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(54) **ZINC-ALUMINUM-MAGNESIUM COATED STEEL PLATE AND PRODUCTION PROCESS THEREFOR**

(57) Disclosed are a zinc-aluminum-magnesium coated steel sheet and a production process therefor, belonging to the technical field of alloys. A plating solution for the coating consists of the following components in percentage by mass: 1.5-2.0% of Mg, 0.2-3.5% of Al and the balance being Zn and inevitable impurities. The production process for the zinc-aluminum-magnesium coated steel sheet comprises the procedures of smelting, continuous casting, hot rolling, acid rolling, annealing, hot dipping, coiling and high-temperature forming, wherein in the hot dipping process, the temperature of the plating solution is controlled to be 440-480°C, and the time

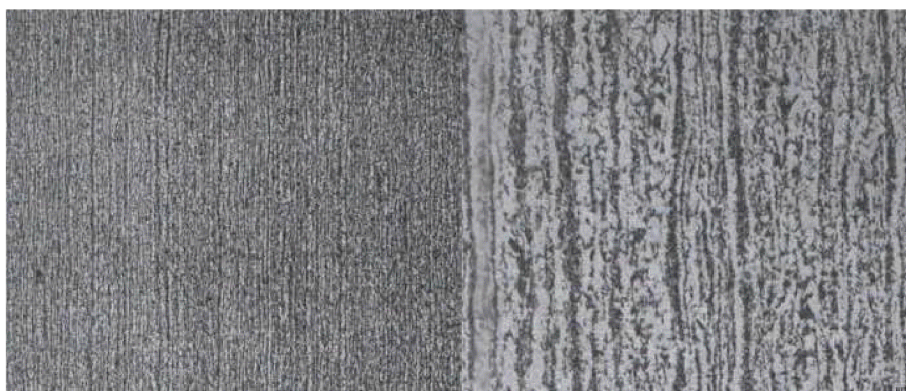
period for dipping is controlled to be 5-10s, and after the hot dipping, the steel sheet is cooled to room temperature at more than 30C/s. In view of the problem of liquid metal embrittlement resulting from the hot forming procedure of the zinc-based coated steel sheet, the microstructure composition of the coating is improved by changing the composition ratio of chemical elements in the plating solution and process optimization, the reaction of the zinc-aluminum-magnesium coated steel sheet and Zn is inhibited, and a cracking problem on the surface of the steel sheet during high-temperature forming is alleviated.

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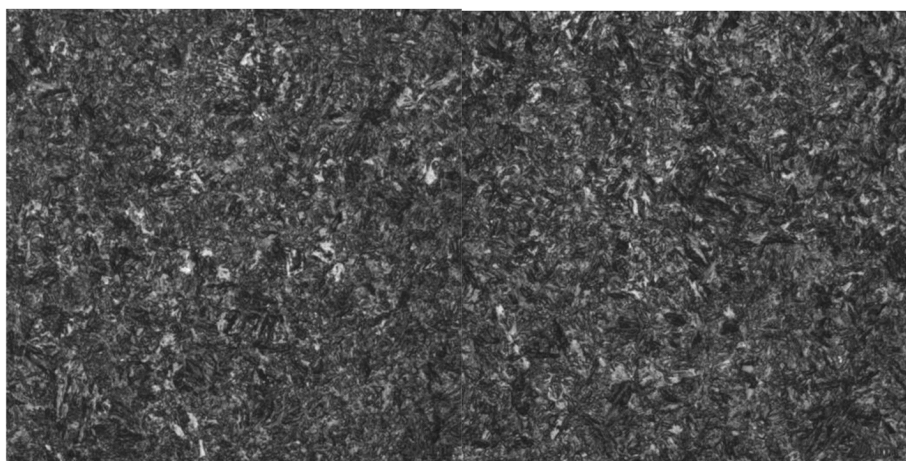
(a)

(b)



(c)

(d)



(e)

(f)

Fig.1

Description**FIELD OF THE INVENTION**

[0001] The present invention relates to the technology field of alloys, in particular to zinc-aluminum-magnesium coated steel sheets and a production process therefor.

BACKGROUND OF THE INVENTION

[0002] The international environment of energy conservation and emission reduction has driven significant changes in the automotive industry. Therefore, it is of great significance to reduce the overall weight of vehicles to achieve goals of energy conservation and emission reduction. By applying hot forming technologies, it is possible to reduce the weight of vehicle bodies, improve fuel economy, and achieve the objectives of energy conservation and emission reduction, while also ensuring automotive safety to some extent. Commonly used hot forming steels includes uncoated steel, aluminum-silicon coated steel, and zinc-based coated steel. Uncoated steel sheets are prone to surface oxidation and decarburization during hot forming, and exhibit poor corrosion resistance and reduced surface fatigue strength in service. The application of aluminum-silicon coatings can prevent steel surfaces from being oxidized and decarburized, but their production costs are high and their corrosion resistance at perforations and cut edges is poor. Researchers are progressively placing their focuses on zinc-based coatings which have lower production costs and can provide excellent cathodic protection. However, zinc-based coating steels faces issues such as volatilization of zinc-based coatings during hot forming and brittle fractures caused by infiltration of liquid metal from the coating into the substrate. These problems limits their use in the automotive field.

[0003] According to literature 1 (Bi Wenzhen, Hong Jiyao, Wang Li. Research status on zinc-coated press-hardness steel [J]. Baogang Technology, 2019(06):38-44.), the causes of liquid metal cracking are as follows. During the high-temperature forming process of pure Zn coated steel sheets, the coating consists of a solid α -Fe(Zn) phase and a liquid Zn phase, with the liquid Zn phase existing among the α -Fe(Zn) phases. Cracks initiate at the interface between the solid α -Fe(Zn) phase and the liquid Zn phase. The liquid Zn continuously diffuses along austenite grain boundaries and reacts with the substrate to form an α -Fe(Zn) layer. Under the action of external stress, cracks continuously extend into the substrate along the interface between the α -Fe(Zn) phase and the liquid Zn phase, thereby causing substrate fracture. Based on the above cause, it is possible to add alloying elements such as Al and Ni to a pure zinc plating bath to form an alloy inhibition layer such as Fe_2Al_5 at a coating-substrate interface, which hinders the diffusion of zinc element into the substrate, in other words, inhibits reactions between zinc and Fe in the substrate and prevents the formation of brittle Fe-Zn phases.

[0004] According to literature 2 (Qiao Changle. Research on the Microstructural Evolution and Cracking Control of 22MnB5 Steel's Zn-Based Coating [D]. Northeastern University, 2018. DOI:10.27007/d.cnki.gdbeu.2018.000250.), it is known that Al elements added to the pure zinc plating bath react with Fe elements prior to Zn elements during the hot dipping to form an Fe_2Al_5 alloy inhibition layer, which hinders the reaction between Fe and Zn. The existence of the inhibition layer effectively inhibits the formation of brittle phases. However, as the temperature rises, the Al elements at an alloy layer transfer to the coating surface during austenitization of a coated sheet, the Fe_2Al_5 alloy inhibition layer is destroyed and loses its inhibitory effect, and a large amount of Zn elements in the coating diffuse into the substrate, forming α -Fe(Zn) phase, which causes substrate fractures.

[0005] To address the above issues, literature 3 (*Ultra-High Strength Zinc-Aluminum-Magnesium Coated Steel Sheet For High-Temperature Forming And Manufacturing Method Therefore*, CN107099748B[P]. 2019) discloses an ultra-high-strength zinc-aluminum-magnesium coated steel sheet for high-temperature forming and its manufacturing method. The coating compositions includes 1.5%~20% of Al, 1.3%~7% of Mg, and the balance of Zn and inevitable impurities. Its coating, before high-temperature forming, includes three microstructures of Zn/Al eutectic structure, Zn/Al/ Zn_2Mg ternary eutectic structure, and pure Zn particle. However, this technology still has the following problems: the content of the Zn/Al/ Zn_2Mg ternary eutectic structure in an alloy coating and the enrichment of zinc are difficult to control, and the high-temperature reaction between the locally-enriched Zn and the steel substrate is difficult to inhibit, causing high-temperature cracking of hot-dip galvanized steel sheets. Moreover, in order to form Zn/Al and Zn/Al/ Zn_2Mg eutectics, it is necessary to add Al and Mg to a higher content. For hot forming steels, the volatilization of Mg and Zn during the high-temperature austenitization can easily lead to the formation of pores and cracks on the coating surface, resulting in a decrease in the surface quality of steel sheets.

SUMMARY OF THE INVENTION

[0006] An objective of the present invention is to provide a low-aluminum, low-magnesium zinc-aluminum-magnesium coated steel sheet for high-temperature forming and a production process therefor. Aiming at the problem of liquid metal embrittlement induced in zinc-based coated steel sheets during high-temperature forming, the present invention improves

the microstructure of the coating and inhabits the reaction between the zinc-aluminum-magnesium coated steel sheet and Zn by modifying the chemical composition ratio of the plating bath and optimizing the production process, alleviating the problem of surface cracking of steel sheets during high-temperature forming. The present invention also elaborates on the chemical composition design of steel sheets, the entire production process, and the preparation method of the low-aluminum, low-magnesium zinc-aluminum-magnesium coated steel sheet, ensuring efficient production.

[0007] An aspect of the present invention is to provide a zinc-aluminum-magnesium coated steel sheet, including a coating made from a plating bath including the following components in percentage by mass: 1.5~2.0% of Mg, 0.2~3.5% of Al, and the balance of Zn and inevitable impurities.

[0008] Prior to high-temperature forming, the microstructure of the coating of the zinc-aluminum-magnesium coated steel sheet according to the present invention varies slightly depending on the Al content in the plating bath. The coating contains a large number of dendritic structure, with Mg/Zn eutectic phase distributed in interdendritic spaces. When the Al content in the plating bath is at 0.2%~1.5%, a Mg/Zn eutectic region mainly contains Mg_2Zn_{11} phases, and when in the Al content in the plating bath is at 1.5%~3.5%, the Mg/Zn eutectic region mainly contains $MgZn_2$ phases. In an intermediate layer at an interface between the coating and a steel substrate, Al and Fe elements form Fe_2Al_5 phases, which more obviously enrich in the intermediate layer as the Al content increases, making the intermediate layer denser. Meanwhile, the increase in Al content further refines the grains in the coating. When the Al content ranges from 2.5% to 3.5%, the steel sheet exhibits the best resistance to corrosion and liquid metal cracking.

[0009] Preferably, the plating bath includes the following components in percentage by mass: 1.5~2.0% of Mg, 2.5~3.5% of Al, and the balance of Zn and inevitable impurities.

[0010] The coating in the present invention contains a large number of dendritic structure, with Mg/Zn eutectic phase distributed in interdendritic spaces. The Mg/Zn eutectic in the present invention is $MgZn_2$ eutectic or Mg_2Zn_{11} eutectic.

[0011] Furthermore, when the Al content in the plating bath is at 0.2%~1.5%, the Mg/Zn eutectic region mainly contains Mg_2Zn_{11} phases, and when the Al content in the plating bath is at 1.5%~3.5% (excluding 1.5%), the Mg/Zn eutectic region mainly contains $MgZn_2$ phases.

[0012] The microstructure of the coating of the zinc-aluminum-magnesium coated steel sheet according to the present invention varies depending on the Al content in the plating bath. In the case that the Al content in the plating bath is at 0.2%~1.5%, the primary phase in the Mg/Zn eutectic region is the Mg_2Zn_{11} phase, with a small amount of $MgZn_2$ phase; in the case that the Al content in the plating bath exceeds 1.5%, the primary phase in the Mg/Zn eutectic region is the $MgZn_2$ phase, with a small amount of Mg_2Zn_{11} phase.

[0013] As to the coating of the present invention, in an intermediate layer at an interface between the coating and a steel substrate, Al and Fe elements form Fe_2Al_5 phases, which more obviously enrich in the intermediate layer as the Al content increases, making the intermediate layer denser and grains in the coating finer.

[0014] The zinc-aluminum-magnesium coated steel sheet of the present invention comprises a steel substrate including the following components in percentage by mass: 0.30~0.36% of C, 0.05~0.5% of Si, 1.0~2.0% of Mn, not more than 0.050% of P, not more than 0.008% of S, not less than 0.080% of Al, 0.020~0.040% of Ti, 0.1~0.3% of V, not more than 0.005% of O, not more than 0.005% of N, 0.01~0.05% of rare earth (RE), 0.0005~0.0040% of B, and the balance of Fe and inevitable impurities.

[0015] The zinc-aluminum-magnesium coated steel sheet of the present invention has a thickness of 1.0mm-2.5mm, a width of 850mm-1800mm, a tensile strength of ≥ 1900 MPa after high-temperature forming, and an elongation of $\geq 6\%$.

[0016] Another objective of the present invention is to provide a production process for the above-mentioned zinc-aluminum-magnesium coated steel sheet.

[0017] The production process includes steps of smelting, continuous casting, hot rolling, pickling and cold rolling, annealing, hot dipping, coiling, and high-temperature forming. During the hot dipping, a temperature of the plating bath is controlled within 440 °C to 480 °C, a dipping time ranges from 5 s to 10 s, and a resulting steel sheet after dipping is then cooled to room temperature at a cooling rate of more than 30°C/s to obtain a coated steel sheet.

[0018] Furthermore, during the annealing, a dew point is controlled within -50 °C to -20 °C, a heating temperature is controlled within 700 °C to 850 °C, then a resulting heated steel sheet is cooled to 420 °C to 500 °C at a cooling rate ranging from 30°C/s to 50°C/s to obtain an annealed steel sheet.

[0019] Furthermore, the high-temperature forming employs a direct forming process, including heating the coated steel sheet to a temperature ranging from 850 °C to 900 °C and holding it for 4-10 minutes, followed by quickly transferring it to a mold for stamping, with a mold-closing temperature controlled within 700 °C to 750 °C to obtain a part. After stamping, the part is quickly cooled to room temperature in the mold at a cooling rate ranging from 100°C/s to 140 °C/s.

[0020] Furthermore, the steel substrate is smelted by the following method: a pretreated molten iron is smelted in a converter, followed by refining in LF and RH furnaces. The pretreatment of molten iron ensures that a sulfur content of finished products meets internal control requirements. The phosphorus content of the molten iron entering the converter is $\leq 0.1\%$. A tapping temperature from the converter is ≥ 1600 °C. In the LF furnace, nitrogen increase is controlled to ≤ 20 ppm. After refining in RH furnace, the resulting molten iron is held for ≥ 15 minutes.

[0021] Furthermore, during the continuous casting, a casting speed is controlled within 1.6 m/min to 2.4 m/min and

maintained constant to obtain a continuous casting slab.

[0022] Furthermore, during the hot rolling, the continuous casting slab is heated to 1250 °C to 1300 °C, and subjected to finishing rolling at a temperature ranging from 780 °C to 900 °C, and a coiling temperature is 600 °C to 720 °C.

[0023] [The present invention has the following beneficial effects:

Upon inspection, in the salt spray test of the Zn-Al-Mg coating, SEM and EDS characterization shows that the $MgZn_2$ phase in the Mg-containing coating of the present invention corrodes first, thereby protecting the Zn dendritic, and the Mg-Zn phase enhances the corrosion resistance of the coating. Compared with pure zinc phase, two Mg-Zn phases of $MgZn_2$ and Mg_2Zn_{11} exhibit lower anodic dissolution currents, which also improves the corrosion resistance of the coating. After the completion of hot dipping, Al elements in the coating are enriched at the interface between the coating and the steel substrate, reacting with Fe elements to form an Fe-Al intermetallic alloy layer, which hinders the diffusion of Zn into the steel substrate and prevents the formation of fine cracks. After austenitization, the coated steel sheet exhibits a continuous and intact coating without cracks diffusing into the steel substrate. Since Mg is prone to oxidation upon heating, it quickly diffuses to the surface of the coating at high temperatures, forming an oxide layer that prevents Zn volatilization, and the position of the Fe-Al alloy layer migrates toward the surface of the coating. In addition, the hot stamping forming process for the steel sheet in the present invention, compared with conventional processes, lowers the heating temperature and reduces energy consumption while meeting performance requirements, aligning with the requirements of green and low-carbon development.

BRIEF DESCRIPTION OF THE DRAWINGS

[0024]

Fig. 1 shows microstructural photographs of the steel sheets after continuous annealing and hot stamping forming in Example 1, in which panels (a) and (b) are microstructural photographs of Sample 1 at 100x and 500x magnifications, respectively, panels (c) and (d) are microstructural photographs of Sample 2 at 100x and 500x magnifications, respectively, and panels (e) and (f) are microstructural photographs of Sample 3 and Sample 4 at a 500x magnification, respectively.

Fig. 2 shows a microstructural photograph of the Zn-Al-Mg alloy coating of the steel sheet after hot dipping in Example 1.

Fig. 3 shows a microstructural photograph of a cross-section of the coating of the steel sheet after austenitization in Example 1.

Fig. 4 shows a microstructural photograph of a cross-section of the coating of the steel sheet after hot dipping in Comparative Example 1.

Fig. 5 shows a microstructural photograph of a cross-section of the coating of the steel sheet after austenitization in Comparative Example 1.

Fig. 6 shows crack morphologies at bent parts of the steel sheets in Example 1 and Comparative Example 1, in which panel (a) is a crack morphology at a bent part of the Zn-3.5Al-1.8Mg coated steel sheet after hot stamping forming in Example 1, and panel (b) shows a crack morphology at a bent part of the pure Zn coated steel sheet after hot stamping forming in Comparative Example 1.

Fig. 7 shows a microstructural photograph of a cross-section of the coating of the steel sheet after austenitization in Example 2.

Fig. 8 shows a microstructural photograph of a cross-section of the coating of the steel sheet after austenitization in Example 2.

Fig. 9 shows microstructural photographs of the steel sheet after hot stamping forming in Example 3, in which panels (a) and (b) are microstructural photographs of Sample 5 and Sample 6 at 500x magnification, respectively.

Fig. 10 shows an energy dispersive spectroscopy (EDS) analysis of a cross-section of the coating of the steel sheet after hot stamping forming in Example 4.

Fig. 11 shows microstructural photographs of the steel sheet after hot stamping forming in Example 4, in which panels

(a) and (b) are microstructural photographs of Sample 7 and Sample 8 at 500x magnification, respectively.

Fig. 11 shows microstructural photographs of the steel sheet after hot stamping forming in Example 4, in which panels (a) and (b) are microstructural photographs of Sample 7 and Sample 8 at 500x magnification, respectively.

Fig. 12 shows an EDS analysis of a cross-section of the coating of the steel sheet after hot stamping forming in Example 5.

Fig. 13 shows microstructural photographs of the steel sheet after hot stamping forming in Example 5, in which panels (a) and (b) are microstructural photographs of Sample 9 and Sample 10 at 500x magnification, respectively.

Fig. 14 shows salt spray test results of the steel sheets with different coatings, in which panels (a) to (c) are test results at 81 hours for the Zn-3.5Al-1.8Mg coating, pure Zn coating, and Al-Si coating, respectively; panels (d) and (e) are test results at 157 hours for the Zn-3.5Al-1.8Mg coating and Al-Si coating, respectively; panels (f) and (g) are test results at 229 hours for the Zn-3.5Al-1.8Mg coating and Al-Si coating, respectively; panels (h) and (i) are test results at 283 hours for the Zn-3.5Al-1.8Mg coating and Al-Si coating, respectively; and panels (j) and (k) are test results at 396 hours for the Zn-3.5Al-1.8Mg coating and Al-Si coating, respectively.

Fig. 15 shows polarization curve test results of the steel sheets with different coatings, in which panels (a) and (b) are test results of the Zn-3.5Al-1.8Mg coating, panels (c) and (d) are test results of the pure Zn coating, and panels (e) and (f) are test results of the Al-Si coating.

DETAILED DESCRIPTION OF SOME EMBODIMENTS

[0025] The non-limiting embodiments hereinafter can enable those skilled in the art to more fully understand the present invention, but do not pose any limitations on the present invention.

[0026] The test methods described in the following embodiments, unless otherwise specified, are conventional methods. The reagents and materials used herein, unless otherwise specified, are commercially available.

[0027] The Chinese patent ZL201610535069.3 discloses an uncoated hot-stamped steel sheet, which is a 2GPa hot-stamped steel product developed by Bengang steel sheets Co., Ltd. in 2017. It has been industrially applied to door anti-collision beams of BAIC New Energy vehicles, achieving a global first launch. To further improve oxidation resistance, break free from constraints of foreign patents, and promote localization process of hot stamping steels, the present invention is based on the aforementioned uncoated hot-stamped steel product, makes a modification on the process route and develops a low-aluminum, low-magnesium zinc-aluminum-magnesium coated hot-stamped steel sheet for automobiles and a production process therefor.

[0028] The present invention provides a low-aluminum, low-magnesium zinc-aluminum-magnesium coated steel sheet for high-temperature forming and its production process.

[0029] A steel substrate of the steel sheet includes the following components in percentage by mass: 0.30~0.36% of C, 0.05~0.5% of Si, 1.0~2.0% of Mn, not more than 0.050% of P, not more than 0.008% of S, not less than 0.080% of Al, 0.020~0.040% of Ti, 0.1~0.3% of V, not more than 0.005% of O, not more than 0.005% of N, 0.01~0.05% of RE, 0.0005~0.0040% of B, and the balance of Fe and inevitable impurities.

[0030] The plating bath for preparing the coating includes the following components in percentage by mass: 1.5~2.0% of Mg, 0.2~3.5% of Al, and the balance of Zn and inevitable impurities.

[0031] The production process for the low-aluminum, low-magnesium zinc-aluminum-magnesium coated steel sheet for high-temperature forming includes steps of smelting, continuous casting, hot rolling, pickling and cold rolling, annealing, hot dipping, coiling, and high-temperature forming.

[0032] During the annealing, a dew point is controlled within -50 °C to -20 °C, a heating temperature is controlled within 700 °C to 850 °C. During the hot dipping, a temperature of the plating bath is controlled within 440 °C to 480 °C, a dipping time ranges from 5s to 10s, and a resulting steel sheet after dipping is cooled to room temperature at a cooling rate more than 30°C/s to obtain a coated steel sheet. The high-temperature forming employs a direct forming process, including heating the coated steel sheet to a temperature ranging from 850 °C to 900 °C, then holding it for 4~10 minutes. After stamping, the part is quickly cooled to room temperature.

[0033] The steel substrate is smelted by the following method: a pretreated molten iron is smelted in a converter, followed by refining in LF and RH furnaces. The pretreatment of molten iron ensures that a sulfur content of finished products meets internal control requirements. The phosphorus content of the molten iron entering the converter is $\leq 0.1\%$. A tapping temperature from the converter is $\geq 1600^\circ\text{C}$. In the LF furnace, nitrogen increase is controlled to ≤ 20 ppm. After refining in RH furnace, the resulting molten iron is held for ≥ 15 minutes.

[0034] During the continuous casting, a casting speed is controlled within 1.6 m/min to 2.4 m/min and maintained

constant, and a resulting continuous casting slab is required to be free of burrs.

[0035] During the hot rolling, the continuous casting slab is heated in a heating furnace to 1250 °C to 1300 °C, and is subjected to finishing rolling at a temperature ranging from 780 °C to 900 °C, and a coiling temperature ranges from 600 °C to 720 °C. A holding time for the continuous casting slab in the heating furnace is more than 110 minutes, and a temperature of the continuous casting slab is below 1150°C when entering the finishing rolling.

[0036] During the annealing, a heating temperature is controlled within 700 °C to 850 °C, and a resulting heated steel sheet is then cooled to 420 °C to 500 °C at a cooling rate ranging from 30 °C/s to 50 °C/s.

[0037] The coated steel sheet produced by the above method has a thickness of 1.0mm-2.5mm, a width of 850mm-1800mm, a tensile strength after high-temperature forming of $\geq 1900\text{MPa}$, and a elongation of $\geq 6\%$.

EXAMPLE 1

[0038] A production process for the low-aluminum, low-magnesium zinc-aluminum-magnesium coated steel sheet for high-temperature forming was prepared, including the following steps:

S1, The raw materials were smelted into molten iron according to a designed chemical composition, followed by pretreatment, converter smelting, secondary, and continuous casting to obtain a casting slab. The casting slab included components in percentage by mass as follows: 0.33% of C, 0.2% of Si, 1.45% of Mn, not more than 0.03% of P, not more than 0.006% of S, 0.08% of Al, 0.02% of Ti, 0.18% of V, not more than 0.005% of N, 0.03% of RE, 0.002% of B, and the balance of Fe and inevitable impurities. The casting during the continuous casting was controlled at 2 m/min.

S2, The casting slab was subjected to heating, rolling, controlled cooling, and coiling, with a finishing rolling of 880°C and a coiling temperature of 680°C, and a steel coil was obtained.

S3, The steel coil from step S2 was uncoiled, then subjected to processes of pickling, cold rolling, and continuous annealing, with a pickling speed of 200 mpm, a rolling reduction rate of 65%, a continuous annealing temperature of 780 °C, and a dew point of -30 °C.

S4, The steel sheet after continuous annealing from step S3 was subjected to hot dipping in a plating bath including 3.5% of Al, 1.8% of Mg, and the balance of Zn and inevitable impurities, where the temperature of the plating bath was 460 °C, and the dipping time was 10s. The steel sheet after hot dipping was cooled to room temperature at a cooling rate more than 30 °C/s, followed by coiling to obtain a steel coil.

S5, The steel coil from step S4 was uncoiled and subjected to high-temperature forming, involving heating the uncoiled steel sheet to 875 °C and holding it for 5 minutes to achieve complete austenitization, and then stamping the steel sheet in a mold, followed by cooling to room temperature to obtain a zinc-aluminum-magnesium coated steel sheet based on hot dipping and high-temperature forming.

[0039] Metallographic structure examination was carried out on the continuous annealed steel sheet prepared in step S3, from which two samples were taken and designated as No.1 and No.2. Metallographic structure examination was also carried out on the hot-stamped steel sheet prepared in step S5, from which two samples were taken and designated as No.3 and No.4. The examination results are shown in Fig. 1.

[0040] Mechanical property tests were carried out on the steel sheets after hot rolling, continuous annealing, and hot stamping, respectively. Three samples were taken from each process and subjected to tensile tests. The mechanical property results of the steel sheet after hot rolling are shown in Table 1, the results of the steel sheet after continuous annealing are shown in Table 2, and the results of the steel sheet after hot stamping are shown in Table 3.

Table 1 Mechanical property test results of the steel sheet after hot rolling

No.	Yield Strength /MPa	Tensile Strength /MPa
Standard	≥ 350	≥ 600
1	681	883
2	684	920
3	678	869

Table 2 Mechanical property test results of the steel sheet after continuous annealing

No.	Thickness	Yield Strength /MPa	Tensile Strength /MPa	Elongation /%
Standard		≥400	≥580	≥8
1	1.2	441	682	20
2	1.2	501	720	19
3	1.2	532	716	19

Table 3 Mechanical property test results of the steel sheet after hot stamping

No.	Yield Strength /MPa	Tensile Strength /MPa	Elongation /%
1	1290	2080	6.0
2	1250	2010	6.2
3	1220	1970	6.5

[0041] From the microstructure photograph (Fig. 1) and the mechanical property test results of the steel sheets prepared in this example after each process, it can be found that the microstructure of the steel sheet after continuous annealing consists of ferrolite and pearlite (F+P), and the steel sheet has relatively low overall strength relatively high elongation, meeting the standard requirements and being able to satisfy users' service conditions. Furthermore, during the hot stamping forming process, since the steel sheet is completely austenitized at high temperature, the microstructure transforms from F+P into a fully martensitic structure during cooling to room temperature, resulting a significant increase in strength, with the tensile strength reaching over 2 GPa and the elongation greater than 6%, presenting good performance.

[0042] Microstructure observation was carried out on the cross-section of the zinc-aluminium-magnesium coated steel sheet after hot dipping and before stamping, as shown in Fig. 2. The coating thickness is about 30-40 μm , the alloy layer is relatively thin, almost invisible. There is a large number of dendritic structure in the coating, and dense Zn-MgZn₂ eutectic structures are distributed in the interdendritic spaces. The MgZn₂ phase contributes to improving the coating in corrosion resistance.

[0043] Microstructure observation was carried out on the cross-section of alloy coating of the zinc-aluminum-magnesium coated steel sheet after austenitization at 910°C, as shown in Fig. 3. The cross-section is composed of a uniform alloyed coating, in which Zn, Al, and Mg are uniformly distributed in the coating, no layering phenomenon is observed, and the coating structure is dense without voids or cracks.

COMPARATIVE EXAMPLE 1

[0044] In order to verify the effects of Al and Mg elements in the plating bath on the coating performance, Comparative Example 1 was provided. A pure Zn coated steel sheet was prepared in Comparative Example 1 by a plating bath consisting entirely of zinc element without any Al or Mg elements. Unless otherwise specified, the preparation steps were the same as those in Example 1. Since the other process parameters remain unchanged, no microstructure or property tests were carried out on the steel sheet prepared in comparative example 1.

[0045] Microstructure observation was carried out on the cross-section of coating of the pure Zn coated steel sheet after hot dipping, as shown in Fig.4. The coating has a thickness of about 30 μm , and is divided into two layers, where the upper layer is a Zn phase, and the lower layer is an Fe-Zn phase, without existence of eutectic structures. There are many microcracks in the Fe-Zn phase, but they do not extend into the substrate.

[0046] Microstructure observation was carried out on the cross-section of the pure Zn coated steel sheet after austenitization, as shown in Fig.5. The coating has a thickness of about 30 μm , and contains a large number of single-phase structures. Many cracks exist at the interface between the coating and the steel substrate and extend into the steel substrate. Due to the continuous diffusion of Fe from the substrate into the coating, the coating mainly includes α -Fe(Zn) phase and FeZn₇ phase that exists at the interface between the steel substrate and the coating.

[0047] Compared with Comparative Example 1, the addition of Mg element in Example 1 enables the formation of a Mg/Zn eutectic structure in the coating, improving the corrosion resistance of the coating. Furthermore, the addition of Al and Mg elements enable the coating to form more uniform and dense structures after austenitization, reducing the formation of microcracks.

[0048] Conventional pure Zn coated steel sheets have been encountering the problem of liquid metal embrittlement (LEM) during hot stamping. In order to verify whether the zinc-aluminum-magnesium coated steel sheet designed in the present invention can effectively solve the LEM problem, U-die stamping tests were carried out on the steel sheets of

Example 1 and Comparative Example 1, and the cracks at bent positions after the tests were analyzed. The results are shown in Fig. 6. From the microstructure observation, it can be seen that in the Zn-Al-Mg coated steel sheet, cracks terminate at the substrate and do not extend into the substrate, whereas in the pure Zn coated steel sheet, there are a small number of cracks extending into the substrate. The design of the present invention can serve a purpose in reducing the LEM problem.

EXAMPLE 2

[0049] In order to investigate the influence of adjusting the Al content on the liquid metal embrittlement problem in high-strength steel coated with the Zn-Al-Mg alloy coating, Example 2 was provided, Example 2 prepared a Zn-Al-Mg coated steel sheet in which the mass percentages of the chemical elements contained in the plating bath differed from those in Example 1. Unless otherwise specified, the preparation steps were the same as those in Example 1, except that the plating bath composition was 0.2% of Al, 1.8% of Mg, and the balance of Zn and inevitable impurities. Since the other process parameters remain unchanged, no microstructure property tests were carried out on the steel sheet prepared in Example 2.

[0050] Microstructure observation was carried out on the cross-section of the zinc-aluminum-magnesium coated steel sheet after hot dipping, as shown in Fig. 6. A large number of dendritic structure is present in the coating, and the dendrite size is larger than that in Example 1, which is mainly attributed to the decrease in Al content leading to deviation from the eutectic composition. Dense Mg/Zn eutectic structures are distributed in the interdendritic spaces, and the eutectic regions are composed of Mg_2Zn_{11} and $MgZn_2$ phases, where the Mg_2Zn_{11} phase has a higher content, and both phases contribute to improving the coating in corrosion resistance. In addition, the intermetallic compound layer at the interface between the coating and the steel substrate is relatively continuous. Since the oxidation of Mg elements upon contact with air, Mg is enriched at the outermost layer of the coating.

[0051] Microstructure observation was carried out on the cross-section of coating of the zinc-aluminum-magnesium coated steel sheet after austenitization, as shown in Fig. 7. The backscattered electron (BSE) image shows that Fe, Zn, Al, and Mg elements are uniformly distributed.

[0052] By comparing Example 1 and Example 2, it can be seen that the increase in Al content in the coating refines the coating grain structure, increases the amount of dendritic structure, and reduce the thickness of the diffusion layer after austenitization, indicating that the increase in Al content in the coating can inhibit the diffusion of Zn elements from the coating into the substrate.

EXAMPLE 3

[0053] A low-aluminum, low-magnesium zinc-aluminum-magnesium coated steel sheet for high-temperature forming was prepared, and its steel substrate included components in percentage by mass as follows: 0.35% of C, 0.4% of Si, 1.8% of Mn, not more than 0.03% of P, not more than 0.006% of S, 0.1% of Al, 0.04% of Ti, 0.28% of V, not more than 0.005% of N, 0.045% of RE, 0.004% of B, and the balance of Fe and inevitable impurities. The chemical element composition of the plating bath used are the same as that in Example 1.

[0054] During the hot rolling process, the finishing rolling temperature was controlled at 850°C and the coiling temperature at 700°C. During the continuous annealing process, the annealing temperature was 750°C and the dew point was -40°C. During the hot dipping process, the plating bath temperature was 440°C, and the dipping time was 10 seconds. During the high-temperature forming process, the steel sheet was heated to 870°C and held for 3 minutes, then subjected to stamping forming followed by cooling to room temperature. Metallographic structure examination and mechanical property tests were carried out on the steel sheet after hot stamping forming. Two samples were taken for metallographic structure examination, designated as No.5 and No.6, and the results are shown in Fig. 8. Two samples were taken for mechanical property tests, and the results are shown in Table 4.

Table 4 Mechanical property test results after hot stamping

No.	Yield Strength /MPa	Tensile Strength /MPa	Elongation /%
1	1290	2050	6.6
2	1260	2040	6.8

[0055] The plating bath composition of this example was unchanged compared with that of Example 1, and therefore no tests were carried out on the coating of the samples. The metallographic structure examination results show that the microstructure of the coated steel sheet prepared in this example is entirely martensite after hot stamping forming, thus the coated steel sheet has high strength. The mechanical property test results show that the coated steel plate prepared in this

example has a tensile strength greater than 1800 MPa and an elongation of more than 6%, exhibiting good performance and meeting service requirements.

EXAMPLE 4

[0056] A low-aluminum, low-magnesium zinc-aluminum-magnesium coated steel sheet for high-temperature forming was prepared, and its steel substrate included components in percentage by mass as follows: 0.31% of C, 0.1% of Si, 1.2% of Mn, not more than 0.03% of P, not more than 0.006% of S, 0.09% of Al, 0.02% of Ti, 0.1% of V, not more than 0.005% of N, 0.015% of RE, 0.001% of B, and the balance of Fe and inevitable impurities. The chemical element composition of the plating bath used included 1.5% of Al, 1.8% of Mg, and the balance of Zn and inevitable impurities.

[0057] During the hot rolling process, the finishing rolling temperature was controlled at 800°C, and the coiling temperature at 660°C. During the continuous annealing process, the annealing temperature was 820°C and the dew point was -30°C. The hot dipping process was the same as that of Example 1. During the high-temperature forming process, the steel sheet was heated to 890°C and held for 10 minutes, then subjected to stamping forming followed by cooling to room temperature. Microstructure observation was carried out on the cross-section of the zinc-aluminum-magnesium coated steel sheet after hot dipping, and the results are shown in Fig.9. EDS analysis was carried out on the cross-section of the zinc-aluminum-magnesium coated steel sheet, and the results are shown in Fig.10. Metallographic structure examination and mechanical property tests were carried out on the steel sheet after hot stamping forming. Two samples were taken for metallographic structure examination, designated as No.7 and No.8, and the results are shown in Fig.11. Two samples were taken for mechanical property tests, and the results are shown in Table 5.

Table 5 Mechanical property test results after hot stamping

No.	Yield Strength /MPa	Tensile Strength /MPa	Elongation /%
1	1300	2015	6.5
2	1270	2035	6.0

[0058] The morphology of the coating of the steel sheet after hot dipping is similar to that of Example 1. There is a large number of dendritic structure in the coating. In addition, dense Mg/Zn eutectic structures are distributed in the interdendritic spaces, and Mg_2Zn_{11} and $MgZn_2$ phases are present in the eutectic regions, with a higher content of the Mg_2Zn_{11} phase, which contributes to improvement in corrosion resistance. In the intermediate layer at the interface between the coating and the steel substrate, the ratio of Fe to Al atoms is close to that of the Fe_2Al_5 phase, making it possible to prevent the LME problem. The microstructure of the steel sheet after hot stamping is fully martensitic, and the mechanical properties are good, similar to the results of Examples 1, 2, and 3.

EXAMPLE 5

[0059] A low-aluminum, low-magnesium zinc-aluminum-magnesium coated steel sheet for high-temperature forming was prepared, and its steel substrate included components in percentage by mass as follows: 0.32% of C, 0.3% of Si, 1.6% of Mn, not more than 0.03% of P, not more than 0.006% of S, 0.09% of Al, 0.03% of Ti, 0.15% of V, not more than 0.005% of N, 0.02% of RE, 0.0025% of B, and the balance of Fe and inevitable impurities. The chemical element composition of the plating bath used included 2.5% of Al, 1.8% of Mg, and the balance of Zn and inevitable impurities.

[0060] During the hot rolling process, the finishing rolling temperature was controlled at 900°C and the coiling temperature at 620°C. During the continuous annealing process, the annealing temperature was 800°C and the dew point was -30°C. During the hot dipping process, the plating bath temperature was 480°C, and the dipping time was 10 seconds. During the high-temperature forming process, the steel sheet was heated to 910°C and held for 10 minutes, then subjected to stamping forming followed by cooling to room temperature. Microstructure observation was carried out on the cross-section of the zinc-aluminum-magnesium coated steel sheet after hot dipping, and the results are shown in Fig.12. EDS analysis was carried out on the cross-section of the zinc-aluminum-magnesium coated steel sheet, and the results are shown in Fig.13. Metallographic structure examination and mechanical property tests were carried out on the steel sheet after hot stamping forming. Two samples were taken for metallographic structure examination, designated as No.9 and No.10, and the results are shown in Fig.14. Two samples were taken for mechanical property tests, and the results are shown in Table 6.

Table 6 Mechanical property test results after hot stamping

No.	Yield Strength /MPa	Tensile Strength /MPa	Elongation /%
1	1280	2010	6.9
2	1310	2040	6.2

[0061] It can be seen that, with the increase of Al content in the plating bath, the grain size of the steel sheet after hot dipping is refined. In the eutectic region, both Mg_2Zn_{11} and $MgZn_2$ phases are present, and the $MgZn_2$ phase has a higher content. The two phases help to improve the corrosion resistance of the coated steel sheet. In the intermediate layer at the interface between the coating and the steel substrate, Fe_2Al_5 phases are present. As the Al content in the plating bath increases, the enrichment of Al in the intermediate layer becomes more obvious, the intermediate layer becomes denser, presenting stronger resistance to liquid metal cracking. After hot stamping, the coated steel sheet exhibits good mechanical properties and relatively high elongation.

[0062] In order to more intuitively demonstrate the improvement in corrosion resistance of the low-aluminum, low-magnesium zinc-aluminum-magnesium coated steel sheet, salt spray tests and polarization curve tests were carried out on the Zn-3.5Al-1.8Mg coated steel sheet, the pure Zn coated steel sheet, and the Al-Si coated steel sheet commonly available on the market. The results of salt spray tests are shown in Fig. 14. Based on the analysis of the surface conditions of the steel sheets with different coatings in the same time duration were analyzed according to the salt spray test results. It can be intuitively seen that after 81 hours, the surface of the pure Zn coated steel sheet has begun to exhibit red rust, while the surfaces of the zinc-aluminum-magnesium coated steel sheet and the Al-Si coated steel sheet still remain in good condition. Therefore, the corrosion resistance of the pure Zn coated steel sheet is inferior to that of the other two coated steel sheets. Subsequently, further observation of the surfaces of the zinc-aluminum-magnesium coated steel sheet and the aluminum-silicon coated steel sheet was continued. As time progressed, after 157 hours, the surface quality of the Al-Si coated steel sheet deteriorated significantly; after 229 hours, the surface quality of the Al-Si coated steel sheet was visibly inferior to that of the Zn-3.5Al-1.8Mg coated steel sheet; after 283 hours, red rust faintly appeared on the surface of the Al-Si coated steel sheet; after 396 hours, red rust on the surface of the Al-Si coated steel sheet became very obvious, while the surface of the Zn-3.5Al-1.8Mg coated steel sheet still remained in good condition without any visible red rust formation, indicating that the zinc-aluminum-magnesium coated steel sheet, compared with both the pure Zn coated steel sheet and the Al-Si coated steel sheet, has a significant improvement in corrosion resistance.

[0063] The results of the polarization curve tests are shown in Fig. 15. According to the results of the polarization curve tests, it can be seen that the potential of the Zn-3.5Al-1.8Mg coated steel sheet was far below that of the Al-Si coated steel sheet, indicating that the Zn-3.5Al-1.8Mg coated steel sheet, compared with the Al-Si coated steel sheet, can provide cathodic protection, which is beneficial to improving the corrosion resistance of materials.

Claims

1. A zinc-aluminum-magnesium coated steel sheet, comprising a coating made from a plating bath comprising the following components in percentage by mass: 1.5~2.0% of Mg, 0.2~3.5% of Al, and the balance of Zn and inevitable impurities.
2. The steel sheet according to claim 1, wherein the plating bath comprises the following components in percentage by mass: 1.5~2.0% of Mg, 2.5~3.5% of Al, and the balance of Zn and inevitable impurities.
3. The steel sheet according to claim 1, wherein the coating contains a large number of dendritic structure, with Mg/Zn eutectic phase distributed in interdendritic spaces.
4. The steel sheet according to claim 3, wherein when the Al content in the plating bath is 0.2%~1.5%, a Mg/Zn eutectic region mainly contains Mg_2Zn_{11} phases, or when the Al content in the plating bath is 1.5%~3.5%, the Mg/Zn eutectic region mainly contains $MgZn_2$ phases.
5. The steel sheet according to claim 1, wherein in an intermediate layer at an interface between the coating and a steel substrate, Al and Fe elements form Fe_2Al_5 phases, which more obviously enrich in the intermediate layer as the Al content increases, making the intermediate layer denser and grains in the coating finer.
6. The steel sheet according to claim 1, comprising a steel substrate comprising the following components in percentage by mass: 0.30~0.36% of C, 0.05~0.5% of Si, 1.0~2.0% of Mn, not more than 0.050% of P, not more than 0.008% of S,

not less than 0.080% of Al, 0.020~0.040% of Ti, 0.1~0.3% of V, not more than 0.005% of O, not more than 0.005% of N, 0.01~0.05% of rare earth, 0.0005~0.0040% of B, and the balance of Fe and inevitable impurities.

7. The steel sheet according to claim 1, wherein the steel sheet has a thickness of 1.0 mm-2.5 mm, a width of 850 mm-1800 mm, a tensile strength of ≥ 1900 MPa after high-temperature forming, and an elongation of $\geq 6\%$.
8. A production process for the zinc-aluminum-magnesium coated steel sheet according to any one of claims 1 to 7, comprising steps of smelting, continuous casting, hot rolling, pickling and cold rolling, annealing, hot dipping, coiling, and high-temperature forming, wherein during the hot dipping, a temperature of the plating bath is controlled within 440 °C to 480 °C, a dipping time ranges from 5 s to 10 s, and a resulting steel sheet after dipping is cooled to room temperature at a cooling rate of more than 30°C/s to obtain a coated steel sheet.
9. The production process according to claim 8, wherein during the annealing, a dew point is controlled within -50 °C to -20 °C, a heating temperature is controlled within 700 °C to 850 °C, then a resulting heated steel sheet is cooled to 420 °C to 500 °C at a cooling rate ranging from 30 °C/s to 50 °C/s to obtain an annealed steel sheet.
10. The production process according to claim 8, wherein the high-temperature forming employs a direct forming process, including heating the coated steel sheet to a temperature ranging from 850 °C to 900 °C, then holding it for 4-10 minutes, followed by quickly transferring it to a mold for stamping, with a mold-closing temperature controlled within 700 °C to 750 °C, and after stamping, a resulting part is quickly cooled to room temperature in the mold at a cooling rate ranging from 100 °C/s to 140 °C/s;

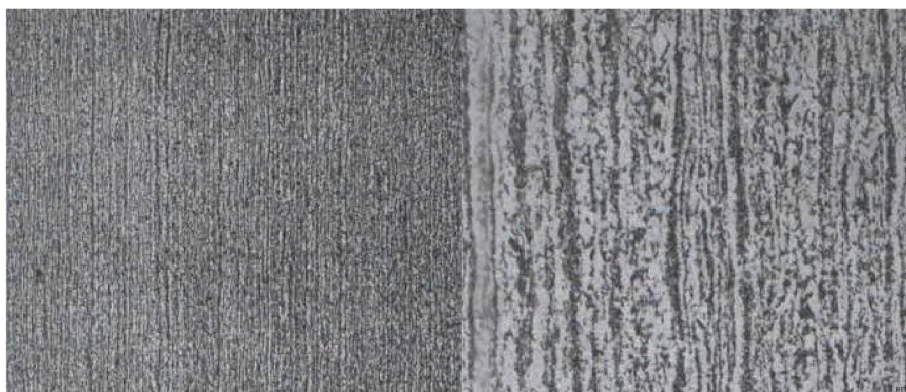
during the hot rolling, a continuous casting slab from the continuous casting is heated to 1250 °C to 1300 °C, and is subjected to finishing rolling at a temperature ranging from 780 °C to 900 °C, and a coiling temperature ranges from 600°C to 720°C; and

during the continuous casting, a casting speed is controlled within 1.6 m/min to 2.4 m/min and maintained constant.



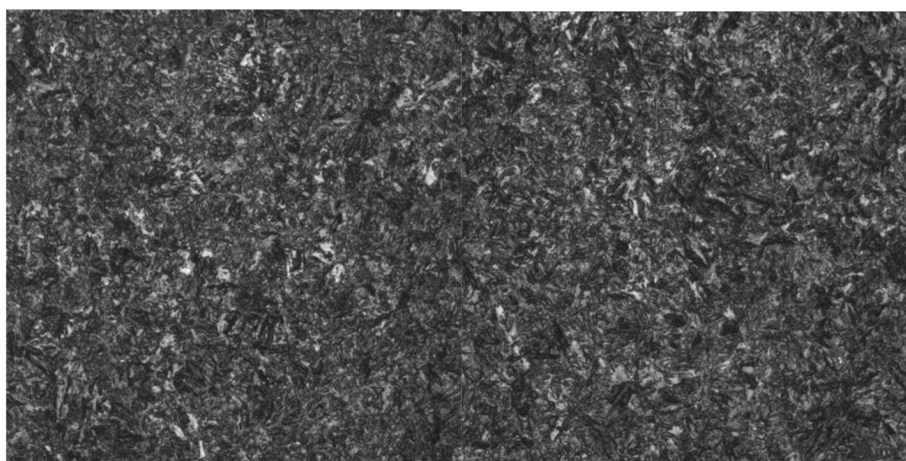
(a)

(b)



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(e)

(f)

Fig.1

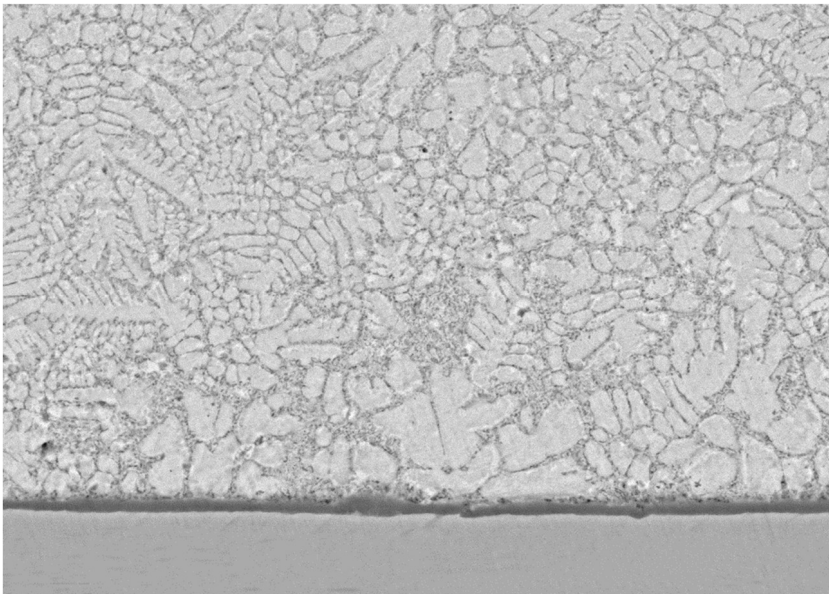


Fig.2

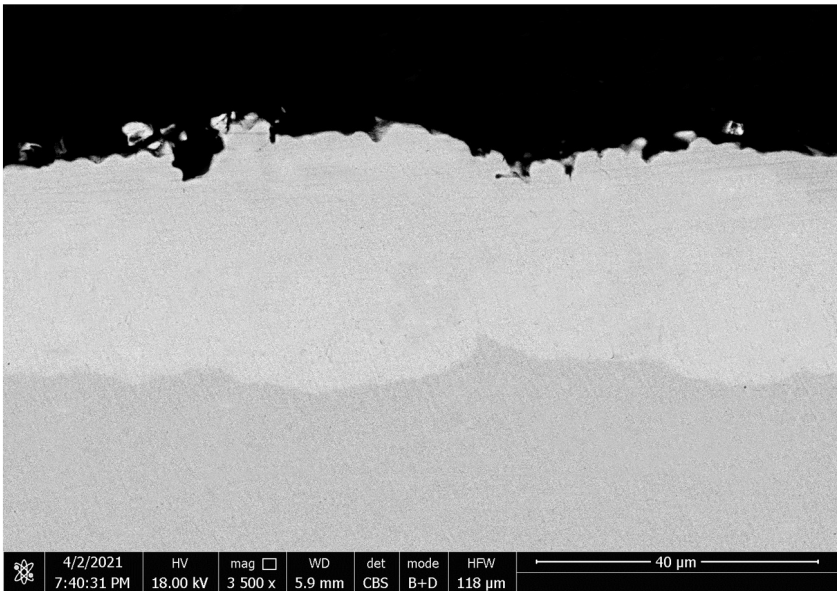


Fig.3

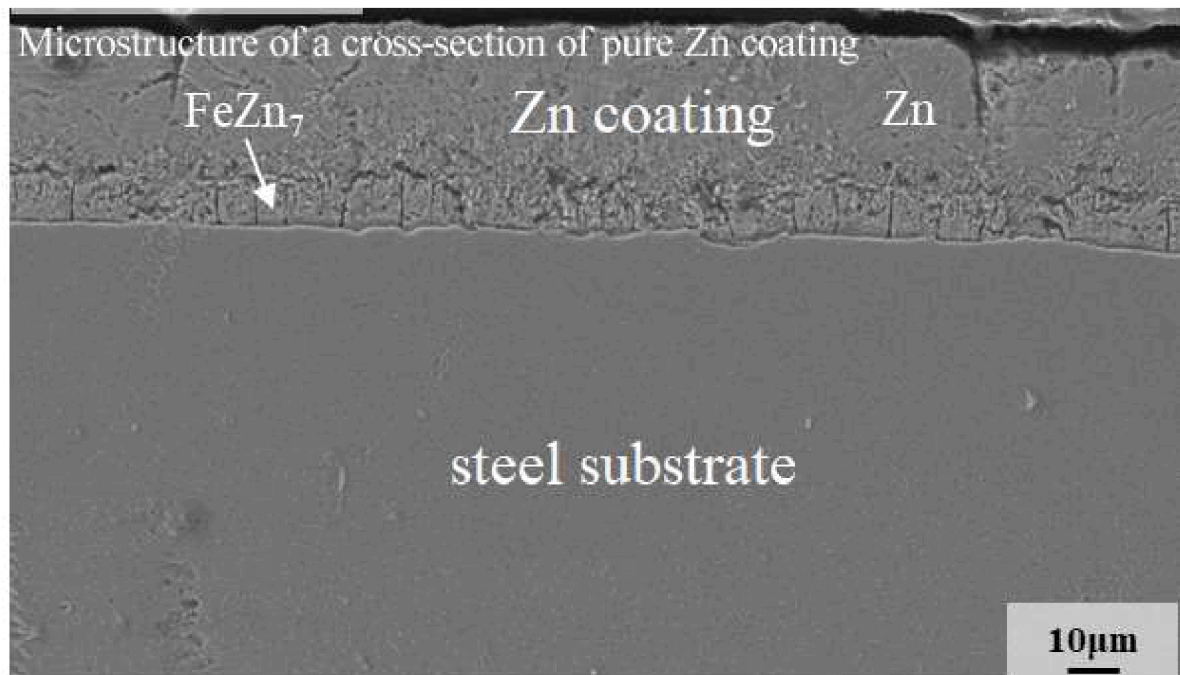


Fig.4

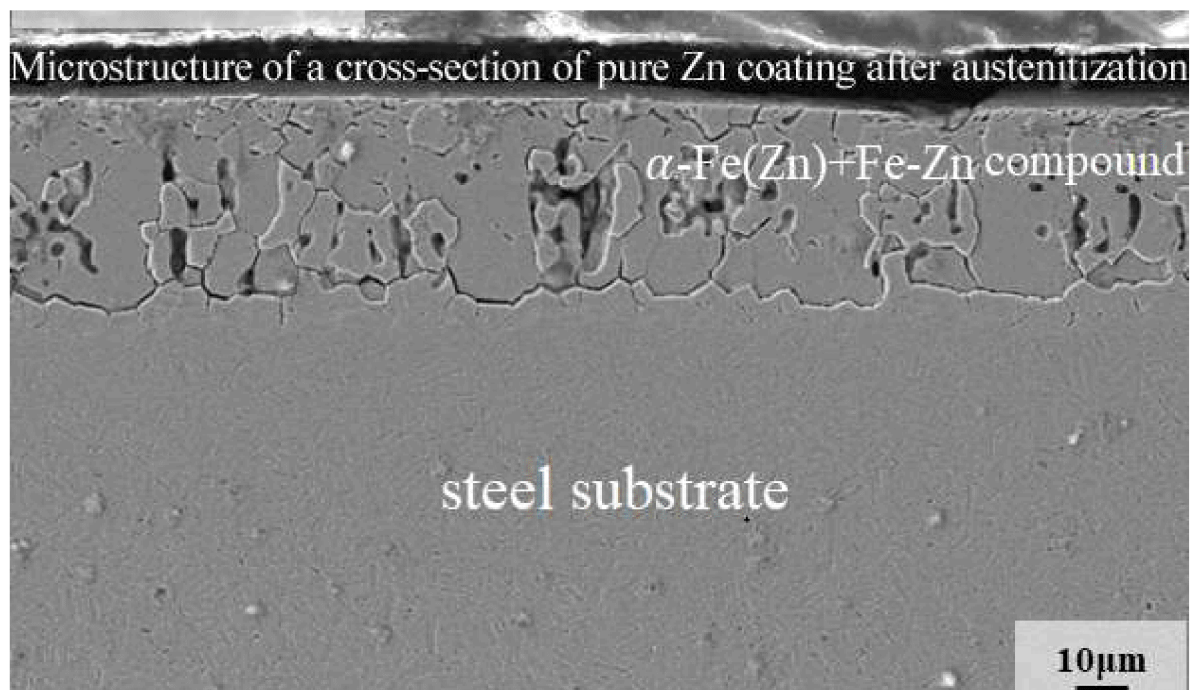
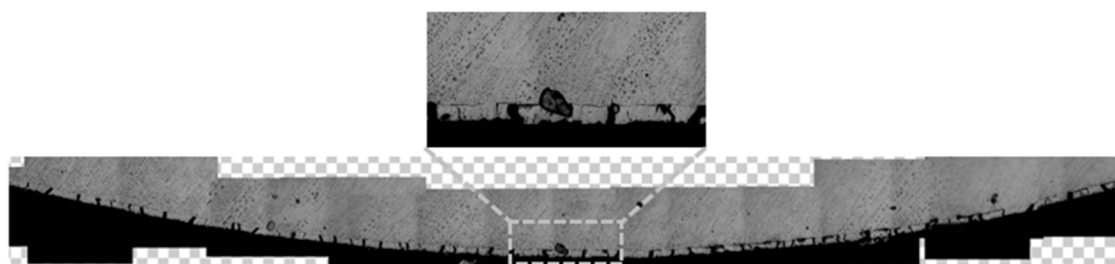
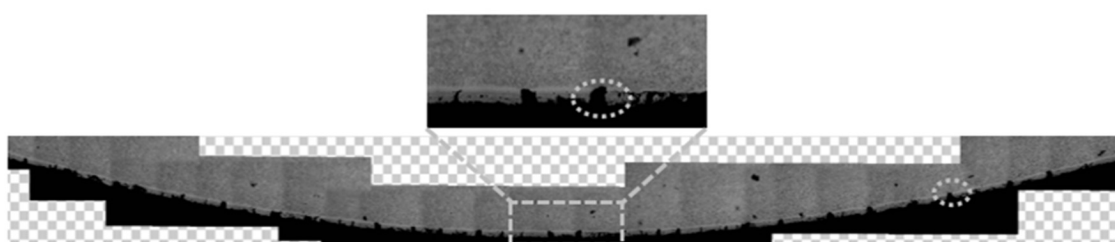


Fig.5



(a)



(b)

Fig.6

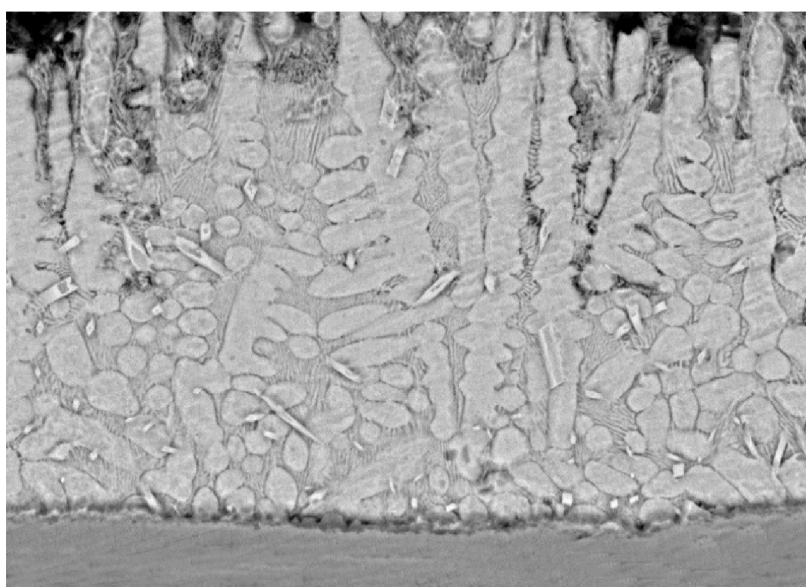


Fig.7

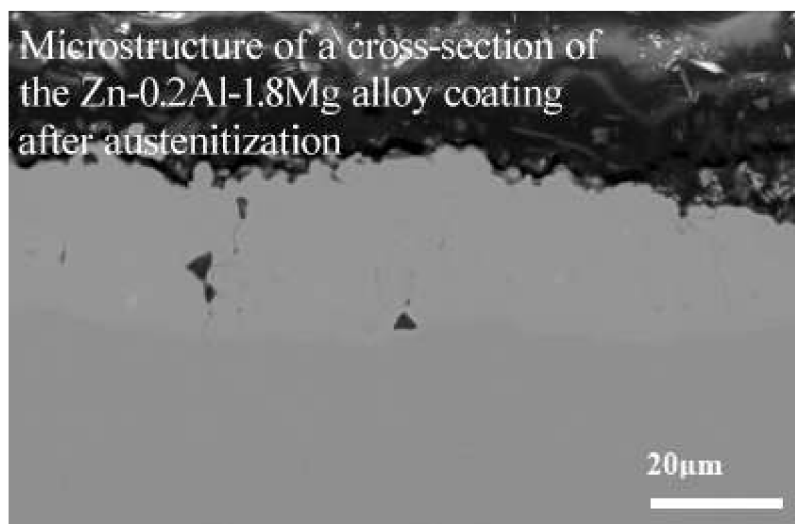


Fig.8

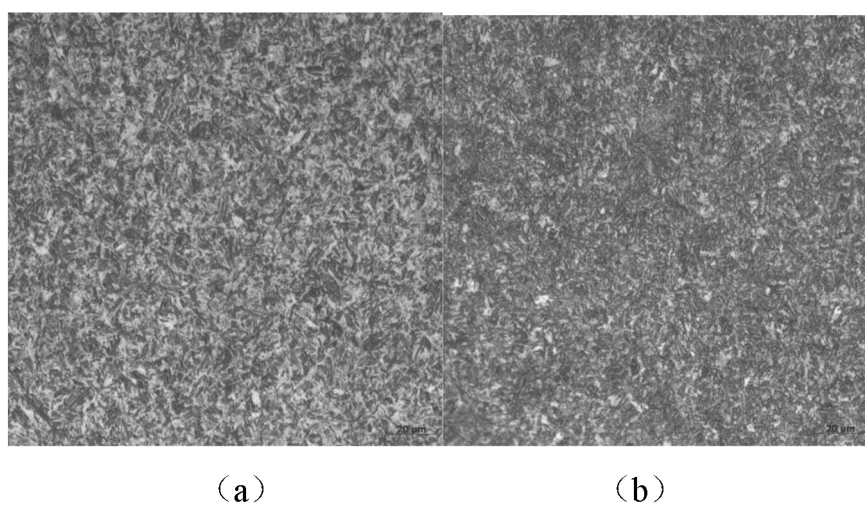


Fig.9

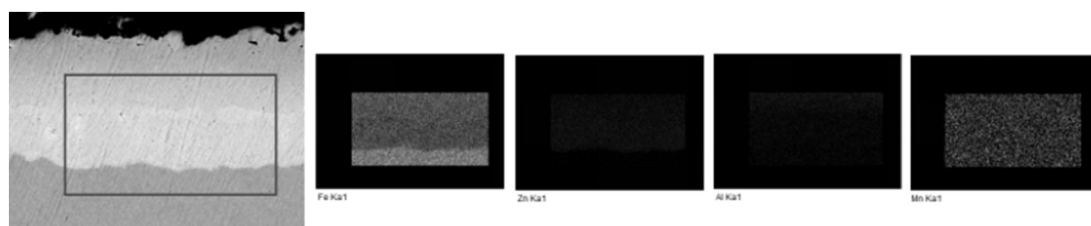


Fig.10

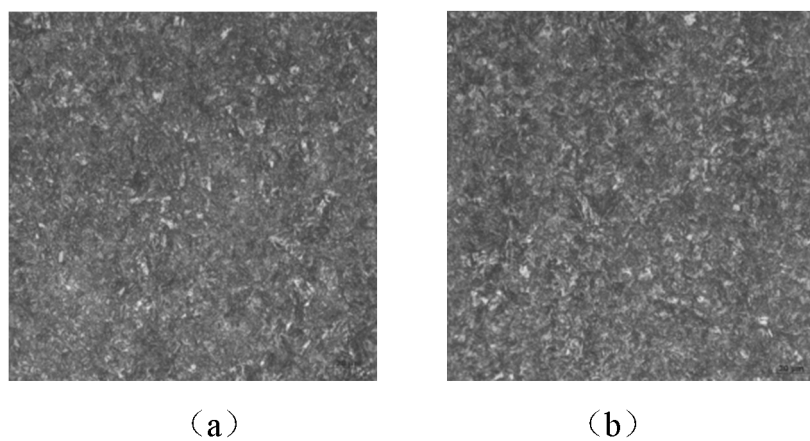


Fig.11

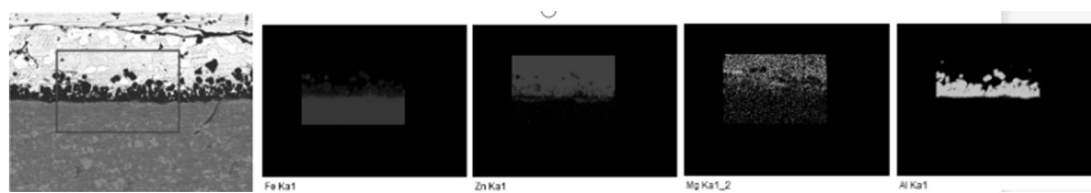


Fig.12

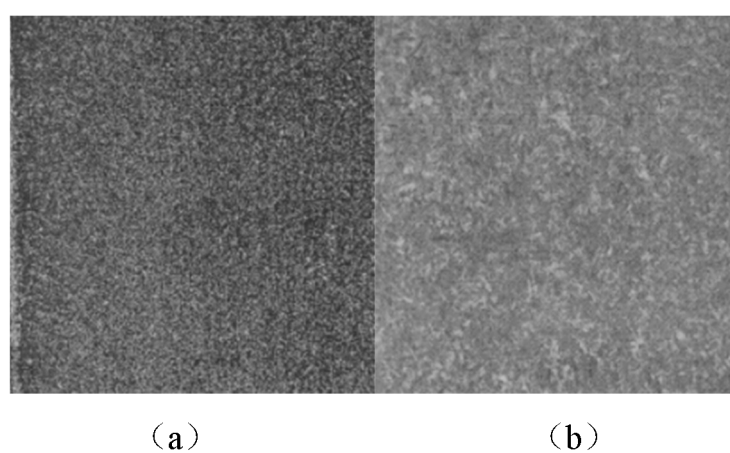
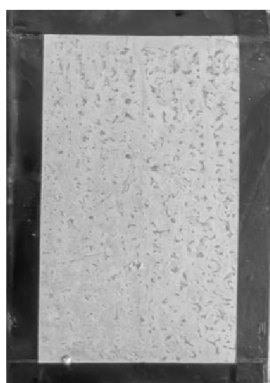


Fig.13



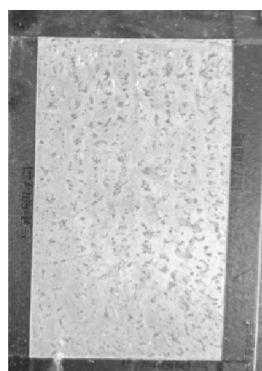
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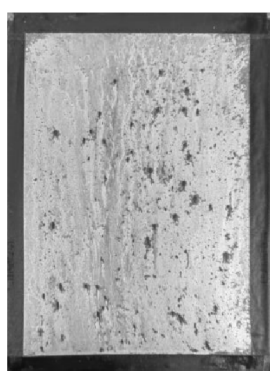
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(c)



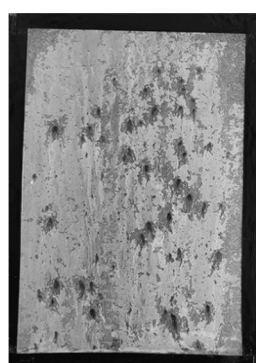
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(e)



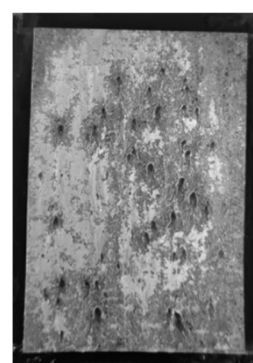
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(g)



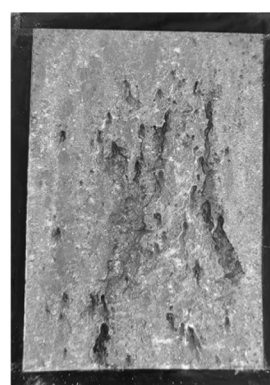
(h)



(i)



(j)



(k)

Fig.14

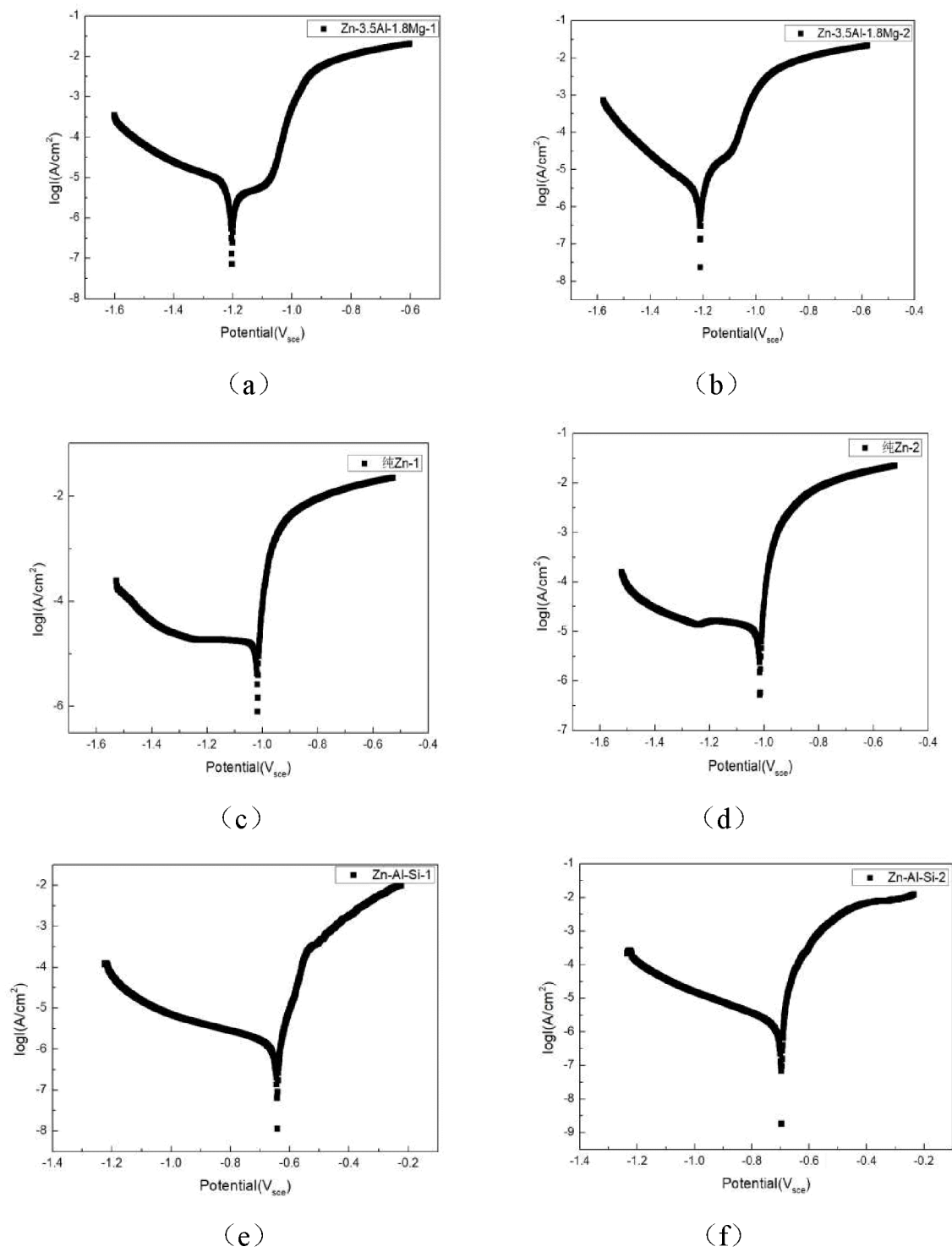


Fig.15

INTERNATIONAL SEARCH REPORT

International application No.

PCT/CN2023/138291

A. CLASSIFICATION OF SUBJECT MATTER

C23C2/06(2006.01)i; C22C18/04(2006.01)i; C22C18/00(2006.01)i; C23C2/40(2006.01)i; C23C2/28(2006.01)i; C23C2/02(2006.01)i; C22C38/02(2006.01)i; C22C38/04(2006.01)i; C22C38/06(2006.01)i; C22C38/14(2006.01)i; C22C38/12(2006.01)i; C21D8/02(2006.01)i; B21C37/02(2006.01)i; B21D22/02(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)

IPC: C23C C22C C21D B21C B21D

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)

WPABSC; CNTXT; ENTXT; DWPI; USTXT; JPTXT: 本钢, 刘宏亮, 锌, 铝, 镁, 镀层, 钢板, 热浸镀, 树枝晶, 共晶, 高温, 成型, Zn, zinc, Al, aluminium, Mg, magnesium, coat+, steel, plate, hot, dip, plating, dendrite, eutectic, high, temperature, forming

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	CN 110983224 A (SHOUGANG GROUP CO., LTD.) 10 April 2020 (2020-04-10) description, paragraphs 10-22, and embodiment 13 of table 2	1-5
Y	CN 110983224 A (SHOUGANG GROUP CO., LTD.) 10 April 2020 (2020-04-10) description, paragraphs 10-22, and embodiment 13 of table 2	6-10
Y	CN 107099748 A (WUHAN IRON & STEEL CO., LTD.) 29 August 2017 (2017-08-29) description, paragraphs 7-15 and 29-31, and embodiment 3 of table 1	6-10
X	CN 104419867 A (ANGANG STEEL CO., LTD.) 18 March 2015 (2015-03-18) table 2, and embodiment 18	1-5
X	CN 103361588 A (ANGANG STEEL CO., LTD.) 23 October 2013 (2013-10-23) claim 1	1-5
X	CN 103507324 A (ANGANG STEEL CO., LTD.) 15 January 2014 (2014-01-15) abstract	1-5

☒ Further documents are listed in the continuation of Box C.

☒ See patent family annex.

* Special categories of cited documents:	"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
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"D" document cited by the applicant in the international application	"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
"E" earlier application or patent but published on or after the international filing date	"&" document member of the same patent family
"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	
"O" document referring to an oral disclosure, use, exhibition or other means	
"P" document published prior to the international filing date but later than the priority date claimed	

Date of the actual completion of the international search

15 July 2024

Date of mailing of the international search report

23 July 2024

Name and mailing address of the ISA/CN

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Authorized officer

Telephone No.

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CN2023/138291

C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2002285311 A (SUMITOMO METAL INDUSTRIES, LTD.) 03 October 2002 (2002-10-03) description, paragraph 38	1-5
A	CN 116641008 A (BENGANG STEEL PLATES CO., LTD.) 25 August 2023 (2023-08-25) entire document	1-10

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.

PCT/CN2023/138291

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Patent document cited in search report			Publication date (day/month/year)	Patent family member(s)			Publication date (day/month/year)
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CN	107099748	A	29 August 2017	CN	107099748	B	26 March 2019
CN	104419867	A	18 March 2015	CN	104419867	B	07 September 2016
CN	103361588	A	23 October 2013	CN	103361588	B	06 April 2016
CN	103507324	A	15 January 2014	CN	103507324	B	03 June 2015
JP	2002285311	A	03 October 2002	JP	3580261	B2	20 October 2004
CN	116641008	A	25 August 2023	None			

Form PCT/ISA/210 (patent family annex) (July 2022)

REFERENCES CITED IN THE DESCRIPTION

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Patent documents cited in the description

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