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(54) **CORROSION-RESISTANT MIRROR DIE STEEL AND MANUFACTURING METHOD THEREFOR**

(57) Corrosion-resistant mirror die steel and a manufacturing method therefor. The components of the die steel are: 0.35-0.45% of C, 12-15% of Cr, less than or equal to 0.05% of Co, 0.4-0.7% of Mn, 0.35-0.55% of Si, 0.08-0.20% of Mo, 0.10-0.30% of Ni, 0.08-0.30% of W, 0.10-0.30% of V, 0.01-0.05% of Ti, less than or equal to

0.020% of P, less than or equal to 0.012% of S, and the balance is Fe. The manufacturing method comprises: smelting to obtain liquid steel, casting a steel billet, annealing before forging, forging, annealing after forging, and thermal processing.

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Description

Field of the Invention

[0001] The present invention relates to corrosion resistant mirror die steel and a manufacturing method therefor, and belongs to the technical field of die steel products.

Background of the Invention

[0002] The die molding is a common practice for electric appliance parts, electromechanical industrial parts, rubber products, ceramic products and plastic products; so, the quality, efficiency and development ability of the products are largely determined by the die. However, die failure occurs frequently in shape change and dimensional overrun, due to some complex factors such as high temperature, pressure and stress for a long time. Basic failure modes are manifested as surface wear and corrosion, fracture, deformation and accidental damage of the die. Therefore, the die steel should present high wear resistance, corrosion resistance, strength, hardness and other properties. With the most productive and largest proportion in the die steel, plastic die steel has developed rapidly in recent years, which makes a higher demand for product quality.

[0003] Corrosion resistant plastic die steel has medium-high carbon content and high chromium content, and belongs to a higher grade of plastic die steel. It reveals good corrosion resistance against chlorine, fluorine and other gases, and presents good strength, hardness and wear resistance. In China, common steel grades are 2Cr13, 3Cr13, 4Cr13, 9Cr18, 9Cr18Mo and 1Cr17Ni2; whereas, overseas steel grades are represented by Krupp's GS-083 series; of which 4Cr13 is a typical medium carbon high chromium martensitic corrosion resistant steel with good machining property, which can obtain high strength and wear resistance, good polishing property and excellent corrosion resistance after heat treatment (quenching and tempering).

[0004] CN103060698A discloses a preparation process of corrosion resistant die steel, wherein the die steel comprises the following compositions in percentage by weight: 1.0-1.2% of C, 16-19% of Cr, 1.3-1.8% of Co, 0.2-0.6% of Mn, 0.2-0.7% of Si, 0.9-1.4% of Mo, 0.05-0.2% of V, 0.05-0.4% of Ti, 0.05-0.4% of rare earth, and the balance of iron. The preparation method is as follows: treating with Ti and rare earth while melting in an electric furnace; preparing 100-300 kg of ingot, electroslag remelting, and rolling to obtain flat steel with rolling deformation of 50-70%; then heating the flat steel to 650-760 °C, holding for 5-6 h, furnace cooling to 280-320 °C, holding for 3-5 h, then heating to 650-690 °C, holding for 32 h, cooling to 400 °C at 40 °C/h, and then cooling to 120 °C at 18 °C/h; carrying out heat treatment on the flat steel obtained above, heating to 1,000 °C, holding for 1-2 h, oil cooling to not more than 100 °C, reheating

to the temperature range of 680 °C-710 °C, holding for 3 h, and then water cooling; after tempering, heating the head of flat steel to 320-400 °C, holding for 4-5 h, then spray cooling, keeping the tail of flat steel at 900-1,020 °C, holding for 6-8 h, then air cooling, finally reheating the middle of flat steel to 160-190 °C, holding for 2-3 h, and placing in an iron box for stacking and cooling.

[0005] The above preparation process requires the addition of many alloying elements and rare earth elements La and Ce to ensure high hardness and toughness of die steel. Particularly, due to active chemical property, rare earth elements can neutralize the impurities such as oxygen and sulfur in the steel resulting in violent reactions, so as to purify the steel and significantly improve the overall performance of steel. However, the process causes a great waste of precious resources and a significant increase in production costs. In addition, the ingot prepared by this method has small weight, limiting its scope of promotion and application.

Summary of the Invention

[0006] The purpose of the present invention is to provide corrosion resistant mirror die steel and a manufacturing method therefor, aiming at solving the problem of high production cost in the prior art, resulting from the addition of rare earth elements and a large number of alloying elements to improve the overall performance of the die steel.

[0007] The present invention provides a manufacturing method of corrosion resistant mirror die steel, comprising the following steps: smelting into molten steel, casting into billets, pre-forging annealing, forging, post-forging annealing, and heat treating; wherein, the molten steel consists of the following chemical components in percentage by weight: 0.35%-0.45% of C, 12%-15% of Cr, $\text{Co} \leq 0.05\%$, 0.4%-0.7% of Mn, 0.35%-0.55% of Si, 0.08%-0.20% of Mo, 0.10%-0.30% of Ni, 0.08%-0.30% of W, 0.10%-0.30% of V, 0.01-0.05% of Ti, $\text{P} \leq 0.020\%$, $\text{S} \leq 0.012\%$, and the balance of Fe; the heat treating comprises the following steps: heating to 1,120 °C-1,200 °C, holding for 12-20 h, quenching, and tempering at 500-590 °C for 5-20 h.

[0008] Further, the quenching comprises the following steps: discharging, air cooling for 2-3 min, spray cooling for 3-5 min, water cooling to the surface temperature of 690-710 °C, air cooling for 3-5 min, proceeding to water cooling to the surface temperature of 390-410 °C, air cooling for 3-5 min, proceeding to water cooling to the surface temperature of 190-210 °C, and removing from the heat treatment tank for air cooling.

[0009] Further, the spray cooling pressure is 5-8 MPa; the water cooling pressure is 7-16 MPa; the air cooling speed is 2-4 m/s.

[0010] Further, the casting comprises the step of obtaining steel ingots by die casting, wherein the die casting adopt bottom pouring with inert gas protection at the casting nozzle.

[0011] Further, the inert gas is argon.

[0012] Further, electroslag remelting is performed on the billets.

[0013] Further, the slag system used for the electroslag remelting comprises CaF_2 , CaO , Al_2O_3 , MgO and SiO_2 .

[0014] Further, the slag system comprises the following components in parts by weight: 50 parts of CaF_2 , 30 parts of CaO , 10 parts of Al_2O_3 , 5 parts of MgO and 5 parts of SiO_2 .

[0015] Further, the heating temperature is not less than $1,200^\circ\text{C}$, and the holding time is 12-15 h in the pre-forging annealing step.

[0016] Further, the heating temperature is $1230-1250^\circ\text{C}$ in the forging step, and the billets are forged into the specifications with thickness of 300-600 mm, width of 800-1350 mm, length of being more than 3000mm, and total rolling deformation of 60-80%.

[0017] Further, the post-forging annealing step comprises the following sub-steps: heating the forged module to $600-650^\circ\text{C}$, holding for 4-8 h, furnace cooling to $280-350^\circ\text{C}$, holding for 2-6 h, heating to $650-700^\circ\text{C}$, holding for 25-35h, cooling to $390-410^\circ\text{C}$ at $30-60^\circ\text{C/h}$, and cooling to $140-160^\circ\text{C}$ at $15-20^\circ\text{C/h}$.

[0018] The present invention provides corrosion resistant mirror die steel obtained according to the manufacturing method.

[0019] According to the present invention, novel corrosion resistant mirror die steel of optimized Changzhi steel 4Cr13 is developed by adopting a vanadium-titanium micro-alloyed composition route, and the die steel mainly has the following advantages: 1, high hardness, almost HRC35-50; 2, small hardness fluctuation range, $\leq 1.5\text{HRC}$; 3, high corrosion resistance, the surface of the specimen is bright as before after the specimen is soaked in 50% high concentration nitric acid at 50°C for 120 h and in 15% acetic acid for 48 h; additionally, the surface of the specimen has not lost its metallic luster and no pitting corrosion is observed after soaking in hydrochloric acid medium at room temperature for 48 h; 4, high toughness, the impact value can reach 31J at room temperature; and 5, large module die steel can be obtained (thickness: 300-600mm, width: 800-1350mm, length > 3000mm).

Detailed Description of the Preferred Embodiments

[0020] The raw materials and equipments used in specific embodiments of the present invention are all well-known products, which can be purchased in the market.

[0021] The molten steel obtained by smelting in the present invention has the following chemical components in percentage by weight: 0.35%-0.45% of C, 12%-15% of Cr, $\text{Co} \leq 0.05\%$, 0.4%-0.7% of Mn, 0.35%-0.55% of Si, 0.08%-0.20% of Mo, 0.10%-0.30% of Ni, 0.08%-0.30% of W, 0.10%-0.30% of V, 0.01-0.05% of Ti, $\text{P} \leq 0.020\%$, $\text{S} \leq 0.012\%$, and the balance of Fe.

[0022] Wherein, the Cr content is controlled to be not less than 12%, so as to ensure good corrosion resistance

of the die steel.

[0023] Role of Mo: on the one hand, Mo forms M_6C -type carbide in steel, which precipitates to increase the solid solubility of Cr and improve wear resistance; on the other hand, the addition of Mo can cause dispersion hardening after tempering, which is conducive to improving the secondary hardness and thermal stability of steel, increasing the tempering brittleness temperature, and avoiding the occurrence of tempering brittleness.

[0024] Role of Ni: the addition of a small amount of Ni can improve the toughness of steel, the thermal fatigue performance of modules and the hardenability.

[0025] Role of V: 1, vanadium can improve the thermal strength, creep resistance and high-temperature durability of steel; 2, vanadium can improve the stability of steel in high-temperature and high-pressure hydrogen, so that the stability of steel to hydrogen can be more than 600°C at high pressure; 3, in pearlite low-alloy steel, vanadium can prevent the graphitization of molybdenum steel at high temperature; and 4, through the precipitation in the final ferrite structure, VN precipitates are formed in a dispersed and finely distributed manner to improve the strength, toughness and fatigue resistance.

[0026] Role of Ti: the addition of trace Ti forms Ti (CN) precipitates, which play a role in refining the grain during the heating process of the slab; whereas fine and diffusely distributed TiC precipitates in the final ferrite organization play a role in strengthening precipitation, and also improve the welding performance of the finished products.

[0027] In summary, the present invention achieves the effect of improving the comprehensive mechanical properties of the die steel, especially the corrosion resistance and wear resistance of the steel, mainly by controlling the content of Cr, Mo, Ni, V, Ti and other elements, thereby avoiding the use of rare earth metals, reducing the total addition amount of alloy elements and significantly reducing the production cost.

[0028] In addition, impurity elements S and P have a detrimental effect on the toughness of the die steel. In high-temperature service, the dynamic polarization of S, P and other impurity elements toward the grain boundary will damage the high-temperature plasticity and toughness of dies, resulting in their high-temperature embrittlement cracking. Relevant studies have shown that the reduction of S and P content helps to improve the cold and hot fatigue properties of steel.

[0029] The present invention is able to control the P content below 0.020% and the S content below 0.012% through the refining process by avoiding the use of rare earth metals and reducing the total addition of alloying elements and the introduction of impurities such as S and P in general, thereby reducing or even eliminating the harm of trace impurity elements, improving the quality of die steel, and providing them with excellent corrosion resistance, wear resistance, hardness, toughness and other properties.

[0030] The manufacturing method of corrosion resist-

ant mirror die steel of the present invention comprises a heat treating step, which is the key to ensure the comprehensive mechanical properties of the die steel. The specific process comprises the following steps: heating to 1,120 °C-1,200 °C, holding for 12-20 h, quenching, and tempering at 500-590 °C for 5-20 h. The quenching + high-temperature tempering treatment of steel by using this method can fully strengthen micro-alloy and control uniform precipitation, eliminate white spots, improve the transverse properties of the die, refine grains, and achieve uniform structure, so as to meet the requirements of mirror polishing of die steel and ensure its corrosion resistance, wear resistance and other properties.

[0031] Another advantage of the above heat treatment process lies in tempering treatment of large modules (thickness: 300-600 mm, width: 800-1,350 mm, length>3000mm). The heat treatment process for large modules of corrosion resistant and wear-resistant mirror plastic die steel (hardness range: HRC35-50, hardness fluctuation $\leq 3\text{HRC}$) has not yet been reported.

Example 1 Manufacturing of corrosion resistant mirror die steel of the present invention

[0032] The production process of the die steel of the present invention is as follows: primary smelting in electric furnace → vacuum treatment outside the refining furnace (LF+VD furnace refining) → molten steel die casting → electroslag remelting → large module heating → forging processing (thickness: 300-600 mm, width: 800-1350 mm, length >3000mm) → finished module annealing → heat treatment (quenching + tempering) → packaging and warehousing.

[0033] Smelting: a) obtaining molten steel with nitrogen content being not more than 60 ppm through initial smelting in an eccentric hearth tapping electric furnace; carrying out large slag refining in a refining furnace; performing alloying quantitative adjustment and deep P and S removal from white slag to ensure that the oxidation temperature is more than 1,580 °C and there is a certain carbon content of molten steel at the end of oxidation, when the method of increasing oxidation flow is used for rapid decarburization; preferably, the steel temperature is controlled to be not less than 1,650 °C; and b) carrying out the LF furnace refining after the electric furnace melting, so as to achieve the purposes of deoxidation, reducing slag oxidation, improving alloy yield, adjusting the composition of the slag system, forming a slag system with low melting point and effectively absorbing inclusions in the molten steel, cleaning the ladle, improving the alkalinity of steel ladle and removing harmful impurity sulfur from molten steel; afterwards, degassing the molten steel in a vacuum state by melting in a vacuum degassing furnace to reduce [H] and [N] in the molten steel. The molten steel obtained by smelting has the following chemical components in percentage by weight: 0.35%-0.45% of C, 12%-15% of Cr, $\text{Co} \leq 0.05\%$, 0.4%-0.7% of Mn, 0.35%-0.55% of Si, 0.08%-0.20% of Mo, 0.10%-

0.30% of Ni, 0.08%-0.30% of W, 0.10%-0.30% of V, 0.01-0.05% of Ti, $\text{P} \leq 0.020\%$, $\text{S} \leq 0.012\%$, and the balance of Fe.

[0034] Die casting: based on bottom pouring, casting in the presence of argon to avoid oxidation at the casting nozzle, and weak blowing argon for being not less than 15 min before VD furnace tapping. Casting temperature: 1,530-1,540 °C; air in the ingot die is discharged by blowing argon in the ingot die before casting steel to prevent nitrogen in air from entering the molten steel, resulting in nitrogen increase.

[0035] Electroslag remelting: the method of electroslag smelting is used to control the quality of steelmaking considering that the steel structure should be uniform and the hard spots like internal oxide inclusions should be as little as possible. The specific process is as follows: one tip of the consumable electrode is inserted into the molten slag that is contained in the copper water-cooled crystallizer. The consumable electrode, slag bath, metal melt pool, ingots, bottom water tank are formed into a circuit through a short-networked conductors and transformers. In the process of energizing, the slag bath emits Joule heat to gradually melt the consumable electrode tip, and the molten metal converges into liquid droplets, which pass through the slag bath and fall into the crystallizer, forming a metal melt pool, where it is rapidly solidified to form ingot by water cooling. In the stages of forming electrode tip droplets and dropping droplets through the slag bath, steel and slag are in full contact and non-metallic inclusions in steel are absorbed by the slag. Harmful elements in the steel (sulfur, lead, antimony, bismuth and tin) are removed relatively effectively by means of the steel-slag reaction and high temperature gasification. Liquid metal is covered by the slag bath and will not be reoxidized bacially. The melting, refining and solidification process occurs in a copper water-cooled crystallizer, which eliminates the pollution of steel by refractory materials. The slag system is a five-element slag system, which is based on CaF_2 and mixed with proper oxides such as CaO , Al_2O_3 , MgO and SiO_2 . The ratio of the five element slag system is 50% of CaF_2 , 30% of CaO , 10% of Al_2O_3 , 5% of MgO , 5% of SiO_2 , which is well adaptative to the special steel electroslag remelting, and has high resistance and melting speed.

[0036] Pre-forging high-temperature diffusion annealing: the heating temperature should be not less than 1,200 °C and the holding time is 12-15 h.

[0037] Forging: a) at the forging heating temperature of 1,230-1,250 °C, forging large module die steel ingots into the required size with thickness of 300-600 mm, width of 800-1,350mm, length of being more than 3000mm and rolling deformation of 60-80%; and placing the forged large module for holding for 4-8 h, and furnace cooling to 280-350 °C and holding for 2-6 h; and b) reheating the ingots obtained in step a to 650-700 °C, holding for 25-35 h, cooling to 400 °C at 30-60 °C/h, and then cooling to 150 °C at 15-20 °C/h.

[0038] Heat treatment: a) heating the large module die

steel obtained in steps 1), 2) and 3) to 1,120 °C-1,200 °C, and holding for 12-20 h; quenching: discharging, air cooling for 2-3 min, spray cooling for 3-5 min, water cooling to the surface temperature of 700 °C, air cooling for 3-5 min, proceeding to water cooling to 400 °C, air cooling for 3-5 min, proceeding to water cooling to 200 °C, and removing from the heat treatment tank for air cooling; wherein the spray cooling pressure is 5-8 MPa, the air cooling speed is 2-4 m/s, and the water cooling pressure is 7-16 MPa; and (b) tempering process: the tempering temperature is 500-590 °C, and tempering time is 5-20 h.

[0039] For the prepared die steel 510×1,080×3,500mm, three different locations including the surface edge, 1/4 thickness and the heart are tested, indicating that the hardness values are HRC47.5, HRC46.5 and HRC46, with a hardness fluctuation range not more than 1.5HRC, and the impact value is 31J at room temperature (20°C). The surface of the specimen is bright as before and the quality thereof remains the same after the specimen is soaked in 50% high concentration nitric acid at 50 °C for 120 h and in 15% acetic acid for 48 h; additionally, the surface of the specimen has not lost its metallic luster and no pitting corrosion is observed after soaking in hydrochloric acid medium at room temperature for 48 h.

Claims

1. A manufacturing method of corrosion resistant mirror die steel, **characterized by** comprising the following steps: smelting into molten steel, casting into billets, pre-forging annealing, forging, post-forging annealing, and heat treating; wherein, the molten steel consists of the following chemical components in percentage by weight: 0.35%-0.45% of C, 12%-15% of Cr, $\text{Co} \leq 0.05\%$, 0.4%-0.7% of Mn, 0.35%-0.55% of Si, 0.08%-0.20% of Mo, 0.10%-0.30% of Ni, 0.08%-0.30% of W, 0.10%-0.30% of V, 0.01-0.05% of Ti, $\text{P} \leq 0.020\%$, $\text{S} \leq 0.012\%$, and the balance of Fe; the heat treating comprises the following steps: heating to 1,120 °C-1,200 °C, holding for 12-20 h, quenching, and tempering at 500-590 °C for 5-20 h.
2. The manufacturing method according to claim 1, **characterized in that** the quenching comprises the following steps: discharging, air cooling for 2-3 min, spray cooling for 3-5 min, water cooling to the surface temperature of 690-710 °C, air cooling for 3-5 min, proceeding to water cooling to the surface temperature of 390-410 °C, air cooling for 3-5 min, proceeding to water cooling to the surface temperature of 190-210 °C, and removing from the heat treatment tank for air cooling.
3. The manufacturing method according to claim 2, **characterized in that** the spray cooling pressure is

5-8 MPa; the water cooling pressure is 7-16 MPa; the air cooling speed is 2-4 m/s.

4. The manufacturing method according to claim 1, **characterized in that** the casting comprises the step of obtaining steel ingots by die casting, wherein the die casting adopt bottom pouring with inert gas protection at the casting nozzle.
5. The manufacturing method according to claim 4, **characterized in that** the inert gas is argon.
6. The manufacturing method according to claim 1, **characterized in that** electroslag remelting is performed on the billets.
7. The manufacturing method according to claim 6, **characterized in that** the slag system used for the electroslag remelting comprises CaF_2 , CaO, Al_2O_3 , MgO and SiO_2 .
8. The manufacturing method according to claim 7, **characterized in that** the slag system comprises the following components in parts by weight: 50 parts of CaF_2 , 30 parts of CaO, 10 parts of Al_2O_3 , 5 parts of MgO and 5 parts of SiO_2 .
9. The manufacturing method according to claim 1, **characterized in that** the heating temperature is not less than 1,200 °C, and the holding time is 12-15 h in the pre-forging annealing step.
10. The manufacturing method according to claim 1, **characterized in that** the heating temperature is 1230-1250 °C in the forging step, and the billets are forged into the specifications with thickness of 300-600 mm, width of 800-1350 mm, length of being more than 3000mm, and total rolling deformation of 60-80%.
11. The manufacturing method according to claim 1, **characterized in that** the post-forging annealing step comprises the following sub-steps: heating the forged module to 600-650 °C, holding for 4-8 h, furnace cooling to 280-350 °C, holding for 2-6 h, heating to 650-700 °C, holding for 25-35 h, cooling to 390-410 °C at 30-60 °C/h, and cooling to 140-160 °C at 15-20 °C/h.
12. The corrosion resistant mirror die steel obtained by the manufacturing method according to any one of claims 1 to 11.

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2019/097543

A. CLASSIFICATION OF SUBJECT MATTER

C21D 1/18(2006.01)i; C22C 38/50(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)

C21D 1, C22C 38

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)

VEN; CNABS: 模具钢, 耐腐蚀, 锻造, 铬, 钨, 镍, 钒, mould steel, corrosion resist+, forg+, Cr, chrome, W, tungsten, Ni, nickel, V, vanadium

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
PX	CN 108866444 A (PANZHIHUA IRON AND STEEL RESEARCH INSTITUTE, PANGANG GROUP) 23 November 2018 (2018-11-23) claims 1-10	1-12
X	JP 2015045071 A (SANYO SPECIAL STEEL CO., LTD.) 12 March 2015 (2015-03-12) description, paragraphs [0017]-[0029] and [0033]-[0042], and table 2	1-12
A	CN 1876881 A (DAIDO STEEL CO., LTD.) 13 December 2006 (2006-12-13) entire document	1-12
A	KR 20160041869 A (DOOSAN HEAVY INDUSTRIES & CONSTRUCTION CO., LTD.) 18 April 2016 (2016-04-18) entire document	1-12

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Date of the actual completion of the international search

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Name and mailing address of the ISA/CN

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Telephone No.

Form PCT/ISA/210 (second sheet) (January 2015)

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/CN2019/097543

Patent document cited in search report	Publication date (day/month/year)	Patent family member(s)	Publication date (day/month/year)
CN 108866444 A	23 November 2018	None	
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CN 1876881 A	13 December 2006	None	
KR 20160041869 A	18 April 2016	None	

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REFERENCES CITED IN THE DESCRIPTION

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