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(54) **HIGH-NITROGEN AND HIGH-CHROMIUM PLASTIC DIE STEEL, AND SMELTING METHOD AND THERMAL PROCESSING METHOD THEREFOR**

(57) High-nitrogen and high-chromium plastic die steel, and a smelting method and a thermal processing method therefor. The high-nitrogen and high-chromium plastic die steel comprises the chemical components in percentage by weight: 0.25-0.35% of C, 0.45-0.8% of Si, 0.4-0.7% of Mn, 16.5-17.5% of Cr, 0.1-0.3% of Ni,

0.1-0.5% of Mo, 0.06-0.10% of N, and the balance being Fe and inevitable impurity elements. The thermal processing comprises spheroidizing annealing and isothermal quenching-tempering. The plastic die steel has good corrosion resistance.

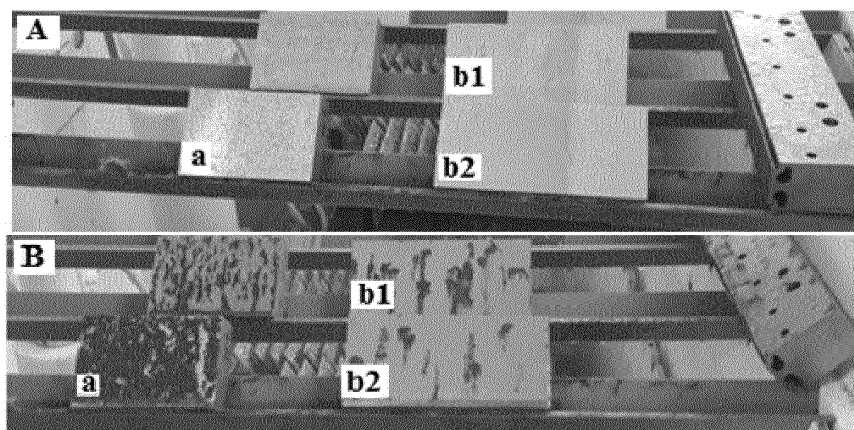


Fig. 5

Description**Field of the Invention**

5 **[0001]** The present invention relates to the field of metal materials, in particular to a high-nitrogen and high-chromium plastic mold steel and a smelting method and a heat treatment method thereof.

Background of the invention

10 **[0002]** With the rapid development of the plastic mold industry, higher corrosion resistance is required for plastic mold steel in addition to high strength and toughness, especially in the production of plastic products made of PVC, fluoroplastics and flame retardant ABS. Hydrogen chloride, hydrogen fluoride and sulfur dioxide decomposed from plastics in a molten state have a strong corrosive effect on mold cavities.

15 **[0003]** In order to meet the above service conditions, plastic mold steel 3Cr17NiMo has been developed in China. The steel is developed on the basis of comprehensive analysis of the advantages and disadvantages of S136 steel from Sweden and 1.2316 steel from Germany, and is widely applied in the production of PVC and other plastic products with a strong corrosive effect.

20 **[0004]** However, with the increasing demand for plastic products in the market and cost problems resulting from fierce competition, it is required that plastic molds have higher corrosion resistance to meet the long life of plastic mold cavities.

Summary of the Invention

25 **[0005]** The first technical problem to be solved by the present invention is to provide a high-nitrogen and high-chromium plastic mold steel. The high-nitrogen and high-chromium plastic mold steel comprises the following chemical components by weight: 0.25 - 0.35% of C, 0.45 - 0.8% of Si, 0.4 - 0.7% of Mn, 16.5 - 17.5% of Cr, 0.1 - 0.3% of Ni, 0.1 - 0.5% of Mo, 0.06 - 0.10% of N, and the rest of Fe and inevitable impurity elements.

[0006] Further, in the high-nitrogen and high-chromium plastic mold steel, the impurity elements comprise $Al \leq 0.02\%$, $P \leq 0.025\%$, $S \leq 0.005\%$, $O \leq 0.003\%$ and $H \leq 0.0002\%$.

30 **[0007]** The second technical problem to be solved by the present invention is to provide a smelting method of the high-nitrogen and high-chromium plastic mold steel. The smelting method comprises the following steps:

- A. smelting: smelting an IF steel in an electric furnace until the IF steel is completely melted;
- B. refining: pouring the completely melted molten steel into an LF furnace for slagging and alloying;
- C. nitriding: blowing nitrogen for nitrogen alloying; and
- 35 D. tapping: tapping and casting into ingots after nitrogen blowing.

[0008] Further, the step D of the smelting method of the high-nitrogen and high-chromium plastic mold steel further comprises forging or rolling into 180 - 250 mm thick flat steel after casting into ingots.

40 **[0009]** Specifically, in the step A of the smelting method of the high-nitrogen and high-chromium plastic mold steel, the IF steel is an IF steel billet or IF steel scrap.

[0010] Specifically, in the step A of the smelting method of the high-nitrogen and high-chromium plastic mold steel, the smelting temperature is 1630 - 1650°C. The pressure in the furnace is 1 standard atmospheric pressure.

[0011] Preferably, in the step C of the smelting method of the high-nitrogen and high-chromium plastic mold steel, the nitrogen blowing time is 10 - 20min.

45 **[0012]** Preferably, in the step D of the smelting method of the high-nitrogen and high-chromium plastic mold steel, the tapping control temperature of molten steel is 1540 - 1560°C.

[0013] The third technical problem to be solved by the present invention is to provide a heat treatment method of the high-nitrogen and high-chromium plastic mold steel. The heat treatment method comprises the following steps:

- 50 A. spheroidizing annealing: heating the flat steel below 500°C to 940 - 960°C and holding, then cooling to 760 - 780°C and holding, cooling below 500°C, discharging and air-cooling to room temperature;
- B. isothermal quenching-tempering: heating the flat steel below 450°C obtained in the step A to 810 - 830°C and holding, and then heating to 1030 - 1060°C and holding; then putting the heated flat steel in a salt bath for salt bath treatment, taking the treated flat steel out for tempering at 550 - 650°C, discharging and air-cooling to room temperature.
- 55 C. tempering: heating the flat steel below 450°C obtained in the step A to 810 - 830°C and holding, and then heating to 1030 - 1060°C and holding; then putting the heated flat steel in a salt bath for salt bath treatment, taking the treated flat steel out for tempering at 550 - 650°C, discharging and air-cooling to room temperature.

[0014] In the step A of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the heating rate for heating to 940 - 960°C is 90 - 100°C/h.

[0015] In the step A of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the temperature is held at 940 - 960°C for 8 - 12h.

[0016] In the step A of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the cooling rate for cooling to 760 - 780°C is 40 - 50°C/h.

[0017] In the step A of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the cooling rate for cooling below 500°C is 40 - 50°C/h.

[0018] In the step A of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the temperature is held at 760 - 780°C for 7 - 10h.

[0019] In the step B of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the heating rate for heating to 810 - 830°C is 90 - 100°C/h.

[0020] In the step B of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the temperature is held at 810 - 830°C for 5 - 8h.

[0021] In the step B of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the heating rate for heating to 1030 - 1060°C is 90 - 100°C/h.

[0022] In the step B of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the temperature is held at 1030 - 1060°C for 1 - 1.5h.

[0023] In the step B of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the salt bath consists of NH_4NO_2 and KNO_2 which are mixed at a mass ratio of 1:1.5 - 2.0, preferably at a mass ratio of 1:1.5.

[0024] In the step B of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the temperature of the salt bath is 210 - 230°C.

[0025] Specifically, in the step B of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the salt bath treatment lasts for 15 - 20min.

[0026] In the step B of the heat treatment method of the high-nitrogen and high-chromium plastic mold steel, the tempering at 550 - 650°C lasts for 5 - 8h.

[0027] The present invention puts forward the design idea of adding N for alloying and reducing the use of Mo, and nitrogen alloying is achieved directly by blowing nitrogen to provide excellent corrosion resistance, together with the heat treatment process of spheroidizing annealing-isothermal quenching-tempering.

Brief Description of the Drawings

[0028]

Fig. 1 shows a metallographic structure after spheroidizing annealing according to example 1;

Fig. 2 shows a metallographic structure after tempering according to example 1;

Fig. 3 shows a metallographic structure after spheroidizing annealing according to example 2;

Fig. 4 shows a metallographic structure after tempering according to example 2;

Fig. 5 shows physical comparison of salt spray corrosion; in which, a is the conventional 3Cr17NiMo, b1 is a product in example 1 of the present invention, b2 is a product in example 2 of the present invention, Fig. A shows the result after corrosion for 1h, and Fig. B shows the result after corrosion for 24h.

Detailed Description of the Preferred Embodiments

[0029] The high-nitrogen and high-chromium plastic mold steel of the present invention comprises the following chemical components by mass: 0.25 - 0.35% of C, 0.45 - 0.8% of Si, 0.4 - 0.7% of Mn, 16.5 - 17.5% of Cr, 0.1 - 0.3% of Ni, 0.1 - 0.5% of Mo, 0.06 - 0.10% of N, and the rest of Fe and inevitable impurity elements.

[0030] Preferably, in the high-nitrogen and high-chromium plastic mold steel, the other impurity elements comprise Al $\leq 0.02\%$, P $\leq 0.025\%$, S $\leq 0.005\%$, O $\leq 0.003\%$ and H $\leq 0.0002\%$.

[0031] The chemical elements are: C, Si, Mn, Cr, N, Fe, Ni, Al, P, S, O and H.

[0032] According to the smelting method of the high-nitrogen and high-chromium plastic mold steel, the smelting process mainly comprises the following steps:

(1) selecting an IF steel billet or IF steel scrap based on the above chemical components, and smelting in an electric furnace at a smelting temperature of 1630 - 1650°C and pressure in the furnace of 1 standard atmospheric pressure;

(2) pouring the molten steel into an LF furnace after the metal material in the electric furnace is completely melted, starting to blow argon, and adding a slagging agent made up of lime, fluorite, lime sand and aluminum oxide 5 - 10min later;

(3) adding a prepared carburant and an alloy successively after slagging and desulfurization, and inserting an electrode bar for heating and smelting;

- (4) taking samples and fine-tuning components of the alloy based on test results 15 - 30min later;
- (5) stopping blowing argon, blowing high-purity nitrogen instead for 10 - 20min, and adjusting the pressure based on the rolling state at the liquid level;
- (6) at the end of blowing nitrogen, taking samples, fine-tuning the nitrogen blowing time based on component results and adjusting the temperature of the molten steel at 1540 - 1560°C; and
- (7) tapping, casting into ingots, and forging or rolling into finished products.

[0033] The heat treatment method of the high-nitrogen and high-chromium plastic mold steel mainly comprises the following steps:

A. spheroidizing annealing: placing the flat steel below 500°C obtained by smelting into an annealing furnace, heating to 940 - 960°C at a rate of 90 - 100°C/h and holding for 8 - 12h, then cooling to 760 - 780°C at a cooling rate of 40 - 50°C/h and holding for 7 - 10h, and finally cooling to $\leq 500^\circ\text{C}$ at a cooling rate of 40 - 50°C/h, discharging and air-cooling to room temperature; and

B. isothermal quenching-tempering: putting the mold steel subject to spheroidizing annealing into a quenching furnace at a temperature lower than 450°C, heating to 810 - 830°C at a rate of 90 - 100°C/h and holding for 5 - 8h, keeping heating to 1030 - 1060°C at a rate of 90 - 100°C/h and holding for 1-1.5h, and placing the heated mold steel quickly into a 210 - 230°C salt bath consisting of NH_4NO_2 and KNO_2 which are mixed at a mass ratio of 1:1.5, and holding for 15 - 20min; then taking out the steel subject to salt bath treatment quickly, putting the steel into a tempering furnace heated to 550 - 650°C and holding for 5 - 8h, discharging and air-cooling to room temperature.

[0034] In order to completely release residual stress during deformation, the temperature of the flat steel put into the annealing furnace is controlled below 500°C in the step A.

[0035] In order to prevent thermal cracks or prevent affecting the production efficiency, the mold steel is heated to 940 - 960°C at a controlled rate of 90 - 100°C/h in the step A.

[0036] In order to ensure the spheroidizing effect, in the step A, the mold steel is cooled to 760 - 780°C at a controlled cooling rate of 40 - 50°C/h and held for 7 - 10h, and then cooled below 500°C at a cooling rate of 40 - 50°C/h.

[0037] In the step A of the present invention, the formation of martensite is avoided by speeding up cooling. In order to prevent cracks during heating, the temperature of the mold steel put into the heating furnace is controlled below 450°C in the step B.

[0038] In order to prevent thermal cracks, in the step B, the mold steel is heated to 810 - 830°C at a controlled rate of 90 - 100°C/h and held for 5 - 8h, and then continuously heated to 1030 - 1060°C at a rate of 90 - 100°C/h and held for 1 - 1.5h.

[0039] In order to obtain the products with excellent mechanical properties and corrosion resistance, the tempering temperature is controlled at 550 - 650°C.

[0040] According to the present invention, users can take the following approaches: spheroidizing annealing in the step A, then processing into the corresponding molds, and isothermal quenching-tempering in the step B; or spheroidizing annealing in the step A, then isothermal quenching-tempering in the step B, and processing into the corresponding molds, based on their own processing capability and requirements for comprehensive mechanical properties and corrosion resistance of finished mold products.

Example 1

[0041] Smelting of a high-nitrogen and high-chromium plastic mold steel in the following steps:

(1) placing an IF steel billet mainly containing the following elements by mass: 0.002% of C, 0.01% of Si, 0.1% of Mn, 0.012% of P, 0.010% of S, 0.015% of Al, and the rest of Fe and other inevitable impurities into an electric furnace for primary smelting at a temperature of $1640 \pm 10^\circ\text{C}$;

(2) pouring the molten steel into an LF furnace after the metal material in the electric furnace is completely melted, starting to blow argon, and adding a slagging agent (lime, fluorite, lime sand and aluminum oxide) 5min later;

(3) adding a prepared carburant and an alloy successively after slagging and desulfurization, and inserting an electrode bar for heating and smelting;

(4) taking samples and fine-tuning components of the alloy based on test results 30min later;

(5) stopping blowing argon, blowing high-purity nitrogen instead for 15min, and adjusting the pressure based on the rolling state at the liquid level;

(6) at the end of blowing nitrogen, taking samples, fine-tuning the nitrogen blowing time based on the component results and adjusting the temperature of molten steel at $1550 \pm 10^\circ\text{C}$; and

(7) tapping, casting into ingots, then upsetting and stretching for two times into finished products after high-temperature homogenization treatment.

[0042] Heat treatment of a new high-nitrogen and high-chromium plastic mold steel in the following steps:

- (1) spheroidizing annealing: putting the mold steel into an annealing furnace at a temperature lower than 500°C, heating the mold steel to $950 \pm 10^\circ\text{C}$ at a rate of 95°C/h and holding for 12h, then cooling the mold steel to 760°C at a cooling rate of 40°C/h and holding for 9h, finally cooling the mold steel to $\leq 500^\circ\text{C}$ at a cooling rate of 45°C/h , discharging and air-cooling to room temperature, with the metallographic structure as shown in Fig. 1.
- (2) Isothermal quenching-tempering

[0043] Putting the mold steel subject to spheroidizing annealing into a quenching furnace at a temperature lower than 450°C , heating to 830°C at a rate of 100°C/h and holding for 8h, keeping heating to 1060°C at a rate of 100°C/h and holding for 1.5h, placing the heated mold steel quickly into a 220°C salt bath consisting of NH_4NO_2 and KNO_2 which are mixed at a ratio of 1:1.5, and holding for 20min; then taking out the mold steel subject to salt bath treatment quickly, putting the treated mold steel into a tempering furnace heated to 580°C and holding for 8h, discharging and air-cooling to room temperature, with the metallographic structure as shown in Fig. 2.

[0044] The components and content of the product prepared in Example 1 are shown in Table 1 below.

Table 1 Chemical Components (wt%)

C	Si	Mn	Cr	Ni	Mo	N	Al	P	S	O	H
0.33	0.6	0.65	17.2	0.25	0.2	0.09	0.02	0.021	0.004	0.0028	0.0002

Example 2

[0045] Smelting of a new high-nitrogen and high-chromium plastic mold steel in the following steps:

- (1) placing an IF steel billet mainly containing the following elements by mass: 0.0015% of C, 0.011% of Si, 0.12% of Mn, 0.011% of P, 0.010% of S, 0.015% of Al, and the rest of Fe and other inevitable impurities into an electric furnace for primary smelting at a temperature of $1640 \pm 10^\circ\text{C}$;
- (2) pouring the molten steel into an LF furnace after the metal material in the electric furnace is completely melted, starting to blow argon, and adding a slagging agent (lime, fluorite, lime sand and aluminum oxide) 5min later;
- (3) adding a prepared carburant and an alloy successively after slagging and desulfurization, and inserting an electrode bar for heating and smelting;
- (4) taking samples and fine-tuning components of the alloy based on test results 30min later;
- (5) stopping blowing argon, blowing high-purity nitrogen instead for 15min, and adjusting the pressure based on the rolling state at the liquid level;
- (6) at the end of blowing nitrogen, taking samples, fine-tuning the nitrogen blowing time based on the component results and adjusting the temperature of molten steel at $1550 \pm 10^\circ\text{C}$; and
- (7) tapping, casting into ingots, then upsetting and stretching for two times into finished products after high-temperature homogenization treatment.

[0046] Heat treatment of a new high-nitrogen and high-chromium plastic mold steel in the following steps:

- (1) spheroidizing annealing: putting the mold steel into an annealing furnace at a temperature lower than 500°C , heating the mold steel to $950 \pm 10^\circ\text{C}$ at a rate of 95°C/h and holding for 12h, then cooling the mold steel to 760°C at a cooling rate of 40°C/h and holding for 9h, finally cooling the mold steel to $\leq 500^\circ\text{C}$ at a cooling rate of 45°C/h , discharging and air-cooling to room temperature, with the metallographic structure as shown in Fig. 3.
- (2) Isothermal quenching-tempering

[0047] Putting the mold steel subject to spheroidizing annealing into a quenching furnace at a temperature lower than 450°C , heating to 820°C at a rate of 95°C/h and holding for 8h, keeping heating to 1050°C at a rate of 100°C/h and holding for 1.5h, and placing the heated mold steel quickly into a 230°C salt bath consisting of NH_4NO_2 and KNO_2 which are mixed at a ratio of 1:1.5, and holding for 20min; then taking out the mold steel subject to salt bath treatment quickly, putting the treated mold steel into a tempering furnace heated to 590°C and holding for 8h, discharging and air-cooling to room temperature, with the metallographic structure as shown in Fig. 4.

[0048] The components and content of the product prepared in Example 2 are shown in Table 2 below.

Table 2 Chemical Components (wt%)

C	Si	Mn	Cr	Ni	Mo	N	Al	P	S	O	H
0.31	0.45	0.62	16.9	0.22	0.24	0.08	0.02	0.022	0.004	0.0029	0.0002

[0049] The results of samples prepared in examples 1 and 2 of the present invention and the conventional 3Cr17NiMo under the same salt spray corrosion conditions are shown in Fig. 5. As can be seen from the figure, the conventional 3Cr17NiMo is severely corroded, while the plastic mold steel of the present invention is slightly corroded, indicating that the plastic mold steel of the present invention has better corrosion resistance.

Claims

1. A high-nitrogen and high-chromium plastic mold steel, **characterized by** comprising the following chemical components by weight: 0.25 - 0.35% of C, 0.45 - 0.8% of Si, 0.4 - 0.7% of Mn, 16.5 - 17.5% of Cr, 0.1 - 0.3% of Ni, 0.1 - 0.5% of Mo, 0.06 - 0.10% of N, and the rest of Fe and inevitable impurity elements.
2. The high-nitrogen and high-chromium plastic mold steel according to claim 1, **characterized in that** the impurity elements comprise $Al \leq 0.02\%$, $P \leq 0.025\%$, $S \leq 0.005\%$, $O \leq 0.003\%$ and $H \leq 0.0002\%$.
3. A smelting method of the high-nitrogen and high-chromium plastic mold steel, **characterized by** comprising the following steps:
 - A. smelting: smelting an IF steel in an electric furnace until the IF steel is completely melted;
 - B. refining: pouring the completely melted molten steel into an LF furnace for slagging and alloying;
 - C. nitriding: blowing nitrogen for nitrogen alloying; and
 - D. tapping: tapping and casting into ingots after nitrogen blowing.
4. The preparation method of the high-nitrogen and high-chromium plastic mold steel according to claim 3, **characterized in that** step D further comprises forging or rolling into 180 - 250 mm thick flat steel after casting into ingots.
5. The smelting method of the high-nitrogen and high-chromium plastic mold steel according to claim 3 or 4, **characterized in that** the smelting temperature is 1630 - 1650°C in step A.
6. The smelting method of the high-nitrogen and high-chromium plastic mold steel according to claim 3 or 4, **characterized in that** the nitrogen blowing time is 10 - 20min in step C.
7. The smelting method of the high-nitrogen and high-chromium plastic mold steel according to claim 3 or 4, **characterized in that** the tapping control temperature of the molten steel is 1540 - 1560°C in step D.
8. A heat treatment method of the high-nitrogen and high-chromium plastic mold steel, **characterized by** comprising the following steps:
 - A. spheroidizing annealing: heating the flat steel below 500°C obtained by the smelting method of the high-nitrogen and high-chromium plastic mold steel according to any one of claims 4 to 7 to 940 - 960°C and holding, then cooling to 760 - 780°C and holding, cooling below 500°C, discharging and air-cooling to room temperature;
 - B. isothermal quenching-tempering: heating the flat steel below 450°C obtained in the step A to 810 - 830°C and holding, and then heating to 1030 - 1060°C and holding; then putting the heated flat steel in a salt bath for salt bath treatment, taking the treated flat steel out for tempering at 550 - 650°C, discharging and air-cooling to room temperature.
9. The heat treatment method of the high-nitrogen and high-chromium plastic mold steel according to claim 8, **characterized in that** in step A, at least any one of the following is satisfied:
 - the heating rate for heating to 940 - 960°C is 90 - 100°C/h;
 - the temperature is held at 940 - 960°C for 8-12h;
 - the cooling rate for cooling to 760 - 780°C is 40 - 50°C/h;

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the cooling rate for cooling below 500°C is 40-50°C/h; and
the temperature is held at 760 - 780°C for 7-10h.

- 5 **10.** The heat treatment method of high-nitrogen and high-chromium plastic mold steel according to claim 8, **characterized in that** in step B, at least any one of the following is satisfied:

10 the heating rate for heating to 810 - 830°C is 90 - 100°C/h;
the temperature is held at 810 - 830°C for 5 - 8h;
the heating rate for heating to 1030 - 1060°C is 90 - 100°C/h;
15 the temperature is held at 1030 - 1060°C for 1 - 1.5h;
the salt bath consists of NH_4NO_2 and KNO_2 which are mixed at a mass ratio of 1:1.5 - 2.0, preferably at a mass ratio of 1:1.5;
the temperature of the salt bath is 210 - 230°C;
the salt bath treatment lasts for 15 - 20min; and
15 the tempering at 550 - 650°C lasts for 5 - 8h.

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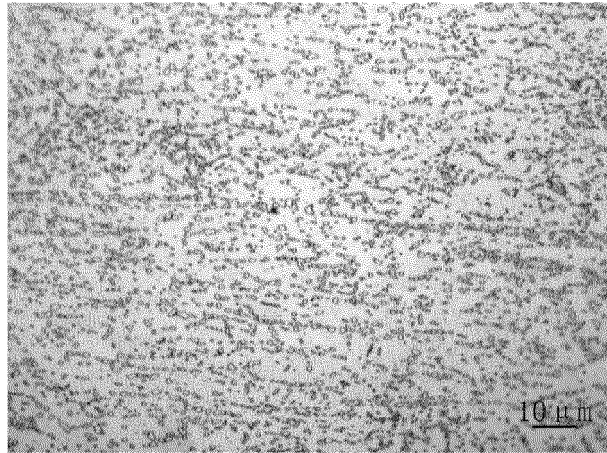


Fig. 1

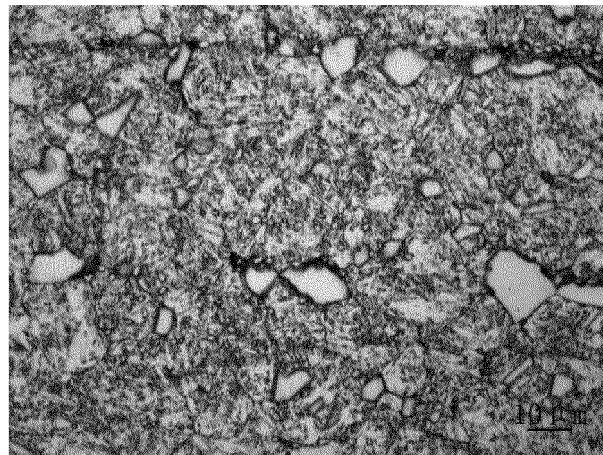


Fig. 2

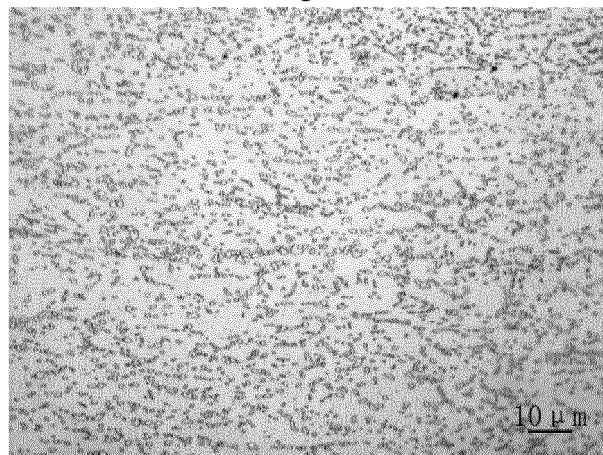


Fig. 3

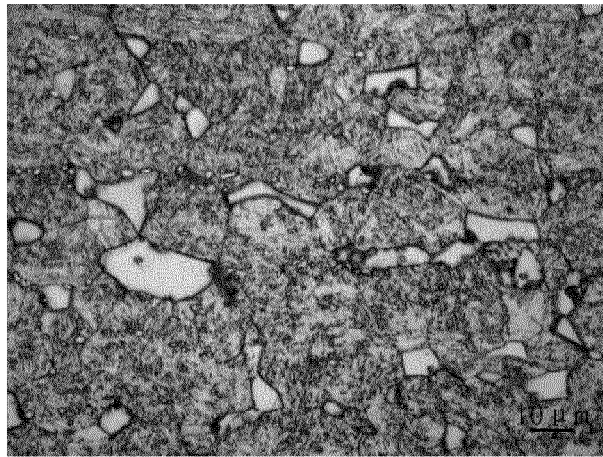


Fig. 4

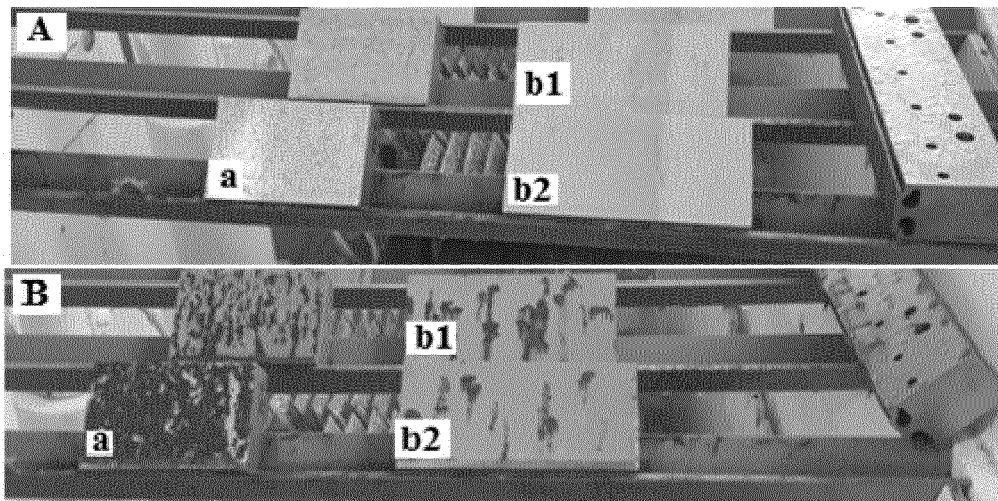


Fig. 5

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2019/110868

A. CLASSIFICATION OF SUBJECT MATTER

C22C 38/02(2006.01)i; C22C 38/44(2006.01)n; C21D 1/20(2006.01)n; C22C 33/04(2006.01)n; C21D 6/00(2006.01)n; C21D 1/32(2006.01)n

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)

DWPI, SIPOABS, CNABS, CNTXT, CNKI, STN; 锰, 铬, 模具钢, Si, C, N, Mo, 碳, Mn, 氮, Ni, 硅, Cr, 铜, 镍, 球化退火, 等温淬火, 回火, mould w steel, anneal, quench, temper

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 11335782 A (DAIDO STEEL CO., LTD.) 07 December 1999 (1999-12-07) description, paragraphs 5 and 19	1-7
Y	JP 11335782 A (DAIDO STEEL CO., LTD.) 07 December 1999 (1999-12-07) description, paragraphs 5 and 19	8-10
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☒ Further documents are listed in the continuation of Box C.☒ See patent family annex.

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Date of the actual completion of the international search

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INTERNATIONAL SEARCH REPORT

International application No. PCT/CN2019/110868

C. DOCUMENTS CONSIDERED TO BE RELEVANT		
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