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(54) **SPRING STEEL HAVING SUPERIOR FATIGUE LIFE, AND MANUFACTURING METHOD FOR SAME**

(57) A spring steel having a superior fatigue life, and a manufacturing method for the same. The chemical components thereof are as follows in weight percentage: C: 0.52-0.62%, Si: 1.20-1.45%, Mn: 0.25-0.75%, Cr: 0.30-0.80%, V: 0.01-0.15%, Nb: 0.001-0.05%, N: 0.001-0.009%, O: 0.0005-0.0040%, P:  $\leq 0.015\%$ , S:  $\leq 0.015\%$ , and Al:  $\leq 0.0045\%$ , with the remainder being Fe and incidental impurities, wherein the following condition is also met  $0.02 \leq (2Nb+V)/(20N+C) \leq 0.40$ . The spring steel of the present invention has a microstructure of tem-

pered troostite + tempered sorbite, a prior austenite grain size less than 80  $\mu\text{m}$ , a size of alloy nitride and carbide precipitates being 5-60 nm, and a maximum width of single-grain inclusions being less than 30  $\mu\text{m}$ . The spring steel has a handling strength greater than 2020 MPa, superior ductility and toughness (the reduction of area  $\geq 40\%$ ), and a fatigue life  $\geq 800,000$  times, thereby meeting application requirements of high-stress springs in industries, such as automobiles, machinery, and the like.

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**Description****Technical Field**

**[0001]** The present disclosure relates to a spring steel and a method for manufacturing the same, in particular to a spring steel having a superior fatigue life and a method for manufacturing the same, wherein the steel may be used to manufacture automotive springs having a machining strength of at least 2020 MPa, an area reduction rate  $\geq 40\%$ , a fine structure, a high steel purity, as well as a low cost and a superior fatigue life.

**Background Art**

**[0002]** As an important shock absorption and functional component, springs are widely used in various aspects of social production and people's lives, such as transportation, machinery manufacturing, automobile industry, military industry and daily life. The spring is used within its elastic range and should return to its original state after unloading. It is desired to have plastic deformation as small as possible. Hence, steel wire should have high elastic limit, yield strength and tensile strength. The higher the yield ratio, the closer the elastic limit is to the tensile strength, and thus the higher the strength utilization rate, resulting in a spring having higher elastic force. The spring relies on elastic deformation to absorb impact energy. Hence, the spring steel wire does not have to have high plasticity, but it must at least have plasticity that can endure spring forming and sufficient toughness that can endure impact energy. The spring usually works for a long time under alternating stress, so they must have high fatigue limit, and good creep and relaxation resistance.

**[0003]** Along with the progress of the technologies in the automobile and machinery industries, higher requirements are imposed on the strength and fatigue life of spring parts. Development of materials with high strength, good plasticity, and high fatigue resistance for manufacturing springs has become the focus of advanced steel companies in various countries.

**[0004]** At present, the conventional Cr-V family, Cr-Mn family, and Si-Mn family spring steel materials cannot meet the requirements of the high-strength spring production. On the other hand, the commonly used Si-Cr family spring steel with higher strength and better yield ratio has already reached the limits of the strength and fatigue life.

**[0005]** Chinese Patent No. CN101787493B discloses a high-strength spring steel alloy comprising: 0.56%-0.64%C, 0.80%-1.10%Si, 0.80%-1.20%Mn,  $P \leq 0.035\%$ ,  $S \leq 0.03\%$ , 0.80%-1.20%Cr, 0.60%-1.00% Mo, 0.20%-0.30% V, 0.05%-0.12% Nb, 0.01%-0.060% N, 0.02%-0.07% RE, and a balance of Fe. Mn, Cr, Mo alloying elements are added to the above designed material in relatively high amounts, wherein Mo is mainly used to improve the tempering stability, long-lasting creep resistance and heat resistance of the steel.

**[0006]** Chinese Patent No. CN100455691C discloses a spring steel alloy comprising 0.4-0.6% C, 1.7-2.5% Si, 0.1-0.4 Mn, 0.5-2.0% Cr, 0-0.006% N, and 0.021-0.07% Al. A design route featuring a high-carbon, high-silicon and low-manganese alloy is adopted. A main consideration is to control the amount, size and shape of the residual austenite to enhance the hydrogen embrittlement resistance of the steel. High requirements are imposed on the quenching and tempering process for the material. At the same time, the high content of alloying AL increases the difficulty in controlling the inclusions in smelting, and the hard and brittle alumina can easily lead to reduction of the fatigue life of the spring.

**[0007]** Chinese Patent No. CN1279204C discloses a spring steel alloy having a compositional design as follows: 0.30-0.50% C, 0.80-2.0% Si, 0.50-1.0% Mn, 0.40-1.0% Cr, 0.01-0.5% W, 0.08-0.30% V, 0.005-0.25% of rare earth elements, and optional 0.001-0.10% of B. A design featuring low carbon is mainly adopted for this alloy. The content of the Si element is increased to enhance the strength. At the same time, the W element is used to improve the hardenability of the steel, improve deformation resistance and prevent decarburization. However, it is difficult to control smelting and heat treatment in the presence of W and the rare earth elements.

**[0008]** Chinese patent CN1039725C discloses a low-decarburized, high-toughness spring steel for automobile suspension springs. In this kind of steel, the content of the Si element is increased without reducing the C content. The steel comprises 0.5-0.7% C, 1.0-3.5% Si, 0.3-1.5% Mn, 0.3-1.0% Cr, 0.05-0.5% V and/or Nb, less than 0.02% of P, less than 0.02% of S, 0.5-5.0% Ni and other unavoidable impurities, the remainder being Fe. In order to solve the decarburization problem and improve the toughness of the material, a relatively large amount of the Ni element is added, and thus the alloy cost is high.

**[0009]** The existing technical solutions involving alloying mainly increase material strength by adjustment of the C, Si and Mn elements. If the Si content is too low, the elastic limit of the material will be reduced, and the elasticity attenuation resistance will become poor. If the Si content is too high, the plasticity of the material will be deteriorated; at the same time, the difficulty in controlling decarburization will be increased, affecting the fatigue life of the spring. The addition of alloying elements in excessively high amounts will lead to higher material costs and affect the precipitation size at the same time, resulting in degraded fatigue performance of the material. The designed strength of the material is still low, and the fatigue life of the spring is not considered much.

**[0010]** The lightweight development of automobiles and the technological progress in the machinery industry have prompted the continuous improvement of the strength of spring materials. At present, the commonly used Cr-V family, Cr-Mn family, Si-Mn family, and Cr-Si family spring steels have reached the limits of the materials.

## 5 Summary

**[0011]** One object of the present disclosure is to provide a spring steel having a superior fatigue life and a method for manufacturing the same. The spring steel has a machining strength  $\geq 2020$  MPa, good plastic toughness (an area reduction rate  $\geq 40\%$ ) and a fatigue life  $\geq 800000$  cycles. It can meet the application requirements of high-stress springs in the industries such as automobiles and machinery.

**[0012]** To achieve the above object, the technical solution of the present disclosure is as follows:

A spring steel having a superior fatigue life, wherein its chemical composition based on weight percentage is:

C: 0.52-0.62%;

Si: 1.20-1.45%;

Mn: 0.25-0.75%;

Cr: 0.30-0.80%;

V: 0.01-0.15%;

Nb: 0.001-0.05%;

N: 0.001-0.009%;

O: 0.0005-0.0040%;

P:  $\leq 0.015\%$ ;

S:  $\leq 0.015\%$ ;

Al:  $\leq 0.0045\%$ ;

a balance of Fe and unavoidable impurities, wherein the following relationship is satisfied:  $0.02 \leq (2Nb+V)/(20N+C) \leq 0.40$ .

**[0013]** The microstructure of the spring steel according to the present disclosure is a tempered troostite + sorbite structure. The original austenite grain size is  $\leq 80 \mu\text{m}$ ; the size of alloying nitride and carbide precipitates is 5-60 nm; and the maximum width of a monoparticle inclusion is  $\leq 30 \mu\text{m}$ .

**[0014]** In the compositional design of the spring steel according to the present disclosure:

C is an essential component for ensuring the room temperature strength and hardenability of the spring steel, and it is also an element for the spring steel to achieve a high elastic limit and good elasticity attenuation resistance. When the C content is less than 0.52%, the strength of the alloy spring steel cannot be guaranteed to achieve 2020 MPa or higher, and it is also undesirable for precipitation of carbides/nitrides of microalloying elements. However, when the C content is too high, the carbide size will be too large in the tempering process, and the plasticity of the material deteriorates, which is undesirable for maintaining good plastic toughness under high strength, and thus affects the fatigue life of the material. Hence, the content of the C element must be less than 0.62%.

**[0015]** Si is a non-carbide forming element. It is mainly solid-dissolved in the ferrite phase to play a strengthening role. Increasing the alloying silicon content is desirable for improving the elastic limit and elasticity attenuation resistance of the material, thereby optimizing the spring performances. However, if the Si content is too high, the plasticity of the material will be deteriorated, which is undesirable for spring forming, and affects the life of the finished spring. At the same time, the high content of Si will increase the tendency of decarburization during the production and heat treatment of the material, resulting in increased processing cost. Upon comprehensive consideration, the Si content in the present material is controlled in the range of 1.2-1.45%.

**[0016]** Mn is an additive element commonly used in steel. It can effectively improve hardenability and strength while having little influence on the plasticity of the steel. To ensure the strength and hardenability of the alloy, the Mn content cannot be less than 0.25%. When the Mn content is too high, it will cause serious segregation, and at the same time, it will cause grain growth. Hence, Mn in the steel needs to be controlled, and the allowable range is 0.25-0.75%.

**[0017]** Cr has the effect of improving the hardenability of the spring steel, and allows for precipitation of alloy cementite in the tempering process to increase the strength of the material. The Cr element also has the effect of refining the structure. Therefore, in order to utilize the effect of Cr on solid solution strengthening and precipitation strengthening while improving the material structure, the Cr content should be controlled within 0.30-0.80% in the design of the present material.

**[0018]** The V and Nb elements are commonly added to steel as microalloying elements. These two types of elements have a strong tendency to form nitrides and carbides, thereby increasing the precipitation and nucleation rate of carbides/nitrides during tempering, and refining the structure. The carbides/nitrides of V and Nb are precipitated during the wire rod rolling process, which is desirable for reducing the austenite grain size in the material, and improving the strength

and plasticity of the material. Nano-sized precipitates are beneficial to the improvement of the material strength, plasticity and fatigue life. When the contents of V and Nb in the alloy are too high, the size of the precipitates will increase. At the same time, with the mutual influence of these two elements taken into account, after several runs of testing, it's verified that desirable effects can be resulted when the V content is controlled at 0.01-0.15%, and the Nb content is 0.001-0.05%.

An increased N content will increase the brittleness of the material. At the same time, with the effect of N on the precipitation of alloying elements taken into consideration, it is necessary to control the N content in the steel in the range of 0.001-0.009%. At the same time, in order to fulfil the purpose of refining precipitates,  $(2\text{Nb}+\text{V})/(20\text{N}+\text{C})$  in the steel is controlled in the range of 0.02-0.40, preferably in the range of 0.045-0.37. In some embodiments,  $(2\text{Nb}+\text{V})/(20\text{N}+\text{C})$  in the steel is in the range of 0.15-0.37. In order to achieve high strength, good plasticity and long fatigue life of the finished spring, the original austenite grain size in the material is  $\leq 80\text{ }\mu\text{m}$  after quenching and tempering treatment, and the size of the precipitates in the steel is controlled in the range of 5-60 nm.

**[0019]** Al mainly has a deoxygenation effect in the steel. However, alumina formed by deoxygenation with Al is a hard and brittle phase which has a significant influence on the fatigue life of the spring. Large brittle inclusions are one of the main factors that cause abnormal spring fracture. In order to have an effective control over the alumina inclusions in the steel, the Al content is controlled to be  $\leq 0.0045\%$  in the steel, and the oxygen content is controlled in the range of 0.0005-0.0040%. In order to prolong the fatigue life of the spring under high strength, the width of a monoparticle inclusion in the steel needs to be controlled at  $\leq 30\text{ }\mu\text{m}$ .

**[0020]** In order to ensure the toughness of the material and prevent defects such as hot brittleness and cold brittleness in the production process, the contents of the harmful P and S elements in the steel are controlled at 0.015% or less and 0.015% or less respectively to increase the purity of the steel.

**[0021]** The method for manufacturing the spring steel having a superior fatigue life according to the present disclosure includes: smelting, continuous casting, rough rolling, high-speed wire rolling, Stelmor controlled cooling, wire rod drawing, and quenching and tempering treatment, wherein,

an electric furnace or a converter is used for the smelting; after the smelting, secondary refining is performed with the use of an LF furnace plus VD or RH degassing treatment; during the LF refining, the composition and basicity of a synthetic slag are adjusted to control the contents of the P and S elements in the steel to be lower than 0.015% and 0.015%; stirring in the presence of argon is performed to allow for full reaction between a refining slag and inclusions in the molten steel to realize denaturation and removal of the inclusions; the VD or RH vacuum degassing time should be more than 30 minutes to ensure sufficient gas removal and control a final O content at 0.0005-0.0040%, a final N content at 0.0010-0.0090%, and a H content of less than 2 ppm; killing time of the ladle is more than 15 min at the end of the refining to facilitate floating of large particle inclusions, so that the size of inclusions in molten steel can be controlled at  $\leq 30\text{ }\mu\text{m}$ .

**[0022]** In the high-speed wire rolling, the heating of the heating furnace is controlled at 920-1150 °C, and the holding time is 1.0-3.0 h. The rolling speed is controlled at 15-115 m/s in the high-speed wire rod rolling process. A preferred scheme for online temperature control is as follows: an inlet temperature of a finishing rolling unit is 880-1050 °C, an inlet temperature of a reducing-sizing unit is 840-970 °C, and a silking temperature is 800-950 °C.

**[0023]** Preferably, a continuous casting machine is used to cast a round or square billet having a size of 320-500 mm. During the continuous casting process, the drawing speed range is controlled in the range of 0.5-0.8 m/min, and the tail end soft reduction is controlled to be greater than 10 mm, so as to control the carbon segregation in the core of the billet to achieve the target of lower than 1.08, prevent secondary oxidation during the casting process of the molten steel, and at the same time, facilitate floating and removal of inclusions larger than 30  $\mu\text{m}$ .

**[0024]** Preferably, the rough rolling adopts a twice-heating production process, wherein the cast billet is bloomed into a 115-170 mm square or round blank at a temperature of 1050-1270 °C, and the total rolling reduction is higher than 40%.

**[0025]** Preferably, when the wire rod is drawn, the drawing speed is not higher than 3.5 m/min.

**[0026]** Preferably, in the quenching and tempering treatment, the heating temperature before the quenching and tempering treatment of the drawn steel wire is controlled in the range of 850-1100 °C; the quenching medium is oil or water; the temperature of the quenching medium is controlled at 15-40 °C; and the tempering temperature is controlled at 370-550 °C, so that the size of nitride and carbide precipitates in the finished steel wire is controlled in the range of 5-60 nm.

**[0027]** Preferably, in the Stelmor controlled cooling, the air volumes of 14 fans on the Stelmor line are adjusted in the following ranges: fans F1-F7 have an air volume of 10-100%, fans F8-F12 have an air volume of 0-50%, and fans F13-F14 have an air volume of 0-50%.

**[0028]** In the method for manufacturing the spring steel according to the disclosure:

an electric furnace or a converter is used for the smelting; after the smelting, secondary refining is performed with the use of an LF furnace plus VD or RH degassing treatment; during tapping of the molten steel from the electric furnace or converter, furnace slag is prevented from entering the steel ladle; during the LF refining, the composition and basicity of the synthetic slag are adjusted to control the contents of the P and S elements in the steel to be lower than 0.015% and 0.015%; stirring in the presence of argon is performed to allow for full reaction between the refining slag and the

inclusions in the molten steel to realize denaturation and removal of the inclusions; the VD or RH vacuum degassing time should be more than 30 minutes to ensure sufficient gas removal and control a final O content at 0.0005-0.0040%, a final N content at 0.0010-0.0090%, and a H content of less than 2 ppm. The killing time of the ladle is more than 15 min at the end of the refining to facilitate floating of the large particle inclusions, so that the size of the inclusions in the molten steel is controlled at  $\leq 30\mu\text{m}$ .

**[0029]** The smelted alloy is cast with a continuous casting machine. Round or square billets may be cast. The size of the round or square billets is 320-500 mm. By adjusting the drawing speed and the tail end soft reduction parameter during the continuous casting process, the carbon segregation in the core of the billet can be controlled to achieve the target of less than 1.08. Secondary oxidation in the molten steel casting process is prevented, and at the same time, the floating and removal of inclusions larger than  $30\mu\text{m}$  are facilitated. A twice-heating production process is used, wherein the cast billet is bloomed into a 115-170 mm square or round blank at a temperature of 1050-1270 °C; the total rolling reduction is required to be higher than 40%; and the structure is refined.

**[0030]** Heating is performed with the heating furnace, wherein the heating is controlled at 920-1150 °C, and the holding time is 1.0-3.0 h. In the high-speed wire rod rolling process, the rolling speed is controlled to be 15-115 m/s. A preferred scheme for online temperature control is as follows: the inlet temperature of the finishing rolling unit is 880-1050 °C, the inlet temperature of the reducing-sizing unit is 840-970 °C, and the silking temperature is 800-950 °C. By adjusting the rolling process temperature and the silking temperature, the original austenite grains in the material are refined to  $\leq 80\mu\text{m}$ , and the size of the precipitates is controlled to be 5-60 nm.

**[0031]** The size of the rolled wire rod is  $\Phi 5-28\text{mm}$ . After the wire rod is rolled, the structure change of the wire rod is controlled by adjusting the fan components of the Stelmor line. The air volumes of 14 fans on the Stelmor line are adjusted in the following ranges: fans F1-F7 have an air volume of 10-100%, fans F8-F12 have an air volume of 0-50%, and fans F13-F14 have an air volume of 0-50%.

**[0032]** The wire rod needs to be drawn before heat treatment, and the drawing speed should be controlled to be not higher than 3.5 m/min during drawing. The heating temperature of the drawn steel wire is controlled in the range of 850-1100 °C before quenching and tempering treatment. The quenching medium may be oil or water, and its temperature is controlled at 15-40 °C. The tempering temperature is controlled at 370-550 °C, so as to control the size of the precipitates in the finished steel wire at 5-60 nm.

**[0033]** The beneficial effects of the present disclosure include:

The strength of the spring steel produced using the steel composition and manufacturing method according to the present disclosure can reach 2020 MPa or higher. The cost of this alloy is low. The material strengthened by the nano-sized precipitates has good plastic toughness and good spring formability at the same time, and cracking during the processing is prevented. With the refinement of the structure and the control over the composition and size of the inclusions, the finished spring has a high fatigue life, which can meet the requirements of automotive lightweight as well as high strength and long service life in the machinery industry. This is desirable for promoting the technical level of the industry, and brings about favorable economic benefits.

## Detailed Description

**[0034]** The chemical compositions of Examples A1-10# according to the present disclosure and three Comparative Steel Grades B1-3# are shown in Table 1 below, and the specific manufacturing methods are as follows:

Examples A1-5# according to the present disclosure, and Comparative Steel Grades B1 and B2 alloys were smelted with the use of an electric furnace, and Example A6-10# and Comparative Steel Grade B3 alloys were smelted with the use of a converter. Then, secondary refining was performed, wherein Examples A1-3#, A6-8#, and Comparative Steel Grade B1 alloys were treated with an LF furnace plus VD refining, while Examples A4-5#, A9-10#, Comparative Steel Grade B2, and B3 alloys were treated with LF plus RH. The structure and basicity of a synthetic slag were optimized. A1-6#, and B1 were vacuum degassed for 30 minutes, and A7-10#, B2, and B3 were vacuum degassed for 35 minutes. The final O content was controlled at 0.0005-0.0040%, the N content was 0.001-0.009%, and the H content was less than 2 ppm.

**[0035]** After smelting, A1-4# and B1 were cast into 300 mm round billets, A5-6# were cast into 450 mm round billets, A7-9# and B2 were cast into 320\*420 mm square billets, and A10# and B3 were cast into 500mm square billets. A tundish covering agent and a casting mold with good sealing performance were used to protect the slag in the casting process. The blooming temperature for the A1-5# and B1 continuously cast billets was 1050 °C, and the end face size of the rolled small square blanks was 115 mm. The heating temperature for A6-7# and B2 square billets was 1270 °C, and the size of the rolled blanks was 125 mm. The heating temperature for A8-10# and B3 square billets was 1100 °C, and the size of the rolled blanks was 170 mm.

**[0036]** The furnace temperature of the heating furnace for A1-4# and B1 was controlled at 920 °C, and the holding time was 1.0 h. The temperature of the heating furnace for A5-10#, B2 and B3 was controlled at 1150 °C, and the holding time was 3.0 h. In the high-speed wire rod rolling process, the rolling speed was controlled to be 15-115 m/s. The online

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temperature control scheme: for the A1-6# and B1 alloys, the inlet temperature of the finishing rolling unit was 880-950 °C, the inlet temperature of the reducing and sizing unit was 840-950 °C, and the silking temperature was 800-890 °C; for the A7-10#, B2 and B3 alloys, the inlet temperature of the finishing rolling unit was 950-1050 °C, the inlet temperature of the reducing and sizing unit was 940-970 °C, and the silking temperature was 870-950 °C.

**[0037]** The dimensions of the A1-5#, B1 and B2 alloy rolled wire rods were  $\Phi 5$ -15mm respectively, and the rolling specifications of the A6-10# and B3 alloy wire rods were  $\Phi 16$ -28mm. After the rolling of A1-5# and B1 alloy wire rods, the Stelmor cooling process was: the air volume was 40% for fans F1-F4, 10% for fans F5-F7, 5% for fans F8-F12, and 40% for fans F13-F14. After the rolling of A6-10#, B2 and B3 alloy wire rods, the Stelmor cooling process was: the air volume was 50% for fans F1-F4, 20% for fans F5-F7, 15% for fans F8-F12, and 35% for fans F13-F14. The structure of the wire rods after the Stelmor cooling was sorbite plus a very small amount of ferrite.

**[0038]** The wire rods were drawn prior to heat treatment. The quenching and tempering treatment temperatures for the drawn steel wires were divided into three groups, wherein the heating temperature was 850 °C and the tempering temperature was 550 °C for A1-2# and B1; the heating temperature was 980 °C and the tempering temperature was 470 °C for A3-7# and B2; and the heating temperature was 1100 °C and the tempering temperature was 370 °C for A8-10# and B3.

**[0039]** The mechanical properties of the high-strength springs of Examples A1-A10 and the Comparative Steel Grades B1-B3 are shown in Table 2 below. As can be seen from the table, the strength of the alloys all reach 2020 MPa or higher, higher than that of the samples of Comparative Examples B1-B3. At the same time, the area reduction rate of the materials can still reach 40% or higher. A good combination of plasticity and toughness is obtained. The high-strength springs according to the present disclosure and the comparative alloys were made into the same type of helical springs, and the fatigue life of the helical springs was measured using a spring fatigue testing machine according to GBT16947-2009 "Helical Spring Fatigue Testing Standard". The results are shown in Table 3. Under the same conditions, the fatigue life of the high-strength spring steel according to the present disclosure is superior to that of the comparative steel.

Table 1: Chemical compositions (wt%) of Examples A1-10# according to the present disclosure and Comparative Steel Grades B1-3#

Steel No.	C	Si	Mn	Cr	V	Nb	Al	N	O	P	S
A1	0.60	1.40	0.75	0.80	0.15	0.03	0.0030	0.001	0.004	0.015	0.008
A2	0.62	1.45	0.75	0.75	0.15	0.03	0.0027	0.001	0.003	0.010	0.008
A3	0.55	1.30	0.75	0.70	0.15	0.03	0.0030	0.001	0.0015	0.010	0.008
A4	0.58	1.35	0.60	0.60	0.10	0.03	0.0045	0.001	0.0015	0.010	0.008
A5	0.54	1.35	0.55	0.70	0.05	0.05	0.0045	0.001	0.0015	0.010	0.008
A6	0.56	1.35	0.30	0.67	0.02	0.05	0.0010	0.009	0.0015	0.008	0.008
A7	0.55	1.35	0.25	0.70	0.02	0.008	0.0010	0.009	0.0005	0.008	0.008
A8	0.52	1.35	0.60	0.65	0.02	0.05	0.0010	0.009	0.001	0.008	0.015
A9	0.53	1.20	0.60	0.30	0.01	0.05	0.0020	0.005	0.001	0.008	0.004
A10	0.52	1.45	0.60	0.60	0.05	0.001	0.0020	0.005	0.001	0.008	0.004
B1	0.55	1.50	0.70	0.75	0	0	0.0020	0.005	0.001	0.008	0.004
B2	0.65	1.35	0.70	1.05	0.2	0	0.0045	0.001	0.004	0.017	0.001
B3	0.5	1.6	0.55	0.8	0.15	0.08	0.003	0.001	0.006	0.015	0.015

Table 2: Alloy steel structure according to the present disclosure

Steel No.	Original austenite grain size ( $\mu\text{m}$ )	Nitride-carbide precipitate size (nm)	Maximum width of monoparticle inclusions ( $\mu\text{m}$ )
A1	75	10-60	28
A2	60	5-55	30
A3	55	30-55	15

(continued)

Steel No.	Original austenite grain size (μm)	Nitride-carbide precipitate size (nm)	Maximum width of monoparticle inclusions (μm)
A4	54	5-45	19
A5	67	10-55	25
A6	80	7-45	18
A7	60	10-56	30
A8	34	23-60	10
A9	56	12-55	25
A10	77	12-60	30
B1	90	--	45
B2	50	15-100	50
B3	45	25-145	25

Table 3: Properties of alloy steel examples according to the present disclosure and comparative steel grades

Steel No.	Tensile strength MPa	Area reduction rate %	Fatigue life Number of cycles
A1	2080	46	$85 \times 10^4$
A2	2110	42	$90 \times 10^4$
A3	2050	43	$105 \times 10^4$
A4	2090	40	$99 \times 10^4$
A5	2110	44	$90 \times 10^4$
A6	2075	41	$98 \times 10^4$
A7	2095	42	$100 \times 10^4$
A8	2130	44	$110 \times 10^4$
A9	2097	40	$95 \times 10^4$
A10	2089	45	$97 \times 10^4$
B1	1905	45	$70 \times 10^4$
B2	2080	37	$57 \times 10^4$
B3	2055	35	$60 \times 10^4$

## Claims

1. A spring steel having a superior fatigue life, wherein its chemical composition based on weight percentage is:

C: 0.52-0.62%;  
 Si: 1.20-1.45%;  
 Mn: 0.25-0.75%;  
 Cr: 0.30-0.80%;  
 V: 0.01-0.15%;  
 Nb: 0.001-0.05%;  
 N: 0.001-0.009%;  
 O: 0.0005-0.0040%;  
 P: ≤0.015%;  
 S: ≤0.015%;

Al:  $\leq 0.0045\%$ ;

a balance of Fe and unavoidable impurities, wherein the following relationship is satisfied:  $0.02 \leq (2Nb+V)/(20N+C) \leq 0.40$ .

2. The spring steel having a superior fatigue life according to claim 1, wherein the spring steel has a microstructure that is a tempered troostite + sorbite structure, an original austenite grain size  $\leq 80 \mu\text{m}$ , a size of alloying nitride and carbide precipitates in the range of 5-60 nm, and a maximum width of monoparticle inclusions  $\leq 30\mu\text{m}$ .

3. The spring steel having a superior fatigue life according to claim 1 or 2, wherein the spring steel has a machining strength  $\geq 2020 \text{ MPa}$ , an area reduction rate  $\geq 40\%$ , and a fatigue life  $\geq 800000$  cycles.

4. A method for manufacturing the spring steel having a superior fatigue life according to claim 1 or 2 or 3, comprising: smelting, continuous casting, rough rolling, high-speed wire rolling, Stelmor controlled cooling, wire rod drawing, and quenching and tempering treatment, wherein

an electric furnace or a converter is used for the smelting; after the smelting, secondary refining is performed with the use of an LF furnace plus VD or RH degassing treatment; during the LF refining, the composition and basicity of a synthetic slag are adjusted to control the contents of the P and S elements in the steel to be lower than 0.015% and 0.015%; stirring in the presence of argon is performed to allow for full reaction between a refining slag and inclusions in the molten steel to realize denaturation and removal of the inclusions; VD or RH vacuum degassing time is more than 30 minutes to control a final O content at 0.0005-0.0040%, a final N content at 0.0010-0.0090%, and a H content of less than 2 ppm; killing time of the ladle is more than 15 min at the end of the refining to facilitate floating of large particle inclusions, so that the size of inclusions in molten steel is smaller than  $30 \mu\text{m}$ ;

in the high-speed wire rolling, heating of a heating furnace is controlled at 920-1150 °C, and holding time is 1.0-3.0 h; a rolling speed is controlled at 15-115 m/s in the high-speed wire rod rolling process; an online temperature control scheme is as follows: an inlet temperature of a finishing rolling unit is 880-1050 °C, an inlet temperature of a reducing-sizing unit is 840-970 °C, and a silking temperature is 800-950 °C.

5. The method for manufacturing the spring steel having a superior fatigue life according to claim 4, wherein a continuous casting machine is used to cast a round or square billet having a size of 320-500 mm; during the continuous casting process, a drawing speed is controlled in the range of 0.5-0.8 m/min, and a tail end soft reduction is controlled to be greater than 10 mm, so as to control carbon segregation in a core of the billet to achieve a target of lower than 1.08.

6. The method for manufacturing the spring steel having a superior fatigue life according to claim 4, wherein the rough rolling adopts a twice-heating production process, wherein a cast billet is bloomed into a 115-170 mm square or round blank at a temperature of 1050-1270°C, and a total rolling reduction is higher than 40%.

7. The method for manufacturing the spring steel having a superior fatigue life according to claim 4, wherein when the wire rod is drawn, a drawing speed is not higher than 3.5 m/min.

8. The method for manufacturing the spring steel having a superior fatigue life according to claim 4, wherein in the quenching and tempering treatment, a heating temperature prior to the quenching and tempering treatment of the drawn steel wire is controlled in the range of 850-1100 °C; oil or water is used as a quenching medium; a temperature of the quenching medium is controlled at 15-40 °C; and a tempering temperature is controlled at 370-550 °C, so that a size of nitride and carbide precipitates in a finished steel wire is controlled in the range of 5-60 nm.

9. The method for manufacturing the spring steel having a superior fatigue life according to claim 4, wherein in the Stelmor controlled cooling, air volumes of 14 fans on a Stelmor line are adjusted in the following ranges: fans F1-F7 have an air volume of 10-100%, fans F8-F12 have an air volume of 0-50%, and fans F13-F14 have an air volume of 0-50%.



## INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2019/096726

## A. CLASSIFICATION OF SUBJECT MATTER

C22C 38/00(2006.01)i; C22C 38/24(2006.01)i; C21D 7/06(2006.01)i; C21D 9/02(2006.01)i

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

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C22C; C21D

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

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

CNABS, CNKI, EPODOC, VEN: 碳, 硅, 锰, 铬, 钒, 铌, 弹簧, 钢, 精炼, 连铸, 轧, C, carbon, Si, silicon, Mn, manganese, Cr, chromium, V, vanadium, Nb, niobium, spring, steel, refinement, continuous, casting, rolling

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CN 101397629 A (NANJING IVECO AUTOMOBILE CO.) 01 April 2009 (2009-04-01) claims 1-4d, and description, embodiment 15	1-9
X	CN 108239726 A (POSCO CORPORATION) 03 July 2018 (2018-07-03) claims 1-8	1-9
X	CN 1118611 A (POHANG IRON & STEEL CO., LTD. ET AL.) 13 March 1996 (1996-03-13) claims 1-4	1-9
X	US 4711675 A (AICHI STEEL WORKS LTD. ET AL.) 08 December 1987 (1987-12-08) claim 1	1-9
A	CN 1764733 A (KOBE STEEL LTD.) 26 April 2006 (2006-04-26) entire document	1-9
A	EP 1985721 A1 (KOBE STEEL LTD.) 29 October 2008 (2008-10-29) entire document	1-9

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

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“&amp;” document member of the same patent family

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**INTERNATIONAL SEARCH REPORT**  
**Information on patent family members**

International application No.

**PCT/CN2019/096726**

Patent document cited in search report	Publication date (day/month/year)	Patent family member(s)	Publication date (day/month/year)
CN 101397629 A	01 April 2009	CN 101397629 B	08 September 2010
CN 108239726 A	03 July 2018	KR 20180074008 A	03 July 2018
CN 1118611 A	13 March 1996	WO 9518243 A1	06 July 1995
		JP H08506623 A	16 July 1996
		KR 950018545 A	22 July 1995
		KR 960005230 B1	23 April 1996
		CN 1039725 C	09 September 1998
		US 5575973 A	19 November 1996
		DE 4480344 T1	22 February 1996
US 4711675 A	08 December 1987	None	
CN 1764733 A	26 April 2006	EP 1612287 A1	04 January 2006
		WO 2004087977 A1	14 October 2004
		US 7615186 B2	10 November 2009
		EP 1612287 A4	21 November 2007
		KR 20050103981 A	01 November 2005
		US 2007163680 A1	19 July 2007
		EP 1612287 B1	01 June 2016
EP 1985721 A1	29 October 2008	US 2010224287 A1	09 September 2010
		CA 2632407 C	03 April 2012
		US 8038934 B2	18 October 2011
		BR PI0706549 A2	29 March 2011
		KR 101029431 B1	14 April 2011
		KR 20080080210 A	02 September 2008
		WO 2007083808 A1	26 July 2007
		CA 2632407 A1	26 July 2007
		EP 1985721 B1	27 October 2010
		DE 602007010102 D1	09 December 2010
		ES 2352856 T3	23 February 2011
		BR PI0706549 B1	08 September 2015
		CN 101365820 B	27 March 2013
		AT 486147 T	15 November 2010
		JP 4027956 B2	26 December 2007
		CN 101365820 A	11 February 2009
		EP 1985721 A4	24 March 2010
		JP 2007191776 A	02 August 2007

Form PCT/ISA/210 (patent family annex) (January 2015)

**REFERENCES CITED IN THE DESCRIPTION**

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**Patent documents cited in the description**

- CN 101787493 B [0005]
- CN 100455691 C [0006]
- CN 1279204 C [0007]
- CN 1039725 C [0008]