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### (54) High-strength steel sheets and processes for production of the same

(57) A high strength steel sheet with both excellent elongation and stretch-flanging performance is provided.

The high strength steel sheet of the present invention comprises, in percent by mass, C : 0.05 to 0.3%, Si: 0.01 to 3.0%, Mn: 0.5 to 3.0%, Al: 0.01 to 0. 1%, and Fe and inevitable impurities as the remainder, and has a structure mainly composed of tempered martensite and annealed bainite. The space factor of the tempered martensite is 50 to 95%, the space factor of the annealed bainite is 5 to 30%, and the mean grain size of the tempered martensite is 10  $\mu m$  or smaller in terms of the equivalent of a circle diameter. The steel sheet has a tensile strength of 590 MPa or higher.

The high strength steel sheet of the present invention has a space factor of the martensite phase which is a

main component of the metal structure is 80% or higher; the mean grain size of the martensite phase is 10  $\mu m$  or smaller in terms of the equivalent of a circle diameter; in the martensite phase, the space factor of the martensite phase having a grain size of 10  $\mu m$  or larger in terms of the equivalent of a circle diameter is 15% or lower; and the space factor of the retained austenite phase in the metal structure is 3% or lower.

The high strength steel sheet of the present invention is a dual phase steel sheet mainly composed of a ferrite phase and martensite, and the space factor of the ferrite phase is 5 to 30%, and the space factor of the martensite phase is 50 to 95%. Moreover, the ferrite phase is annealed martensite.

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### Description

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[Technical Field]

**[0001]** The present invention relates to a high strength steel sheet for which high press formability is required, typically including steel sheets for automobiles, particularly to a high strength steel sheet with both elongation and stretch-flanging performance and a method for manufacturing the same.

[Background Art]

[0002] High strength steel sheets, which are generally used by being press-molded, are used in industrial product such as automobiles, electric devices and industrial machines- Since high strength steel sheets are used for the purpose of lightening industrial products, they need not only have high strength, but also have the ability to form various configurations of the products. Accordingly, it is required for high strength steel sheets to have excellent press formability. To meet this requirement, high-strength steel sheets having excellent elongation and stretch-flanging performance, which are necessary for improving press formability, are required-[0003] Examples of known steels having such characteristics include dual phase steel (DP steel) whose metal structure is composed of a ferrite phase and a martensite phase, as described in Patent document 1. Since this DP steel can ensure ductility (elongation) due to its soft ferrite and strength due to its rigid martensite, it has both strength and elongation (in particular, uniform elongation). However, because of the coexistence of soft ferrite and rigid martensite, distortion (stress) is concentrated at the interface of the two phases when deformed, and therefore the interface is likely to serve as the starting point of rupture, thereby disadvantageously preventing ensuring stretch-flanging performance (local elongation).

**[0003]** Examples of steel sheets which expectedly have ductility (especially, uniform elongation) higher than those of DP steels include TRIP steels utilizing the TRIP (Transformation Induced Plasticity) phenomenon, as described in Patent document 2. This TRIP steel is a steel sheet in which uniform elongation is increased by transforming retained austenite into martensite during deformation (working-induced transformation). However, since martensite which has been transformed from retained austenite in the TRIP steel is extremely hard, it likely serves as the starting point of rupture, lowering the stretch-flanging performance of the steel sheet.

**[0004]** other methods of improving the stretch-flanging performance of the high strength steel sheets include that in which the metal structure is single-phase structure and localization of process distortion is suppressed by homogenizing the in the metal structure, and that in which a difference in strength between a soft phase having a multi-phase metal structure and a hard phase is reduced.

Since martensite single-phase structure steel sheet has a uniform structure, it is known as a steel sheet which has both strength and stretch-flanging performance. However, the martensite single-phase structure steel sheet disadvantageously has low ductility, and insufficient elongation.

[0005] Patent document 3 discloses a high-stretch-strength cold-rolled steel sheet in which martensite single-phase structure is achieved by justifying the composition and heat treatment conditions of the steel sheet, and tensile strength is 880 to 1170 MPa. That is, the high-stretch-strength cold-rolled steel sheet of Patent document 3 is produced by heating and retaining a steel sheet having a predetermined composition range at 850°C, which is normally reachable temperature industrially, to transform the steel sheet into austenite, and then rendering it a martensite single-phase structure. A steel sheet of a martensite single-phase structure produced by this invention has a tensile strength of 880 to 1170 MPa, and thus has excellent stretch-flanging performance. However, it has elongation EL (%) lower than 8% and thus has low ductility. In the high strength steel sheet of the invention of Patent document 3, if ductility is improved, press formability can be further improved.

**[0006]** Moreover, Patent document 4 discloses a method for manufacturing a high tensile strength steel sheet, in which a steel sheet in which the ratio by volume of a low-temperature transformation phase comprising a martensite phase and others and a retained austenite phase is 90% or higher of the entire metal structure is heated and retained to produce a two phase region: a ferrite phase and an austenite phase, a metal structure comprising a fine ferrite phase which has succeeded the laths of the low-temperature transformation phase and the austenite phase is provided, and finally the steel sheet is given such a metal structure that comprises ferrite and the low-temperature transformation phase finely dispersed in the form of laths.

**[0007]** However, since the steel sheet produced by the steelmaking method disclosed in Patent document 4 has a relatively high cooling stop temperature in the steelmaking process, a large amount of bainite is deposited, while a large amount of retained austenite also remains therein, and therefore the steel sheet has excellent ductility, but has insufficient stretch-flanging performance. By the steelmaking method of Patent document 4, a steel sheet which is excellent in both elongation and stretch-flanging performance cannot be produced.

[Patent document 1] Japanese Unexamined Patent Application Publication (JP-A) No. S55-122820

[Patent document 2] JP-A-S60-43425 [Patent document 3] Japanese Patent No. 3729108 [Patent document 4] JF-A-2005-272954

5 [Disclosure of the Invention]

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[Problem to be Solved by the invention]

**[0008]** As mentioned above, since DP steel sheets, TRIP steel sheets, and martensite single-phase structure steel sheets have their advantages and disadvantages, a steel sheet which has high strength and excellent elongation and stretch-flanging performance at the same time is required. The present invention has been made to solve such a problem, and an object thereof is to provide a high strength steel sheet excellent in both elongation and stretch-flanging performance and a method for manufacturing the same.

**[0009]** Another object of the present invention is to provide a high strength steel sheet having a tensile strength of 780 MPa or higher, in which elongation and stretch-flanging performance are both improved, and a method for manufacturing the same.

[Means for Solving the Problem]

20 [0010] The high strength steel sheet of the present invention is constituted of, in percent by mass, C: 0.05 to 0.3%, Si: 3% or less (not including 0%.), Mn: 0.5 to 3.0%; Al: 0.01 to 0.1%, and the remainder comprising iron and inevitable impurities, has a space factor of a martensite phase which is a main component of a metal structure of 50% or higher, and has a tensile strength of 590 MPa or higher.

**[0011]** To this end, the inventors of the present invention have studied various structures that can ensure high strength and improve elongation, especially stretch-flanging performance at the same time. As a result, the inventors found the following: by annealed bainite, which is a fine lath-shaped structure, as an initial structure in a two phase temperature region of ferrite +austenite (hereinafter referred to as "two-phase region annealing".), fine annealed bainite produced in a base material acts in a manner of suppressing the growth of austenite, fine tempered martensite is produced from austenite by the following hardening and tempering, and the entire structure is formed from these microstructures. Therefore, elongation and stretch-flanging performance are improved. The inventors accomplished the present invention based on these findings.

**[0012]** That is, the high strength steel sheet of the present invention has a structure mainly comprising tempered martensite and finely dispersed annealed bainite, a space factor of the tempered martensite of 50 to 95%, a space factor of the annealed bainite of 5 to 30%, and a mean grain size of the tempered martensite in terms of the equivalent of a circle diameter of 10  $\mu$ m or lower. The term "equivalent of a circle diameter" means the diameter of an anticipated circle having the same area as the grains of tempered martensite, and is determined by subjecting a structure picture to image analysis. Moreover, the term "space factor" means the percentage by volume, and is determined by corroding a structure observation test piece with nital, observing the test piece with an optical microscope (1000 times), and by subjecting the observed structure picture to image analysis. Moreover, annealed bainite is observed as a body centered cubic structure in terms of a crystal structure.

[0013] The method for manufacturing a high strength steel sheet with excellent elongation and stretch-flanging performance according to the present invention comprises using a steel sheet having a space factor of bainite in the entire metal structure of 90% or higher as a material steel sheet; heating and retaining the steel sheet at a temperature of ( $Ac_3$  point -100°C) or higher but not higher than  $Ac_3$  point for 0 to 2400 seconds (including 0 seconds), and then cooling to a transformation start temperature of martensite, Ms point, or lower at an average cooling rate of 10°C/sec. or higher, subsequently heating and retaining the steel sheet at a temperature of 300 to 550°C for 60 to 1200 seconds. The high strength steel sheet of the present invention is thus produced. The material steel sheet can be produced by hot rolling a steel piece having the above-mentioned chemical component or further by cold rolling the same.

Herein,  $Ac_3$  point is a temperature at which a two-phase region comprising an austenite phase and a ferrite phase transforms into an austenite single-phase region that is stable at high temperatures in a temperature raising step.

**[0014]** The inventors of the present invention have also invented a high strength steel sheet having a limited ratio by volume of the retained austenite phase of 3%or lower, which does not affect stretch-flanging performance, and a metal structure in which a large part of the metal structure is a fine martensite phase.

That is, in the high strength steel sheet of the present invention, the space factor of the martensite phase which is a main component of the metal structure is 80% or higher; the mean grain size of the martensite phase is 10  $\mu$ m or smaller in terms of the equivalent of a circle diameter; the space factor of a martensite phase having a grain size of 10  $\mu$ m or larger in terms of the equivalent of a circle diameter in the martensite phase is 15% or lower, and the space factor of the retained austenite phase in the metal structure is 3% or lower.

[0015] Herein, space factor means a ratio by volume of each phase constituting the metal structure in the steel material to the entire metal structure the space factors of the martensite phase and ferrite phase were determined by subjecting the steel material to repeller corrosion, observing the material by an optical microscope and an SEM (1000 times), and then subjecting the material to image analysis. The space factor of the retained austenite phase was determined by the saturation magnetization method (refer to "Netsushori" (heat treatment), Vol.136, (1996)). Moreover, the mean grain size of the martensite phase is the mean value of the crystal grain size of the martensite phase, and is determined by structure analysis using a FE/SEM-EBSP at step intervals of 100 nm in the present invention.

[0016] In the metal structure of the high strength steel sheet, the space factor of the fine tempered martensite phase having a mean grain size of 10  $\mu$ m or smaller is 80% or higher, and therefore a tensile strength of 780 MPa or higher and excellent ductility are ensured. Moreover, when the space factor of the retained austenite phase is high, stretch-flanging performance is lowered. However, the space factor of the retained austenite phase is limited to 3% at the highest in the present invention, and therefore stretch-flanging performance is not lowered.

**[0017]** In the high strength steel sheet, it is preferable that the martensite phase is a tempered martensite phase; an annealed martensite phase is contained as the metal structure other than the martensite phase and the retained austenite phase; and that the space factor of the annealed martensite phase is 3 to 20%.

Such features suppress combination between the crystal grains of the austenite phase and the growth of the same by the finely dispersed annealed martensite phase. As a result, the final structure is micronized, and the processability of the high strength steel sheet is ensured.

[0018] The method for manufacturing the high strength steel sheet according to the present invention is for manufacturing a high strength steel sheet of the present invention by using a steel sheet in which the total space factor of the martensite phase and/or of the retained austenite phase in the entire metal structure is 90% or higher as a material steel sheet, heating and retaining the steel sheet at a temperature of (Ac<sub>3</sub> point-100°C) or higher, Ac<sub>3</sub> point or lower for 30 to 1200 seconds, cooling the steel sheet to a transformation start temperature of martensite, Ms point, or lower at an average cooling rate of 10°C/sec- or higher, and further conducting a heat treatment in which the steel sheet is heated and retained at a temperature of 300 to 500°C for 60 to 1200 seconds.

**[0019]** In the high strength steel sheet of the present invention, the structure which is the main part of the metal structure is a martensite phase and a ferrite phase; the space factor of the martensite phase is 50 to 95% (meaning "% by volume", and so on); the space factor of the ferrite phase is 5 to 30%; and the mean grain size of the martensite phase is 10  $\mu$ m or smaller in terms of the equivalent of a circle diameter.

[0020] The ferrite phase is preferably an annealed martensite.

[0021] The method for manufacturing the high strength steel sheet according to the present invention is for manufacturing a high strength steel sheet of the present invention by using, as a material steel sheet, a steel sheet in which the total space factor of the martensite phase and/or bainite phase in the entire metal structure is 90% or higher and the grain size of the former austenite is 20  $\mu$ m or smaller in terms of the equivalent of a circle diameter, heating and retaining the steel sheet at a temperature of (Ac<sub>3</sub> point -100°C) or higher but not higher than Ac<sub>3</sub> point for 1 to 2400 seconds, then cooling the steel sheet to a transformation start temperature of martensite, Ms point, or lower at an average cooling rate of 10°C/sec. or higher, and subsequently conducting a heat treatment in which the steel sheet is heated and retained at a temperature of 300 to 550°C for 60 to 1200 seconds.

**[0022]** The high strength steel sheet according to the present invention may comprise, in addition to the above-mentioned basic components, any of the element groups (a) to (e) described below, or one or more elements selected from a plurality of groups within a range defined for each element group.

- (a) an element selected from Ti, Nb, V and Zr: 0.01 to 1% by mass in total
- (b) Ni and/or Cu: 1% by mass or less in total
- (c) Cr: 2% by mass or less and/or Mo: 1% by mass or less
- (d) 0.0001 to 0.005% by mass of B
- (e) Ca and/or REM: 0.003% by mass or less in total

[Effect of the Invention]

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[0023] In the present invention, a structure which is mainly composed of especially tempered martensite and finely dispersed annealed bainite is provided, wherein the space factors thereof are defined to have predetermined amounts, and the mean grain size of tempered martensite is defined 10  $\mu$ m or smaller. Accordingly, a high strength steel sheet which has strength as high as 590 MPa or higher, excellent elongation and stretch-flanging performance, and thus excellent press formability can be provided.

**[0024]** According to the present invention, it is also possible to provide a high strength steel sheet in which the space factor of the retained austenite phase is 3% or lower and the space factor of the fine martensite phase is 80% or higher by a relatively simple heat treatment step. Since this high strength steel sheet has a tensile strength of 780 MPa or

higher, and also has excellent elongation and stretch-flanging performance, it is excellent in press formability.

**[0025]** According to the present invention, it is also possible to achieve a high strength steel sheet which has excellent elongation and stretch-flanging performance at the same time by designing the steel sheet especially for a dual phase steel sheet mainly composed of a ferrite phase and martensite, ensuring high strength of the steel sheet as a whole, and appropriately controlling the space factors of especially the ferrite phase and martensite and the mean grain sizes of the same.

[Best Mode for Carrying out the Invention]

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[0026] The best mode for carrying out the invention will be described below in detail.

A high strength steel sheet according to one embodiment of the present invention is designed to have a structure, as the main body, in which annealed bainite is finely dispersed in tempered martensite, a space factor of the tempered martensite of 50 to 95%, a space factor of the annealed bainite of 5 to 30%, a mean grain size of the tempered martensite of 10  $\mu$ m or smaller in terms of the equivalent of a circle diameter, and a tensile strength of 590 MPa or higher. The reasons for limitation of the structure will be described below.

[0027] When the space factor of the annealed bainite is 5% or lower, the pinning effect, which suppresses the growth of austenite, is weak, and austenite grains grow so that martensite grains become large, thereby preventing ensuring good elongation. In contrast, when the space factor is higher than 30%, stretch-flanging performance is lowered. For this reason, the lower limit of annealed bainite is 5%, and preferably 7%, while its upper limit is 30%, and preferably 25%. [0028] When the space factor of tempered martensite is lower than 50%, strength and stretch-flanging performance are lowered. In contrast, when the space factor is higher than 95%, the steel sheet becomes too hard and thus elongation is lowered. For this reason, the lower limit of the tempered martensite phase is 50%, and more preferably 70%, while its upper limit is 95%, and preferably 85%.

**[0029]** The mean grain size of the tempered martensite varies depending on the amount of annealed bainite finely dispersed. When the grain size is larger than 10  $\mu$ m in terms of the equivalent of a circle diameter, elongation and stretch-flanging performance are lowered. For this reason, the upper limit is 10  $\mu$ m.

**[0030]** The structure in which the tempered martensite and annealed bainite coexist constitutes the main part of the structure of the high strength steel sheet of the present invention. Herein, the main part means 90% or higher, and preferably 95% or higher, and other structures contained in an amount of less than about 10% are permitted because they hardly affect elongation, especially stretch-flanging performance. Examples of other structures include ferrite, pearlite, retained austenite and the like. Of course, the less these structures, the better.

**[0031]** Chemical component (unit: % by mass) which is preferable for obtaining the structure and strength of the steel sheet according to the present invention will be described now. Examples of such a chemical component include that comprises the followings: C: 0.05 to 0.3%, Si: 0.01 to 3.0%, Mn: 0.5 to 3.0%, and Al: 0.01 to 0.1%, and Fe and inevitable impurities as the remainder. The reasons for component limitation will be described below.

[C: 0.05 to 0-3%]

**[0032]** C is an important element in producing martensite, and increasing the strength of the steel sheet. When the amount of C is lower than 0.05%, such an effect is excessively lowered. In contrast, from the perspective of increasing strength, the higher the amount of C, the more preferable. However, when the amount of C is higher than 0.3%, a large amount of retained austenite is produced and stretch-flanging performance is lowered. Moreover, weldability is also deteriorated. For this reason, the lower limit of the amount of C is 0.05%, and preferably 0.07%, while its upper limit is 0.3%, and preferably 0.25%.

[Si: 0.01 to 3.0%]

**[0033]** Si acts as a deoxidizing element when steel is melted, and is an element effective in increasing strength without deteriorating the ductility of steel. Si also acts to suppress deposition of coarse carbide which deteriorates stretch-flanging performance. When the amount of Si is lower than 0.01%, these actions are excessively lowered, while addition of the same in an amount higher than about 3.0% saturates the effect. For this reason, the lower limit of the amount of Si is 0.01%, and preferably 0.1%, while its upper limit is 3.0%, and preferably 2.5%.

[Mn: 0.5 to 3%]

[0034] Mn is an element useful in increasing the hardening characteristics of steel to ensure high strength, but when

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its amount is lower than 0.5%, such an action is excessively lowered. In contrast, when its amount is higher than 3%, ductility is lowered and processability is thus adversely affected. For this reason, the lower limit of the amount of Mn is 0.5%, and preferably 0.7%, while its upper limit is 3%, and preferably 2.5%.

5 [Al: 0.01 to 0.1%]

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**[0035]** Al is an element which has a deoxidation effect, and needs to be added in an amount of 0.01% or higher to perform the effect. In contrast, even if it is added in an amount higher than 0.1%, the deoxidation effect is saturated, and it becomes a source of non-metallic mediators to deteriorate physical properties and surface properties. For this reason, the lower limit of the amount of Al is 0.01%, and preferably 0.03%, while its upper limit is 0.1%, and preferably 0.08%.

**[0036]** Preferable chemical components of the steel sheet the present invention include, in addition to the above-mentioned basic components, Fe and impurities which inevitably get in, for example, P, S, N and O. However, to improve the mechanical characteristics of the steel sheet, any of the auxiliary element groups (a) to (e) described below, or one or more element selected from a plurality of groups may be added within the additional permissible range of each group.

- (a) One or more elements selected from Ti, Nb, V and Zr in a total amount of 0.01 to 1%
- (b) One or more elements selected from Ni and Cu in a total amount of 1% or lower
- (c) One or more elements of Cr: 2% or lower, Mo: 1% or lower
- (d) B in an amount of 0.0001 to 0.005%
- (e) One or more elements selected from Ca and REM in a total amount of 0.003% or lower

[One or more member of Ti, Nb, V and Zr: in a total amount of 0.01 to 1%]

**[0037]** These elements form precipitates such as carbides, nitrides, and carbonitrides together with C and N, and contribute to the improvement of strength. They also have an action to increase elongation and stretch-flanging performance bymicronizing crystal grains during hot rolling. When the total amount of these elements added is 0.01%, such an action is excessively lowered. In contrast, when the amount is higher than 1%, elongation and stretch-flanging performance are lowered rather than increased. For this reason, the lower limit of the total amount of one or more of these elements is 0.01%, and preferably 0.03%, while its upper limit is 1.0%, and preferably 0.7%.

[One or more members of Ni and Cu: in a total amount of 1% or lower]

**[0038]** These elements are effective in maintaining the balance of strength and ductility high and realizing high strength at the same time. To effectively exhibit such an effect, it is preferable to add the elements in an amount of 0.05% or higher. Meanwhile, the higher the amount of these elements contained, the higher the above-mentioned effect, but when the total amount of one or more of these elements is higher than 1%, such an effect is saturated, and cracks may occur during hot rolling. For this reason, the upper limit of the total amount of these elements is 1.0%, and preferably 0.7%.

40 [One or more members of Cr: 2% or lower, Mo: 1% or lower]

**[0039]** These elements are both effective in stabilizing the austenite phase, and facilitating the generation of bainite in the course of cooling. The higher the amount of the elements contained, the higher the effect, but when they are contained in an excessive amount, ductility is deteriorated rather than improved. For this reason, the amount of Cr is 2.0% or lower, and more preferably 1.5% or lower, while the amount of Mo is 1.0% or lower, and more preferably 0.7% or lower.

[B: 0.0001 to 0.005%]

[0040] B is an element effective in improving hardening characteristics, and increasing the strength of the steel sheet when added in a minute amount. To perform such an effect, it is preferable that the element is contained in an amount of 0.0001% or higher. However, when the amount of B contained is excessive and higher than 0.005%, crystal grain boundaries maybe embrittled and cracks may occur during rolling. For this reason, the upper limit of the amount of B is 0.005%.

[One or more members of Ca and REM: in a total amount of 0.003% or lower]

[0041] These elements are effective in controlling the form of sulfide in the steel and improving processability. The

higher the amount of the elements contained, the higher such an effect, but when they are contained in an excessively high amount, the above-mentioned effect is saturated. Therefore, the upper limit of the total amount of one or more members of these elements is 0.003%.

[0042] The method for manufacturing the high strength steel sheet according to an embodiment of the present invention will be now described. First, a material steel sheet which has the above-mentioned chemical components and a space factor of bainite to the entire structure of 90% or higher is prepared. Second, this material steel sheet is retained at a temperature of (Ac<sub>3</sub> point-100) °C or higher but not higher than Ac<sub>3</sub> for 0 sec. or longer but nor longer than 2400 sec., and then an annealing heat treatment is carried out, in which the material steel sheet is cooled to the martensite transformation start temperature, Ms point, or lower at an average cooling rate of 10°C/sec. or higher. Subsequently, a tempering heat treatment is carried out, in which the material steel sheet is retained at 300°C or higher but not higher than 550°C for 60 sec. or longer but not longer than 1200 sec., whereby a microstructure steel sheet mainly composed of the tempered martensite and annealed bainite and having a tensile strength of 590 MPa or higher is obtained.

[0043] The material steel sheet can be produced by the steps described below. First, steel having the above-mentioned chemical components is melted, By using the steel slab, hot rolling is terminated in such a manner that the finishing temperature is not lower than  $Ar_3$  point. Second, the steel slab is cooled at an average cooling rate of  $10^{\circ}$ C/sec. or higher to the bainite transformation temperature (about 350 to 450°C), and is wound up at the same temperature. When the finishing temperature is lower than  $Ar_3$  point or the cooling rate after the hot rolling is lower than  $10^{\circ}$ C/sec., a ferrite phase is likely to be produced in the hot-rolled steel sheet, and the space factor of bainite of the material steel sheet becomes lower than 90%. The material steel sheet used may be a cold-rolled steel sheet produced by hot rolling steel and then subj ecting the steel to an acid cleaning process and cold rolling. In the steel types which contain Ti, Nb, V and Zr, to re-solutionize precipitates containing the elements produced before hot rolling, it is preferable to heat and retain the steel piece at a relatively high temperature during hot rolling.

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**[0044]** As for the material steel sheet, the space factor of bainite can be made 90% or higher by subjecting a hot-rolled steel sheet which does not meet the above hot rolling condition and cooling condition to preliminary annealing. This preliminary annealing is a heat treatment in which a hot-rolled steel sheet is retained in a temperature range of  $Ac_3$  point or higher for about 5 seconds, and then the steel sheet is cooled at an average cooling rate of  $10^{\circ}$ C/sec. or higher to the bainite transformation temperature. When the retaining temperature is lower than  $Ac_3$  point, the ferrite phase is likely to be produced in the steel sheet, and the space factor of bainite is lowered. Even when the steel sheet is retained at a temperature  $Ac_3$  point or higher, if the retaining time is shorter than about 5 seconds, transformation into austenite is insufficient, and therefore the space factor becomes lower than 90%. Even when the steel sheet is subjected to the preliminary annealing, it can be cold-rolled thereafter to prepare as a cold-rolled steel sheet, and this can be used as the material steel sheet.

[0045] After the material steel sheet is prepared, the material steel sheet is retained at a temperature (Ac<sub>3</sub> point -100) °C or higher but not higher than Ac<sub>3</sub> for 0 sec. or longer (including 0 sec.) but not longer than 2400 sec., and then two-phase region annealing is carried out, in which the material steel sheet is cooled to the martensite transformation start temperature, Ms point, or lower at an average cooling rate of 10°C/sec. or higher, followed by tempering. By such a heat treatment, the structure of the high strength steel sheet according to the present invention is obtained. First, the conditions of the two-phase region annealing will be described below.

[0046] The reason why the annealing temperature of the two-phase region annealing is set to  $(Ac_3 \text{ point } -100)$  °C or higher but not higher than  $Ac_3$  is as follows: When the annealing temperature is set to a temperature range higher than  $Ac_3$  point in which the austenite single phase is stable, the crystal grains of austenite grow in the material steel sheet and combine with each other to become coarse, and the growth inhibitory effect (pinning effect) of austenite by finely dispersed annealed bainite cannot be obtained. For this reason, a fine dual phase steel sheet cannot be obtained, and the stretch-flanging performance of the high strength steel sheet is lowered. In contrast, if the steel sheet is annealed at a temperature lower than  $(Ac_3 \text{ point } -100)$  °C, transformation into austenite does not proceed sufficiently, and the space factor of martensite after the heat treatment becomes lower than 50%, and the stretch-flanging performance of the steel sheet is thus lowered.

**[0047]** As for the annealing time (heating and retaining time), austenite having a space factor of about 50% and thus martensite can be obtained simply by heating the steel sheet to the annealing temperature, but the time is preferably 1 sec. or longer, and more preferably 5 seconds or longer. In contrast, if the material steel sheet is retained longer than necessary, austenite grains become coarse, and fine martensite cannot be obtained. Therefore, it is preferable that the retaining time is limited to 2400 sec. or shorter, and preferably 1200 sec. or shorter.

**[0048]** When the average cooling rate after heating and retaining is lower than 10°C/sec. or the cooling stop temperature is higher than the martensite transformation start temperature, Ms point, a retained austenite phase, a pearlite phase and a ferrite phase are produced, a cementite phase is deposited, and structures other than martensite are formed from austenite in large amounts, whereby elongation and stretch-flanging performance are lowered.

**[0049]** Tempering (reheating treatment) is carried out after the two-phase region annealing, which is a process for improving elongation and stretch-flanging performance by softening hard martensite, and decomposing retained austen-

ite which produces martensite by working-induced transformation. Tempering conditions are as follows: the material steel sheet is retained at a temperature of 300°C or higher but not higher than 550°C for 60 sec. or longer but not longer than 1200 sec. The cooling rate after retaining is not especially limited.

**[0050]** When the tempering temperature is lower than 300°C, softening of martensite is insufficient, and the elongation and stretch-flanging performance of the steel sheet are lowered. In contrast, when the temperature is higher than 550°C, a coarse cementite phase is deposited, and the stretch-flanging performance of the steel sheet is lowered. For this reason, tempering is carried out at a temperature of 300°C or higher but not higher than 550°C.

**[0051]** When the retaining time of tempering is shorter than 60 sec., softening of martensite is insufficient, while when the time is longer than 1200 sec., martensite is too softened, which makes ensuring strength difficult, and deposition of cementite lowers the stretch-flanging performance of the steel sheet. For this reason, the lower limit of the retaining time during tempering is 60 sec., preferably 90 sec. or longer, and more preferably 120 sec., and the upper limit is 1200 sec., preferably 900 sec., and more preferably 600 sec.

**[0052]** The present invention will be described in more detail below with reference to Examples, but the present invention should not be interpreted as being restricted by such Examples.

(Example 1)

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[0053] Steel slabs having chemical compositions shown in Table 1 below were melted, and the steel slabs were heated to about 1000 to 1100°C. The steel slabs were subjected to hot rolling or further preliminary annealing under the conditions described in Table 2 below, producing material steel sheets. The average cooling rate after the hot rolling was 50°C/sec. test pieces for observing structures were collected from the material steel sheets, and the space factors of bainite were determined by observing structure constitutions with a microscope and subjecting microscope structure pictures after being corroded with nital to image analysis. The values of Ac<sub>3</sub> point and Ms point calculated from the components by a known equation are also shown in Table 1 for reference. Moreover, the results of structure observation are also shown in Table 2. The obtained material steel sheets were subjected to final annealing (two-phase region annealing) and tempering under the conditions shown in Table 3 below, producing sample steel sheets.

[Table 1]

Steel symbol		C	Chemica	compon	ents (% b	y mass)			ormation tures (°C)	Remarks
Symbol	С	Si	Mn	Р	S	Al	Others	Ac <sub>3</sub>	Ms	
А	0.11	1.21	1.62	0.011	0.001	0.044	-	873	448	Component of invention
В	0.18	1.54	2.06	0.120	0.002	0.048	-	934	406	Component of invention
С	0.01	0.88	1.56	0.012	0.002	0.044	-	908	487	Component of comparison
D	0.08	1.86	2.29	0.016	0.001	0.039	-	894	433	Component of invention
E	0.25	1.55	2.01	0.020	0.002	0.034	-	845	382	Component of invention
F	0.35	1.51	2.01	0.012	0.002	0.033	-	819	346	Component of comparison
G	0.18	0.05	2.05	0.009	0.001	0.031	-	783	406	Component of invention
Н	0.16	2.63	1.22	0.009	0.002	0.034	-	930	446	Component of invention
I	0.21	3.52	1.99	0.011	0.001	0.038	-	938	398	Component of comparison

(continued)

	Steel		С	hemical	compone	ents (% b	y mass)			ormation tures (°C)	Remarks
5	symbol	С	Si	Mn	Р	S	Al	Others	Ac <sub>3</sub>	Ms	
	J	0.14	1.54	0.38	0.009	0.003	0.039	-	913	486	component of comparison
10	К	0.13	1.56	0.62	0.009	0.001	0.038	-	909	480	Component of invention
	L	0.21	1.24	2.78	0.006	0.002	0.033	Zr:0.021	806	367	Component of invention
15	М	0.19	1.53	3.49	0.013	0.001	0.033	ı	808	346	Component of comparison
20	Z	0.17	1.38	2.02	0.015	0.002	0.005	V:0.018	842	409	component of invention
20	0	0.19	1.32	1.97	0,011	0.003	0.089	ı	865	407	Component of invention
25	Р	0.17	1.42	2.06	0.012	0.001	0.167	1	903	413	component of comparison
	Q	0.17	1.39	2.00	0.009	0.002	0.019	Ni:0.2	842	411	Component of invention
30	R	0.16	1.56	1.93	0.010	0.001	0.031	Cu:0.1	860	418	component of Invention
	S	0.17	1.33	2.19	0.012	0.002	0.042	Cr:0.35	841	397	Component of invention
35	Т	0.16	1.27	2.03	0.015	0.003	0.042	Mo:0.1	855	414	Component of invention
	U	0.18	1.36	1.93	0.016	0.003	0.045	B:0.0002	856	411	Component of invention
40	٧	0.17	1.40	1.97	0.014	0.002	0.039	Ca+REM: 0.001	855	413	Component of invention
	(Note) Rer	nainder	is Fe an	d inevita	able impu	rities					

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[Table 2]

		Hot-rollina	conditions	F	Preliminary anne	ealing condition	S	Material steel	sheet structure	
Sample No.	Steel symbol	Finishing temperature °C	Winding temperature °C	Heating temperature °C	Retaining time sec.	Cooling rate	Cooling stop temperature °C	Phase constitution	Space factor of bainite %	Remarks
1	А	930	550	900	120	50	400	Β+ γ	97	Conditions of invention
2	В	950	550	950	120	50	400	Β+ γ	94	Conditions of invention
3	С	870	500	930	90	20	420	Β+α+γ	85	Conditions of comparison
4	D	860	550	930	240	50	400	Β+ γ	96	Conditions of invention
5	E	890	550	900	180	50	370	Β+ γ	92	Conditions of invention
6	F	850	500	930	120	50	350	Β+ γ	91	Conditions of comparison
7	G	800	500	910	60	40	400	Β+ γ	100	Conditions of invention
8	Н	850	600	930	120	50	400	Β+ γ	91	Conditions of invention
9	-	900	550	930	120	50	400	Β+ γ	87	Conditions of comparison
10	J	900	500	930	60	50	400	Β+ γ	95	Conditions of comparison
11	К	900	550	930	120	50	430	Β+ γ	96	Conditions of invention
12	L	900	550	930	360	50	400	Β+ γ	94	Conditions of invention
13	М	850	500	900	120	50	400	B+r	92	Conditions of comparison

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(continued)

		Hot-rolling	conditions	F	Preliminary anne	ealing condition	s	Material steel	sheet structure	
Sample No.	Steel symbol	Finishing temperature °C	Winding temperature °C	Heating temperature °C	Retaining time sec.	Cooling rate	Cooling stop temperature °C	Phase constitution	Space factor of bainite %	Remarks
14	N	900	500	860	120	50	400	Β+ γ	95	Conditions of invention
15	0	900	500	880	30	50	430	Β+ γ	94	Conditions of invention
16	Р	900	550	930	180	50	400	Β+ γ	96	Conditions of comparison
17	Q	870	550	870	120	40	400	Β+ γ	93	Conditions of invention
18	R	880	550	890	120	50	400	Β+ γ	94	Conditions of invention
19	S	900	550	880	10	50	400	Β+ γ	94	Conditions of invention
20	Т	900	550	870	120	50	430	Β+ γ	96	Conditions of invention
21	U	900	550	900	120	50	400	Β+ γ	93	Conditions of invention
22	V	900	550	890	120	50	400	Β+ γ	94	Conditions of invention
23	В	930	400	_	1		1	Β+ γ	92	Conditions of invention
24	В	930	420					Β+ γ	91	Conditions of invention
25	А	900	400	_	_	_	_	Β+ γ	96	Conditions of invention
26	В	930	350	_	_	_	_	Β+ γ	96	Conditions of invention

(continued)

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		Hot-rolling	conditions	F	Preliminary anne	ealing condition	Material steel			
Sample No.	Steel symbol	Finishing temperature °C	Winding temperature °C	Heating temperature °C	Retaining time sec.	Cooling rate	Cooling stop temperature °C	Phase constitution	Space factor of bainite %	Remarks
27	А	930	430	_	_	_	_	В+ү	98	Conditions of Invention
(Note) α: Fer	rite. B: Bainite. 1	/: Austenite	•	•		•	•	•	•	

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[Table 3]

				[Iab	ie oj			
			Final annealing	g conditions		Tempering co	onditions	
Sample No.	Steelsymbol	Heating temperature	Retaining time	Cooling rate	Cooling stop temperature	Heating temperature	Retaining time	Remarks
		°C	sec.	°C/sec.	°C	°C	sec.	
1	Α	850	180	500	20	400	180	Conditions of invention
2	В	850	180	500	20	400	120	Conditions of invention
3	С	850	200	100	20	500	180	Conditions of comparison
4	D	870	180	200	20	500	180	Conditions of invention
5	E	815	80	300	20	520	120	Conditions of invention
6	F	810	220	300	20	350	180	Conditions of comparison
7	G	750	120	300	100	400	120	Conditions of invention
8	Н	910	350	300	50	500	180	Conditions of invention
9	I	870	100	200	20	350	120	Conditions of comparison
10	J	800	100	200	20	450	180	Conditions of comparison
11	К	850	180	500	20	520	180	Conditions of invention
12	L	770	120	300	20	500	180	Conditions of invention
13	М	770	180	200	20	400	180	Conditions of comparison

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				(conti	nued)			
			Final annealing	g conditions		Tempering of	onditions	
Sample No.	Steel symbol	Heating temperature	Retaining time	Cooling rate	Cooling stop temperature	Heating temperature	Retaining time	Remarks
		°C	sec.	°C/sec.	°C	°C	sec.	
14	N	820	120	500	20	500	180	Conditions of invention
15	0	850	180	300	20	500	180	Conditions of invention
16	Р	880	120	100	20	400	120	Conditions of comparison
17	Q	825	180	500	20	500	120	Conditions of invention
18	R	830	120	500	20	500	180	Conditions of invention
19	S	810	120	300	20	500	180	Conditions of invention
20	Т	850	60	300	20	500	180	Conditions of invention
21	U	820	180	500	20	500	180	Conditions of invention
22	V	830	120	500	20	500	180	Conditions of invention
23	В	880	180	300	20	450	180	Conditions of invention
24	В	900	120	300	20	500	120	Conditions of invention
25	А	850	180	300	20	450	180	Conditions of invention
26	В	850	180	300	20	500	180	Conditions of invention

(continued)

			Final annealing	g conditions	Tempering co			
Sample No. Steel symb		Heating temperature °C	Retaining time	Cooling rate	Cooling stop temperature °C	Heating temperature °C	Retaining time	Remarks
_					-	-		Conditions of
27	Α	800	120	500	20	500	180	invention

**[0057]** The structures (space factors of annealed bainite, space factors and mean grain sizes of tempered martensite), and mechanical characteristics (tensile strength TS, elongation EL and stretch-flanging performance) of the sample steel sheets were determined in the manner described below.

Test pieces for observing structures were collected from the sample steel sheets, and the space factors of annealed bainite and tempered martensite were determined by subjecting microscope structure pictures after being corroded with natal to image analysis. Moreover, the mean grain sizes of tempered martensite were determined by measuring the areas of the grains by structure analysis using FE/SEM-EBSP, determining the diameters of circles corresponding to the grains, and averaging the diameters.

Among the mechanical properties, tensile strength and elongation were determined by using a universal tensile tester manufactured by Instron and JIS No. 5 tensile test piece. Stretch-flanging performance was determined by measuring a hole expansion rate ( $\lambda$ ) by using a 20-ton hole expansion tester manufactured by Tokyo Koki, according to The Japan Iron and Steel Federation standard (JFST 1001-1996), and was evaluated based on this. The results of these measurements are also shown in Table 4. In Table 4, as for "evaluation", tensile strength (TS) of 590 MPa or higher, elongation (EL) of 10% or higher, and hole expansion rate ( $\lambda$ ) of 80% or higher were rated excellent characteristics. The samples which were excellent in all three characteristics were rated o; those which were excellent in two characteristics out of three were rated  $\lambda$ ; and those which were excellent in only one characteristic out of three were rated  $\lambda$ .

[0058]

### [Table 4]

					[Table	4]				
20			Stru	cture parame	eters		/lechanic			
25	Sample No.	Steel symbol	Space factor of annealed B	Space factor of tempered M	Mean grain size of tempered M	TS	EL	λ	Evaluation	Remarks
			%	%	μm	MPa	%	%		
30	1	Α	12	86	7.4	984	13.5	127.0	0	Example of invention
	2	В	29	70	8.3	689	32.1	80.8	0	Example of invention
35	3	С	19	80	8.1	554	31.9	81.5	Δ	Comparative example
	4	D	12	86	7.3	992	11.9	114.2	0	Example of invention
40	5	Е	13	84	7.8	1108	12.1	107.9	0	Example of invention
	6	F	10	89	8.3	1388	6.7	53.2	×	Comparative example
45	7	G	16	83	8.9	782	18.1	106.8	0	Example of invention
	8	Н	12	86	7.9	1022	12.9	104.0	0	Example of invention
50	9	I	25	76	9.1	1382	5.8	27.4	×	Comparative example
	10	J	35	65	8.8	588	28.8	64.9	×	Comparative example
55	11	K	22	75	8.2	603	28.3	86.3	0	Example of invention
	12	L	14	85	7.9	1109	12.5	100.5	0	Example of invention

(continued)

			Stru	cture parame	eters		/lechanic aracteris			
5	Sample No.	Steel symbol	Space factor of annealed B	Space factor of tempered M	Mean grain size of tempered M	TS	EL	λ	Evaluation	Remarks
10	40	N.4	% 12	%	μm	MPa	%	%		Comen amatica
	13	M	12	85	8.1	1299	8.1	58.7	×	Comparative example
15	14	Z	11	88	7.3	1031	13.9	124.0	0	Example of invention
	15	0	10	89	7.2	1017	14.7	127.9	0	Example of invention
20	16	Р	13	86	8.1	1031	10.3	61.1	Δ	Comparative example
	17	Q	10	89	8.0	1022	14.3	122.8	0	Example of invention
25	18	R	13	86	7.9	1098	12.9	121.4	0	Example of invention
	19	Ø	12	86	8.5	1139	10.9	114.9	0	Example of invention
30	20	Т	7	92	8.8	1222	10.7	98.7	0	Example of invention
	21	U	12	87	8.1	1154	11.1	104.8	0	Example of invention
<i>35</i>	22	V	12	86	7.9	1095	11.9	106.3	0	Example of invention
	23	В	13	86	7.9	989	13.2	112.8	0	Example of invention
40	24	В	12	87	7.7	981	14.3	127.3	0	Example of invention
,,	25	Α	12	86	7.5	789	17.5	117.7	0	Example of invention
45	26	В	24	75	8.7	708	19.8	103.2	0	Example of invention
45	27	Α	19	80	8.3	737	18.9	128.3	0	Example of invention
	(Note) B:	Bainite, M:	Martensite							

**[0059]** It can be seen from Table 4 that the sample steel sheets (examples of the invention) sample Nos. 1, 2, 4, 5, 7, 8, 11, 12, 14, 15 and 17 to 27 in which the conditions of the present invention were met in terms of all of chemical components, material steel sheet structures, final annealing conditions and tempering conditions all have tensile strengths as high as 590 MPa or higher, elongations of 10% or higher, and stretch-flanging performances of hole expansion rates of 80% or higher. That is, it can be seen that these samples have high strength and yet excellent elongation and stretch-flanging performance, and excellent press formability.

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[0060] other embodiments of the present invention will be described below in detail.

First, the composition of constituents of the high strength steel sheet material of this embodiment will be described. The elements which constitute the composition of the high strength steel sheet of this embodiment are C, Si, Mn, Al, Cr, Mo, Nb, Ti and V, and the remainder is Fe and inevitable impurities. Among these constituent elements, Cr, Mo, Nb, Ti and V are not essentially necessary constituent elements, but are the elements which are added to further increase the effect of the present invention. The actions of the elements will be described below. In the description provided below, the proportions of compositional ranges are indicated by % by mass.

[0061] Among the constituent elements mentioned above, the compositional range of C is limited within the range of 0.05% to 0.3%. C is an element effective in producing the tempered martensite phase, and increasing the strength of the steel sheet material. The lower limit value, i.e., 0.05% is an amount which is minimally necessary to obtain desired strength. The upper limit value, i.e., 0.3%, is limited for the following reason. When C is added in an amount higher than the upper limit value, 0.3%, the concentrations of C in the tempered martensite phase and the retained austenite phase are increased, and the strength of these phases is increased. A difference in strength between these phases and the ferrite phase having a low concentration of C is increased. Since rupture is likely to occur at the interface of these phases having a difference in strength, stretch-flanging performance is lowered. Meanwhile, when the concentration of C in the steel sheet is increased, weldability is significantly deteriorated.

**[0062]** The compositional range of Si is limited within the range higher than 0% but not higher than 3%. Si has the action to inhibit the generation of relatively coarse carbide which lowers stretch-flanging performance, and also improve ductility. However, this action to improve ductility is saturated in an amount of Si added of about 3%. Moreover, since Si has the action to retard softening by tempering of the tempered martensite phase, when the amount of Si contained is high, the tempered martensite phase is not sufficiently tempered and thus strength is retained high, whereby a difference in strength between the martensite phase and the ferrite phase is increased and stretch-flanging performance is lowered. Accordingly, the upper limit of the amount of Si added is 3%.

**[0063]** The compositional range of Mn is limited within the range of 0.5% or higher but not higher than 3%. Mn has the effect to increase the tensile strength of the steel sheet by solid solubility reinforcement, improve the hardening characteristics of the steel sheet, and promote generation of the martensite phase. Such an action of Mn is found in steel having containing Mn in an amount of 0.5% or higher. Preferably, the amount of Mn contained is 1% or higher. In contrast, when the amount of Mn contained is higher than 3%, adverse effects such as cracks in the slab occur. The amount of Mn contained is preferably 2.5% or lower.

[0064] The compositional range of AI is limited within the range of 0.01% or higher not higher than 0.1%. AI is used for deoxidation of steel in the steelmaking process. When there is no solid solution of AI present in the metal structure of steel, deoxidation of steel may not be completed. When oxygen is remaining in steel, remaining oxygen is bonded to Si and Mn. Since these oxidation products of Si and Mn are likely to separate and float from the cast, the composition of steel becomes non-uniform and processability is lowered. Moreover, when the amount of AI solutionized in the metal structure of steel is higher than 0.1%, deoxidation products are reduced by AI again, and metal-like AI is produced. this metal-like AI serves as a relatively large mediator, and creates material defects or surface flaws. Therefore, the upper limit value of AI is 0.1%.

**[0065]** Cr and Mo are not elements essential to the high strength steel sheet of the embodiment, but their addition acts effectively. Cr and Mo act to inhibit the generation of carbide which lowers stretch-flanging performance, and promote the generation of the martensite phase in the metal structure of the steel sheet. Therefore, they can be added as needed. The compositional range of Cr and Mo is such that at least one or more elements selected from Cr and Mo is contained, and the total compositional ratio of these elements is 0.5% or lower. In order to effectively perform the action of Cr and Mo, it is recommended that the compositional proportions of Cr and Mo are 0.05% or higher (more preferably 0.1% or higher), respectively. However, even if Cr and Mo are added, whether singly or in combination of both, in an amount higher than 0.5%, the action mentioned above is saturated, and an action which is worth the amount of Cr and Mo contained cannot be obtained.

**[0066]** None of Nb, Ti and V is an element which is essential to the high strength steel sheet of this embodiment, but their addition acts effectively. Nb, Ti and V have the action to form carbonitride, increase the tensile strength of steel by enhancing deposition, and micronize crystal grains in the metal structure of the steel sheet. Accordingly, these elements are added as needed. When the total amount added of one or more members selected from Nb, Ti and V is lower than 0.01%, the action of Nb, Ti and V mentioned above is not effective. In contrast, when the above-mentioned amount added is higher than 0.1% in total, too much precipitate is produced, and stretch-flanging performance is thus significantly lowered. Therefore, the upper limit of the total amount added mentioned above is 0.1%.

**[0067]** The high strength steel sheet of this embodiment may be composed to contain Ni or Cu in an amount of 1% by mass or lower in place of Cr, Mo, Nb, Ti and V. Moreover, it may be composed to contain B in an amount of 0.0001% by mass or higher but 0.0010% by mass or lower. Further, it may be composed to contain 0.003% by mass or less of

Ca and/or REM in total.

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[0068] The material of the high strength steel sheet of this embodiment is composed of Fe and inevitable impurities, in addition to the above-mentioned components. P and S are present as inevitable impurities, but they do not adversely affect the characteristics of the high strength steel sheet of this embodiment as long as the amount of P is 0.05% or lower (not including 0%) and the amount of S is 0.02% or lower (including 0%). The less the amount of P and S contained, the better the processability of the steel sheet. Especially, when the amount of S contained is high, MnS which serves as a mediator is increased in steel, whereby the stretch-flanging performance of the steel sheet is significantly lowered. [0069] Second, the metal structure of the high strength steel sheet of this embodiment will be described. The metal structure of the high strength steel sheet of this embodiment comprises a tempered martensite phase having a space factor of 80% or higher and a retained austenite phase having a space factor of 3% or lower, and the rest is mainly composed of a ferrite phase.

Among these constituent phases, the tempered martensite phase will be described first. When the space factor of the tempered martensite phase is 80% or higher, combination between austenite crystal grains and growth of the same can be suppressed by the annealed martensite phase remaining finely in part of the ferrite phase after the annealing step employed in the method for manufacturing the high strength steel sheet of the embodiment described later. When the space factor of the tempered martensite phase is lower than 80%, the tempered martensite phase is divided into ferrite phases, and therefore stretch-flanging performance is lowered. In contrast, when the phase becomes a substantially single-phase structure of tempered martensite having a space factor of the tempered martensite phase of 100%, ductility is lowered. For this reason, the case where the space factor is 100% is not included in the present invention.

[0070] In the tempered martensite phase of the high strength steel sheet of this embodiment, the mean grain size is 10  $\mu$ m or smaller, and the space factor of the tempered martensite phase having a grain size larger than 10  $\mu$ m is 15% or lower. When the mean grain size is larger than 10 $\mu$ m, or when the space factor of the tempered martensite phase having a grain size larger than 10  $\mu$ m is higher than 15%, the interfaces of the tempered martensite phase which act as the starting point of rupture are unevenly distributed, and therefore sufficient stretch-flanging performance cannot be obtained.

**[0071]** In the metal structure of the high strength steel sheet of this embodiment, the space factor of the retained austenite phase is 3% or lower. The retained austenite phase undergoes induced transformation in which it transforms into a tempered martensite phase during processing. Accordingly, the retained austenite phase lowers stretch-flanging performance. Therefore, in order to improve stretch-flanging performance, the space factor of the retained austenite phase needs to be limited to a low level. The space factor of the retained austenite phase is preferably 2% or lower, and more preferably 1% or lower.

[0072] The high strength steel sheet of the embodiment as described above has a fine tempered martensite phase formed therein and has a sufficiently low space factor of the retained austenite phase. Hence, it has excellent characteristics: it not only has high tensile strength, but also high elongation and stretch-flanging performance at the same time.

[0073] The method for manufacturing the high strength steel sheet of this embodiment will be now described.

First, materials of the high strength steel sheet of this embodiment will be described. The high strength steel sheet of this embodiment is obtained by subjecting a steel sheet material which meets predetermined conditions to a heat treatment comprising a predetermined annealing step and a tempering step.

The steel sheet material of the high strength steel sheet of this embodiment needs to meet the conditions of the composition of constituents mentioned above and also the conditions of the following metal structure. The steel sheet material of the high strength steel sheet of this embodiment needs to have a space factor of the martensite phase and the retained austenite phase of 90% or higher. Preferably, the space factor of the martensite phase and the retained austenite phase is 95% or higher. If the space factor of these constituent phases is lower than 90%, when the steel sheet is heated to a 2-phase region of the ferrite phase and austenite phase, a coarse austenite phase is produced. Therefore, the fine tempered martensite phase mentioned above cannot be obtained. This prevents improvement in stretch-flanging performance.

**[0074]** The steel sheet material of the high strength steel sheet of this embodiment having a space factor of the martensite phase and the retained austenite phase of 90% or higher is produced in the manner described below.

The process for manufacturing a steel sheet material of the high strength steel sheet of this embodiment (hereinafter referred to as "steel sheet material") comprises the following steps: A steel slab adjusted to meet the composition of constituents of the high strength steel sheet material mentioned above is hot-rolled at such a temperature that the finishing rolling temperature is  $Ac_3$  point or higher. this hot-rolled steel sheet is then cooled at a cooling rate of  $10^{\circ}$ C/sec. or higher to a cooling stop temperature, which is lower than Ms point at which the austenite phase starts to transform into the martensite phase (about 350°C or lower), and is wound up. When the finishing rolling temperature is  $Ac_3$  point or lower or the cooling rate after the hot rolling is  $10^{\circ}$ C/sec. or lower, the ferrite phase is likely to be produced during cooling after the hot rolling, and the space factor of the low-temperature transformation phase after the hot rolling does not become 90% or higher.

[0075] Even a steel sheet produced under conditions which do not meet those of the hot rolling and cooling rate

mentioned above from a steel slab adjusted to meet the composition of constituents of the steel sheet material can be modified into a steel sheet material having a space factor of its low-temperature transformation phase of 90% or higher by carrying out the following preliminary annealing. This preliminary annealing is a heat treatment in which the hot-rolled steel sheet is retained in a temperature range of  $Ac_3$  point or higher for 5 seconds or longer, and is cooled at a cooling rate of  $10^{\circ}$ C/sec. or higher to a cooling stop temperature of  $350^{\circ}$ C or lower. When the steel sheet mentioned above is retained in a temperature range of  $Ac_3$  point or lower, a ferrite phase is produced, and the space factor becomes no greater than 90%. Moreover, even when the steel sheet mentioned above is retained in a temperature range of  $Ac_3$  point or higher, transformation of the metal structure into austenite is insufficient in case where the retaining time is shorter than 5 seconds, and the space factor thus becomes no greater than 90%. As long as the conditions of this preliminary annealing are met, the upper limit of the annealing temperature, retaining time and cooling rate and the lower limit of the cooling stop temperature are not specifically determined.

[0076] The heat treatment step of the high strength steel sheet of this embodiment will be described now. The high strength steel sheet of this embodiment is obtained by subjecting a steel sheet material to a heat treatment comprising a predetermined annealing step and a tempering step. This annealing step is a heat treatment in which the steel sheet material is heated to a temperature of  $Ac_3$  point or lower but not lower than  $Ac_3$  point -50°, retained for 30 seconds or longer but not longer than 1200 seconds, and is then cooled at a cooling rate of 10°C/sec. or higher to Ms point or lower. By conducting this annealing step, the above-mentioned martensite phase having a space factor of 80% or higher is formed. Moreover, the size of austenite crystal grains produced when the steel sheet material is heated to and retained at a temperature of  $Ac_3$  point or lower but not lower than  $Ac_3$  point -50°affects the crystal grain size of the tempered martensite phase of the high strength steel sheet of the embodiment. That is, to obtain a fine tempered martensite having a mean grain size of 10  $\mu$ m or smaller and the space factor of the tempered martensite phase having a grain size larger than 10  $\mu$ m is 15% or lower phase as the high strength steel sheet of this embodiment, the steel sheet material needs to be heated to and retained at a temperature of  $Ac_3$  point or lower but not lower than  $Ac_3$  point -50°. A steel sheet having the metal structure in which such a fine tempered martensite phase is formed is characterized by high strength and high ductility.

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can be.

In this annealing step , when the steel sheet material is retained in a temperature range higher than  $Ac_3$  point at which the austenite single-phase is stable, crystal grains of austenite grow and combine with each other to be coarse. Therefore, the steel sheet material cannot be imparted a metal structure having a fine tempered martensite phase as the high strength steel sheet of this embodiment. As a result, the stretch-flanging performance of the high strength steel sheet is lowered. Moreover, when the steel sheet material is retained at a temperature lower than  $Ac_3$  point -50°C, transformation into austenite does not proceed sufficiently, and the space factor of the tempered martensite phase of the high strength steel sheet after the heat treatment becomes lower than that of the high strength steel sheet of this embodiment. As a result, the stretch-flanging performance of the high strength steel sheet is lowered. Therefore, the retaining temperature was set to  $Ac_3$  point or lower but not lower than  $Ac_3$  point -50°C.

[0077] When the retaining time in this annealing step is shorter than 30 seconds, the austenite phase is not sufficiently produced, and thus a fine martensite phase cannot be obtained after this annealing step. When the retaining time is longer than 1200 seconds, produced austenite crystal grains become coarse, and therefore the fine tempered martensite phase mentioned above cannot be obtained. Accordingly, the retaining time is to be in the range of 30 seconds or longer but not longer than 1200 seconds, Preferably, it is in the range of 120 seconds or longer but not longer than 600 seconds.

[0078] In this annealing step, when the cooling rate is 10°C/sec. or lower, or the cooling stop temperature is higher than Ms point at which the transformation from the austenite phase into the tempered martensite phase starts, generation of a bainite phase, retained austenite phase, pearlite phase and ferrite phase and deposition of a cementite phase are caused, and a number of phases other than the martensite phase are formed, whereby the space factor of the martensite phase cannot be increased. Accordingly, the stretch-flanging performance of the steel sheet is lowered. The higher the cooling rate, and the lower the cooling stop temperature, the higher the space factor of the tempered martensite phase

**[0079]** The tempering step will be described now. The steel sheet material which has undergone the annealing step is retained at a temperature of 300°C to 550°C for 60 seconds to 1200 seconds. A fine martensite phase is formed in the metal structure of the steel sheet material which has undergone the annealing step. The steel sheet material is softened by tempering this martensite phase to reduce a difference in hardness from the annealed martensite phase and ferrite phase, whereby excellent stretch-flanging performance, as well as ductility, can be obtained.

**[0080]** When the retaining temperature in this tempering step is lower than 300°C, the hardness of the tempered martensite phase is too high, and the stretch-flanging performance of the steel sheet is thus lowered. In contrast, when the retaining temperature is higher than 550°C, the cementite phase produced by the decomposition of the retained austenite phase becomes coarse, whereby the stretch-flanging performance of the steel sheet is lowered.

When the retaining time in this tempering step is shorter than 60 seconds, the hardness of the tempered martensite phase is too high, and therefore the elongation and stretch-flanging performance of the steel sheet are lowered. In contrast, When the retaining time is longer than 1200 seconds, the cementite phase become coarse, and the stretch-

flanging performance of the steel sheet is lowered. The retaining time in this tempering step is 60 seconds or longer but not longer than 1200 seconds, but it is preferably 90 seconds or longer but not longer than 900 seconds, and more preferably 120 seconds or longer but not longer than 600 seconds.

**[0081]** The steel sheet material which has been subjected to the annealing step and this tempering step becomes the high strength steel sheet of this embodiment, and is characterized by high stretch-flanging performance, in addition to high tensile strength and high ductility. Accordingly, this high strength steel sheet is used for various industrial products typically including automobiles as a steel sheet having excellent press formability.

(Example 2)

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**[0082]** The actions and effects of the high strength steel sheet of this embodiment and method for manufacturing the same will be described below with reference to Examples.

First, a method for preparing test steel sheets tested in these Examples will be described. In these Examples, Steel slabs having the compositions of constituents represented by steel symbols A to Y having the compositions of constituents shown in Table 5 were tested. As shown in Table 6 and 7, 56 types of test steel sheets were prepared under different hot rolling conditions, preliminary annealing conditions, and in different annealing steps and tempering steps from the steel slabs having the compositions of constituents of these A to Y, and the tensile strength, ductility, stretch-flanging performance and other characteristics of the test steel sheets were determined. Among the steel slabs having the compositions of constituents of A to Y, B, C, E, F, I, J, L, N to Y are the steel slabs having the compositions of constituents which fall within the Examples of the embodiment. Steel slabs having other compositions of constituents do not fall within the compositions of constituents of this embodiment. As can be seen from Tables 6 and 7, the test steel sheets prepared from these steel slabs are Comparative Examples. The steel slabs having the compositions of constituents of these A to Y, respectively, were hot-rolled at a finishing temperature of 850°C to give 56 types of test steel sheets having a thickness of 3 mm (Nos.1 to 56), which were then wound up at predetermined temperatures shown in Table 6. Furthermore, the test steel sheets No.1 to 45 were washed with acid to remove scales, and were cold-rolled to a thickness of 1.2 mm. The test steel sheets excluding test steel sheets 2 and 11 were then subjected to preliminary annealing under predetermined conditions shown in Table 6. Thereafter, test steel sheets Nos.1 to 56 were subjected to the heat treatment comprising the annealing step and the tempering step under predetermined conditions shown in Table 7, and were used as test steel sheets for measurements.

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[Table 5]

Steel symbol					Chem	ical comp	onents		ass)					on temperature C)	Remarks
	С	Si	Mn	Р	S	Al	Мо	Cr	Ti	Nb	V	Others	Ac <sub>3</sub>	Ms	
А	0.02	1.52	1.90	0.012	0.002	0.027							911	489	Comparative example
В	0.06	1.51	1.93	0.003	0.001	0.026							882	469	Example of invention
С	0.22	1.51	2.05	0.006	0.002	0.031							837	389	Example of invention
D	0.35	1.49	1.98	0.005	0.002	0.032							813	330	Comparative example
Е	0.18	0.05	2.03	0.008	0.001	0.029							782	409	Example of invention
F	0.16	2.88	2.05	0.008	0.002	0.033							915	418	Example of invention
G	0.21	3.25	2.08	0.018	0.001	0.028							924	393	Comparative example
Н	0.19	1.51	0.41	0.009	0.003	0.026							893	457	Comparative example
I	0.20	1.49	0.56	0.009	0.001	0.028							887	448	Example of invention
J	0.22	1.48	2.95	0.004	0.002	0.031							808	359	Example of invention
К	0.19	1.50	3.25	0.007	0.001	0.033							809	364	Comparative example
L	0.21	1.48	1.94	0.016	0.002	0.088							871	397	Example of invention
М	0.20	1.49	1.98	0.009	0.003	0.110							877	401	Comparative example

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(continued)

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Steel symbol					Chem	ical comp	onents	(% by m	ass)				Transformation temperature (°C)		Remarks
	С	Si	Mn	Р	S	Al	Мо	Cr	Ti	Nb	V	Others	Ac <sub>3</sub>	Ms	
N	0.18	1.50	2.08	0.015	0.001	0.031	0.20						858	403	Example of invention
0	0.19	1.52	1.92	0.012	0.002.	0.026	0.10	0.20					852	402	Examples of invention
Р	0.22	1.51	2.95	0.005	0.002	0.027			0.05				828	359	Examples of invention
Q	0.21	1.51	2.05	0.006	0.002	0.028				0.025			838	394	Example of invention
R	0.20	1.48	1.90	0.006	0.001	0.031					0.10		855	404	Example of invention
S	0.19	1.52	2.10	0.008	0.001	0.031			0.05		0.05		870	402	Example of invention
Т	0.20	1.47	2.04	0.015	0.003	0.027				0.024	0.05		850	399	Example of invention
U	0.18	1.49	1.99	0.009	0.002	0.048							856	409	Example of invention
V	0.17	1.43	2.03	0.011	0.003	0.051						Ni:0.2	854	408	Example of invention
W	0.16	1.52	1.96	0.012	0.003	0.047						Cu:0.1	863	416	Example of invention
Х	0.17	1.49	2.11	0.008	0.002	0.040						B: 0.003	851	408	Example of invention
Υ	0.18	1.38	1.89	0.011	0.003	0.045						Ca+REM: 0.001	855	413	Example of invention

(Note) Remainder is Fe and inevitable impurities

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[0084] As can be seen from Table 6, the steel sheets corresponding to Examples all have a space factor of the low-

	temperature transformation phase of 90% or higher, which falls within the conditions of the steel sheet material.
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[Table 6]

					[Table o]					
		Hot-rolling	conditions	F	Preliminary ann	ealing condition	s	Structure befo	ore annealing	
Test steel sheet No.	Steel symbol	Finish temperature	Winding temperature	Heating temperature	Retaining time (sec.)	Cooling rate (°C/sec.)	Cooling stop temperature (°C)	Phase constitution	Spacefactor of M+ retained γ (%)	Remarks
		( C)	( C)	( C)	(Sec.)	( C/Sec.)	( C)		(%)	
1	Α	850	500	930	120	600	20	M	100	Comparative example
2	В	850	300	-	-	-	-	α+M	85	Comparative example
3	В	850	500	930	120	600	20	M	100	Example of invention
4	С	850	500	800	120	600	20	α+M	80	Comparative example
5	С	850	500	930	120	600	20	M	100	Example of invention
6	С	850	500	930	2	600	20	α+Μ	80	Comparative example
7	С	850	500	930	10	600	20	α+M	90	Example of invention
8	С	850	500	930	600	600	20	M	100	Example of invention
9	С	850	500	930	120	5	20	α+Μ	70	Comparative example
10	С	850	500	930	120	600	500	В	0	Comparative example
11	С	850	300	-	-	-	-	α+Μ	95	Example of invention
12	D	850	500	930	120	600	20	М	100	Comparative example

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		Hot-rolling	conditions	F	Preliminary ann	ealing condition	s	Structure befo	ore annealing	
Test steel sheet No.	Steel symbol	Finish temperature	Winding temperature	Heating temperature	Retaining time (sec.)	Cooling rate (°C/sec.)	Cooling stop temperature (°C)	Phase constitution	Spacefactor of M+ retained γ (%)	Remarks
		( C)	( C)	( C)	(Sec.)	( C/Sec.)	( C)		(%)	
13	E	850	500	930	120	600	20	М	100	Example of invention
14	F	850	500	930	120	600	20	M	100	Example of invention
15	G	850	500	930	120	600	20	M	100	Comparative example
16	Н	850	500	930	120	600	20	M	100	Comparative example
17	I	850	500	930	120	600	20	M	100	Example of invention
18	J	850	500	930	120	600	20	M	100	Example of invention
19	К	850	500	930	120	600	20	M	100	Comparative example
20	L	850	500	930	120	600	20	M	100	Example of invention
21	М	850	500	930	120	600	20	M	100	Comparative example
22	N	850	500	930	120	600	20	M	100	Comparative example
23	N	850	500	930	120	600	20	M	100	Example of Invention
24	N	850	500	930	120	600	20	M	100	Example of invention
25	N	850	500	930	120	600	20	M	100	Comparative example

(continued)										
		Hot-rolling	conditions	F	Preliminary ann	ealing condition	s	Structure befo		
Test steel sheet No.	Steel symbol	Finish temperature	Winding temperature	Heating temperature	Retaining time	Cooling rate	Cooling stop temperature	Phase constitution	Spacefactor of M+ retained γ	Remarks
		(°C)	(°C)	(°C)	(sec.)	(°C/sec.)	(°C)		(%)	
26	N	850	500	930	120	600	20	М	100	Comparative example
27	N	850	500	930	120	600	20	M	100	Example of invention
28	N	850	500	930	120	600	20	M	100	Example of invention
29	N	850	500	930	120	600	20	M	100	Comparative example
30	N	850	500	930	120	600	20	M	100	Comparative example
31	N	850	500	930	120	600	20	M	100	Comparative example
32	N	850	500	930	120	600	20	M	100	Comparative example
33	N	850	500	930	120	600	20	M	100	Example of invention
34	N	850	500	930	120	600	20	M	100	Example of invention,
35	N	850	500	930	120	600	20	M	100	Comparative example
36	N	850	500	930	120	600	20	M	100	Comparative example
37	N	850	500	930	120	600	20	M	100	Example of invention
38	N	850	500	930	120	600	20	М	100	Example of invention

(continued)

					(continued)					
		Hot-rolling	conditions	F	Preliminary ann	ealing condition	S	Structure befo	ore annealing	
Test steel sheet No.	Steel symbol	Finish temperature	Winding temperature (°C)	Heating temperature	Retaining time (sec.)	Cooling rate	Cooling stop temperature	Phase constitution	Spacefactor of M+ retained γ (%)	Remarks
39	N	850	500	930	120	600	20	M	100	Comparative example
40	0	850	500	930	120	600	20	М	100	Example of invention
41	Р	850	500	930	120	600	20	M	100	Example of invention
42	Q	850	500	930	120	600	20	M	100	Example of invention
43	R	850	500	930	120	600	20	M	100	Example of invention
44	S	850	500	930	120	600	20	M	100	Example of invention
45	Т	850	500	930	120	600	20	M	100	Example of invention
46	U	900	500	930	780	700	20	M	100	Example of invention
47	V	900	500	930	120	700	20	M	100	Example of invention
48	W	900	500	930	120	700	20	M	100	Example of invention
49	Х	900	500	930	180	700	20	M	100	Example of invention
50	Y	900	500	930	240	700	20	M	100	Example of invention
51	U	900	250	-	-	-	-	M	100	Example of invention

(continued)

	Hot-rolling	conditions	F	Preliminary ann	ealing condition	S	Structure befo		
Steel symbol	Finish temperature	Winding temperature (°C)	Heating temperature	Retaining time (sec.)	Cooling rate (°C/sec.)	Cooling stop temperature (°C)	Phase constitution	Spacefactor of M+ retained γ (%)	Remarks
U	950	300	-	-	-	-	М	100	Example of invention
U	900	200	-	-	-	-	М	100	Example of invention
V	920	280	-	-	-	-	М	100	Example of invention
W	920	300	-	-	-	-	М	100	Example of invention
Х	920	300	-	-	-	-	М	100	Example of invention
	symbol  U  V  W	Steel symbol Finish temperature (°C)  U 950  U 900  V 920  W 920	symbol         temperature (°C)         temperature (°C)           U         950         300           U         900         200           V         920         280           W         920         300	Steel symbol         Finish temperature         Winding temperature         Heating temperature           (°C)         (°C)         (°C)           U         950         300         -           U         900         200         -           V         920         280         -           W         920         300         -	Steel symbol         Finish temperature         Winding temperature         Heating temperature         Retaining time           U         950         300         -         -           U         900         200         -         -           V         920         280         -         -           W         920         300         -         -	Steel symbol         Finish temperature         Winding temperature         Heating temperature         Retaining time         Cooling rate           U         950         300         -         -         -         -           U         900         200         -         -         -         -           V         920         280         -         -         -         -           W         920         300         -         -         -         -	Steel symbol         Finish temperature         Winding temperature         Heating temperature         Retaining time         Cooling rate         Cooling stop temperature           U         950         300         -         -         -         -         -         -           U         900         200         -         -         -         -         -           V         920         280         -         -         -         -         -           W         920         300         -         -         -         -         -	Steel symbol         Finish temperature         Winding temperature         Heating temperature         Retaining time         Cooling rate         Cooling stop temperature         Phase constitution           U         950         300         -         -         -         -         -         M           U         900         200         -         -         -         -         M           V         920         280         -         -         -         -         M           W         920         300         -         -         -         -         M	Steel symbolFinish temperature (°C)Winding temperature (°C)Heating temperature (°C)Retaining time (sec.)Cooling rate (°C/sec.)Cooling stop temperature (°C/sec.)Phase constitution of M+ retained γ (%)U950300M100U900200M100V920280M100W920300M100

(Note) M: Martensite phase,  $\alpha$ : Ferrite phase, B: Bainite phase, Retained  $\gamma$ : Retained austenite phase

[0085] As shown in Table 7, among the 56 types of test steel sheets prepared, 33 types correspond to Examples

	corresponding to the present embodiment, while others are Comparative Examples.
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[Table 7]

				[Table /]				
			Annealing of	conditions		Tempering	conditions	
Test steel sheet No.	Steel symbol	Heating temperature	Retaining time	Cooling rate	Cooling stop temperature	Heating temperature	Retaining time	Remarks
		(°C)	(sec)	(°C/sec.)	(°C)	(°C)	(sec.)	
1	А	880	180	600	20	450	180	Comparative example
2	В	850	180	600	20	450	180	Comparative example
3	В	850	180	600	20	450	180	Examples of invention
4	С	800	180	600	20	450	180	Comparative example
5	С	800	180	600	20	450	180	Example of invention
6	С	800	180	600	20	450	180	Comparative example
7	С	800	180	600	20	450	180	Example of invention
8	С	800	380	600	20	450	180	Example of invention
9	С	800	180	600	20	450	180	Comparative example
10	С	800	180	600	20	450	180	Comparative example
11	С	800	180	600	20	450	180	Examples of invention
12	D	780	180	600	20	450	180	Comparative example
13	Е	750	180	600	20	450	180	Example of invention

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(continued)

		Annealing conditions				Tempering conditions		
Test steel sheet No.	Steel symbol	Heating temperature	Retaining time	Cooling rate	Cooling stop temperature	Heating temperature	Retaining time	Remarks
		(°C)	(sec)	(°C/sec.)	(°C)	(°C)	(sec.)	
14	F	880	180	600	20	450	180	Example of invention
15	G	880	180	600	20	450	180	Comparative example
16	Н	850	180	600	20	450	180	Comparative example
17	I	850	180	600	20	450	180	Example of invention
18	J	780	180	600	20	450	180	Example of invention
19	К	780	180	600	20	450	180	Comparative example
20	L	850	180	600	20	450	180	Example of invention
21	М	850	180	600	20	450	180	Comparative example,
22	N	900	180	600	20	450	180	Comparative example
23	N	840	180	600	20	450	180	Example of invention
24	N	820	180	600	20	450	180	Example of invention
25	N	780	180	600	20	450	180	Comparative example
26	N	820	20	600	20	450	180	Comparative example

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		Annealing conditions				Tempering conditions		
Test steel sheet No.	Steel symbol	Heating temperature	Retaining time	Cooling rate	Cooling stop temperature	Heating temperature	Retaining time	Remarks
		(°C)	(sec)	(°C/sec.)	(°C)	(°C)	(sec.)	
27	N	820	50	600	20	450	380	Example of invention
28	N	820	1000	600	20	450	180	Example of invention
29	N	820	1500	600	20	450	180	Comparative example
30	N	820	180	5	20	450	180	Comparative example
31	N	820	180	600	500	450	180	Comparative example
32	N	820	180	600	20	250	180	Comparative example
33	N	820	180	600	20	350	180	Example of invention
34	N	820	180	600	20	550	180	Example of invention
35	N	820	180	600	20	650	180	Comparative example
36	N	820	180	600	20	450	40	Comparative example
37	N	820	180	600	20	450	60	Examples of invention
38	N	820	180	600	20	450	1000	Example of invention
39	N	820	180	600	20	450	1500	Comparative example

(continued)

				(continued	)			
		Annealing conditions				Tempering conditions		
Test steel sheet No.	Steel symbol	Heating temperature	Retaining time	Cooling rate	Cooling stop temperature	Heating temperature	Retaining time	Remarks
		(°C)	(sec)	(°C/sec.)	(°C)	(°C)	(sec.)	
40	0	820	180	600	20	450	180	Example of invention
41	Р	800	180	600	20	450	180	Example of invention
42	Q	800	180	600	20	450	180	Example of invention
43	R	820	180	600	20	450	180	Example of invention
44	S	850	180	600	20	450	180	Example of invention
45	Т	820	180	600	20	450	180	Examples of invention
46	U	840	120	700	20	500	180	Example of invention
47	V	840	120	700	20	500	180	Example of invention
48	W	850	180	300	20	400	120	Example of invention
49	X	820	120	700	20	500	180	Example of invention
50	Y	820	180	500	20	450	120	Example of invention
51	U	840	120	700	20	500	180	Example of invention
52	U	820	240	500	50	500	240	Example of invention

# (continued)

		Annealing conditions				Tempering conditions		
Test steel sheet No.	Steel symbol	Heating temperature	Retaining time	Cooling rate	Cooling stop temperature	Heating temperature	Retaining time	Remarks
		(°C)	(sec)	(°C/sec.)	(°C)	(°C)	(sec.)	
53	U	820	180	500	20	500	180	Example of invention
54	V	840	120	500	20	400	180	Example of invention
55	W	850	180	500	20	400	180	Example of invention
56	Х	820	120	500	20	400	180	Example of invention

[0086] The 56 types of the test steel sheets prepared by these steps were tested for their tensile strength and stretch-flanging performance.

The tensile strength test was performed in such a manner that the direction perpendicular to the rolling direction of each of the test steel sheets is the direction of pulling during testing by using a JIS No. 5 test piece collected from each test steel sheet according to JIS Z2241. In this test, yield strength YS, tensile strength TS and elongation EL were determined. The stretch-flanging performance test was performed according to Japan Iron and Steel Federation standard (JFST 1001-1996), and hole expansion rates  $\lambda$  were determined.

[0087] The measurement results of the characteristics of the 56 types of test steel sheets are shown in Tables 8 and 9. In Tables 8 and 9, AM represents an annealed martensite phase; TM represents a tempered martensite phase; and retained  $\gamma$  represents a retained austenite phase. The space factor of the retained austenite phase was indicated 0% when it was below the detection limit.

In the results of this test, when the tensile strength is 780 MPa or higher, it is a sufficient strength characteristic in practical use, and meets the conditions of the tensile strength of the present invention. Moreover, elongation (ductility) and stretch-flanging performance are considered excellent when they are 10% or higher and 80% or higher, respectively. The stretch-flanging performance is judged especially excellent characteristics when it is 100% or higher.

Test steel sheets which meet all the following conditions are considered to correspond to high strength steel sheets according to the present invention: tensile strength: TS $\geq$ 780 MPa, elongation: EL $\geq$ 10%, hole expansion rate:  $\lambda \geq$ 80%. A test steel sheet which meets all these three conditions and has especially good hole expansion rate ( $\lambda \geq$ 100%) was rated  $\odot$ ; a test steel sheet which meets all the conditions was rated O; a test steel sheet which meets two conditions out of three was rated  $\Delta$ ; and a test steel sheet which meets only one condition or less out of three conditions was rated  $\times$ .

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# [Table 8]

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			Metal structu	re parameters		Dyı	namic cha	racterist	ics		
Test steel sheet No.	Steel symbol	Mean grain size of TM	Space factor of TM	Space factor of retained γ	Space factor of TM having grain size of 10 µm or larger	YS (MDa)	TS (MDs)	EL	λ	Judgment	Remarks
		(μm)	(%)	(%)	(%)	(MPa)	(MPa)	(%)	(%)		
1	A	6.8	89	0	8	365	608	32.2	70.1	×	Comparative example
2	В	8.8	87	0	18	523	752	28.7	82.7	Δ	Comparative example
3	В	7.0	89	0	7	598	801	26.5	122.2	<b>©</b>	Example of invention
4	С	9.2	88	0	18	961	1011	8.5	61.2	×	Comparative example
5	С	6.2	88	0	11	953	1052	11.6	105.2	0	Example of invention
6	С	11.4	88	0	20	811	1084	7.6	45.4	×	Comparative example
7	С	9.7	88	0	12	862	1098	12.1	83.4	0	Example of invention
8	С	7.0	88	0	10	873	1065	12.7	103.8	0	Example of invention
9	С	9.8	88	0	18	795	1035	8.9	65.2	×	Comparative example
10	С	11.0	88	0	20	888	1069	8.6	75.8	×	Comparative example
11	С	8.4	88	0	12	996	1127	12	98.9	0	Examples of invention
12	D	6.6	89	0	7	887	1220	11.2	76 2	Δ	Comparative example

(continued)

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			Metal structu	re parameters		Dyı	namic cha	racterist	ics		
Test steel sheet No.	Steel symbol	Mean grain size of TM	Space factor of TM	Space factor of retained γ	Space factor of TM having grain size of 10 µm or larger	YS (MDa)	TS (MBo)	EL (%)	λ	Judgment	Remarks
		(μm)	(%)	(%)	(%)	(MPa)	(MPa)	(%)	(%)		
13	E	7.6	89	0	11	778	967	13.9	90.5	0	Examples of invention
14	F	6.6	89	0	8	898	1263	15.8	114.2	0	Examples of invention
15	G	8.0	87	0	8	1091	1313	10.3	67.7	Δ	Comparative example
16	Н	7.2	87	6	11	932	1011	8.3	46.2	×	Comparative example,
17	I	6.2	88	3	6	963	1081	10.3	89.1	0	Examples of invention
18	J	7.4	90	0	8	1011	1122	11.3	81.1	0	Examples of invention
19	К	6.8	90	0	11	1194	1271	7.3	48.3	×	Comparative example,
20	L	7.6	91	0	7	785	823	34.7	106.4	0	Example of invention
21	M	8.2	78	0	10	763	845	36.2	65.4	Δ	Comparative example
22	N	12.7	100	0	62	1120	1265	8.1	81.7	Δ	Comparative example
23	N	7.0	92	0	13	996	1063	12.2	88.3	0	Example of invention
24	N	8.6	88	0	11	902	1003	15.7	86.9	0	Example of invention

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# (continued)

			Metal structu	re parameters		Dyı	namic cha	racterist	ics		
Test steel sheet No.	Steel symbol	Mean grain size of TM	Space factor of TM	Space factor of retained γ	Space factor of TM having grain size of 10 µm or larger	YS	TS	EL	λ	Judgment	Remarks
		(μm)	(%)	(%)	(%)	(MPa)	(MPa)	(%)	(%)		
25	N	8.4	72	0	12	855	980	17.8	58.7	Δ	Comparative example
26	N	7.0	62	0	9	913	969	15.2	61.7	Δ	Comparative example
27	N	8.0	81	0	10	909	1029	12.2	91.5	0	Example of invention
28	N	9.4	88	0	13	1056	1101	10.4	85.4	0	Example of invention
29	Ν	11.7	88	0	24	1101	1248	7.2	96.0	Δ	Comparative example
30	N	8.0	72	0	7	678	758	16.7	80.2	Δ	Comparative example
31	N	8.2	23	8	10	798	866	20.2	34.4	Δ	Comparative example
32	N	7.2	88	6	5	1229	1499	8.2	39.3	×	Comparative example
33	N	7.4	88	2	6	1129	1225	10.2	83.8	0	Example of invention
34	N	7.2	88	0	12	1078	1158	11.4	89.2	0	Example of invention
35	N	9.6	88	0	16	597	778	16.7	45.0	Δ	Comparative example
36	N	7.0	88	7	6	826	1205	12.3	40.6	Δ	Comparative example

# (continued)

			Metal structu	re parameters		Dyı	namic cha	racterist	ics		
Test steel sheet No.	Steel symbol	Mean grain size of TM	Space factor of TM	Space factor of retained γ	Space factor of TM having grain size of 10	YS	TS	EL	λ	Judgment	Remarks
		(µm)	(%)	(%)	(%)	(MPa)	(MPa)	(%)	(%)		
37	N	6.9	88	3	7	972	1258	10.3	85.6	0	Example of invention
38	N	7.2	88	0	7	1023	1181	11.5	91.3	0	Example of invention
39	N	10.2	88	0	17	606	798	16.5	51.6	Δ	Comparative example
40	0	6.2	89	0	7	1014	1150	12.3	86.5	0	Example of invention
41	Р	6.4	90	0	6	987	1097	11.9	82.2	0	Example of invention
42	Q	6.2	88	0	6	960	983	10.3	98.3	0	Example of invention
43	R	6.4	88	0	8	936	952	10.9	96.2	0	Example of invention
44	S	6.6	92	0	8	1023	1095	10.4	88.5	0	Example of invention
45	Т	6.3	89	0	7	1008	1109	13.3	83.4	0	Example of invention

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[Table 9]

			Meta	al structure paran	neters		Dynami	c charac	teristics		
Test steel sheet No	Steel symbol	Space factor of AM	Mean grain size of TM	Space factor of TM	Space factor of retained γ	Space factor of TM having grain size of 10 µm or larger	TS	EL	λ	Judgment	Remarks
		(%)	(μm)	(%)	(%)	(%)	(MPa)	(%)	(%)		
46	а	11	8.3	89	0	10	813	31.5	108.3	0	Example of invention
47	b	10	8.5	90	0	9	837	30.8	98.9	0	Example of invention
48	С	10	8.6	90	0	8	870	283	92.1	0	Example of invention
49	d	15	7.2	85	0	12	860	28.3	93.7	0	Example of invention
50	е	16	7.4	84	0	11	887	21.9	97.5	0	Example of invention
51	а	11	8.3	89	0	9	844	28.9	92.4	0	Example of invention
52	а	15	7.4	85	0	13	825	30.1	89.3	0	Example of invention
53	а	16	7.6	84	0	13	838	26.9	96.6	0	Example of invention
54	b	9	8.1	91	0	7	913	17.3	115.0	0	Example of invention
55	С	10	8.4	90	0	8	942	15.3	109.3	0	Example of invention
56	d	14	7.5	86	0	11	938	14.9	107.4	0	Example of invention

[0090] The results of the measurement of test steel sheet characteristics in Table 8 will be described below.

The test steel sheets No.3, 5, 7, 8, 11, 13, 14, 17, 18, 20, 23, 24, 27, 28, 33, 34, 37, 38 and 40 to 45 are all prepared from steel slabs (B, C, E, F, I, J, L, N to T in Table 5) corresponding to the compositions of constituents of the high strength steel sheet of this embodiment. Moreover, as can be seen from Tables 6 and 7, the space factor of the martensite phase and the retained austenite phase of the metal structures of these test steel sheets before the annealing step, the annealing step and the tempering step correspond to the conditions of the high strength steel sheet of this embodiment. All of these test steel sheets meet the conditions of the tensile strength, elongation and stretch-flanging performance of the present invention.

**[0091]** The test steel sheets (Nos.46 to 56) of Table 9 all meet the conditions of the tensile strength, elongation and stretch-flanging performance of the present invention.

[0092] Among the test steel sheets which correspond to the high strength steel sheet of the embodiment, Nos. 3, 5, 8, 14 and 20 have especially good stretch-flanging performance. The space factor of the retained austenite phase of these test steel sheets is 0%. The tempered martensite phases of these test steel sheets have relatively small mean grain sizes, and the space factor of the tempered martensite phase having a crystal grain size of 10  $\mu$ m or larger is relatively low.

[0093] The reason why the test steel sheets of Comparative Examples did not meet the conditions of the high strength steel sheet according to the present invention will be described.

Since the test steel sheet No.1 was prepared from steel slab A having low level of C, it has low tensile strength.

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Test steel sheet No.2 had low space factors of the martensite phase and the retained austenite phase in the metal structure in a state that it is yet to be annealed were low. Therefore, crystal grains of the tempered martensite phase became coarse, and the strength and stretch-flanging performance were lowered.

Test steel sheet No.4 was subjected to the preliminary annealing at a temperature lower than  $Ac_3$  point, and therefore the space factor of the low-temperature transformation phase in the metal structure in a state that it is yet to be annealed was lowered. This caused crystal grains of the tempered martensite phase to be coarse, whereby ductility and stretch-flanging performance are low.

**[0094]** The test steel sheet No.6 had low space factors of the martensite phase and the retained austenite phase in the metal structure in a state that it was yet to be annealed because the retaining time in the preliminary annealing was short, and therefore crystal grains in the tempered martensite phase became coarse. As a result, it had low elongation and stretch-flanging performance.

The test steel sheet No.9 had low space factors of the martensite phase and the retained austenite phase in the metal structure in a state that it was yet to be annealed because cooling after the preliminary annealing was delayed, and therefore the tempered martensite phase became coarse. As a result, it had low elongation and stretch-flanging performance.

The test steel sheet No.10 had low space factors of the martensite phase and the retained austenite phase in the metal structure in a state that it was yet to be annealed because the cooling stop temperature after the preliminary annealing was high, and therefore the tempered martensite phase became coarse. As a result, it had low elongation and stretch-flanging performance.

Although the test steel sheet No.12 has a metal structure after the tempering step corresponding to that of the high strength steel sheet of the embodiment, the difference in strength between the annealed martensite phase which is a part of the ferrite phase and the tempered martensite phase has not been sufficiently reduced because this test steel sheet was prepared from steel slab D having a high level of C. As a result, it had low stretch-flanging performance.

**[0095]** Although the test steel sheet No.15 has a metal structure after the tempering step corresponding to that of the high strength steel sheet of the embodiment, this test steel sheet was prepared from steel slab G having a high level of Si. Accordingly, the tempered martensite phase was not sufficiently tempered, and the difference in strength between the annealed martensite phase which is a part of the ferrite phase and the tempered martensite phase has not been sufficiently reduced. As a result, it had low stretch-flanging performance.

Since the test steel sheet No.16 was prepared from steel slab H having a low level of Mn, it has insufficient hardening characteristics, and therefore a large amount of retained austenite remained after the annealing step. As a result, it had low elongation and stretch-flanging performance.

Since the test steel sheet No.19 was prepared from steel slab K having a high level of Mn, uneven distribution of Mn occurred although the space factors and the size of the martensite phase and the retained austenite phase in the metal structure after the tempering step correspond to that of the high strength steel sheet of the embodiment. As a result, it had low elongation and stretch-flanging performance.

The test steel sheet No. 21 was prepared from steel slab M having a high amount of Al added. Accordingly, it had a number of flaws on the surface of the steel material. As a result, it had low stretch-flanging performance.

**[0096]** The test steel sheet No. 22 had coarse crystal grains of the austenite phase since it was heated to a temperature higher than  $Ac_3$  point in the annealing step. As a result, its ductility was lowered.

In the test steel sheet No. 25, the austenite phase was not sufficiently produced because the heating and retaining

temperature in the annealing step was lower than  $Ac_3$  point -50°C. As a result, it had low space factor of the tempered martensite phase, and low stretch-flanging performance.

In the test steel sheet No. 26, the austenite phase was not sufficiently produced because the retaining time at a temperature of  $Ac_3$  point or lower but not higher than  $Ac_3$  point -50°C in the annealing step was too short. As a result, it had low space factor of the martensite phase and low stretch-flanging performance.

In the test steel sheet No. 29, the crystal grains of the austenite phase became coarse because the retaining time at a temperature of  $Ac_3$  point or lower but not higher than  $Ac_3$  point -50°C in the annealing step was too long. As a result, it had large crystal grain size of the martensite phase, and low ductility.

**[0097]** In the test steel sheet No. 30, the tempered martensite phase was not sufficiently generated because cooling after the annealing step was too late and thus tempered phases other than the martensite phase were produced. As a result, it had low tensile strength.

In the test steel sheet No. 31, the martensite phase was not sufficiently produced because the cooling stop temperature after the annealing step was higher than Ms point. As a result, it had low space factor of the tempered martensite phase, and low stretch-flanging performance.

In the test steel sheet No. 32, the dislocation density of the tempered martensite phase was not lowered because the heating and retaining temperature in the tempering step was lower than the lower limit value, and distortion was not sufficiently mitigated. As a result, it had low elongation and stretch-flanging performance.

**[0098]** In the test steel sheet No. 35, cementite was deposited because the heating and retaining temperature in the tempering step was higher than the upper limit value. As a result, it had low stretch-flanging performance.

In the test steel sheet No. 36, the space factor of the retained austenite phase was not sufficiently lowered because the heating and retaining time in the tempering step was too short. Moreover, the dislocation density of the tempered martensite phase was not lowered, and distortion was not

sufficiently mitigated. As a result, it had low stretch-flanging performance.

In the test steel sheet No. 39, cementite was deposited because the heating and retaining time in the tempering step was too long. As a result, it had low stretch-flanging performance.

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[0099] Still another embodiment of the present invention will be described in detail below.

The inventors of the present invention studied the requirements for obtaining both strength and elongation, which are the features of a dual phase steel sheet (DP steel sheet), and also stretch-flanging performance by presupposingly using this DP steel sheet comprising the ferrite phase and martensite, from various angles. As a result, the inventors of the present invention found that a very fine ferrite + martensite dual structure can be obtained by subjecting a steel sheet having a fine lath-shaped structure (martensite and/or bainite) as a material steel sheet (that is, as an initial structure) to annealing (hereinafter referred to as "dual-phase range annealing") in a dual-phase range (ferrite +austenite range). Moreover, the inventors of the present invention also found that a steel sheet having such a structure has good elongation and stretch-flanging performance.

**[0100]** In a steel sheet having the fine lath-shaped structure (martensite and/or bainite) as mentioned above, ferrite produced by the dual-phase range annealing is finely dispersed, and the growth of austenite during the dual-phase range annealing is suppressed by its pinning effect. Accordingly, its structure after hardening becomes a very fine ferrite + martensite structure. In addition, crystal grain micronizing elements such as Ti, Nb, V and Zr are added to the steel sheet as chemical components, whereby further micronization of the structure can be achieved. The thus-obtained dual phase steel sheet is imparted further improved elongation and stretch-flanging performance.

**[0101]** The high strength steel sheet of the present invention is a dual phase steel sheet which is mainly composed of a ferrite phase and martensite. In order to achieve the above object, it is necessary that the space factors of these phases to the entire structure are adjusted appropriately. That is, in the high strength steel sheet of the present invention, the space factors of the ferrite phase and martensite are 5 to 30% and 50 to 95%, respectively.

**[0102]** When the space factor of the ferrite phase is 5% or lower, good elongation cannot be ensured, and the pinning effect which suppresses the growth of austenite becomes weak. When the space factor is higher than 30%, the stretch-flanging performance is deteriorated. A preferable space factor of the ferrite phase is 7% or higher but not higher than 25%. **[0103]** When the space factor of martensite is lower than 50%, the stretch-flanging performance is lowered. When the space factor is higher than 95%, elongation is lowered. A preferable space factor of the martensite phase is 70% or higher but not higher than 85%.

**[0104]** It should be noted that the term "space factor" mentioned above means the ratio (% by volume) of each phase constituting a metal structure in the steel material to the entire structure, and the space factors of the ferrite phase and martensite can be determined by corroding a steel material with nital, observing the material with an optical microscope (1000 times), and then subjecting the material to image analysis.

[0105] In the high strength steel sheet of the present invention, it is preferable that the mean grain size of the above

ferrite phase is 3  $\mu$ m or smaller in terms of the equivalent of a circle diameter, and that the mean grain size of the martensite phase is 6  $\mu$ m or smaller in terms of the equivalent of a circle diameter. If these sizes are increased, elongation and stretch-flanging performance are lowered. The "mean grain sizes" of these phases are determined, for example, by measuring the grain sizes of twenty grains by observing the structure using an optical microscope and FE/SEM-EBSP, and averaging the measurements.

**[0106]** The dual phase steel sheet according to the present invention is composed of a ferrite phase and martensite as its main structure, but it is not necessarily 100% composed of these phases, and it is also allowed that at least the total sum is 70% or higher, preferably 80% or higher, in terms of space factor, due to the intention that it is merely the main part, and that bainite, pearlite, retained austenite and the like are contained. However, the less these structures, the better, from the standpoint of not reducing stretch-flanging performance.

**[0107]** In the steel sheet of the present invention, the structure is controlled in the above-mentioned manner, whereby good elongation and stretch-flanging performance are exhibited. A preferable composition of constituents considering the strength (590 MPa or higher as tensile strength TS) and other points is as follows: C: 0-05 to 0.3%; Si: 0.01 to 3%; Mn: 0.5 to 3.0%; Al: 0.01 to 0.1%; at least one element selected from the group consisting of Ti, Nb, V and Zr: 0.01 to 1% in total; and iron and inevitable impurities as the remainder. The reason for the definition of these preferable ranges is as follow:

[C: 0.05 to 0.3%]

20 [0108] C is an important element in producing martensite to increase the strength of the steel sheet. To perform such an effect, the amount of C contained is preferably 0.05% or higher. From the perspective of increasing strength, the higher the amount of C contained, the better. However, if the amount of C is excessively high, a large amount of retained austenite which deteriorates stretch-flanging performance is produced, and weldability is also adversely affected. Therefore, the amount is preferably 0.3% or lower. A more preferable lower limit of the amount of C contained is 0.07%, and a more preferable upper limit is 0.25%.

[Si: 0.01 to 3%]

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**[0109]** Si is an element which effectively acts as a deoxidizing element when steel is melted, and effectively increases strength without deteriorating the ductility of steel. It also acts to suppress deposition of coarse carbide which deteriorates stretch-flanging performance. In order to perform these effects effectively, it is preferably contained in an amount of 0.01% or higher. However, since the effect of adding Si is saturated in an amount of about 3%, A preferable upper limit is set to 3%. A more preferable lower limit of the amount of Si contained is 0.1%, and a more preferable upper limit is 2.5%.

[Mn: 0.5 to 3.0%]

**[0110]** Mn is an element useful in increasing the hardening characteristics of the steel sheet to ensure high strength. To perform such an effect, it is preferably contained in an amount of 0.5% or higher. However, when the amount of Mn contained is excessively high, ductility is lowered and therefore processability is adversely affected. For this reason, the upper limit is set to 3.0%. A more preferable amount of Mn contained is 0.7% or higher but not higher than 2.5% or lower.

[Al: 0.01 to 0.1%]

**[0111]** Al is an element having a deoxidation effect, and when Al deoxidation is performed, it needs to be added in an amount of 0.01% or higher. However, when the amount of Al contained is excessively high, the above effect is saturated, and it also becomes a source of non-metallic mediators to deteriorate physical properties and surface properties. Therefore, the upper limit is set to 0.1%. a more preferable amount of Al contained is 0.03% or higher but not higher than 0.08%.

[One or more members selected from the group consisting of Ti, Nb, V and Zr: 0.01 to 1% in total]

**[0112]** These elements have the action to form precipitates such as carbide, nitride and carbonitride together with C and N to contribute to increased strength, and micronize crystal grains during hot rolling to increase elongation and stretch-flanging performance. Such effects are effectively performed when they are added in an amount of 0.01% or higher in total (of one or more members). A more preferable amount of these elements contained is 0.03% or higher. however, when the amount is excessively high, elongation and stretch-flanging performance are deteriorated rather than improved. Therefore, the amount is to be limited to 1% or lower, and more preferably 0.7% or lower.

**[0113]** Preferable basic components in the dual phase steel sheet of the present invention are as stated above, and the remainder is iron and inevitable impurities. Examples of inevitable impurities include steel raw materials, and P, S,

N, O and others which can get into steel during the manufacturing process of the materials, among others.

[0114] It is also effective to add to the steel sheet of the present invention, as needed, the following substances: (a) Ni and/or Cu: 1% or lower (not including 0%) in total; (b) Cr: 2% or lower (not including 0%) and/or Mo: 1% or lower (not including 0%); (c) B: 0.0001 to 0.005%; and (d) Ca and/or REM: 0.003% or lower (not including 0%) in total, among others. The characteristics of the steel sheet are further improved depending on the types of the components contained. The reason for setting the range of these elements when contained is as follows.

[Ni and/or Cu: 1% or lower (not including 0%) in total]

**[0115]** These elements are effective in maintaining the balance of strength and ductility high and realizing high strength at the same time. such effects increase as their amount contained is increased, but if they are contained in an amount higher than 1% in total (of one or more members), the above effects may be saturated and cracks may be produced during hot rolling. A more preferable lower limit of the amount of these elements contained is 0.05%, and a more preferable upper limit is 0.7%.

[Cr: 2% or lower (not including 0%) and/or Mo: 1% or lower (not including 0%) ]

**[0116]** Both Cr and Mo are elements effective in stabilizing the austenite phase, and facilitating the generation of the low-temperature transformation phase in the course of cooling. Although their effects increase as their amount contained is increased, if they are contained in an excessively high amount, ductility is deteriorated. Therefore, the amount of Cr is to be limited to 2% or lower (more preferably 1.5% or lower), and the amount of Mo is to be limited to 1% or lower (more preferably 0.7% or lower).

[B: 0.0001 to 0.005%]

**[0117]** B is an element effective in improving hardening characteristics, and increasing the strength of the steel sheet when added in a minute amount. To exhibit such an effect, it is preferably contained in an amount of 0.0001% or higher. However, when the amount of B contained is excessively high and is higher than 0.005%, crystal grain boundaries may be embrittled and cracks may occur during rolling.

[Ca and/or REM: 0.003% or lower (not including 0%) in total]

[0118] Ca and REM (rare earth element) are elements effective in controlling the form of sulfide in the steel and improving processability. The higher their amount contained, the higher the effects. However, when they are contained in an excessively high amount, the above-mentioned effect is saturated. Therefore, the amount is to be 0.003% or lower. [0119] The method for producing the high strength steel sheet having a structure as mentioned above will be described now. In order to produce a high strength steel sheet as mentioned above, it is necessary to conduct a predetermined heat treatment by using a steel sheet in which the space factor of martensite and/or bainite (hereinafter these two phases may be referred to as "low-temperature transformation phases") in total to the entire structure is 90% or higher and the former the grain size of austenite is 20  $\mu$ m or smaller in terms of the equivalent of a circle diameter.

**[0120]** The material steel sheet used in the present invention has a space factor of the low-temperature transformation phase of 90% or higher. This low-temperature transformation phase may be constituted only by martensite or bainite. When the space factor of the low-temperature transformation phase is lower than 90%, and the material steel sheet is heated to a 2-phase range of the ferrite phase and austenite phase (dual-phase range annealing) in the annealing step (final annealing step) described later, a coarse ferrite phase and an austenite phase are produced. Therefore, the fine ferrite phase and martensite mentioned above cannot be obtained in the final structure. As a result, stretch-flanging performance cannot be improved.

[0121] A material steel sheet having space factor of the low-temperature transformation phase of 90% or higher can be produced by the following steps: First, a steel slab adjusted to meet the composition of chemical constituents as mentioned above is hot-rolled in such a manner that the finishing rolling temperature is higher than  $Ac_3$  point. Second, the material steel sheet is cooled to a temperature lower than a martensite transformation start temperature, Ms point (temperature at which the austenite phase starts to transform into martensite), at an average cooling rate of  $10^{\circ}$ C/sec. or higher, and is then wound up, giving a material steel sheet having a space factor of martensite of 90% or higher. Moreover, a material steel sheet which is mainly composed of bainite and has a space factor of the low-temperature transformation phase of 90% or higher is obtained by cooling the material steel sheet to a bainite transformation temperature after the hot rolling, at an average cooling rate of  $10^{\circ}$ C/sec. or higher and winding up the same. When the finishing rolling temperature is  $Ac_3$  point or lower or the cooling rate after the hot rolling is  $10^{\circ}$ C/sec. or lower, a ferrite phase is likely to be produced during cooling after the hot rolling, and therefore the space factor of the low-temperature

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transformation phase after the hot rolling is not 90% or higher.

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**[0122]** In the above hot rolling step, from the perspective of micronization of the structure, it is preferable to appropriately adjust a predetermined heating temperature, and the time for retaining at the heating temperature (retaining time). In the present invention, the grain size of austenite is micronized by utilizing the pinning effect by finely depositing a microalloy (Ti, Nb, V, Zr, etc.). In order to do so, it is necessary to re-solutionize the deposition of the coarse micro-alloy produced prior to the hot rolling step. Accordingly, the heating temperature and its retaining time is preferably 1000°C or higher, and 600 seconds or longer, respectively, to perform the solutionization effect of the micro-alloy (Ti, Nb, V, Zr and the like). When the heating temperature is 1400°C or higher and its retaining time is longer than 1000 seconds, the grain size of austenite becomes undesirably coarse.

[0123] The material steel sheet used in the present invention needs to have the grain size of the former austenite of  $20~\mu m$  or smaller. This is from the perspective of improvement of elongation and stretch-flanging performance due to the micronization of the structure. That is, by subjecting a basis steel sheet having a grain size of the former austenite of  $20~\mu m$  or smaller to the final annealing step and tempering step, the final structure becomes finer than in the case where the grain size is larger than  $20~\mu m$ , and elongation and stretch-flanging performance are significantly improved. [0124] Even a steel sheet produced from a steel slab adjusted so as to meet the chemical components as mentioned above under conditions which do not meet the hot rolling and cooling rate conditions mentioned above can be imparted a space factor of the low-temperature transformation phase of 90% or higher by subjecting to the following preliminary annealing (Experiments No.5, 6 in Table 14 described later).

**[0125]** This preliminary annealing is a treatment in which the above steel sheet is retained in a temperature range of  $Ac_3$  point or higher for 5 seconds or longer, and is then cooled at an average cooling rate of  $10^{\circ}$ C/sec. or higher to a temperature of Ms point or lower or to the bainite transformation temperature range and retained. When the retaining temperature of the steel sheet is lower than  $Ac_3$  point, a ferrite phase is likely to be produced, and a space factor of the low-temperature transformation phase of 90% or higher is not attained. Moreover, even in the case where the steel sheet is retained in a temperature range of  $Ac_3$  point or higher, if the retaining time is shorter than 5 seconds, transformation of the metal structure into austenite is insufficient, and a space factor of 90% or higher is not attained.

**[0126]** By subjecting the material steel sheet whose structure and grain size of the former austenite have been adjusted in the manner described above to a heat treatment (final annealing step and tempering step) as described below, a high strength steel sheet whose space factors and grain sizes of the ferrite phase and martensite are appropriately adjusted can be obtained. At this time, the case where not only the preliminary annealing step but also acid cleaning, a cold rolling step and other processes are carried out between the hot rolling step and the heat treatment step described below also falls within the scope of the present invention. The actions and effects under the heat treatment conditions at this time are as follow:

[0127] First, a material steel sheet is subjected to a heat treatment in which it is heated to and retained at a temperature range of (Ac $_3$  point -100°C) or higher but not higher than Ac $_3$  point for 1 second or longer but not longer than 2400 seconds, and is then cooled at a cooling rate of 10°C/sec. or higher to Ms point or lower (cooling stop temperature). By performing such an annealing step, a steel sheet having the structure (the space factor of ferrite: 5 to 30%, the space factor of martensite: 50 to 95%) mentioned above is obtained. Moreover, the mean crystal grain diameters of the ferrite phase and martensite in the high strength steel sheet which is finally obtained are determined by the sizes of the crystal grains of the ferrite phase and austenite produced when the material steel sheet is heated to and retained at a temperature range of (Ac $_3$  point -100°C) or higher but not higher than Ac $_3$  point. That is, in order to obtain a fine dual phase steel sheet in which the mean grain size of the ferrite phase is 3  $\mu$ m or smaller and the mean grain size of martensite is 6  $\mu$ m or smaller, it is necessary to heat the material steel sheet to a temperature range of (Ac $_3$  point -100°C) or higher but not higher than Ac $_3$  point and retain at the same.

**[0128]** In this annealing step, when the material steel sheet is heated and retained at a temperature range higher than Ac<sub>3</sub> point in which the austenite single phase is stable, crystal grains of austenite grow and combine with each other to become coarse, and the pinning effect of fine ferrite is not produced, whereby a fine dual phase steel sheet cannot be obtained. As a result, the stretch-flanging performance of the high strength steel sheet is lowered.

[0129] The "pinning effect" mentioned above is as follows: the basis steel sheet has a structure form mainly composed of a highly micronized lath-shaped low-temperature transformation phase due to the micronization effect of the microalloy. When such a steel sheet is heated to the high temperature side of the dual-phase range, a finely dispersed ferrite phase having a low space factor is produced. The term "ferrite phase" used in this invention denotes annealed martensite or annealed bainite produced when martensite or bainite is annealed at a high temperature (dual-phase range). Since such a ferrite phase suppresses the growth and combination of the austenite phase, the final structure obtained in the following hardening and tempering steps becomes a structure mainly composed of a very fine ferrite phase and martensite. When the material steel sheet is heated and retained at a temperature lower than (Ac<sub>3</sub> point -100°C), transformation into austenite does not proceed sufficiently, and the space factor of martensite after the heat treatment becomes lower than 50%, thereby lowering the stretch-flanging performance of the steel sheet.

[0130] In this annealing step, if the heating and retaining time is shorter than 1 second, the austenite phase is not

sufficiently produced, and therefore a space factor of martensite of 50% or higher cannot be attained after this annealing step. When the heating and retaining time is longer than 2400 seconds, the crystal grains of austenite produced become coarse, and therefore the fine dual structure mentioned above cannot be obtained. From such a perspective, the heating and retaining time in the final annealing needs to be in the range of 1 second or longer but not longer than 2400 seconds. It is preferably 5 seconds or longer but shorter than 1200 seconds.

**[0131]** When the cooling rate after heating and retaining is 10°C/sec. or lower or the cooling stop temperature is higher than Ms point, generation of bainite, retained austenite phase and pearlite, generation of more ferrite phase than necessary, and deposition of a cementite phase are caused, and structures other than martensite are formed in large amounts. Therefore, the space factor of martensite is lowered, and the space factor and mean crystal grain size of the ferrite phase are excessively increased, leading to lowered elongation and stretch-flanging performance. The higher the cooling rate at this time, and the lower the cooling stop temperature, the higher the space factor of martensite is likely to be. However, since the temperature and time of the above dual-phase range annealing are suitably controlled, the space factor becomes no greater than 95%.

**[0132]** After the annealing step as mentioned above is performed, it is necessary to carry out tempering (reheating treatment) in which the material steel sheet is retained at a temperature range of 300 to 550°C for 60 seconds or longer but not longer than 1200 seconds. In the steel sheet which has undergone the annealing step as mentioned above, fine (ferrite phase +martensite) is formed in its metal structure. However, martensite in an annealed state is very hard, which lowers elongation. Moreover, since martensite is hard, a difference in hardness between martensite and soft ferrite is large, which leads to lowered stretch-flanging performance. In order to obtain excellent elongation and stretch-flanging performance, the hardness of martensite needs to be reduced than in an annealed state, which is why it is subjected to the tempering step.

**[0133]** When the retaining temperature in this tempering step is lower than 300°C, softening of martensite is insufficient, and therefore elongation and stretch-flanging performance of the steel sheet are lowered. In contrast, when the retaining temperature is higher than 550°C, a coarse cementite phase is deposited, whereby the stretch-flanging performance of the steel sheet is lowered.

**[0134]** When the retaining time of the tempering step is shorter than 60 seconds, softening of martensite is insufficient, and therefore elongation and stretch-flanging performance of the steel sheet are lowered. In contrast, when the retaining time is longer than 1200 seconds, martensite is too softened so that ensuring strength is made difficult, and the stretch-flanging performance of the steel sheet is lowered by the deposition of cementite. This retaining time is preferably 90 seconds or longer but not longer than 900 seconds, and more preferably 120 seconds or longer but not longer than 600 seconds.

**[0135]** By subjecting the material steel sheet as mentioned above to annealing (final annealing) and tempering as mentioned above, a steel sheet in which the space factors and grain sizes of the ferrite phase and martensite are suitably adjusted can be obtained, and a tensile strength as high as 590 MPa and excellent elongation and stretch-flanging performance are achieved. Such a high strength steel sheet can be used as a steel sheet with excellent press formability as a material for various steel products typically including automobiles.

(Example 3)

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[0136] Now, the present invention will be described more specifically by referring to Examples. The present invention is not restricted in itself by the following Examples. Therefore, it is possible to carry out the invention by properly modifying the Examples within the above described or later describe spirit of the invention, and such modifications are all to be included in the technical scope of the present invention.

**[0137]** steel slabs having compositions of chemical constituents shown in Tables 10 and 11 below were prepared, and material steel sheets were prepared from the steel slabs under the hot rolling conditions and preliminary annealing conditions shown in Tables 12 and 13 below. Tables 10 and 11 also show the Ac<sub>3</sub> point (Ac<sub>3</sub> transformation point) and martensite transformation start temperature, Ms point, for each steel type determined by equations (1) and (2).

$$Ac_{3}(^{\circ}C) = 910-203 \cdot \sqrt{[C]-15.2 \cdot [Ni]+44.7 \cdot [Si]+104 \cdot [V]+31.5 \cdot [Mo]+13.1 \cdot [W]-330 \cdot [Mn]+11 \cdot [Cr]+20 \cdot [Cu]-720 \cdot [P]-400 [A1]-120 \cdot [As]-400 \cdot [Ti] ...(1)$$

$$Ms(^{\circ}C) = 550-361 \cdot [C]-39 \cdot [Mn]-35 \cdot [V]-20 \cdot [Cr]-17 \cdot [Ni]-10 \cdot [Cu]-5 \cdot [Mo]-5 [W]+15 \cdot [Co]+30 \cdot [A1] ...(2)$$

	However, [C], [Ni], [Si], [V], [Mo], [W], [Mn], [Cr], [Cu], [P], [Al], [As], [Ti] and [Co] represent the amounts contained of C, Ni, Si, V, Mo, W, Mn, Cr, Cu, P, Al, As, Ti and Co (% by mass), respectively.  [0138]
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[Table 10]

Ctool tupo			(	Composit	ion of che	emical co	mponents	s* (% by r	nass)			Transform	ation point
Steel type	С	Si	Mn	Р	S	Al	Ti	Nb	V	Zr	Others	Ac <sub>3</sub> (°C)	Ms(°C)
Α	0.12	1.23	1.58	0.010	0.001	0.045	-	-	-	-	-	872	446
В	0.25	1.57	2.24	0.013	0.002	0.041	-					837	374
С	0.01	0.87	1.53	0.012	0.002	0.042	-	-	0.015	-	-	909	487
D	0.08	1.87	2.21	0.016	0.001	0.037	-	-	0.020	-	-	898	435
E	0.25	1.51	2.05	0.020	0.002	0.031	-	-	0.016	-	-	843	380
F	0.35	1.49	1.98	0.012	0.002	0.032	-	-	0.012	-	-	820	347
G	0.18	0.05	2.03	0.009	0.001	0.029	0.022	-	-	-	-	797	407
Н	0.16	2.63	1.20	0.009	0.002	0.033	0.037	-	-	-	-	945	446
I	0.21	3.54	2.08	0.011	0.001	0.038	-	0.011	-	-	-	936	394
J	0.13	1.51	0.38	0.009	0.003	0.039	-	0.015	-	-	-	915	489
K	0.12	1.49	0.62	0.009	0.001	0.031	-	0.021	-	-	-	906	483
L	0.22	1.21	2.78	0.006	0.002	0.031	-	-	-	0.021	-	802	363
М	0.20	1.50	3.49	0.013	0.001	0.033	-	-	-	0.022	-	804	343
N	0.17	1.35	2.02	0.015	0.002	0.005	-	-	0.018	-	-	840	409
0	0.19	1.32	1.99	0.011	0.003	0.087	-	-	0.017	-	-	865	406
Р	0.18	1.43	2.04	0.012	0.001	0.162	-	-	0.012	-	-	901	410
Q	0.17	1.32	1.92	0.012	0.002	0.040	0.005	-	-	-	-	854	415
R	0.18	1.28	2.12	0.014	0.002	0.038	-	0.001	0.001	-	-	- 843	403

Remainder: Iron and inevitable impurities other than P and S

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[Table 11]

Stool tuno				Com	position o	of chemic	al compo	nents* (%	by mass	s)		Transform	ation point
Steel type	С	Si	Mn	Р	S	Al	Ti	Nb	V	Zr	Others	Ac <sub>3</sub> (OC)	Ms(°C)
A1	0.17	1.40	2.05	0.009	0.002	0.028	0.015	-		-	-	851	410
B1	0.17	1.20	2.20	0.010	0.001	0.031	-	0.018	-	-	-	833	404
C1	0.19	1.45	2.10	0.009	0.001	0.031	-	-	0.021	-	-	844	400
D1	0.20	1.37	2.04	0.015	0.003	0.027	-	-	-	0.015	-	841	399
E1	0.17	1.35	2.00	0.011	0.002	0.035	0.016	0.012	-	-	-	855	412
F1	0.18	1.37	2.13	0.010	0.002	0.037	0.011	-	0.022	-	-	850	402
G1	0.19	1.34	2.06	0.010	0.002	0.031	0.005	0.015	0.004	-	-	841	402
H1	0.16	1.26	1.78	0.014	0.002	0.035	-	-	0.150	-	-	872	419
I1	0.17	1.34	1.99	0.009	0.002	0.028	-	-	0.014	-	Ni:0.2	845	411
J1	0.16	1.41	1.90	0.010	0.001	0.031	-	-	0.023	-	Cu:0.1	857	418
K1	0.17	1.32	2.18	0.012	0.002	0.040	-	-	0.025	-	Cr:0.35	843	397
L1	0.16	1.26	2.00	0.015	0.003	0.042	-	-	0.019	0.03	Mo:0.1	858	414
M1	0.19	1.32	1.95	0.016	0.003	0.045	-	-	0.025	-	B:0.0002	854	406
N1	0.16	1.39	1.95	0.013	0.002	0.039	-	-	0.017	-	Ca+REM:0.001	859	41 7
01	0.19	1.33	1.89	0.009	0.003	0.039	-	1.135	-	-	-	846	409
P1	0.21	1.35	2.08	0.011	0.003	0.037	0.154	-	0.404	0.551	-	941	380

<sup>\*</sup> Remainder: Iron and inevitable impurities other than P and S

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[Table 12]

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			Hot-rolling	conditions		Preliminary annealing conditions				
Experiment No.	Steel type	Heating temperature	Retaining time	Hot finishing temperature	Wingding temperature	Heating temperature	Retaining time	Cooling rate	Cooling stop temperature	
		(°C)	(sec.)	(°C)	(°C)	(°C)	(sec.)	(°C/sec.)	(°C)	
1	Α	1300	1800	930	550	850	120	500	20	
2	В	1250	1800	950	550	880	240	300	20	
3	С	1300	2400	850	600	900	90	300	20	
4	D	1300	1800	850	550	930	240	200	20	
5	E	1200	1800	880	250	-	-	-	-	
6	F	1200	1200	850	300	-	-	-	-	
7	G	1300	2400	800	500	900	60	200	20	
8	Н	1250	1800	850	600	930	120	300	20	
9	I	1300	1800	900	550	930	120	100	50	
10	J	1300	7800	900	500	910	60	100	20	
11	K	1200	2400	900	550	930	120	500	20	
12	L	1300	1800	850	550	930	360	300	20	
13	M	1200	1800	850	500	850	120	100	20	
14	N	1300	1800	880	500	860	120	300	20	
15	0	1300	2400	900	500	880	10	300	20	
16	Р	1300	1800	900	550	930	180	100	50	
17	Q	1250	1800	850	550	880	120	300	20	
18	R	1250	1800	850	550	930	180	300	20	

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[Table 13]

			Hot-rolling	conditions		Preliminary annealing conditions				
Experiment No.	Steel type	Heating temperature (°C)	Retaining time (sec.)	Hot finishing temperature (°C)	Winding temperature (°C)	Heating temperature (°C)	Retaining time (sec.)	Cooling rate (°C/sec.)	Cooling stop temperature (°C)	
19	A1	1300	1800	850	550	880	120	300	20	
20	B1	1200	1800	850	550	850	60	300	20	
21	C1	1300	1800	850	550	850	0	300	20	
22	D1	1300	1800	850	550	880	120	300	20	
23	E1	1300	1800	900	550	870	30	300	20	
24	F1	1300	1800	900	550	860	90	300	20	
25	G1	1300	1800	900	550	850	60	300	20	
26	H1	1350	1800	900	550	890	120	300	20	
27	I1	1250	1800	850	550	860	120	300	20	
28	J1	1300	1800	870	550	875	120	300	20	
29	K1	1200	1800	850	550	870	10	300	20	
30	L1	1300	2400	870	550	860	120	300	20	
31	M1	1300	1200	850	550	900	120	300	20	
32	N1	1300	1200	850	550	870	120	300	20	
33	01	1200	1800	850	550	930	240	200	20	
34	P1	1300	1200	950	550	950	120	200	20	
35	B1	1300	1800	850	550	900	120	300	20	
36	C1	1300	1800	850	550	880	120	300	20	

[0142] The material steel sheets obtained were subjected to the final annealing and reheating (tempering) under the conditions shown in Tables 14 and 15 below to prepare test steel sheets, and the structures (space factor of ferrite  $\alpha$ , mean grain size of ferrite  $\alpha$ , space factor of martensite M, and mean grain size of martensite M) and mechanical characteristics (tensile strength TS, elongation EL, hole expansion rate  $\lambda$ ) of the test steel sheets were determined by the methods described below. Tables 14 and 15 below also show the structures [phase constitution, space factor of low-temperature transformation phase, grain size of former austenite ( $\gamma$ )] of the test steel sheets before the final annealing.

[Method for measuring structures of test steel sheets]

- 10 **[0143]** The space factors of ferrite  $\alpha$  and martensite M were determined by subjecting the structure pictures of the test steel sheets after being corroded with nital to image analysis. The mean grain sizes of ferrite  $\alpha$  and martensite M were measured by structure analysis using FE/SEM-EBSP, and the measurements were converted into "the equivalent of a circle diameter" described above to determine their mean value.
- 15 [Method for measuring mechanical characteristics of test steel sheets]

## [0144]

- (a) Tensile test: A universal tensile tester manufactured by Instron was used to determine tensile strength (TS) and elongation (total elongation rate: EL) by using JIS No. 5 tensile test pieces.
- (b) Hole expansion test: 20-ton hole expansion tester manufactured by Tokyo Koki was used to determine hole expansion rates ( $\lambda$ ) according to Japan Iron and Steel Federation standard (JFST1001-1996), and stretch-flanging performance was evaluated.

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		Structu	ire before final anr	nealing		Final anneal	ing conditions		Tempering	conditions
Experiment No.	Steel type	Steel type Phase constitution*	constitution* temperature transformation phase	Grainsizeof former γ	Heating temperature	Retaining time	Cooling rate	Cooling stop temperature	Heating temperature	Retaining time
			(% by volume)	(μm)	(°C)	(sec.)	(°C/sec.)	(°C)	(°C)	(sec.)
1	Α	М	100	28	850	120	500	20	400	180
2	В	М	100	29	810	120	300	20	400	120
3	С	M	100	18	850	240	100	20	500	180
4	D	М	100	11	870	120	200	20	500	180
5	Е	М	93	13	815	90	300	20	520	120
6	F	М	97	17	810	240	100	20	350	180
7	G	М	100	12	750	120	200	100	400	120
8	Н	α <b>+</b> M	95	1 1	910	360	300	50	500	180
9	I	α <b>+</b> M	95	16	870	120	100	20	350	120
10	J	α <b>+</b> M	95	14	900	90	100	20	450	180
11	K	М	100	12	850	180	500	20	520	180
12	L	М	100	12	770	120	300	20	500	180
13	М	М	100	13	795	120	100	20	400	180
14	N	М	100	10	820	120	300	20	500	180
15	0	М	100	14	850	120	300	20	500	180
16	Р	М	100	9	880	120	100	50	400	120
17	Q	М	100	24	830	120	300	20	500	180
18	R	М	100	29	830	120	300	20	400	120

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		Structu	ire before final anr	nealing		Final anneal	ing conditions		Tempering conditions	
Experiment No.	Steel type	Phase constitution*	Space factor of low-temperature transformation phase	Grainsize of former γ	Heating temperature	Retaining time	I Cooling rate	Cooling stop temperature	Heating temperature	Retaining time
			(% by volume)	(μm)	(°C)	(sec.)	(°C/sec.)	(°C)	(°C)	(sec.)
19	A1	М	100	12	830	180	300	20	500	180
20	B1	М	100	9	825	120	300	20	500	240
21	C1	М	100	7	800	120	300	20	500	180
22	D1	М	100	12	810	120	300	20	500	180
23	E1	М	100	10	790	180	300	20	520	180
24	F1	М	100	11	810	180	300	20	500	180
25	G1	М	100	9	810	180	300	20	500	180
26	H1	М	100	10	840	240	300	20	500	120
27	I1	М	100	12	825	120	300	20	500	120
28	J1	М	100	11	830	120	300	20	500	180
29	K1	М	100	8	810	120	300	20	500	180
30	L1	М	100	9	850	60	300	20	500	180
31	M1	М	100	13	820	120	300	20	500	180
32	N1	М	100	10	830	120	300	20	500	180
33	01	М	100	16	830	180	200	50	500	180
34	P1	М	100	14	900	120	200	50	500	180
35	B1	М	100	15	730	120	300	20	500	120
36	C1	М	100	13	860	120	300	20	400	120

[0147] The measurement results of the structures (space factor of ferrite  $\alpha$ , mean grain size of ferrite  $\alpha$ , space factor of martensite M, mean grain size of M), and mechanical characteristics (tensile strength TS, elongation EL, hole expansion rate A) of the test steel sheets are shown in Tables 16 and 17 below. As for "evaluation" of the mechanical characteristics, tensile strength (TS) of 590 MPa or higher, elongation (EL) of 10% or higher, and hole expansion rate ( $\lambda$ ) of 80% or higher were rated excellent characteristics. The samples which were excellent in all three characteristics were rated o; those which were excellent in two characteristics out of three were rated  $\Delta$ ; and those which were excellent in only one characteristic out of three were rated x. Only o was rated a pass.

#### [0148]

10			Structure of steel plate			Mechar	nical character	istics		
15	Experiment No.	Steel type	Space factor of α	Mean grain size of α	Space factor of M	Mean grain size of M	Tensile strength TS	Elongation EL	λ	Evaluation
			(% by volume)	(μm)	(% by volume)	(μm)	(MPa)	(%)	(%)	
	1	Α	12	4. 8	88	4.3	609	28. 5	67.3	Δ
20	2	В	13	4. 9	87	4. 5	1341	7. 9	71.4	х
	3	С	19	2. 9	81	2. 5	548	32.1	82. 5	Δ
	4	D	12	2. 1	88	2. 2	993	12. 1	113.0	0
	5	E	13	2. 3	85	2. 0	1107	11.9	108.0	0
25	6	F	11	2. 7	89	2. 1	1398	6.1	54. 2	х
	7	G	16	2. 8	84	2. 4	776	17.9	107.3	0
	8	Н	14	2. 3	86	2. 3	1024	13.2	105.0	0
30	9	I	28	3. 0	72	2. 3	1419	5. 8	27.4	х
	10	J	35	3. 8	65	2. 8	587	29.0	65.9	х
	11	K	25	2. 8	75	2.4	603	28. 3	86. 3	0
	12	L	14	2. 5	86	2. 1	1117	12. 1	100.3	0
35	13	М	12	2. 5	88	2. 2	1311	7. 9	59. 9	×
	14	N	11	1. 6	89	1.7	1024	14.2	125.0	0
	15	0	10	1. 3	90	1.5	1018	15.4	128.3	0
40	16	Р	13	2. 5	87	2.0	1032	10. 1	67.3	Δ
	17	Q	12	4.0	88	3. 9	1098	12. 1	72. 9	Δ
	18	R	11	4.7	89	4. 1	1015	10.8	69. 5	Δ
	* M: Martensi	te, α: Ferr	rite							
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## [0149]

			S	tructure c	f steel plate	,	Mecha	nical characte	ristics	
50	Experiment No.	Steel type	Space factor of α	$\begin{array}{c} \text{Mean} \\ \text{grain} \\ \text{size} \\ \text{of } \alpha \end{array}$	Space factor of M	Mean grain size of M	Tensile strength TS	Elongation EL	λ	Evaluation
55			(% by volume)	(μm)	(% by volume)	(μm)	(MPa)	(%)	(%)	
	19	A1	12	2. 0	88	1. 9	1022	11.9	112.0	0

(continued)

			S	tructure o	f steel plate	)	Mecha	nical characte	ristics	
5	Experiment No.	Steel type	Space factor of α	Mean grain size of $\alpha$	Space factor of M	Mean grain size of M	Tensile strength TS	Elongation EL	λ	Evaluation
10			(% by volume)	(μm)	(% by volume)	(μm)	(MPa)	(%)	(%)	
	20	B1	9	1. 6	91	1.5	995	13. 2	117. 2	0
	21	C1	26	1. 8	74	1. 9	978	14.2	119.9	0
	22	D1	12	2. 1	88	2. 0	1017	12. 5	112.7	0
15	23	E1	22	2. 2	78	1. 9	716	16. 2	97.3	0
	24	F1	16	2. 0	84	2.1	1012	14.9	118.6	0
	25	G1	13	1. 7	87	1.9	1023	13. 6	124.1 1	0
20	26	H1	8	1. 4	92	1.3	1100	13.8	118.8	0
	27	I1	10	1. 7	90	1.9	1025	14.3	123.6	0
	28	J1	11	1. 8	89	1.8	1098	13. 7	121. 8	0
	29	K1	12	1. 9	88	1.9	1167	11.6	112. 1	0
25	30	L1	7	1. 1	93	1.3	1228	10. 9	98. 5	0
	31	M1	12	2.0	88	1. 9	1145	11.0	103.5	0
	32	N1	12	1. 8	88	1. 8	1097	11. 7	105.2	0
30	33	01	13	2. 7	87	2. 1	1212	8. 4	22. 9	×
	34	P1	16	2. 2	84	1.9	1329	5. 3	19. 1	×
	35	B1	78	13.2	22	3.5	578	15.3	34.4	×
	36	C1	0	ı	100	3. 8	1383	5. 9	78. 5	×
35	* M: Martensit	te, α: Ferr	ite							

**[0150]** It is possible to consider as follows from these results: Since the samples of Experiments No.4, 5, 7,8,11,12,14,15 and 19 to 32 all meet the requirements defined in the present invention, they are all provided with excellent characteristics.

**[0151]** In contrast, the test pieces of No.1 to 3, 6, 9, 10, 13, 16 to 18 and 33 to 36 are not provided with satisfactory characteristics as the followings because at least one requirement of their composition of chemical constituents and manufacturing conditions falls outside the scope defined in the present invention.

**[0152]** Since the test pieces of Experiments No.1,2 do not contain Ti, Nb, V, Zr and the like, the grain size of the former  $\gamma$  in the material steel sheet (steel sheet before the final annealing) became coarse, and the desired elongation and stretch-flanging performance could not be obtained.

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**[0153]** The test piece of Experiment No.3 has low tensile strength TS since the amount of C contained does not fall with in the preferable range defined in the present invention. The test piece of Experiment No. 6 has strength higher than necessary because the amount of C contained is higher than the preferable range defined in the present invention, so that ductility is lowered and elongation characteristics are deteriorated.

**[0154]** In the test piece of Experiment No. 9, the amount of Si contained is higher than the preferable range defined in the present invention, and therefore its ductility is lowered, and elongation and stretch-flanging performance are deteriorated.

**[0155]** In the test piece of Experiment No. 10, the amount of Mn contained does not fall with in the preferable range defined in the present invention, and therefore the space factor of ferrite is increased, deteriorating tensile strength and stretch-flanging performance.

[0156] In the test piece of Experiment No. 13, the amount of Mn contained is higher than the preferable range defined in the present invention, and therefore its ductility is lowered, deteriorating elongation and stretch-flanging performance.

[0157] In the test piece of No.16, flaws on the surface of the steel material are increased because the amount of Al

is higher than the preferable range defined in the present invention, whereby ductility of flawed material is lowered, and stretch-flanging performance is deteriorated.

**[0158]** In the test pieces of Experiments Nos.17 and 18, the amounts of Ti, Nb, V, Zr and the like contained are low. Therefore, micronization has not been sufficiently produced, and desired stretch-flanging performance has not been obtained.

**[0159]** In the test pieces of Experiments Nos.33 and 34, the amounts of Ti, Nb, V, Zr and the like contained are too high. Therefore, coarse carbide remains even under predetermined heat treatment conditions, and elongation and stretch-flanging performance are deteriorated.

**[0160]** In the test piece of Experiment No. 35, the heating temperature in the final annealing is much below the range defined in the present invention. Therefore, the space factor and mean grain size of ferrite, the space factor and mean grain size of martensite in the final structure fall outside the range defined in the present invention, and desired tensile strength and stretch-flanging performance have not been obtained.

**[0161]** The test piece of Experiment No. 36, the heating temperature in the final annealing is much above the range defined in the present invention. Therefore, the final structure became a single-phase structure of martensite, and the space factor of ferrite and the space factor and mean grain size of martensite fall outside the range defined in the present invention. Accordingly, desired elongation and stretch-flanging performance have not been obtained.

**[0162]** Although the present invention has been described in detail with reference to specific embodiments, it is obvious for a person of skill in the art that various modifications and alterations can be made without deviating from the spirit and scope of the present invention. the present application is based on Japanese Patent Application (No. 2006-194056) applied on July 14, 2006; Japanese Patent Application (No. 2007-144466) applied on May 31, 2007; Japanese Patent Application No. applied on May 31, 20072007-144705); and Japanese Patent Application (No. 2007-145987) applied on May 31, 2007, and their disclosures are incorporated herein by reference.

#### [Industrial Applicability]

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**[0163]** The high strength steel sheet according to the present invention has excellent elongation and stretch-flanging performance at the same time, and thus has excellent press formability. Therefore, the high strength steel sheet according to the present invention can be processed by press molding to be used for various industrial products such as automobiles, especially for industrial products where weight reduction is necessary.

The present invention further relates to the following embodiments:

[1] A high strength steel sheet which comprises, in percent by mass, C: 0.05 to 0.3%; Si: 3% or less (not including 0%.); Mn: 0.5 to 3.0%; Al: 0.01 to 0.1%; and the remainder comprising iron and inevitable impurities, the high strength steel sheet having a space factor of a martensite phase which is a main component of a metal structure of 50% or higher, and a tensile strength of 590 MPa or higher.

[2] A high strength steel sheet according to embodiment 1, wherein the structure which is a main part of the metal structure is the martensite phase, which is tempered martensite, and finely dispersed annealed bainite; the space factor of the tempered martensite is 50 to 95%; the space factor of the annealed bainite is 5 to 30%; and a mean grain size of the tempered martensite is 10  $\mu$ m or lower in terms of the equivalent of a circle diameter.

[3] A high strength steel sheet according to embodiment 1, wherein the space factor of the martensite phase which is a main part of the metal structure is 80% or higher; the mean grain size of the martensite phase is 10  $\mu$ m or smaller in terms of the equivalent of a circle diameter; the space factor of the martensite phase having a grain size of 10  $\mu$ m or larger in terms of the equivalent of a circle diameter in the martensite phase is 15% or lower; and the space factor of the retained austenite phase in the metal structure is 3% or lower.

[4] A high strength steel sheet according to embodiment 3, wherein the martensite phase is a tempered martensite phase; an annealed martensite phase is contained as the metal structure other than the martensite phase and the retained austenite phase; and the space factor of the annealed martensite phase is 3 to 20%.

[5] A high strength steel sheet according to embodiment 1, wherein the structure which is a main part of the metal structure is the martensite phase and a ferrite phase; the space factor of the martensite phase is 50 to 95%; the space factor of the ferrite phase is 5 to 30%; and the mean grain size of the martensite phase is 10  $\mu$ m or smaller in terms of the equivalent of a circle diameter.

[6] A high strength steel sheet according to embodiment 5, wherein the ferrite phase is annealed martensite.

- [7] A high strength steel sheet according to any one of embodiments 1 to 6, wherein at least an element selected from Ti, Nb, V and Zr is further contained in an amount of 0.01 to 1% by mass in total.
- [8] A high strength steel sheet according to any one of embodiments 1 to 7, wherein Ni and/or Cu are/is further contained in an amount of 1% by mass or lower in total.
- [9] A high strength steel sheet according to any one of embodiments 1 to 8, wherein Cr: 2% by mass or less and/or Mo: 1% by mass or less are/is further contained.
- [10] A high strength steel sheet according to any one of embodiments 1 to 9, wherein 0.0001 to 0.005% by mass of B is further contained.
  - [11] A high strength steel sheet according to any one of embodiments to 10, wherein Ca and/or REM are/is further contained in an amount of 0.003% by mass or lower in total.
  - [12] A method for manufacturing a high strength steel sheet according to embodiment 2, the method comprising using a steel sheet having a space factor of bainite in the entire metal structure of 90% or higher as a material steel sheet; heating and retaining the material steel sheet at a temperature of ( $Ac_3$  point -100°C) or higher but not higher than  $Ac_3$  point for 0 to 2400 seconds (including 0 seconds); then cooling the material steel sheet to a transformation start temperature of martensite, Ms point, or lower at an average cooling rate of 10°C/sec. or higher; and subsequently conducting a heat treatment in which the steel sheet is heated and retained at a temperature of 300 to 550°C for 60 to 1200 seconds.
- [13] A method for manufacturing a high strength steel sheet according to embodiments 3 or 4, the method comprising using a steel sheet in which the total space factor of the martensite phase and/or of the retained austenite phase in the entire metal structure is 90% or higher as a material steel sheet; heating and retaining the steel sheet at a temperature of (Ac<sub>3</sub> point-100°C) or higher but not higher than Ac<sub>3</sub> point for 30 to 1200 seconds; cooling the steel sheet to a transformation start temperature of martensite, Ms point, or lower at an average cooling rate of 10°C/sec. or higher; and further conducting a heat treatment in which the steel sheet is heated and retained at a temperature of 300 to 500°C for 60 to 1200 seconds.
  - [14] A method for manufacturing a high strength steel sheet according to embodiments 5 or 6, the method comprising providing a total space factor of the martensite phase and/or bainite phase in the entire metal structure is 90% or higher; using a steel sheet having a grain size of the former austenite of 20  $\mu$ m or smaller in terms of the equivalent of a circle diameter as a material steel sheet; heating and retaining the steel sheet at a temperature of (Ac<sub>3</sub> point -100°C) or higher but not higher than Ac<sub>3</sub> point for 1 to 2400 seconds; then cooling the steel sheet to a transformation start temperature of martensite, Ms point, or lower at an average cooling rate of 10°C/sec. or higher; and subsequently conducting a heat treatment in which the steel sheet is heated and retained at a temperature of 300 to 550°C for 60 to 1200 seconds.

## Claims

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- 1. A high strength steel sheet which comprises, in percent by mass, C: 0.05 to 0.3%; Si: 3% or less (not including 0%.); Mn: 0.5 to 3.0%; Al: 0.01 to 0.1%; and the remainder comprising iron and inevitable impurities, the high strength steel sheet having a space factor of a martensite phase which is a main component of a metal structure of 50% or higher, wherein the structure which is a main part of the metal structure is the martensite phase and a ferrite phase; the space factor of the martensite phase is 50 to 95%; the space factor of the ferrite phase is 5 to 30%; and the mean grain size of the martensite phase is 10 μm or smaller in terms of the equivalent of a circle diameter, and a tensile strength of 590 MPa or higher.
  - 2. A high strength steel sheet according to claim 1, wherein the ferrite phase is annealed martensite.
- 3. A high strength steel sheet according to any one of claims 1 to 2, wherein at least an element selected from Ti, Nb, V and Zr is further contained in an amount of 0.01 to 1% by mass in total.
- **4.** A high strength steel sheet according to any one of claims 1 to 3, wherein Ni and/or Cu are/is further contained in an amount of 1% by mass or lower in total.

- 5. A high strength steel sheet according to any one of claims 1 to 4, wherein Cr: 2% by mass or less and/or Mo: 1% by mass or less are/is further contained.
- 6. A high strength steel sheet according to any one of claims 1 to 5, wherein 0.0001 to 0.005% by mass of B is further contained.

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- 7. A high strength steel sheet according to any one of claims 1 to 6, wherein Ca and/or REM are/is further contained in an amount of 0.003% by mass or lower in total.
- 10 8. A method for manufacturing a high strength steel sheet according to claims 1 or 2, the method comprising providing a total space factor of the martensite phase and/or bainite phase in the entire metal structure is 90% or higher; using a steel sheet having a grain size of the former austenite of 20 µm or smaller in terms of the equivalent of a circle diameter as a material steel sheet; heating and retaining the steel sheet at a temperature of (Ac<sub>3</sub> point -100°C) or higher but not higher than Ac<sub>3</sub> point for 1 to 2400 seconds; then cooling the steel sheet to a transformation start 15 temperature of martensite, Ms point, or lower at an average cooling rate of 10°C/sec. or higher; and subsequently conducting a heat treatment in which the steel sheet is heated and retained at a temperature of 300 to 550°C for 60 to 1200 seconds.



## **EUROPEAN SEARCH REPORT**

Application Number EP 11 19 3464

	DOCUMENTS CONSID	ERED TO BE RE	ELEVANT		
Category	Citation of document with in of relevant pass		oriate,	Relevant to claim	CLASSIFICATION OF THE APPLICATION (IPC)
X A	JP 2001 192768 A (k 17 July 2001 (2001- * abstract; example * paragraph [0037] * paragraph [0058]	-07-17) es E,2E; table - paragraph [	s 1-6 * 0041] *	1,3,5,6, 8 7	INV. C22C38/00 C22C38/06 C22C38/58 C21D9/46
Х	JP 2005 213603 A (J 11 August 2005 (200 * abstract; tables * paragraph [0039] * paragraph [0044]	05-08-11)   1-5 *  - paragraph [	0041] *	1,8	
А	JP 2004 232022 A (3 19 August 2004 (200 * abstract; example * paragraph [0037]	04-08-19) e 1; table 1 *		1,8	
E	EP 1 808 505 A1 (NI 18 July 2007 (2007- * claim 4; example	-07-18)	/	1	
А	JP 2004 068050 A (S 4 March 2004 (2004- * abstract * * paragraph [0025] tables 1-5 *	-03-04)	,	1,8	TECHNICAL FIELDS SEARCHED (IPC)  C21D  C22C
А	JP 2001 207235 A (k 31 July 2001 (2001- * abstract *		CO)	1	
	The present search report has	•			
	Place of search		etion of the search	127	Examiner
	Munich	10 May	2012	Lil	impakis, Emmanue
X : part Y : part docu A : tech O : non	ATEGORY OF CITED DOCUMENTS icularly relevant if taken alone icularly relevant if combined with another iment of the same category inological background written disclosure mediate document	E her	: theory or principle is earlier patent docu after the filing date of document cited in its document cited for a member of the san document	ment, but publis the application other reasons	hed on, or

## ANNEX TO THE EUROPEAN SEARCH REPORT ON EUROPEAN PATENT APPLICATION NO.

EP 11 19 3464

This annex lists the patent family members relating to the patent documents cited in the above-mentioned European search report. The members are as contained in the European Patent Office EDP file on The European Patent Office is in no way liable for these particulars which are merely given for the purpose of information.

10-05-2012

Patent document cited in search report	Publication date	Patent family member(s)	Publication date
JP 2001192768 <i>F</i>	17-07-2001	JP 3840864 B2 JP 2001192768 A	01-11-200 17-07-200
JP 2005213603 A	11-08-2005	JP 4457681 B2 JP 2005213603 A	28-04-201 11-08-200
JP 2004232022 /	19-08-2004	JP 4085826 B2 JP 2004232022 A	14-05-200 19-08-200
EP 1808505 #	l 18-07-2007	CA 2582409 A1 CN 101035921 A CN 101851730 A EP 1808505 A1 JP 4445365 B2 JP 2006104532 A KR 20070061859 A TW 1305232 B US 2008000555 A1 US 2009314395 A1 WO 2006038708 A1	13-04-200 12-09-200 06-10-201 18-07-200 07-04-201 20-04-200 14-06-200 11-01-200 03-01-200 24-12-200
JP 2004068050 <i>F</i>	04-03-2004	JP 4306202 B2 JP 2004068050 A	29-07-200 04-03-200
JP 2001207235 <i>F</i>	31-07-2001	JP 3587116 B2 JP 2001207235 A	10-11-200 31-07-200

FORM P0459

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## REFERENCES CITED IN THE DESCRIPTION

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## Patent documents cited in the description

- JP S55122820 A **[0007]**
- JP S6043425 A **[0007]**
- JP 3729108 B [0007]
- JP 2005272954 A [0007]

- JP 2006194056 A [0162]
- JP 2007144466 A [0162]
- JP 2007144705 A [0162]
- JP 2007145987 A [0162]

## Non-patent literature cited in the description

• Netsushori, 1996, vol. 136 [0015]