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(54) HIGH-PLASTICITY STEEL AND MANUFACTURING METHOD THEREFOR

(57) Provided in the present invention are a high-plasticity steel and a manufacturing method therefor. The steel comprises the following components in percentage by mass: C: 0.10-0.35%, Si: 0.8-2.0%, Mn: 1.0-3.0%, P: $\leq 0.02\%$, S $\leq 0.005\%$, Al: 0.1-2.0%, N: $\leq 0.005\%$, with the balance being Fe and other inevitable impurities. The steel of the present invention can achieve good matching among a low yield strength, a low yield ratio, a high tensile strength and an ultrahigh elongation rate, and can be widely applied to components with complex shape requirements, or other parts that require thinning while maintaining high strength, such as those in commercial or passenger vehicles.

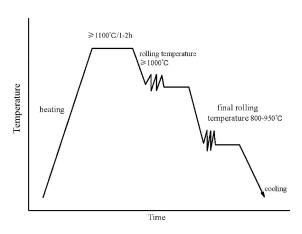


Figure 1

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Description

TECHNICAL FIELD

[0001] The present invention belongs to the field of steel and manufacturing method therefor, and particularly relates to a high-plasticity steel and a manufacturing method therefor.

BACKGROUND

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[0002] At present, as one of the important pillar industries of the national economy, automotive industry has an increasingly urgent demand for advanced high-strength steel, and a rapidly growing demand for low-carbon or even zero-carbon emission products. Thinning while maintaining high strength is not only a development trend in the passenger car industry, but also progress in high-strength thinning, as well as energy conservation and emission reduction, is also gradually accelerating in the commercial vehicle field. This is not only a need for industry development, but also an inevitable requirement for the transformation and upgrading of the automotive industry. Especially, in the commercial vehicle field, traditional large-tonnage vehicles can no longer meet the increasingly stringent legislation requirements. Therefore, designers and manufacturers in the automotive industry are reexamining traditional design and production concepts, reducing weight while maintaining high strength in various aspects such as chassis, body, and seats, and even using other new materials such as aluminum alloy and carbon fiber. Moreover, the commercial vehicle industry is accelerating the process of lightweighting in terms of chassis, compartments, and upper loads. With the continuous strengthening of the enforcement of commercial vehicle policies and regulations, there is enormous potential for lightweighting in the future commercial vehicle field.

[0003] Many parts of passenger cars and commercial vehicles, such as body, collision beams, fuel tank brackets, battery brackets, gas cylinder brackets, and bent pipes, are generally produced with low-strength thick-gauge ordinary steel such as Q235 or Q345, and the process is relatively complex, some require bolt connections and some require welding. With the development of lightweighting, many users hope to use cold stamping for the integrated forming of these parts in commercial vehicles, which reduces procedure and achieves lightweighting. Consequently, this places higher demands on the performance of hot-rolled high-strength steel, requiring it to have higher elongation and better formability while ensuring high strength. When using traditional high-strength steel for stamping parts such as fuel tank brackets, cracks are likely to occur at the large arc of the parts, making smooth stamping impossible. Therefore, it is desirable to develop new high-strength steels having high tensile strength and excellent formability. Additionally, considering the lifespan of the user's molds, the yield strength of the new high-strength steel must not be too high; otherwise, parts will be rebounded severely and be difficult to form during actual stamping.

[0004] For the aforementioned reasons, it is desirable to develop hot-rolled or pickled high-strength steel having low yield strength, high tensile strength, and ultra-high elongation rate. This type of steel should be suitable for stamping complex parts with particularly high requirements for cold-drawing and forming, and has good manufacturability and broad application prospects.

[0005] Many applications disclose steel with ultra-high plasticity, mostly focusing on the field of cold-rolled steel with high-strength, with some relate to hot-rolled steel with ultra-high plasticity.

[0006] For example, Chinese Patent Application CN104233092A discloses a 780 MPa grade steel with ultra-high plasticity, whose composition is designed to have a low carbon content and a high silicon content, and a certain amount of precious alloy elements such as Cr, Mo and Nb, resulting in relatively high alloy costs.

[0007] Chinese Patent Application CN107815593A discloses a steel with low silicon content and high aluminum content and with ultra-high plasticity, whose composition has low silicon content and high aluminum content, and a certain amount of precious element Cu. The production process mainly includes heat treatment in the two-phase zone for 1-3 minutes and then phase transformation in the bainite zone, to obtain 780 MPa grade heat-treated steel with ultra-high plasticity. However, the heat treatment process cannot be applied to existing hot rolling production lines.

SUMMARY OF THE INVENTION

[0008] The objective of the present invention is to provide a high plasticity steel and a manufacturing method therefor. The high plasticity steel has good mechanical properties and can achieve good matching among a low yield strength, a low yield ratio, a high tensile strength and an ultra-high elongation rate. The steel can be widely applied to components with complex shape requirements, or other parts that require thinning while maintaining high strength, such as those in commercial or passenger vehicles.

[0009] In order to achieve the above-mentioned objective, in the first aspect, the present invention provides a steel comprising the following components in percentage by mass: C: 0.10-0.35%, Si: 0.8-2.0%, Mn: 1.0-3.0%, P: \leq 0.005%, Al: 0.1-2.0%, N: \leq 0.005%, with the balance being Fe and other inevitable impurities.

[0010] Preferably, the above-mentioned steel also comprises Ti, and, in percentage by mass, the content of Ti is less than or equal to 0.2%, preferably 0.05-0.2%, more preferably 0.05-0.1%.

[0011] Preferably, the above-mentioned steel also comprises one or more selected from the group consisting of Mo, Nb, V, Cu, Ni, Cr and B; wherein, in percentage by mass, the content of Mo is less than or equal to 0.5%, preferably less than or equal to 0.3%; the content of Nb is less than or equal to 0.1%, preferably less than or equal to 0.06%; the content of V is less than or equal to 0.1%, preferably less than or equal to 0.5%, preferably less than or equal to 0.3%; the content of Ni is less than or equal to 0.5%, preferably less than or equal to 0.3%; the content of Cr is less than or equal to 0.5%, preferably less than or equal to 0.001%, preferably less than or equal to 0.001%, preferably less than or equal to 0.0005%.

10 **[0012]** Preferably, the inevitable impurities of the above-mentioned steel include, in percentage by mass, $O \le 0.003\%$, preferably $O \le 0.002\%$; $S \le 0.003\%$; and/or $N \le 0.004\%$.

[0013] Preferably, the composition in percentage by mass of the above-mentioned steel satisfies one or more of the following: C: 0.15~0.25%, Si: 1.0~1.6%, Mn: 1.5~2.5%, Al: 0.3~1.0%.

[0014] The design concept of each element in the steel of the present invention is as follows.

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[0015] Carbon is a basic element in steel, and is also one of the important elements in the present invention. Carbon expands the austenite phase region and stabilizes austenite. Carbon, as a gap atom in steel, plays a very important role in improving the strength of steel, and has the greatest impact on the yield strength and tensile strength of steel. Additionally, as an effective element for stabilizing residual austenite, carbon usually has a relatively high concentration in steel. In the present invention, in order to obtain high-strength steel having different levels of tensile strength and having a relatively stable residual austenite in the steel microstructure, the content of carbon must be greater than or equal 0.10%. However, the content of carbon cannot exceed 0.35%. Excessive content of carbon can easily lead to higher strength, reduction of elongation rate, and deterioration of welding performance. Therefore, the content of carbon is between 0.10-0.35%.

[0016] Silicon is a basic element in steel, and is also one of the important elements in the present invention. The addition of silicon to steel can lower the non-recrystallization temperature of austenite, expanding the rolling process window of austenite. This allows dynamic recrystallization of the steel to be completed during the finish rolling stage, which is beneficial for improving the differences in transverse and longitudinal properties of the steel. Another function of adding silicon into steel is the inhibition of the formation of the cementite. In the present invention, to ensure that the steel microstructure contains a large amount of residual austenite, it is necessary to add a relatively high amount of silicon to inhibit the formation of the cementite. This inhibitory effect of silicon on carbide formation is evident when the silicon content reaches 0.8% or more. However, the content of silicon should not be too high; otherwise, the rolling force load during actual rolling process will be too large, and there will be a significant amount of red scale on the surface of steel plate, which is not conducive to stable production during rolling. Therefore, the silicon content in the steel should be 0.8-2.0%, preferably 1.0-1.6%.

[0017] Manganese is the most fundamental elements in steel, and is also one of the most important elements in the present invention. Mn expands the austenite phase region, reduces the critical quenching rate of the steel, stabilizes austenite, refines grains, and delays the transition of austenite to pearlite. Additionally, during heat treatment process, Mn undergoes partition and diffuses from bainite into residual austenite, further stabilizing the residual austenite and increasing its content. A manganese content of at least 1.0% is required to achieve these effects. However, the content of manganese should not be too high. If the content of manganese exceeds 3.0%, it may lead to segregation in the continuous casting slab and the formation of a large amount of MnS inclusions. Therefore, the manganese content in the steel should be 1.0-3.0%, preferably 1.5-2.5%.

[0018] Phosphorus is an impurity element in steel. P tends to segregate easily at grain boundaries. When the content of P in steel is relatively high (\geq 0.1%), Fe₂P is formed and precipitated around the grain, reducing the plasticity and toughness of the steel. Thus, the lower the content of P, the better. It is generally preferable to control the content of P to 0.02% or less, as this level does not increase the cost of steel-making.

[0019] Sulfur is an impurity element in steel. In steel, S typically combines with Mn to form MnS inclusions, especially when the contents of S and Mn are both relatively high, a significant amount of MnS will be formed in the steel. MnS itself has a certain degree of plasticity. MnS deforms along the rolling direction during subsequent rolling process, which not only reduces the transverse plasticity of the steel, but also increases structure anisotropy, adversely affecting hole expansion performance. Therefore, the lower the content of S in steel, the better. To minimize the content of MnS, the content of S must be strictly controlled. The S content is required to be controlled to 0.005% or less, preferably 0.003% or less.

[0020] Aluminum is one of the important elements in the present invention. In addition to its basic roles of deoxidation and nitrogen fixation, aluminum also has two other important functions in the present invention. In the present invention, the content of austenite-stabilizing elements such as carbon and manganese is relatively high, so that austenite has strong stability. As a result, it is difficult to form the required amount of ferrite during the short air-cooling stage in staged cooling process after rolling. Therefore, more aluminum than is used in conventional high-strength steel needs to be added to accelerate the transformation of ferrite and ensure sufficient amount of ferrite. On the other hand, in order to obtain highly stable residual austenite, additional aluminum is also necessary. Aluminum is added into steel for accelerating the

transformation of ferrite. Besides, during the bainite transformation process, Al can not only play a role in inhibiting the formation of cementite, but also promote the diffusion of carbon atoms from bainite ferrite to residual austenite, thereby accelerating the diffusion of carbon atoms in residual austenite, increasing the carbon concentration in residual austenite, and obtaining highly stable residual austenite. When the content of aluminum is $\geq 0.1\%$, the above-mentioned various beneficial effects can be achieved. However, when the content of aluminum exceeds 2.0%, its effect of promoting diffusion and enrichment of carbon becomes saturated, and the viscosity of the molten steel increases, which can easily clog the casting nozzle. Therefore, the aluminum content in the steel of the present invention should be 0.1-2.0%, preferably 0.3%-1.0%.

[0021] Nitrogen is an impurity element in the present invention. The lower the N content, the better. However, nitrogen is an inevitable element in the steelmaking process. Although present in small amounts, nitrogen can combines with strong carbide-forming elements such as Ti to form TiN particles, which are detrimental to the performances of steel. Therefore, the content of nitrogen in the present invention is controlled to be 0.005% or less, preferably 0.004% or less.

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[0022] Titanium is one of the optional additive elements in the present invention. Steel with ultra-high plasticity and high-strength comprises a large amount of residual austenite, which is a soft phase with relatively low yield strength. Therefore, to improve the yield strength of the steel, microalloying elements such as titanium can be added under certain conditions. Titanium enhances yield strength through its precipitation strengthening effect in the proeutectoid ferrite. As the titanium content increases, the precipitation strengthening effect is gradually enhanced. When the titanium content reaches 0.20%, the precipitation strengthening effect of titanium becomes saturated. Therefore, the amount of titanium added can be adjusted as needed. The titanium content in the steel of the present invention is controlled to be 0.20% or less, preferably 0.05-0.2%, more preferably 0.05-0.1%.

[0023] Molybdenum is one of the optional additive elements in the present invention. The addition of molybdenum to steel can greatly delay the phase transition of ferrite and pearlite, which is conducive to obtaining bainite structure. In addition, molybdenum has a strong resistance to welding softening. Since the primary objective of the present invention is to obtain a microstructure mainly consisting offerrite, bainite, and residual austenite, and since ferrite and bainite are prone to softening after welding, the addition of an appropriate amount of molybdenum can effectively reduce the degree of welding softening. Considering that molybdenum is a precious metal, adding too much would increase the cost of the alloy. Therefore, the content of molybdenum in the present invention is 0.5% or less, preferably 0.3% or less.

[0024] Oxygen is an inevitable element in the steelmaking process. For the present invention, after deoxidation, the content of O in steel can generally be reduced to 0.003% or less, which does not cause any significant adverse effects on the performance of the steel plate. Therefore, in the present invention, the O content in the steel is controlled to be 0.003% or less, preferably 0.002% or less.

[0025] Copper is one of the optional additive elements in the present invention. The addition of copper to steel can improve the corrosion resistance of the steel, and when combined with phosphorus, the corrosion resistance effect is even better. When the addition amount of Cu exceeds 1%, an ϵ -Cu precipitation phase may be formed under certain conditions, which has a relatively strong precipitation strengthening effect. However, the addition of Cu tends to result in "Cubrittleness" phenomenon during the rolling process. To fully utilize the corrosion resistance benefits of Cu in specific applications while avoiding significant "Cu-brittleness" phenomenon, the content of Cu in the present invention is controlled to be 0.5% or less, preferably 0.3% or less.

[0026] Nickel is one of the optional additive elements in the present invention. The addition of nickel to steel provides a certain level of corrosion resistance, though its corrosion resistance effect is weaker than copper. The addition of nickel in steel has little effect on the tensile properties of the steel, but can refine the structure and precipitation phases of steel and greatly improve the low-temperature toughness of steel. Additionally, in steel added with copper, the addition of a small amount of nickel can inhibit the occurrence of "Cu-brittleness". The addition of relatively high amount of nickel does not have any significant adverse effect on the properties of the steel itself. When both copper and nickel are added, they not only improve corrosion resistance, but also refine the structure and precipitate phases of the steel, greatly improving the low-temperature toughness. However, both copper and nickel are relatively valuable alloying elements. Therefore, to minimize the cost of alloy design, the amount of nickel added in the present invention is 0.5% or less, preferably 0.3% or less.

[0027] Chromium is one of the optional additive elements in the present invention. The addition of chromium to steel can improves its strength mainly through mechanisms such as solid solution strengthening or microstructure refinement. Chromium easily dissolves into ferrite, and plays a role in strengthening ferrite. Additionally, the addition of a small amount of chromium element can also improve corrosion resistance. Therefore, the amount of chromium added in the present invention is 0.5% or less, preferably 0.3% or less.

[0028] Niobium is one of the optional additive elements in the present invention. Similar to titanium, niobium is a strong carbide-forming element in steel. The addition of niobium to steel can greatly increase the non-recrystallization temperature of steel, allowing the formation of deformation austenite with higher dislocation density during the finish rolling stage, and refine the final phase transition structure during the subsequent transformation process. However, the amount of niobium added should not be too much. On one hand, when the amount of niobium added exceeds 0.01%, it is

prone to form a relatively coarse niobium-carbonitride in the structure, which is not conducive to the low-temperature impact toughness of the steel. On the other hand, a large amount of niobium is easy to cause anisotropy in hot-rolled austenite structure. Therefore, the niobium content in the steel of the present invention is 0.10% or less, preferably 0.06% or less.

[0029] Vanadium is one of the optional additive elements in the present invention. Similar to Ti and Nb, vanadium is also a strong carbide-forming element. However, vanadium carbides have a low solubility or precipitation temperature and are typically fully solid dissolved in austenite during the finish rolling stage. Carbides of vanadium begins to form in ferrite only when the temperature decreases and phase transition starts. Since vanadium carbides have a high solid solubility in ferrite compared to niobium and titanium carbides, the size of vanadium carbides formed in ferrite is larger and vanadium carbides are prone to form at grain boundaries, which is not conducive to the toughness of steel. Therefore, the amount of vanadium added in the steel of the present invention is 0.10% or less, preferably 0.06% or less.

[0030] Boron is one of the optional additive elements in the present invention. Boron is an element that is prone to segregation. During rolling in the austenite region, B element can segregate to the austenite grain boundaries, reducing the interfacial energy at the austenite grain boundaries and inhibiting the formation of ferrite during subsequent cooling and phase transformation. Since the desired microstructure of the present invention includes ferrite, bainite and stable residual austenite, it is necessary to strictly control the content of boron element in the steel to prevent the inhibition of ferrite formation due to excessive addition of boron. Therefore, the amount of boron added in the steel of the present invention is 0.001% or less, preferably 0.0005% or less.

[0031] Unless otherwise specified, the content of elements in the steel of the present invention refer to mass fractions. [0032] Preferably, the steel of the present invention has a microstructure includes ferrite, bainite, and residual austenite, wherein the residual austenite content is 5% or more. Specifically, the volume fraction of ferrite in steel is between 30-50%, preferably between 35-45%; the volume fraction of bainite is between 40-60%, preferably between 45-55%; and the volume fraction of residual austenite is between 5-15%, preferably between 10-15%.

[0033] Preferably, the above-mentioned steel has a yield strength of 500 MPa or more, preferably 600 MPa or more, more preferably 700 MPa or more; a tensile strength of 780 MPa or more, preferably 980MPa or more; an elongation rate of 25% or more, preferably 30% or more.

[0034] Preferably, the above-mentioned steel has a hole expansion of 30% or more, preferably 50% or more.

[0035] Preferably, in the steel, when the Ti content is 0.05-0.2% and the C content is 0.10-0.25%, the yield strength of the steel is \geq 600MPa, the tensile strength is \geq 780MPa, the elongation rate is \geq 30%, and the hole expansion rate is \geq 50%; When the content of Ti is 0.05-0.2% and the content of C is 0.25-0.35%, the yield strength of the steel is \geq 700MPa, the tensile strength is \geq 980MPa, the elongation rate is \geq 25%, and the hole expansion rate is \geq 30%.

[0036] The existing 780 MPa grade steel with ultra-high plasticity comprises C-Si-Mn as the main elements, with microalloying elements such as Nb and Ti added when necessary to refine the grains. In these steels, the content of Al is 0.1% or less, mainly to utilize the deoxidation and nitrogen fixation function of Al element.

[0037] In contrast, the present invention adopts a composition design of high content AI, with the AI content of 0.1% or more. The main purposes of adding high content of AI are to promote transformation of ferrite and to further improve the stability of residual austenite.

[0038] In terms of performances, the existing 780 MPa grade steels with ultra-high plasticity have low yield strength or low yield ratio, and the residual austenite is not sufficiently stable. In case of deformation, the residual austenite in the microstructure easily transforms into martensite.

[0039] In contrast, the ultra-high plasticity steel of the present invention can have tensile properties with varying yield strengths and yield ratios. Moreover, the residual austenite in the structure is more stable, and the content of residual austenite is 5% or more. Despite the varying yield strengths, the tensile strength and elongation rate remain at a high level, making the steel more favorable for downstream processing and use. Moreover, the steel of the present invention can also have a relatively high hole expansion rate, making it particularly suitable for stamping processes involving parts with higher requirements for drawing and flanging forming.

[0040] The method for manufacturing the aforementioned steel comprises the following steps:

- 1) smelting and casting
- smelting the components according to the above composition in a converter or an electric furnace, then secondary refining in a vacuum furnace, and then casting it into a casting blank or a casting ingot;
- 2) reheating the casting blank or the casting ingot at a heating temperature of 1100 °C or higher and holding for 1-2 hours;
- 3) hot rolling and cooling the casting blank or the casting ingot

wherein the casting blank or the casting ingot is hot rolled at an initial rolling temperature of 1000 °C or higher, then subjected to 5-7 passes of rolling with a relatively large deformation rate of 50% or more at 1000 °C or higher, then subjected to 3-7 passes of final rolling with a cumulative deformation of 70% or more after an intermediate blank

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reaches \geq 950 °C, to obtain a steel strip; wherein the final rolling temperature is 800~950 °C; wherein the cooling is staged cooling, after the final rolling, the steel strip is water-cooled to a temperature between 600~750 °C at a cooling rate of 30 °C/s or more; after air cooling for 1~10 seconds, the steel strip is then cooled to a temperature between 350~550 °C at a cooling rate of 10 °C/s or more and coiled, and then cooled to room temperature

[0041] Furthermore, the above-mentioned method further includes step 4) pickling, wherein the hot-rolled strip steel is pickled at a running speed of 30~120 m/min, with a pickling temperature of 75~85 °C and a straightening rate of 2% or less, and then rinsed at a temperature in the range of 35~50 °C, and the surface of the hot-rolled strip steel is dried at a temperature of 120~140 °C, and oiled, to obtain a pickled steel with high-strength and ultra-high plasticity.

[0042] In the method for manufacturing the steel of the present invention:

at a cooling rate of 50 °C/h or less, to obtain a hot-rolled strip steel.

The primary purpose of setting the initial rolling temperature of hot rolling at 1000 °C or higher, and performing 5-7 passes of rolling with a relatively large deformation rate of 50% or more at 1000 °C or higher, is to refine the austenite grains.

[0043] After final rolling in the temperature range of 800-950 °C, a staged cooling process is used to control the content of ferrite, bainite and residual austenite in the steel. The water-cooling stop temperature and air-cooling duration in the first stage cooling after rolling determine the content of ferrite, while the coiling temperature after the second cooling stage determines the contents of bainite and residual austenite.

[0044] Through the combination of the staged cooling process and the innovative composition design, the content of ferrite, bainite and residual austenite can be quantitatively controlled. This combination of the innovative composition design and process can obtain ultra-high plasticity steel with exceptionally stable residual austenite.

[0045] The innovation of the present invention are as follows.

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[0046] The present invention obtains hot-rolled or pickled ultra-high plasticity high-strength steel with low yield strength by a composition design of medium-to-low carbon, high silicon and high aluminum, combined with an innovative staged cooling process during hot rolling, a medium-temperature coiling process, and pickling process. The relatively high content of carbon is beneficial for obtaining high strength, and provides a large amount of available carbon atoms that can diffuse into the residual austenite, resulting in highly stable residual austenite. The main purpose of adding a relatively high silicon content is to inhibit the formation of carbides and extend the temperature range for ferrite formation. The addition of relatively high content of aluminum promotes the diffusion of carbon atoms from bainite ferrite to residual austenite, further improving the stability of the residual austenite.

[0047] Preferably, the present invention adds high content Ti into the steel. By combining this with a staged cooling process, nano-sized TiC precipitates are formed within the ferrite grains during the ferrite transformation process, thereby improving the strength of ferrite, reducing the performance differences between ferrite and bainite, and increasing the hole expansion rate of steel.

[0048] In addition, the relatively high manganese content in the steel of the present invention further improves the stability of the residual austenite.

[0049] The microstructure of the steel of the present invention with a high hole expansion rate and ultra-high plasticity consists of ferrite, bainite and residual austenite. Bainite provides the steel with high tensile strength. Ferrite and the relatively high content of metastable residual austenite provide the steel plate with ultra-high elongation rate through the TRIP effect, with the content of residual austenite being $\geq 5\%$. The ferrite containing nano-sized precipitates has improved yield strength through precipitation strengthening, which reduces the hardness difference between ferrite and bainite, and greatly increases its hole expansion rate while achieving ultra-high plasticity. Through the precise combination of the above-mentioned components and processes, an excellent balance of high tensile strength, ultra-high elongation rate and high hole expansion rate has been achieved.

[0050] The main purpose of coiling at 350-550 °C after hot rolling is to obtain bainite and highly stable residual austenite. The microstructure of the ultra-high plasticity steel of the present invention primarily consists of ferrite, bainite and stable residual austenite, with a content of residual austenite content of \geq 5%. Ferrite endows the steel plate with low yield strength, bainite endows the steel plate with high tensile strength, and stable residual austenite endows the steel plate with ultra-high elongation.

[0051] Based on this innovative composition and process design, the present invention can obtain hot-rolled or pickled ultra-high plasticity high-strength steel with low yield strength. The steel has a yield strength of 500MPa or more, preferably 600MPa or more, more preferably 700MPa or more, a tensile strength of 780MPa or more, preferably 980MPa or more. The elongation rate of the hot-rolled or pickled steel coils is greater than or equal 25%, preferably greater than or equal 30%.

[0052] Compared with prior arts, the advantages of the present invention are as follows.

[0053] Compared with existing ultra-high plasticity steels, the present invention adopts a composition design of medium-to-low carbon, high silicon, and high aluminum, which is completely different from the traditional low-carbon, high-silicon or low-silicon, high-aluminum designs of conventional hot-rolled ultra-high plasticity steels.

[0054] In the steel disclosed in Chinese Patent Application CN104233092A, in addition to low carbon and high silicon,

precious metal elements such as Cr, Mo, and Nb are added, resulting in relatively high alloy costs. The composition design of Chinese patent CN107815593A is low silicon and high aluminum with a certain amount of Cu. However, its process route mainly involves heat treatment in a dual-phase zone followed by phase transformation in the bainite zone, which cannot be applied to hot rolling production lines.

[0055] Therefore, the above patent applications not only differ from the present invention in terms of composition design, but also suffer from issues such as high alloy costs and process routes that cannot be adapted for hot rolling production lines.

[0056] The present invention adopts an innovative composition design concept of medium-to-low carbon and high aluminum, which is matched with the innovative staged cooling and medium-temperature coiling processes. Hot-rolled and pickled ultra-high plasticity steels with high tensile strength, ultra-high elongation, and high hole expansion rate can be obtained using an existing continuous hot rolling production line.

[0057] The high-strength and ultra-high plasticity steel manufactured by using the technology provided by the present invention has a yield strength of \geq 500MPa, a tensile strength of \geq 780MPa, a low yield ratio, and ultra-high elongation (A reaching 30% or more), exhibiting excellent combination of low yield strength, low yield ratio, high tensile strength, ultra-high plasticity, and high hole expansion rate. It can be applied to the manufacturing of various complex parts of passenger or commercial vehicles and has promising application prospects.

DESCRIPTION OF THE DRAWINGS

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Figure 1 is a schematic diagram of the rolling process of the steel according to the present invention.

Figure 2 is a schematic diagram of the cooling process of the steel according to the present invention.

Figure 3 shows a metallographic photograph of Example 1 according to the present invention.

Figure 4 shows a metallographic photograph of Example 6 according to the present invention.

Figure 5 shows a metallographic photograph of Example 10 according to the present invention.

Figure 6 shows a metallographic photograph of Example 14 according to the present invention.

Figure 7 shows a metallographic photograph of Example 17 according to the present invention.

Figure 8 shows a metallographic photograph of Example 19 according to the present invention.

Figure 9 shows a metallographic photograph of Example 21 according to the present invention.

Figure 10 shows a metallographic photograph of Example 23 according to the present invention.

DETAILED DESCRIPTION

³⁵ **[0059]** The present invention is further explained below in conjunction with the examples and drawings.

[0060] The compositions of the steels in the examples and comparative examples of the present invention are shown in Table 1. The balance in the table is Fe and other inevitable impurities.

[0061] The process of the example of the present invention are as follows.

1) Smelting and casting:

According to the composition in Table 1, molten steel was smelted in a converter or an electric furnace, then secondary refined in a vacuum furnace, and then casted into a casting blank or a casting ingot.

2) The casting blank or the casting ingot was reheated at a heating temperature of 1100 °C or higher and held for 1-2 hours.

3) Hot rolling and cooling the casting blank or the casting ingot:

The casting blank or the casting ingot was hot rolled at an initial rolling temperature of 1000 °C or higher, then subjected to 5-7 passes of rolling with a relatively large deformation rate of 50% or more at 1000 °C or higher, then subjected to 3-7 passes of final rolling with a cumulative deformation of 70% or more after an intermediate blank reaches \geq 950 °C, to obtain a steel strip. The final rolling temperature was 800~950 °C.

[0062] The cooling was staged cooling. After the final rolling, the steel strip was water-cooled to a temperature between 600~750 °C at a cooling rate of 30 °C/s or more. After air cooling for 1~10 seconds, the steel strip was then cooled to a temperature between 350~550 °C at a cooling rate of 10 °C/s or more and coiled, and then cooled to room temperature at a cooling rate of 50 °C/h or less, to obtain a hot-rolled strip steel.

[0063] In Examples 1-8 and 17-24, the pickling step was not performed and hot-rolled steels were obtained. In Examples 9-16, the pickling step was performed and pickled steels were obtained.

[0064] The specific process for the pickling step is as follows.

4) Pickling: The hot-rolled strip steel was pickled at a running speed of 30~120 m/min, with a pickling temperature of 75~85

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°C and a straightening rate of 2% or less, and then rinsed at a temperature in the range of 35~50 °C, and the surface of the hot-rolled strip steel was dried at a temperature of 120~140 °C, and oiled.

[0065] The specific process of the above-mentioned step 3 is illustrated in Figure 1 and Figure 2.

[0066] The steels of Comparative Example 1-3 are from CN 104233092 A.

[0067] Table 2 shows the specific production process parameters of the steel in the examples of the present invention, and the specific process parameters for pickling are not shown. Table 3 shows the performance parameters of the hot-rolled steels of Examples 1-8 and 17-24 of the present invention. Table 4 shows the performance parameters of the pickled steels of Examples 9-16 of the present invention.

[0068] The performances of the steels in Table 3-4 were measured as follows.

10 **[0069]** The yield strength, tensile strength, and elongation of steel were tested in accordance with GB/T 228.1-2021 "Tensile testing of metallic materials - Part 1: Room temperature test method".

[0070] The hole expansion rate of steel were tested in accordance with GB/T 24524-2021 "Metallic Materials, Thin Plates and Strips, Test Method for Hole Expansion".

[0071] As shown in Table 1, the composition design of the comparative examples is low-carbon, high silicon and low aluminum, while the composition design of the examples of the present invention is medium-to-low carbon, high silicon and high aluminum. The two have completely different composition designs in terms of carbon content and aluminum content. [0072] As can be seen from the performance comparison in Table 3, the hole elongation rate of comparative examples is around 20%, while the hole elongation rate of the examples of the present invention reaches around 30%, indicating that

[0073] As can be seen from Tables 3 and 4, the ferrite content in the microstructure of the comparative examples is 15% or less, while the ferrite content in the steel microstructure of the examples is 25% to 45%. Additionally, the bainite content in the microstructure of the comparative examples is 70% or more, while the bainite content in the microstructure of the examples is 44% to 53%, demonstrating a significant difference in microstructural design between the two.

the ultra-high plasticity steel of the present invention has a better matching of strength and ultra-high plasticity.

[0074] As can be seen from Tables 3 and 4, the hot-rolled or pickled high-strength ultra-high plasticity steel coils or plates according to the present invention have a yield strength of 500 MPa or more, up to 600 MPa or more, and even up to 700 MPa or more; a tensile strength of 780 MPa or more, up to 980 MPa or more; an elongation rate of 25% or more, up to 30% or more; and a hole expansion rate of 30% or more, and even up to 50% or more. The steel has good matching of yield strength, tensile strength, ultra-high plasticity and high hole expansion rate, and is especially suitable for complex forming and cold stretching parts such as automotive chassis structures and has broad application prospects.

[0075] Figures 3 to 6 show the metallographic photographs of Examples 1, 6, 10, and 14, respectively. These figures demonstrate that the composition and process design according to the present invention achieve a microstructure mainly composed of carbide-free lath bainite and retained austenite between bainitic laths. Figures 7 to 10 show typical metallographic photographs of Examples 17, 19, 21, and 23, respectively. These figures clearly demonstrate that the composition and process design according to the present invention achieve a microstructure mainly composed of ferrite with intragranular nanoprecipitates, bainite and retained austenite. This microstructure provides a good match of low yield strength, high tensile strength, ultra-high plasticity and high hole expansion rate, resulting in excellent overall performance. The steel of the present invention has a good match of strength, ultra-high plasticity, and high hole expansion, making it particularly suitable for complex forming parts such as automotive chassis structures and has broad application prospects.

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		В	0.0005	0.0002	0.0008	0.0003	0.0005	9000.0	0.0004	0.0010		0.0005		0.0010	ı	9000.0		0.0008		•	
5		^	0.08	0.04		-	0.10			0.05	0.10				0.05			0.03	-	-	-
10		qN	0.03	-	-	-	0.05	-	0.10	-	-	90.0	-	-	0.02	-	0.04	-	0.024	0.031	0.032
		Ö		0.5		٠	0.3			0.2	0.3			0.1		0.4	0.2	٠	0.46	0.35	0.30
15		Ē			0.1	-		0.5	0.3		0.1			0.5		0.3		0.2	-	-	•
		Cu		0.2			0.5			0.3		0.5			0.1			0.3		-	
20		Мо	0.2			0.4	-	0.3	0.5			0.1	-	0.5	0.2		0.3	-	0.27	0.22	0.20
		Ι		0.20	0.10	ı	0.02	0.14	,	0.05	0.12	0.16	0.13	0.20	60.0	0.10	0.05	0.07	ı	-	-
25	entage)	0	0.0023	0.0022	0.0021	0.0030	0.0025	0.0028	0.0026	0.0024	0.0028	0.0025	0.0020	0.0030	0.0023	0.0024	0.0025	0.0022	ı	-	-
30	Table 1 (Unit: mass percentage)	z	0.0025	0.0028	0.0022	0.0046	0.0042	0.0050	0.0033	0.0030	0.0041	0.0045	0.0037	0.0038	0.0042	0.0033	0.0035	0.0040	-	-	-
	le 1 (Unit:	A	0.75	0.33	1.96	0.45	1.79	0.10	1.28	0.58	1.15	0.83	0.11	0.39	0.68	1.50	1.97	1.74	0.032	0.037	0.033
35	Tab	S	0.0022	0.0025	0.0030	0.0027	0.0028	0.0029	0.0028	0.0024	0.0022	0.0028	0.0031	0.0025	0.0044	0.0023	0.0026	0.0050	0.0030	0.0040	0.004
40		Д	0.013	0.009	0.016	0.019	0.011	0.010	0.014	0.018	0.018	0.014	0.016	0.020	0.016	0.015	0.009	0.010	600.0	0.008	600.0
		Mn	2.03	1.03	1.82	1.27	1.55	2.68	2.97	2.24	1.59	1.86	2.48	2.21	1.38	1.44	1.25	1.03	1.76	1.65	1.45
45		Si	1.54	0.81	1.75	1.86	1.44	1.99	1.62	1.02	1.73	1.48	1.98	1.02	1.54	0.81	1.66	1.35	1.58	1.41	1.56
		С	0.27	0.16	0.35	0.21	0.33	0.10	0.31	0.23	0.25	0.20	0.10	0.15	0.28	0.26	0.35	0.31	0.07	0.09	0.10
50 55			1, 9	2, 10	3, 11	4, 12	5, 13	6, 14	7, 15	8, 16	17	18	19	20	21	22	23	24	Comparative Example 1	Comparative Example 2	Comparative Example 3
55			Example 1,	Example 2, 10	Example 3, 11	Example 4, 12	Example 5, 13	Example 6, 14	Example 7, 15	Example 8, 16	Example 17	Example 18	Example 19	Example 20	Example 21	Example 22	Example 23	Example 24	Comparat	Comparat	Comparat

		Cooling rate of steel coil (°C/h)	25	90	19	11	20	37	23	16	22	90	18	44	20	38	14	30
5		Coilin g Temp eratur e /°C	450	520	400	350	200	430	380	250	530	250	480	200	350	450	420	520
10		Cooling rate in second stage (°C/s)	36	51	44	25	30	47	20	28	25	75	15	99	21	42	10	33
15		Air coolin g durati on /s	3	8	1	2	10	2	4	7	8	_	9	2	3	7	10	4
20		Air cooli ng temp eratur e /°C	049	092	099	089	009	099	089	002	970	052	009	710	069	089	049	029
		Cooling rate in first stage (°C/s)	22	20	20	92	40	35	30	09	20	120	83	140	30	06	20	42
25		Final rolling temper ature /°C	920	800	850	810	950	860	830	880	830	950	820	930	850	006	800	880
30	Table 2	Cumulativ e deformatio n during final rolling /%	94	91	93	86	93	66	94	93	92.3	9.76	92.8	0.86	92.6	2.36	93.6	91.0
35		Temperatu re of intermediat e blank /° C	975	985	096	970	366	086	950	965	965	950	970	1000	086	096	066	962
40		Cumulativ e deformatio n during rough rolling /%	75	20	22	09	99	20	72	65	20	75	09	22	65	72	20	80
45		Initial rollin g tempe rature	1070	1140	1050	1060	1100	1150	1120	1080	1050	1100	1000	1090	1030	1080	1020	1070
50		Heating tempe rature /°C	1260	1200	1150	1210	1180	1230	1100	1270	1200	1300	1150	1230	1100	1260	1170	1280
55			Example 1, 9	Example 2, 10	Example 3, 11	Example 4, 12	Example 5, 13	Example 6, 14	Example 7, 15	Example 8, 16	Example 17	Example 18	Example 19	Example 20	Example 21	Example 22	Example 23	Example 24

Table 3

5		Thickness of Steel Plate /mm	Yield Strength /MPa	Tensile Strength /MPa	Elongation Rate /%	Hole Expansion Rate /%	Ferrite Content /%	Bainite Content /%	Residual Austenite Content /%
	Example 1	3.6	540	821	32.5	45	35	53	12
	Example 2	7.0	565	813	32.0	47	45	44	11
10	Example 3	8.0	600	810	33.0	42	40	50	10
	Example 4	2.2	612	807	31.0	48	44	44	12
	Example 5	5.8	697	806	31.5	53	42	50	8
	Example 6	1.5	719	811	30.5	50	41	50	9
15	Example 7	4.0	755	856	30.0	55	45	47	8
	Example 8	6.0	609	813	31.5	49	38	49	13
	Example 17	5.8	622	794	32.0	73	36	50.5	13.5
20	Example 18	1.5	676	833	31.0	65	31	57.4	11.6
	Example 19	7.2	655	815	31.0	58	40	54.4	5.6
	Example 20	2.3	629	830	30.0	53	43	49.3	7.7
	Example 21	6.5	791	992	27.0	44	33	51.5	15.5
25	Example 22	3.2	830	1005	25.0	38	28	57.9	14.1
	Example 23	8.0	814	1011	26.0	51	30	50.8	19.2
	Example 24	4.5	873	1036	27.0	32	25	57.7	17.3
30	Comparative Example 1	4.5	512	891	18.0	-	11	78	11
	Comparative Example 2	5.0	507	889	19.0	-	10	77	13
35	Comparative Example 3	6.0	494	873	20.5	-	12	73	15

Table 4

40		Thickness of Steel Plate /mm	Yield Strength /MPa	Tensile Strength /MPa	Elongation Rate /%	Ferrite Content /%	Bainite Content /%	Residual Austenite Content /%	
	Example 9	3.6	651	838	31.0	35	53	12	
	Example 10	7.0	734	857	30.5	45	44	11	
45	Example 11	8.0	573	821	32.5	40	50	10	
	Example 12	2.2	694	847	31.0	44	44	12	
	Example 13	5.8	554	833	31.5	42	50	8	
50	Example 14	1.5	592	844	31.5	41	50	9	
	Example 15	4.0	751	862	30.0	45	47	8	
	Example 16	6.0	583	840	30.5	38	49	13	
55	Comparative Example 1 4.5		512	891	18.0	11	78	11	
	Comparative Example 2	5.0	507	889	19.0	10	77	13	

(continued)

5		Thickness of Steel Plate /mm	Yield Strength /MPa	Tensile Strength /MPa	Elongation Rate /%	Ferrite Content /%	Bainite Content /%	Residual Austenite Content /%
	Comparative Example 3	6.0	494	873	20.5	12	73	15

10 Claims

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- 1. A steel comprising the following components in percentage by mass: C: 0.10-0.35%, Si: 0.8-2.0%, Mn: 1.0-3.0%, P: \leq 0.02%, S \leq 0.005%, Al: 0.1-2.0%, N: \leq 0.005%, with the balance being Fe and other inevitable impurities.
- 2. The steel according to claim 1, wherein the steel further comprises Ti; in percentage by mass, the content of Ti is less than or equal to 0.2%, preferably 0.05-0.2%, more preferably 0.05-0.1%.
- 3. The steel according to claim 1 or 2, wherein the steel further comprises one or more selected from the group consisting of Mo, Nb, V, Cu, Ni, Cr and B,

wherein, in percentage by mass,

the content of Mo is less than or equal to 0.5%, preferably less than or equal to 0.3%;

the content of Nb is less than or equal to 0.1%, preferably less than or equal to 0.06%;

the content of V is less than or equal to 0.1%, preferably less than or equal to 0.06%;

the content of Cu is less than or equal to 0.5%, preferably less than or equal to 0.3%;

the content of Ni is less than or equal to 0.5%, preferably less than or equal to 0.3%;

the content of Cr is less than or equal to 0.5%, preferably less than or equal to 0.3%;

the content of B is less than or equal to 0.001%, preferably less than or equal to 0.0005%.

- 4. The steel according to claim 1 or 2, wherein the inevitable impurities includes, in percentage by mass, $O \le 0.003\%$, preferably $O \le 0.002\%$; $S \le 0.003\%$; and/or $N \le 0.004\%$.
 - **5.** The steel according to claim 1 or 2, wherein the components of the steel satisfies one or more of the following: C: 0.15~0.25%, Si: 1.0~1.6%, Mn: 1.5~2.5%, Al: 0.3~1.0% in percentage by mass.
 - **6.** The steel according to any one of claims 1 to 5, wherein the steel has a microstructure of ferrite, bainite, and residual austenite with a content of \geq 5%.
- 7. The steel according to any one of claims 1 to 6, wherein the steel has a yield strength of 500 MPa or more, preferably 600 MPa or more, more preferably 700 MPa or more; a tensile strength of 780 MPa or more, preferably 980 MPa or more; and an elongation rate of 25% or more, preferably 30% or more.
- 8. The steel according to claim 2, wherein in percentage by mass, the content of C is 0.10-0.25%, the content of Ti is 0.05-0.2%, and wherein the steel has a yield strength of 600 MPa or more, a tensile strength of 780 MPa or more, an elongation rate of 30% or more, and a hole expansion rate of 50% or more; or, wherein in percentage by mass, the content of C is 0.25-0.35%, the content of Ti is 0.05-0.2%, and wherein the steel has a yield strength of 700 MPa or more, a tensile strength of 980 MPa or more, an elongation rate of 25% or more, and a hole expansion rate of 30% or more.
 - **9.** The steel according to any one of claims 1 to 8, wherein the hole expansion rate of the steel is 30% or more, preferably 50% or more.
 - 10. A method for manufacturing the steel according to any one of claims 1 to 9, including the following steps:
 - 1) smelting and casting smelting the components according to any one of claims 1-5 in a converter or an electric furnace, then secondary refining in a vacuum furnace, and then casting it into a casting blank or a casting ingot;

2) reheating the casting blank or the casting ingot heating temperature ≥ 1100 °C, holding time: 1-2 hours;

- 3) hot rolling and cooling the casting blank or the casting ingot
- wherein the casting blank or the casting ingot is hot rolled at an initial rolling temperature of 1000 °C or higher, then subjected to 5-7 passes of rolling with a relatively large deformation rate of 50% or more at 1000 °C or higher, then subjected to 3-7 passes of final rolling with a cumulative deformation of 70% or more after an intermediate blank reaches \geq 950 °C, obtaining a steel strip; wherein the final rolling temperature is 800~950 °C;
- wherein the cooling is staged cooling, after the final rolling, the steel strip is water-cooled to a temperature between 600~750 °C at a cooling rate of 30 °C/s or more; after air cooling for 1~10 seconds, the steel strip is then cooled to a temperature between 350~550 °C at a cooling rate of 10 °C/s or more and coiled, and then cooled to room temperature at a cooling rate of 50 °C/h or less, obtaining a hot-rolled strip steel.
- 11. The method according to claim 10, wherein the method further includes step 4) pickling, wherein the hot-rolled strip steel is pickled at a running speed of 30~120 m/min, with a pickling temperature of 75~85 °C and a straightening rate of 2% or less, and then rinsed at a temperature in the range of 35~50 °C, and the surface of the hot-rolled strip steel is dried at a temperature of 120~140 °C, and oiled.

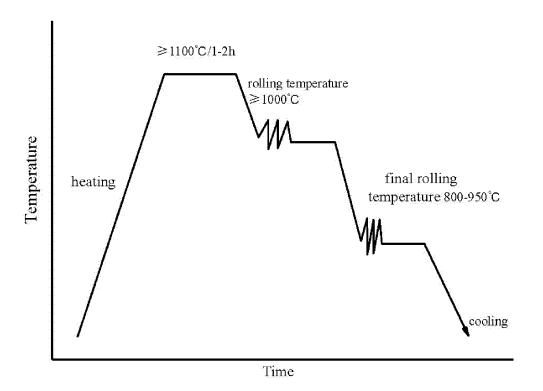


Figure 1

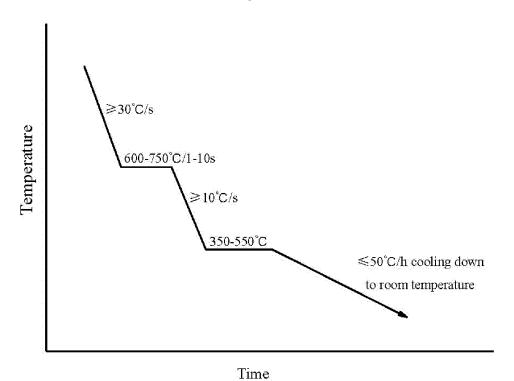


Figure 2

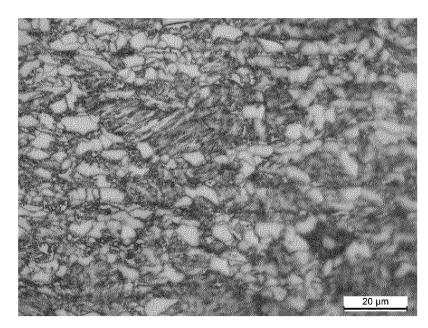


Figure 3

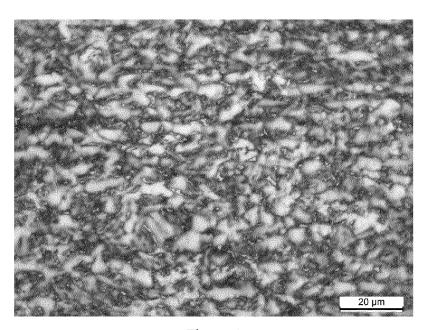


Figure 4

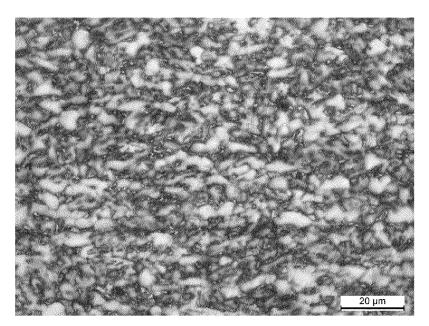


Figure 5

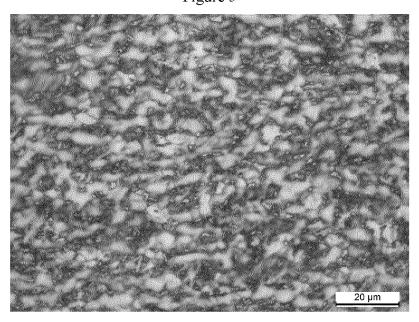


Figure 6

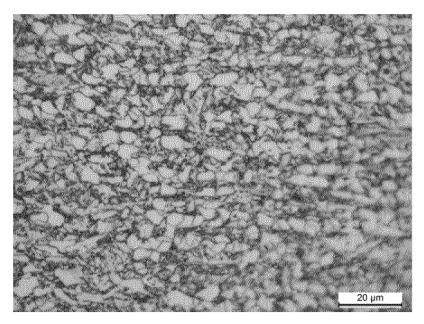


Figure 7

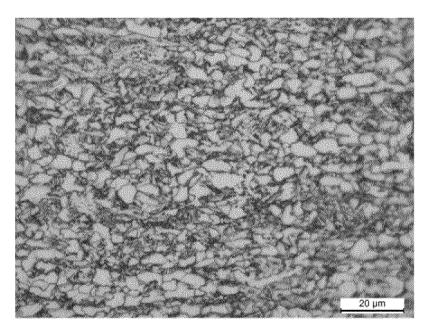


Figure 8

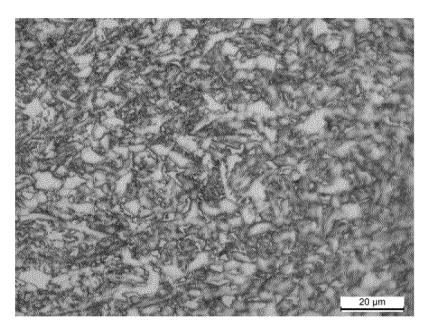


Figure 9

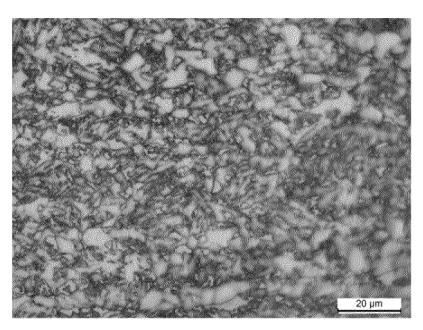


Figure 10

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2023/101832

_			PCT/CN2	2023/101832
5	A. CLAS	SSIFICATION OF SUBJECT MATTER	· · · · · · · · · · · · · · · · · · ·	
	C22C	38/00(2006.01)i; C22C 38/06(2006.01)i; C22C 38/	38(2006.01)i; C22C 38/28(2006.01)i; C2	1D 6/00(2006.01)i
	According to	International Patent Classification (IPC) or to both na	tional classification and IPC	
10		DS SEARCHED		
	Minimum do	cumentation searched (classification system followed	by classification symbols)	
	IPC:C	22C,C21D		
	Documentati	on searched other than minimum documentation to the	e extent that such documents are included in	the fields searched
15				
	CNTX	ta base consulted during the international search (nam T, ENTXT, DWPI, SIPOABS, CNKI: 钢, 碳, C, 硅, S nese, aluminium, titanium	•	· · · · · · · · · · · · · · · · · · ·
20	C. DOC	UMENTS CONSIDERED TO BE RELEVANT		
	Category*	Citation of document, with indication, where a	appropriate, of the relevant passages	Relevant to claim No.
	X	CN 105925887 A (BAOSHAN IRON & STEEL CO description, paragraphs 9-16, 28-34 and 52, and		1-11
25	X	CN 104532126 A (BAOSHAN IRON & STEEL CO description, paragraphs 9-39	., LTD.) 22 April 2015 (2015-04-22)	1-11
	X	CN 108018493 A (BAOSHAN IRON & STEEL CO description, paragraphs 8-34	ı	1-11
30	X	CN 108018498 A (BAOSHAN IRON & STEEL CO description, paragraphs 7-33	ı	1-11
30	Α	CN 106119702 A (BAOSHAN IRON & STEEL CO entire document	ı	1-11
	A	WO 2007132600 A1 (NISSAN MOTOR CO., LTD. entire document	et al.) 22 November 2007 (2007-11-22)	1-11
35			······································	
	Further d	ocuments are listed in the continuation of Box C.	See patent family annex.	
40		ategories of cited documents: t defining the general state of the art which is not considered	"T" later document published after the interna date and not in conflict with the application	ntional filing date or priority on but cited to understand the
	to be of p	articular relevance t cited by the applicant in the international application	principle or theory underlying the inventi "X" document of particular relevance; the cl	on
	"E" earlier ap filing date	plication or patent but published on or after the international e	considered novel or cannot be considered when the document is taken alone	to involve an inventive step
45	cited to e	t which may throw doubts on priority claim(s) or which is establish the publication date of another citation or other	"Y" document of particular relevance; the cl considered to involve an inventive st combined with one or more other such do	ep when the document is
45		eason (as specified) t referring to an oral disclosure, use, exhibition or other	being obvious to a person skilled in the a: "&" document member of the same patent fan	rt
	"P" documen	t published prior to the international filing date but later than y date claimed	or or are state patent tall	<i></i>
	Date of the act	ual completion of the international search	Date of mailing of the international search	report
50		15 September 2023	19 September 202	23
	Name and mai	ling address of the ISA/CN	Authorized officer	
	China Nat CN)	tional Intellectual Property Administration (ISA/		
		6, Xitucheng Road, Jimenqiao, Haidian District,		
55	Deijing 10	0000	Telephone No.	
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Form PCT/ISA/210 (second sheet) (July 2022)

INTERNATIONAL SEARCH REPORT International application No. Information on patent family members PCT/CN2023/101832 Patent document Publication date Publication date Patent family member(s) cited in search report (day/month/year) (day/month/year) 2019194772 CN 105925887 A 07 September 2016 US 27 June 2019 A1US 11220724 B2 11 January 2022 JP 2019523828A 29 August 2019 10 JP 6812465 B2 13 January 2021 WO 2017219938 28 December 2017 A1KR 20190014550 12 February 2019 Α 06 April 2021 KR 102236344 В1 CN 105925887 В 30 January 2018 15 CN 104532126 22 April 2015 WO 2016095664 **A**1 23 June 2016 EP 3235920 25 October 2017 A13235920 27 June 2018 ΕP A4 ΕP 3235920 20 November 2019 B1US 201734252330 November 2017 A120 11180822 US B2 23 November 2021 CN 104532126 В 06 June 2017 108018493 None CN 11 May 2018 CN 108018498 A 11 May 2018 None 25 CN 106119702 16 November 2016 CN 106119702 В 02 October 2018 WO 2007132600 EP 11 February 2009 A122 November 2007 2022865 A1US 2009236015 A124 September 2009 EP 2022865 11 February 2009 US 2009236015 A124 September 2009 30 JP 5220342 **B**2 26 June 2013 JP 2007332456 27 December 2007 Α JP 2007332457 27 December 2007 Α JP 2007332455 27 December 2007 A JP 5220341 B2 26 June 2013 35 5220343 26 June 2013 JP B2 40 45 50 55

REFERENCES CITED IN THE DESCRIPTION

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