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(71) Applicant: **Jiang Yin Xing Cheng Special Steel
Works Co., Ltd
Jiangsu 214434 (CN)**

(72) Inventors:
• **HU, Shaoxin
Wuxi
Jiangsu 214434 (CN)**
• **XU, Xiaohong
Wuxi
Jiangsu 214434 (CN)**

- **SHAO, Shuyan
Wuxi
Jiangsu 214434 (CN)**
- **HUANG, Zhen
Wuxi
Jiangsu 214434 (CN)**
- **NIE, Aichen
Wuxi
Jiangsu 214434 (CN)**
- **Ji, Ling
Wuxi
Jiangsu 214434 (CN)**
- **ZHU, Guorong
Wuxi
Jiangsu 214434 (CN)**

(74) Representative: **Huang, Liwei
Cäcilienstraße 12
40597 Düsseldorf (DE)**

(54) **PROCESS FOR PRODUCING LS-CRNI30C STEEL FORGING USING CONTINUOUS
CASTING BILLET FOR SUBSEA OIL PRODUCTION WELLHEAD DEVICE**

(57) The present invention relates to a process for manufacturing CrNiMo30C forged steel for a wellhead device of deep-sea oil extraction by continuous casting. The forged round steel of $\Phi 300$ -400mm is manufactured by continuous casting billet of $\Phi 600$ mm, the process including steps of molten steel smelting, continuous casting, forging, annealing, cooling and etc, the macrostructure of the continuous casting billet meets the requirements of appendix A of YB/T4149 standard: center porosity is equal to or less than grade 2.0, center crack is equal to or less than grade 1.0, shrinkage cavity is equal to or less than grade 1.0, defects of intermediate cracks, white flakes and upwarping defects are forbidden. The

billet is heated and the heating speed is equal to or less than 200°C per hour, the heating temperature of forging is 1200-1250°C, ensuring that the billet is completely heated, the initial forging temperature is higher than 1150°C, and the final forging temperature is not lower than 850°C; after leaving the production line, the forged steel is at a high temperature of equal to or greater than 500°C and is then slowly cooled in sand; after the temperature drops to 200°C or lower, the forged steel is taken out of pit and slowly cooled. The inner quality of the finished forged steel meets requirements of grade D/d of SEP 1921 standard, and the mechanical property should meet the requirements of APISPEC 6A standard.

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Description

Technical Field

[0001] The present invention relates to a field of manufacturing forged steel, specially relates to a process for manufacturing CrNiMo30C forged steel for a wellhead device of deep-sea oil extraction by continuous casting.

Background Art

[0002] The wellhead device of deep-sea oil extraction consists of casing-head, tubing-head and production tree, and is an important device for producing gas-oil. The wellhead device is mainly used to control the pressure of production wellhead and to regulate the flow rate of oil (gas) and water wells, how good the performance is relates to whether the oil/gas wells can produce safely and efficiently or not.

[0003] At present the wellhead device of deep-sea oil extraction is usually forged by mold casting ingot, because the inner quality of steel ingot in manufacturing is better than that of continuous casting billet (especially the alloy containing a certain amount of Cr, Ni, Mo etc.), the forged bar can meet the higher requirements of ultrasonic flaw detection and mechanical property required by the wellhead device of deep-sea oil extraction. While the casting and solidification process of molten steel of continuous casting billet is different from those of mold casting ingot, the inner quality of continuous casting billet has problems of bad compactness and heavy segregation, therefore, the forged bar by continuous casting billet usually can not meet the requirements of use in the wellhead device of deep-sea oil extraction. However, each forged bar by mold casting ingot has to be cut in heads and tails, the production ratio in forging is only about 80%, whereas the forged bar by continuous casting billet is poured at one time with several stoves of steel. Because the billet yield in continuous casting is high, the production ratio in forging is up to 90% or more, the production efficiency is high and the cost is low. Thus, improving the inner quality of continuous casting billets, controlling the process of forging, and replacing the mold casting ingots by continuous casting billets for manufacturing the wellhead device of deep-sea oil extraction would be a better development tendency.

Disclosure of Invention

[0004] Aiming at above prior arts, the object of the present invention is, by improving the inner quality of continuous casting of $\Phi 600\text{mm}$ CrNiMo30C steel and by adopting a reasonable forging process, to manufacture forged round steel of $\Phi 300\text{--}400\text{mm}$ via replacing mold casting ingot with continuous casting billet. The inner quality meets requirements of an ultrasonic test according to grade D/d of SEP 1921 standard and a mechanical property test according to APISPEC 6A standard.

[0005] The solution of the present invention is as follows:

A process for manufacturing CrNiMo30C forged steel for a wellhead device of deep-sea oil extraction, by continuous casting, including the following steps

1) Smelting molten steel: CrNiMo30C steel of a medium-carbon low-alloy CrNiMo steel, its constituents and mass percentages are as follows: C: 0.25~0.40%, Si: 0.15~0.40%, Mn: 0.80~1.30%, P: $\leq 0.010\%$, S: $\leq 0.008\%$, Cr: 0.85~1.55%, Mo: 0.30~0.65%, Ni: 0.80~1.40%, V: 0.02~0.08%, Nb: 0.02~0.05%, Cu $\leq 0.25\%$, B $\leq 0.0005\%$, H $\leq 0.0002\%$, the balance is Fe and the unavoidable impurity elements, the molten steel is processed by KR pretreatment, BOF converter smelting, LF refining and RH vacuum degassing, thereby the CrNiMo30C steel with low H, S and P is obtained, and the steel is finally controlled according to the requirements of P $\leq 0.010\%$, S $\leq 0.005\%$ and H $\leq 1.5\text{ppm}$;

2) Continuous casting: the molten steel is further processed by argon protection, mixed electromagnetic stirring with mold electromagnetic stirring (M-EMS) and final electromagnetic stirring (F-EMS), in cooperation with secondary cooling water and low speed casting, and strictly controlling a superheat degree of 20-35 °C, thereby to decrease columnar crystals and enlarge equiaxed crystal, to prevent the development of columnar crystals to refine grains, to reduce segregation degree in steel crystallization process, to sufficiently ensure uniformity and compactness of inner structure; a continuous casting billet of $\Phi 600\text{mm}$ leaves the production line and is delivered to a slow cooling pit at a high temperature of equal to or more than 500°C, and is kept warm for equal to or more than 36h in the pit, and then the continuous billet is timely forged after outside the pit; the macrostructure of the continuous casting billet meets the requirements of appendix A of YB/T4149 standard: center porosity is equal to or less than grade 2.0, center crack is equal to or less than grade 1.0, shrinkage cavity is equal to or less than grade 1.0, defects of intermediate cracks, white flakes and upwarping defects are forbidden;

3) Forging: the billet is heated and the heating speed is equal to or less than 200°C per hour, the heating temperature of forging is 1200-1250°C, ensuring that the billet is completely heated, the initial forging temperature is higher than 1150°C, and the final forging temperature is not lower than 850°C, the continuous casting billet is freely forged by a 1400T double-station hydraulic machine, oxide coating of the continuous billet is timely removed during forging, the billet is drawn into square shape by a small reduction amount at initial cogging, thereby to prevent surface cracks, and is drawn into square shape by a large reduction amount at intermediate cogging, thereby to penetrate the forging force into the core so as to

improve the inner quality, and the billet is further drawn and its outside shaped as a circle, and is finally finished to a finished size; after leaving the production line, the forged steel is at a high temperature of equal to or greater than 500°C and is then slowly cooled in sand; after the temperature drops to 200°C or lower, the forged steel is taken out of the pit and slowly cooled;

4) Performing stress relief annealing treatment on forged steel: the annealing temperature is 650-700°C, keeping warm for 12 hours or more, cooling to below 300°C along with the furnace, taking the forged billet outside the furnace, and air-cooling to room temperature;

5) Inspection: cutting head and tail parts of each piece of forged steel, and performing an ultrasonic test according to grade D/d of SEP 1921 standard to each piece of steel; performing a mechanical property test to a batch of the steel, wherein the tensile, the impact and the hardness should meet the requirements of APISPEC 6A standard.

[0006] Compared with the prior art, the advantages of the present invention focus on:

Compared with the prior process of manufacturing CrNiMo30C forged steel by mold casting ingot, the present invention has good benefits:

- Using "KR-BOF-LF-RH-CCM" in smelting and continuous casting, so that the degree of automation and mechanization is high, operating condition is therefore improved, and the production efficiency is improved.
- Using mixed electromagnetic stirring with mold electromagnetic stirring (M-EMS) and final electromagnetic stirring (F-EMS), to improve the inner quality of continuous quality; controlling forging heating process and the reduction amount at cogging, to obtain a forged bar of Cr, Ni, Mo with good inner quality for use in a wellhead device of deep-sea oil extraction; replacing casting ingot by continuous casting, so that the yield ratio is improved, and the cost advantage is significant.

Brief Description of Figures in the Drawings

[0007]

Figure 1 illustrates macrostructure of $\Phi 350$ mm forged round steel by continuous casting billet in embodiment 1;

Figure 2 illustrates macrostructure of $\Phi 390$ mm forged round steel by continuous casting billet in embodiment 2.

Mode(s) for Carrying Out the Invention

[0008] The present invention further describes a proc-

ess for manufacturing CrNiMo30C forged steel for a well-head device of deep-sea oil extraction by continuous casting billet in detail with reference to embodiments.

[0009] Chemical constituents of CrNiMo30C steel are as follows: C: 0.25~0.40%, Si: 0.15~ 0.40%, Mn: 0.80~1.30%, P: $\leq 0.010\%$, S: $\leq 0.008\%$, Cr: 0.85~1.55%, Mo: 0.30~0.65%, Ni: 0.80~1.40%, V: 0.02~0.08%, Nb: 0.02~0.05%, Cu $\leq 0.25\%$, B $\leq 0.0005\%$, H $\leq 0.0002\%$, the balance is Fe and the unavoidable impurity elements.

Embodiment 1

[0010] The steel is selected to be CrNiMo30C steel;

1) Smelting:

the molten steel is processed by KR pretreatment to perform desulphurization and desilication,

before the molten steel is delivered into converter, S=0.005%,

BOF converter smelting mainly performs dephosphorizing, decarbonizing and heating, alloy metal is added after tapping.

In LF refining process, the chemical constituents are fine tuned, deoxidation, desulphurization and removing impurity.

After degassing in RH vacuum and after breaking the vacuum, H is determined to be H=0.8ppm.

2) Continuous casting:

the molten steel is further processed by argon protection, the superheat degree is controlled to be 25°C, a continuous casting billet of $\Phi 600$ mm is produced by mixed electromagnetic stirring with mold electromagnetic stirring (M-EMS) and final electromagnetic stirring (F-EMS), and then it leaves the production line and is delivered to a slow cooling pit at 550°C, and is kept warm for 36h in the pit, then the continuous billet is taken outside the pit and the macrostructure is tested according to appendix A of YB/T4149 standard: center porosity is grade 1.0, center crack is grade 0, shrinkage cavity is grade 0, free of intermediate cracks, white flakes or upwarping defects, etc.

3) Forging:

the continuous casting billet of $\Phi 600$ mm is cold delivered to the furnace and is heated to 1200°C, the heating speed is equal to or less than 200°C per hour, and is kept warm for 5 hours, the initial forging temperature is 1150°C, and the final forging temperature is not lower than 850°C, the continuous casting billet is freely forged by a 1400T double-station hydraulic machine, oxide coating of the continuous billet is timely removed during forging, the billet is drawn into about 550mm square by a small reduction amount of 20-30mm at initial cogging, and the billet

is drawn into about 400mm square by a large reduction amount of 70-80mm at intermedia cogging, thereby to penetrate the forging force into the core, so as to improve the inner quality, and the billet is further drawn and its outside is shaped as a circle of $\Phi 370\text{mm}$, and is finally finished to a finished circle of $\Phi 350\text{mm}$; after leaving the production line, the forged steel is air-cooled to 550°C and then is slowly cooled in sand; after the temperature drops to 200°C or lower, the forged steel is taken out of the pit.

4) Annealing:

after the forged steel is taken out of the pit, performing stress relief annealing treatment, the annealing temperature is 660°C , keeping warm for 12 hours or more, cooling to below 300°C along with the furnace, taking the forged billet outside the furnace, and air-cooling to room temperature, the tested macrostructure is shown in figure 1.

5) Inspection:

cutting head and tail parts of each piece of forged steel, and performing an ultrasonic test according to grade D/d of SEP 1921 standard to each piece of steel; performing a mechanical property test to a batch of the steel, wherein the tensile, the impact and the hardness meet the requirements of APISPEC 6A standard.

Embodiment 2

[0011] The steel is selected to be CrNiMo30C steel.

1) Smelting:

the molten steel is processed by KR pretreatment to perform desulphurization and desilicification, before the molten steel is delivered into converter, $S=0.003\%$, BOF converter smelting mainly performs dephosphorizing, decarbonizing and heating, alloy metal is added after tapping.

In LF refining process, the chemical constituents are fine tuned, deoxidation, desulphurization and removing impurity.

After degassing in RH vacuum and after breaking the vacuum, H is determined to be $H=1.0\text{ppm}$.

2) Continuous casting:

the molten steel is further processed by argon protection, the superheat degree is controlled to be 27°C , a continuous casting billet of $\Phi 600\text{mm}$ is produced by mixed electromagnetic stirring with mold electromagnetic stirring (M-EMS) and final electromagnetic stirring (F-EMS), and then it leaves the production line and is delivered to a slow cooling pit at 580°C , and is kept warm for 40h in the pit, after slowly cooling the continuous billet is taken outside the pit

and the macrostructure is tested according to appendix A of YB/T4149 standard: center porosity is grade 1.5, center crack is grade 0.5, shrinkage cavity is grade 0, free of intermediate cracks, white flakes or upwarping defects, etc..

3) Forging:

the continuous casting billet of $\Phi 600\text{mm}$ is cold delivered to the furnace and is heated to 1220°C , the heating speed is equal to or less than 200°C per hour, and is kept warm for 5 hours, the initial forging temperature is 1120°C , and the final forging temperature is not lower than 850°C , the continuous casting billet is freely forged by a 1400T double-station hydraulic machine, oxide coating of the continuous billet is timely removed during forging, the billet is drawn into about 550mm square by a small reduction amount of 20-30mm at initial cogging, and the billet is drawn into about 410mm square by a large reduction amount of 70-80mm at intermedia cogging, thereby to penetrate the forging force into the core so as to improve the inner quality, and the billet is further drawn and its outside shaped as a circle of $\Phi 400\text{mm}$, and is finally finished to a finished circle of $\Phi 390\text{mm}$; after leaving the production line, the forged steel is air-cooled to 550°C and then is slowly cooled in sand; after the temperature drops to 200°C or lower, the forged steel is taken out of the pit.

4) Annealing:

after the forged steel is taken out of the pit, performing stress relief annealing treatment, the annealing temperature is 660°C , keeping warm for 12 hours or more, cooling to below 300°C along with the furnace, taking the forged billet outside the furnace, and air-cooling to room temperature, the tested macrostructure is shown in figure 2;

5) Inspection:

cutting head and tail parts of each piece of forged steel, and performing an ultrasonic test according to grade D/d of SEP 1921 standard to each piece of steel; performing a mechanical property test to a batch of the steel, wherein the tensile, the impact and the hardness meet the requirements of APISPEC 6A standard.

[0012] Besides above embodiments, the present invention further includes other embodiments, and any technical solution formed by equivalent transformation or equivalent substitution shall fall within the protection scope of claims of the present invention.

Claims

1. A process for manufacturing CrNiMo30C forged steel for a wellhead device of deep-sea oil extraction, by continuous casting, **characterized in** including the following steps

1) Smelting molten steel: CrNiMo30C steel of a medium-carbon low-alloy CrNiMo steel, its constituents and mass percentages are as follows: C: 0.25~0.40%, Si: 0.15~0.40%, Mn: 0.80~1.30%, P: $\leq 0.010\%$, S: $\leq 0.008\%$, Cr: 0.85~1.55%, Mo: 0.30~0.65%, Ni: 0.80~1.40%, V: 0.02~0.08%, Nb: 0.02~0.05%, Cu $\leq 0.25\%$, B $\leq 0.0005\%$, H $\leq 0.0002\%$, the balance is Fe and the unavoidable impurity elements, wherein the molten steel is processed by KR pretreatment, dephosphorizing and decarbonizing in BOF converter smelting and heating, adding alloy after tapping, LF refining and RH vacuum degassing, thereby the CrNiMo30C steel with low H, S and P is obtained, and the steel is finally controlled according to the requirements of P $\leq 0.010\%$, S $\leq 0.005\%$ and H $\leq 1.5\text{ppm}$;

2) Continuous casting: the molten steel is further processed by argon protection, mixed electromagnetic stirring with mold electromagnetic stirring (M-EMS) and final electromagnetic stirring (F-EMS), in cooperation with secondary cooling water and low speed casting, and strictly controlling a superheat degree of 20-35 °C, thereby to decrease columnar crystals and enlarge equiaxed crystal, to prevent the development of columnar crystals to refine grains, to reduce segregation degree in steel crystallization process, to sufficiently ensure uniformity and compactness of inner structure; a continuous casting billet of $\Phi 600\text{mm}$ leaves the production line and is delivered to a slow cooling pit at a high temperature of equal to or more than 500°C, and is kept warm for equal to or more than 36h in the pit, and then the continuous billet is timely forged after outside the pit; the macrostructure of the continuous casting billet meets the requirements of appendix A of YB/T4149 standard: center porosity is equal to or less than grade 2.0, center crack is equal to or less than grade 1.0, shrinkage cavity is equal to or less than grade 1.0, defects of intermediate cracks, white flakes and upwarping defects are forbidden;

3) Forging: the billet is heated and the heating speed is equal to or less than 200°C per hour, the heating temperature of forging is 1200-1250°C, ensuring that the billet is completely heated, the initial forging temperature is higher than 1150°C, and the final forging temperature is not lower than 850°C, the continuous casting billet is freely forged by a 1400T double-station hydraulic machine, oxide coating of the continuous billet is timely removed during forging, the billet is drawn into square shape by a small reduction amount at initial cogging, thereby to prevent surface cracks, and is drawn into square shape by a large reduction amount at intermediate cogging, thereby to penetrate the

forging force into the core so as to improve the inner quality, and the billet is further drawn and its outside shaped as a circle, and is finally finished to a finished size; after leaving the production line, the forged steel is at a high temperature of equal to or greater than 500°C, and is then slowly cooled in sand; after the temperature drops to 200°C or lower, the forged steel is taken out of pit and slowly cooled;

4) Performing stress relief annealing treatment on forged steel: the annealing temperature is 650-700°C, keeping warm for 12 hours or more, cooling to below 300°C along with the furnace, taking the forged billet outside the furnace, and air-cooling to room temperature;

5) Inspection: cutting head and tail parts of each piece of forged steel, and performing an ultrasonic test according to grade D/d of SEP 1921 standard to each piece of steel; performing a mechanical property test to a batch of the steel, wherein the tensile, the impact and the hardness should meet the requirements of APISPEC 6A standard.

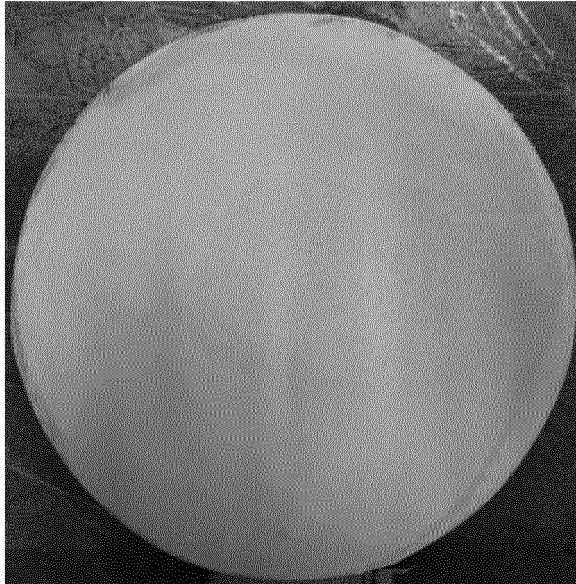


Figure 1

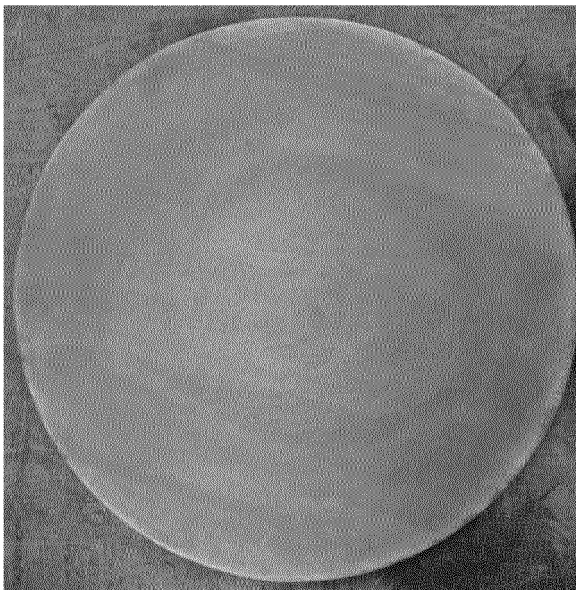


Figure 2

INTERNATIONAL SEARCH REPORT

International application No.
PCT/CN2017/096706

A. CLASSIFICATION OF SUBJECT MATTER

C22C 33/04 (2006.01) i; C22C 38/02 (2006.01) i; C22C 38/04 (2006.01) i; C22C 38/18 (2006.01) i; C22C 38/12 (2006.01) i; B22D 11/115 (2006.01) i

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

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

C22C 38; C22C 33; B22D

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

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

CNABS, SIPOABS, DWPI, CNTXT, CNKI, 铬, 钼, 镍, 压铸, 连续铸造, 锻, chromium, Cr, Mo, molybdenum, nickel, Ni, continu+ cast+, forg+

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
PX	CN 106591680 A (JIANGYIN XINGCHENG SPECIAL STEEL WORKS CO., LTD.), 26 April 2017 (26.04.2017), claim 1	1
A	CN 104894487 A (JIANGSU SHAGANG GROUP HUAIGANG SPECIAL STEEL CO., LTD.), 09 September 2015 (09.09.2015), claims 1 and 3	1
A	CN 102728756 A (JIANGSU JINYUAN FORGING LIMITED CO., LTD.), 17 October 2012 (17.10.2012), entire document	1
A	CN 104975235 A (JIANGYIN XINGCHENG SPECIAL STEEL WORKS CO., LTD.), 14 October 2015 (14.10.2015), entire document	1
A	JP 2002363690 A (DAIDO TOKUSHUKO KK), 18 December 2002 (18.12.2002), entire document	1

☐ 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
"A" document defining the general state of the art which is not considered to be of particular relevance	
"E" earlier application or patent but published on or after the international filing date	"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
"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)	"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
"O" document referring to an oral disclosure, use, exhibition or other means	"&" document member of the same patent family
"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 02 November 2017	Date of mailing of the international search report 17 November 2017
Name and mailing address of the ISA State Intellectual Property Office of the P. R. China No. 6, Xitucheng Road, Jimenqiao Haidian District, Beijing 100088, China Facsimile No. (86-10) 62019451	Authorized officer ZHANG, Fang Telephone No. (86-10) 62084779

INTERNATIONAL SEARCH REPORT
Information on patent family members

International application No.
PCT/CN2017/096706

5	Patent Documents referred in the Report	Publication Date	Patent Family	Publication Date
	CN 106591680 A	26 April 2017	None	
10	CN 104894487 A	09 September 2015	CN 104894487 B	22 February 2017
	CN 102728756 A	17 October 2012	CN 102728756 B	17 December 2014
	CN 104975235 A	14 October 2015	None	
	JP 2002363690 A	18 December 2002	JP 4626092 B2	02 February 2011
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Form PCT/ISA/210 (patent family annex) (July 2009)