							used	used	used
Solid fraction for soft reduction	0.35	0.40	0.45	0.50	0.55	0.60	0.65	0.70	/	0.20	/
Reduction amount for soft reduction [mm]	10	10	8	8	7	7	6	6	/	5	/
Heating temperature of casted slab [°C]	1245	1223	1231	1201	1216	1240	1235	1239	1201	1150	1205
Tapping temperature [°C]	1207	1192	1210	1180	1187	1188	1197	1200	1200	1200	1200
Initial rolling temperature for the first stage [°C]	1088	1091	1154	1063	1076	1101	1103	1099	1089	1040	1070
Initial rolling temperature for the second stage [°C]	922	935	943	920	931	937	940	938	938	900	910
Finishing rolling temperature for the second stage [°C]	828	831	843	821	833	837	830	840	821	850	837
Self-tempering temperature [°C]	430	413	420	428	419	401	425	429	429	429	429
Quenching temperature [°C]	-	-	-	-	-	-	-	-	-	830	-
Holding time for quenching [min]	-	-	-	-	-	-	-	-	-	200	-
Tempering temperature [°C]	611	631	621	623	627	629	615	619	650	640	630
In-furnace time for tempering [min]	90	143	122	114	128	150	107	95	50	150	120
Holding time for tempering [min]	64	90	83	78	85	99	71	67	53	90	82

[0046] The comprehensive performance test results of steel plates in Examples 1-8 are shown in the following table.

[0047] Fatigue Strength: According to GB/T3075 "*Metallic Materials -Fatigue Testing -axial force-controlled method*", specimens were taken from the central part, and specimens with the same nominal size were clamped on the axial force fatigue testing machine. The constant amplitude cyclic stress was applied. The stress ratio was 0.1. The applied force was along the longitudinal axis direction of the specimen and passed through the axis of the cross section of the specimen. The test continued until the specimen failed or until the number of cycles exceeded 10^7 . The maximum stress at which the specimen did not experience fracture failure was the fatigue strength of the material.

[0048] Yield Strength and Tensile Strength: At room temperature of 25 °C, according to GB/T228.1-2010 standard "*Metallic Materials - Tensile Testing - Part 1: Method of test at room temperature*", an axial tension was applied to the tensile specimen, and the specimen was stretched at a certain speed. The maximum stress at which the specimen yielded and before the force first decreased was the yield strength, and the stress corresponding to the maximum tensile force before the material broke was the tensile strength.

[0049] The test method for type, proportion and density of inclusions: a scanning electron microscope (SEM) with an energy dispersive spectrometer (EDS) affiliated thereto was used to scan an area of 20 mm² or more of the material after grinding and polishing. The inclusions were identified according to the different contrast between the inclusions and the steel body. The energy dispersive spectrometer was used for compositional analysis of inclusions to determine the sizes and types of inclusions, to count the numbers of inclusions, and to calculate their proportions.

[0050] The determination of each phase in the microstructure: the metallographic sample was grinded with 1000X sandpaper. After polishing, it was corroded with alcohol containing 4% nitric acid. The metallographic photograph was taken by the metallographic microscope. According to the morphological characteristics of quasi-polygonal ferrite (QF), lath bainite (BF) and pearlite (P), the phase regions were calibrated, and the area and phase ratio of the calibrated regions were calculated respectively.

Table 3: Performance testing results of examples of the present disclosure

Example	Specification [mm]	Yield Strength [MPa]	Tensile Strength [MPa]	Fatigue Strength [MPa]
1	60	533	650	387
2	95	507	608	361
3	81	523	617	367
4	76	521	615	360
5	85	519	623	374
6	100	495	610	356
7	71	517	620	369
8	63	528	644	376
Comparative Example1	70	408	553	241
Comparative Example2	100	455	560	208
Comparative Example3	80	500	605	210

Table 4: The detection results of inclusions in examples of the present disclosure

Example	Specification [mm]	Proportion of oxide inclusions (%)			Density of oxide inclusions (counts/mm ²)	Proportion of oxide inclusions with different sizes (%)		
		Ce ₂ O ₃ +Al ₂ O ₃ type	Ce ₂ O ₃ type	Al ₂ O ₃ type		0.2-2μm	>2-5μm	>5-10μm
1	60	95	4.9	0.1	200	98.2	1.795	0.005
2	95	92	7.7	0.3	300	95.1	4.894	0.006
3	81	90	9.2	0.8	145	99.2	0.793	0.007
4	76	98	1.4	0.6	232	96.2	3.792	0.008
5	85	93	6.6	0.4	495	97	2.994	0.006
6	100	96	3.1	0.9	325	96.2	3.791	0.009

(continued)

Example	Specification [mm]	Proportion of oxide inclusions (%)			Density of oxide inclusions (counts/mm ²)	Proportion of oxide inclusions with different sizes (%)		
		Ce ₂ O ₃ +Al ₂ O ₃ type	Ce ₂ O ₃ type	Al ₂ O ₃ type		0.2-2μm	>2-5μm	>5-10μm
7	71	98	1.4	0.6	225	95.7	4.296	0.004
8	63	97	2.5	0.5	335	99.5	0.497	0.003
Comparative Example 1	70	0	0	100	40	40	45	40
Comparative Example 2	100	0	0	100	45	45	50	30
Comparative Example 3	80	0	0	100	60	60	55	35

Table 5: Detection results of each phase of the present disclosure

Example	Specification [mm]	Phase proportion (%)		
		Quasi-polygonal ferrite	Lath bainite	Pearlite
1	60	30.2	67.3	2.5
2	95	33.3	66.3	0.4
3	81	42.6	56.6	0.8
4	76	45.3	53.2	1.5
5	85	52.9	44.2	2.9
6	100	55.6	44.3	0.1
7	71	56.8	41.2	2
8	63	58.6	40.1	1.3
Comparative Example 1	70	23	75	2
Comparative Example 2	100	17	80	3
Comparative Example 3	80	11	85	4

[0051] From the above tables, it can be seen that the inclusions and microcrystalline structures within the scope of the present disclosure were obtained in Examples of the present disclosure. The fatigue strengths of Examples of the present disclosure were 340 MPa or more, which were significantly higher than that of Comparative Examples, exhibiting a good core fatigue performance.

[0052] The steel plate of the present disclosure is a thick steel plate with a thickness of 60 to 100 mm which has a yield strength of ≥ 490 MPa, a tensile strength of ≥ 600 MPa and a fatigue strength of ≥ 340 MPa at the center in plate thickness. It can be used as supporting parts and components which have certain requirements for the core fatigue performance of steel plates in the buildings, engineering machinery, ocean engineering, etc. The present disclosure has the advantages of simple manufacturing process and can be implemented in various metallurgical enterprises.

[0053] In addition, the combination manners of various technical features are not limited to the combination manners recorded in specific embodiments and examples in this disclosure. All technical features recorded in this disclosure can be freely combined or integrated in any way, unless there is a contradiction between them. All variations directly derived or associated by the technicians in this field from the disclosure of the present application should fall within the protection scope of the present disclosure.

Claims

1. A 490-MPa-grade thick steel plate with high core fatigue strength, comprising the following chemical components in mass percentage:

C: 0.045 to 0.076%, Si: 0.19 to 0.31%, Mn: 0.95 to 1.13%, P: $\leq 0.008\%$, S: $\leq 0.002\%$, Als: 0.010 to 0.040%, Nb: 0.014 to 0.038%, V: 0.025 to 0.041%, Ti: 0.011 to 0.022%, Ni: 1.35 to 1.55%, Ce: 0.020 to 0.040%, and Fe and inevitable impurities.

2. The steel plate according to claim 1, **characterized in that**, the steel plate also comprises one or two of B: 0.0005 to 0.0009% and Mo: 0.15 to 0.25%.
3. A 490-MPa-grade thick steel plate with high core fatigue strength, comprising the following chemical components in mass percentage:
C: 0.045 to 0.076%, Si: 0.19 to 0.31%, Mn: 0.95 to 1.13%, P: $\leq 0.008\%$, S: $\leq 0.002\%$, Als: 0.010 to 0.040%, Nb: 0.014 to 0.038%, V: 0.025 to 0.041%, Ti: 0.011 to 0.022%, Ni: 1.35 to 1.55%, Ce: 0.020 to 0.040%, and optionally one or two of B: 0.0005 to 0.0009% and Mo: 0.15 to 0.25%, the balance being Fe and inevitable impurities.
4. The thick steel plate according to any one of claims 1 to 3, **characterized in that**, the V content in the steel plate satisfies: V: 0.032 to 0.041%.
5. The thick steel plate according to any one of claims 1 to 3, **characterized in that**, the steel plate has a microstructure composed of 30% to 60% of quasi-polygonal ferrite, 40% to 70% of lath bainite and 0.1% to 3% of pearlite, in percentage by area.
6. The thick steel plate according to claim 5, **characterized in that** the microstructure of the steel plate has an average grain size of 8 to 12 μm .
7. The thick steel plate according to any one of claims 1 to 3, **characterized in that**, the steel plate comprises oxide inclusions, wherein the oxide inclusions are mainly $\text{Ce}_2\text{O}_3 + \text{Al}_2\text{O}_3$, Ce_2O_3 , Al_2O_3 and composite inclusions with $\text{Ce}_2\text{O}_3 + \text{Al}_2\text{O}_3$, Ce_2O_3 and Al_2O_3 as cores respectively, with $\text{Ce}_2\text{O}_3 + \text{Al}_2\text{O}_3$ and the composite inclusion with $\text{Ce}_2\text{O}_3 + \text{Al}_2\text{O}_3$ as the core accounting for 90% or more in number, Ce_2O_3 and the composite inclusion with Ce_2O_3 as the core accounting for 1% to 10% in number, and Al_2O_3 and the composite inclusion with Al_2O_3 as the core accounting for 1% or less in number.
8. The thick steel plate according to claim 7, **characterized in that**, the inclusions have a density of 100 to 500 per mm^2 , wherein inclusions with a size of 0.2 to 2 μm account for 95% or more in number, inclusions with a size of greater than 2 μm and not more than 5 μm account for 5% or less in number, inclusions with a size of greater than 5 μm and not more than 10 μm account for 0.01% or less in number, and there are no inclusions with a size of greater than 10 μm .
9. The thick steel plate according to any one of claims 1 to 8, **characterized in that**, the steel plate has a yield strength of ≥ 490 MPa and a tensile strength of ≥ 600 MPa.
10. The thick steel plate according to any one of claims 1 to 8, **characterized in that**, the steel plate has a thickness of 60 to 100 mm, and a fatigue strength of ≥ 340 MPa at the center in plate thickness.
11. A manufacturing method for a 490-MPa-grade thick steel plate with high core fatigue strength, comprising the following steps:
 - 1) smelting and desulfurizing molten steel according to the chemical composition in any one of claims 1 to 4, wherein the molten steel is controlled to satisfy $S \leq 0.002\%$;
 - 2) vacuuming for a time period of ≥ 21 minutes, and continuous casting at a casting speed of 0.5 to 1.0 m/min; wherein a two-stage electromagnetic stirring is used; and for dynamic soft reduction, a solid fraction is 0.35 to 0.70, and a reduction amount is 6 to 10 mm;
 - 3) continuous casting into a slab and heating the casted slab;
 - 4) performing a two-stage rolling;
 - 5) performing tempering heat treatment, wherein a tempering temperature is 611 to 631 $^{\circ}\text{C}$, an in-furnace time is: (product thickness in mm $\times 1.5$) min, and a continuous holding time is not less than (product thickness in mm $\times 0.9$) min after reaching the temperature; and after the tempering is finished, air cooling is performed to room temperature.
12. The manufacturing method according to claim 11, **characterized in that**, in the step 3), a heating temperature is controlled to be 1201 to 1245 $^{\circ}\text{C}$, and a tapping temperature is not lower than 1180 $^{\circ}\text{C}$.

13. The manufacturing method according to claim 11 or 12, **characterized in that**, in the step 4), an initial rolling temperature for the first stage is not lower than 1063 °C; an initial rolling temperature for the second stage is not higher than 943 °C, and a finishing rolling temperature is 821 to 843 °C; and after finishing rolling, cooling is performed at a rate of 0.5 to 5 °C/s, and a self-tempering temperature is controlled to be not higher than 430 °C.

14. The manufacturing method according to claim 13, **characterized in that**, in the step 4), in the first stage of rolling, a reduction amount per pass is ≥ 20 mm, and a reduction amount for the last two passes is ≥ 40 mm; and in the second stage of rolling, a reduction rate for the first two passes is greater than 15%, and a reduction rate for the remaining rolling passes is controlled to be 8% to 10%.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2023/103582

A. CLASSIFICATION OF SUBJECT MATTERC22C 38/02(2006.01)i; C22C 38/04(2006.01)i; C22C 38/06(2006.01)i; C22C 38/08(2006.01)i; C22C 38/12(2006.01)i;
C22C 38/14(2006.01)i; C22C 38/26(2006.01)i; C22C 38/24(2006.01)i; C22C 33/04(2006.01)i; B22D 11/00(2006.01)i

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

IPC: C22C,B22D

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

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

CNTXT, ENTXT, DWPI, SIPOABS, CNKI: 钢, 碳, C, 硅, Si, 锰, Mn, 磷, P, 硫, S, 铝, Al, 铌, Nb, 钒, V, 钛, Ti, 镍, Ni, 铈, Ce, 铁, Fe, steel, carbon, silicon, manganese, phosphorus, sulfur, aluminum, niobium, vanadium, titanium, nickel, cerium, iron, ferrum

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
PX	CN 114875331 A (BAOWU GROUP ECHENG IRON AND STEEL CO., LTD.) 09 August 2022 (2022-08-09) description, paragraphs 10-39	1-14
PX	CN 115572905 A (YANSHAN UNIVERSITY) 06 January 2023 (2023-01-06) description, paragraphs [0008]-[0035]	1-14
X	CN 110241357 A (JIANGYIN XINGCHENG SPECIAL STEEL WORKS CO., LTD.) 17 September 2019 (2019-09-17) description, paragraphs 17-72, 107, and 108	1-14
X	CN 1113391 A (NIPPON STEEL CORP.) 13 December 1995 (1995-12-13) description, p. 8, paragraph 4-p. 13, the last paragraph	1-14
X	JP 2016204690 A (NIPPON STEEL & SUMITOMO METAL CORP.) 08 December 2016 (2016-12-08) description, paragraphs 31-59	1-14

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

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PCT/CN2023/103582

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	JP 2003342672 A (SUMITOMO METAL INDUSTRIES, LTD.) 03 December 2003 (2003-12-03) entire document	1-14

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/CN2023/103582

Patent document cited in search report	Publication date (day/month/year)	Patent family member(s)	Publication date (day/month/year)
CN 114875331 A	09 August 2022	CN 114875331 B	25 November 2022
CN 115572905 A	06 January 2023	CN 115572905 B	04 August 2023
CN 110241357 A	17 September 2019	CN 110241357 B	26 March 2021
CN 1113391 A	13 December 1995	EP 0666332 A1	09 August 1995
		EP 0666332 A4	13 December 1995
		JP 3526576 B2	17 May 2004
		WO 9504838 A1	16 February 1995
		KR 950703661 A	20 September 1995
		KR 0157540 B1	16 November 1998
		NO 951288 A	06 June 1995
		WO 1995004838 A1	07 September 1995
		US 5634988 A	03 June 1997
		CN 1040555 C	04 November 1998
JP 2016204690 A	08 December 2016	JP 6528522 B2	12 June 2019
JP 2003342672 A	03 December 2003	JP 3741078 B2	01 February 2006

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

- CN 107641760 B [0008]
- CN 109161795 A [0008]
- CN 113174535 A [0008]
- CN 112981235 A [0008]