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(54) Title: HIGH STRENGTH CRYOGENIC AUSTENITIC CORROSION RESISTANT WELDABLE CONSTRUCTION STEEL AND PRODUCTION METHOD

(57) Abstract: This invention relates to the metallurgy. The structural high strength cryogenic austenitic corrosion resistant weldable construction steel comprises the following elements in the ratios, wt.%: C - 0.05-0.07, Cr - 18.0-20.0, Ni - 5.0-7.0, Mn - 9.0- 11.0, Mo - 1.8-2.2, Si - 0.25-0.35, N - 0.30-0.38, Cu - 0.6-1.4, B - 0.005-0.015, Al - 0.015-0.035, S≤0.0025, P≤0.015, Sn≤0.005, Pb≤0.005, Bi≤0.005, As≤0.005, Fe - balance. The method of thermal deformation processing of the construction steel comprises: steel ingot heating, ingot deformation to a workpiece in the 1250-1050°C temperature range with a total strain rate of at least 50%, air cooling of the workpiece, straining of the resultant plate in the 1180-1080°C temperature range with a total compression rate of at least 40% and final straining in 2-3 runs with a total compression rate of 30-80% in the 1150- 1080°C temperature range with the finishing rolling temperature of 1050-1080°C, followed by rapid cooling at a 20-100°C/s rate to room temperature.

WO 2018/143837 A1

High Strength Cryogenic Austenitic Corrosion Resistant Weldable Construction Steel and Production Method

Field of the Invention. This invention relates to the metallurgy of construction steels comprising, as the basis, iron with the preset ratio of the alloying and impurity elements, and can be used in various branches of industry, more specifically, for the fabrication of high-strength cryogenic welded structures used for the transportation of liquefied gases.

Prior Art. Known is stainless austenitic steel (RU 2102522 C1, publ. 20.01.1998). said known steel contains carbon, chromium, nickel, manganese, nitrogen, silicon, vanadium, copper, molybdenum, cerium, selenium and iron in the following weight ratio (wt.%): carbon 0,01-0,06, chromium 18-22, nickel 15-18, manganese 2-10, nitrogen 0.2-0.5, silicon 0.01-0.45, vanadium 0.1-0.5, copper 0.1-1.5, molybdenum 0.1-2.5, cerium 0.005-0.25, selenium 0.05-0.25, balance iron, wherein if the manganese content is less than 5% the nitrogen content is approx. 0.3 while if the manganese content is greater than 5% the nitrogen content is 0.4-0.5.

Said known austenitic steel has multiple technical and mechanical properties and the stability of the austenitic structure and can be used for the fabrication of high-load parts of cryogenic machinery and devices.

Said known austenitic steel has the following disadvantages.

This steel is not economic due to high concentrations of expensive elements such as nickel (up to 18%) and molybdenum (up to 2.5%). For example, the nickel content in this steel is higher than in conventional austenitic stainless steel 19-10. Currently, manganese and nitrogen are used for the stabilization of the austenitic structure. A number of steel compositions having the claimed limits of element contents are not feasible. For example, if the manganese content in this steel is higher than 5%, the nitrogen content is acceptable at 0.4-0.5%. In fact at copper contents of above 1.0% and manganese contents of less than 10%, the nitrogen content should be lower than the claimed one, otherwise bubbles will form during the solidification of ingots at nitrogen contents of 0.4-0.5%.

The prototype of the first subject of this invention is high-strength corrosion resistant non-magnetic steel (RU 2392348 C2, publ. 20.06.2010). This steel has the following composition (wt.%): 0.02-0.06 carbon, 0.10-0.60 silicon, 9.5-12.5 manganese, 19.0-21.0 chromium, 4.5-7.5 nickel, 1.2-2.0 molybdenum, 0.08-0.22 vanadium, 0.005-0.010 calcium, 0.005-0.010 sodium, 0.05-0.15 niobium, 0.0005-0.001 magnesium, 0.40-0.60 nitrogen, 0.005-0.01 aluminum, balance iron and impurities, wherein said impurities in this steel are (wt.%) 0.003-0.012 sulfur, 0.004-0.025 phosphorus, 0.0002-0.005 lead, 0.0002-0.005 bismuth, 0.0002-0.005 tin, 0.0002-0.005 arsenic and 0.05-0.2 copper.

The disadvantages of this steel are as follows.

This steel crystallizes with the formation of γ- and δ- phases. Despite the availability of a large number of main element combinations such as Cr, Ni, Mn, V, Nb and Mo in the chemical composition according to this invention, the claimed nitrogen concentrations cannot be obtained using the conventional technology because the suggested nitrogen content of 0.40–0.60% in the steel at these combinations of the chemical composition exceeds the standard solubility of nitrogen in the metal at the melting temperatures, and the quantity of nitrogen that can be introduced in the liquid metal at the melting temperature exceeds its solubility in the γ- and δ- phases precipitating during the solidification, and therefore surplus nitrogen will form a gaseous phase and, hence, bubbles and porosity in the ingot. Furthermore, providing for the simultaneous presence of the active elements V, Nb, Al, Ca, Mg and Na in the steel in the claimed ratios is a complex technological task and is not possible in case of industrial production; off-analysis of these elements and dropout of the finished metal product properties will be unavoidable if these parameters actually depend on the concentrations and ratios of these elements.

The prototype of the second subject of this invention is a method of thermal deformation processing of the abovementioned high-strength corrosion resistant non-magnetic steel (RU 2392348 C2, publ. 20.06.2010).

The method of thermal deformation processing of the high-strength corrosion resistant non-magnetic steel comprises ingot heating, ingot deformation to a plate in the 1240–1000 °C temperature range with a total strain rate of 40–94%, air cooling of the plate for surface quality control and cleaning, deformation of the resultant plate in the 1240–1000 °C temperature range, treatment to the final total strain rate of 45 – 65% by 10–14% at a time to the sheet shape with a thickness of 2.5–3.5 times smaller than the plate thickness, air cooling of the sheet to 1000–950 °C, surface temperature control and final straining in 2-3 runs by 8–12% at a time, followed by rapid cooling at a 10–50 °C/s rate to 100–150 °C at the sheet surface and further air cooling.

Said known steel is melted in furnaces following a standard technology. For providing higher strength, more stable mechanical properties, lower intergranular corrosion susceptibility, higher wear resistance in icing conditions, improved weldability, low magnetic permeability and higher hot processing plasticity, the steel is subjected to thermal deformation processing in a special mode.

Disadvantages of the thermal deformation processing technology is the excessive detalization of process steps which complicates the implementation and control of the technology and, furthermore, for the recommended pre-straining heating mode and at some element ratios, the structure of the steel during heating to ≥ 1200 °C passes through several phases including δ- ferrite. For example, at highest patented Cr, Mn and Nb concentrations, lowest C and Ni concentrations and nitrogen concentrations of 0.40–0.5 % and at 1200–1320 °C the steel has an $\alpha + \gamma +$ (Nb, Cr)N structure. Therefore at the claimed heating temperature according to the patent (1240 °C) it is impossible to obtain a homogeneous γ- structure before rolling and hence no finished austenitic steel will be produced.

Disclosure of the Invention. The technical result of the first subject of the claimed invention relates to high strength and corrosion properties at cryogenic temperatures:

– room temperature strength $\sigma_B \geq 800$ MPa and $\sigma_{0,2} \geq 600$ MPa;

– cryogenic temperature toughness KCU $^{(-170)°C} \geq 1.5$ J/cm^2;

– good welability;

– low cost due to low nickel content;

– stable austenitic structure at -175 – 100 °C;

– corrosion resistance in acidic media and in seawater.

Said technical result of the first subject of the claimed invention is achieved as follows.

The high strength cryogenic austenitic corrosion resistant weldable construction steel comprises the following elements in the ratios as shown below, wt.%:

C: 0.05–0.07;

Cr: 18.0–20.0;

Ni: 5.0–7.0;

Mn: 9.0–11.0;

Mo: 1.8–2.2;

Si: 0.25–0.35;

N: 0.30–0.38;

Cu: 0.6–1.4;

B: 0.005-0.015;

Al: 0.015–0.035;

S: ≤ 0.0025;

P: ≤ 0.015;

Sn: ≤ 0.005;

Pb: ≤ 0.005;

Bi: ≤ 0.005;

As: ≤ 0.005;

Fe: balance,

wherein the nitrogen concentration providing, jointly with copper and boron, in the 1050 – 1300 °C temperature range for the existence of a $\gamma + Cr_2B$ structure and the combination of the claimed properties, is selected from the ratio N = 0.34-0.38% at Cu = 0.6–1.0% and B = 0.005–0.010%; N = 0.30–0.34% at Cu = 1.1–1.4% and B =0.011–0.015%.

The technical result of the second subject of the claimed invention is a simple industrial implementation of said method, good processability of the steel combined with a relatively low manganese concentration and the possibility of obtaining the required nitrogen concentration during melting at a normal pressure in the existing equipment.

The technical result of the second subject of the claimed invention is achieved as follows.

The method of thermal deformation processing of the high strength cryogenic austenitic corrosion resistant weldable construction steel as per Claim 1 comprises ingot heating, ingot deformation to a workpiece in the 1240–1050 °C temperature range with a total strain rate of at least 50%, air cooling of the workpiece, straining of the resultant plate in the 1180–1080 °C temperature range with a total compression rate of at least 40% and final straining in 2-3 runs with a total compression rate of 30–80 % in the 1150–1080 °C temperature range with the finishing rolling temperature of 1050–1080 °C, followed by rapid cooling at a 20–100 °C/s rate to room temperature.

The advantages of the steel suggested in this invention and its treatment method are as follows. At the claimed concentrations of the main elements C, Cr, Ni, Mn, Mo, Cu and B, for nitrogen content N = 0.34–0.38% and copper and boron contents Cu = 0.6–1.0% and B =0.005–0.010%, and for nitrogen content N = 0.30–0.34% and copper and boron contents Cu = 1.1–1.4% и B = 0.011–0.015%, the equilibrium structure of the steel in the 1050 – 1300 °C is austenite with fine chromium boride Cr_2B particles which guarantees the basic austenitic structure and the required combination of properties under actual industrial process conditions.

The suggested steel also has good economic parameters due to a low nickel content and high workability due to a small number of alloying additions, and at their claimed concentrations the required nitrogen content can be obtained by melting at normal pressure in the existing equipment.

Carbon contents in the 0.05–0.07% range favors the formation of an austenitic structure in the steel and, jointly with nitrogen provides for the required steel hardening during thermal and thermal deformation processing combined with good corrosion resistance and weldability. At higher carbon contents in the steel its corrosion resistance decreases, this being accompanied by increasing susceptibility to intergranular corrosion, susceptibility to brittle fracture and decreasing weldability.

Chromium, nickel, manganese, molybdenum and copper in the claimed concentration limits at a boron content of 0.005 – 0.015 wt.% and a nitrogen content of 0.30 – 0.38 wt.% at every possible combination of these element compositions in the composition range delimited in this invention provide the finished steel with a stable austenitic structure with a small quantity of fine boron particles, the required mechanical properties, corrosion resistance in acid media and in seawater and suitability for the fabrication of cold-resistant high-strength welded structure used for liquefied gas transportation.

At the concentrations of the main elements (Cr, Ni, Mn and Mo) below the claimed limits, the required austenitic structure and properties are not achievable, same as the nitrogen concentrations as are required in accordance with this invention. At high concentrations of these elements an austenitic structure forms but the obtained γ- solid solution has a higher hot plastic deformation strength.

Increased Cr and Mo concentrations broaden the α + γ phase existence range at high temperatures and hinders the dissolution of excess phases. Higher manganese concentrations complicate the steel melting process, and at elevated nickel concentrations the steel does not meet the economic requirements.

The claimed concentrations of Cr, Ni, Mn and Mo provide for a high nitrogen solubility in the liquid phase and in the austenite, an das a resultat every possible combination of element concentrations in the composition range claimed in the invention and a nitrogen concentration of 0.30 – 0.38 wt.% the steel crystallized without bubbles or pores in ingots or uninterruptible cast workpieces. At lower nitrogen concentrations the required mechanical properties are not achieved, while at higher nitrogen contents bubbles and pores may form in the ingots.

Copper makes the steel of this composition more corrosion resistant and at the claimed concentration of other elements increases the higher temperature limit of the γ region. At copper concentrations 0.6 – 1.4 wt.% the equilibrium steel structure in the 1050 – 1300 °C temperature range is γ + Cr_2B which guarantees industrial production of an austenite structure and the required combination of properties. At lower copper concentrations the corrosion resistance of the steel decreases in acid media and in seawater. High copper concentrations are also undesirable due to an increase in the lower temperature limit of the austenite region and the melt and the steel may acquire inhomogeneous chemical composition and properties.

Boron in concentrations of 0.005 – 0.015% and the claimed concentrations of alloying elements forms chromium borides in the solid metal below the solidus temperature which prevent grain growth during steel heating in the austenite region before hot straining and before quenching. Lower boron concentrations are inefficient, while at higher boron concentrations chromium borides form in the liquid metal during crystallization, their particles have large sizes and negatively affect the properties of the steel.

Aluminum within the claimed concentration limits 0.015 – 0.035 wt.% provides for the required steel deoxidation degree and oxygen content. At lower aluminum concentrations the required steel deoxidation degree is not achieved and chromium oxides may form, while higher aluminum concentrations lead to the formation of high-temperature aluminum nitrides which negatively affect the properties of the steel.

Silicon within the claimed concentration limits favors efficient steel deoxidation and the removal of nonmetallic inclusions and typically provides for the acceptable equivalent concentration of chromium $Cr_э$. At higher silicon concentrations the $Cr_э$ content increases and δ- ferrites may form in the steel structure. At lower silicon concentrations steel deoxidation is hindered.

The presence of these impurities complicates the achievement of the required structure and properties of the steel and reduces the efficiency of nitrogen addition. Therefore nitrogen alloyed steel are usually melted following the pure steel technology. The impurity concentration limit required in accordance with this invention, i.e. P ≤ 0.015, S ≤ 0.0025, Sn ≤ 0.005, Pb ≤ 0.005, As ≤ 0.005 and Bi ≤ 0.005 in the steel provides for the best steel properties achievable at this composition. At higher impurity concentrations they negatively affect the structure and properties of the steel and the structure formation processes in the steel. Significantly lower impurity concentrations is currently difficultly achievable for technological reasons.

With the thermal deformation processing according to this invention the steel has a basic austenitic structure and the required combination of mechanical and physical properties. Failure to maintain the required heating temperatures before straining and the onset and completion of the thermal deformation processing operations, compression rate and cooling rate at different process steps the required austenitic steel and the properties claimed in the invention cannot be achieved.

Embodiments of the Invention. Example of steel melting and processing technology follows.

Steels of the claimed composition were melted for testing purposes in a vacuum induction furnace with a 50 kg liquid metal capacity. We used pure charge materials: Armco iron, electrolytic copper and nickel, metallic chromium and manganese, nitrated ferrochromium and ferroboron.

The ingots were heated at 1250 °C and forged in the 1250–1050 °C temperature range to 70% strain, and then the forged pieces were air cooled and cleaned.

Then the forged pieces were heated to 1180 °C and rolled to a total strain of 60% (to a 10 mm thickness) in the 1180–1080 °C temperature range in 9 runs with interim heating. After rolling the pieces were air cooled.

The final rolling was following the high-temperature thermomechanical treatment setup. The metal was heated to 1150 °C and rolled to a sheet with a total strain of 60% (to a 6 mm thickness) in the 1150–1080 °C temperature range with interim heating. The final cooling of the rolled pieces was at a 100 °C/s rate in water. Then the rolled pieces were cleaned and cut to the required piece sizes. The chemical composition of the steels is summarized in Table 1. The mechanical properties of the alloys are presented in Table 2.

Table 1. Chemical Composition of the Steels

Ingot	Element Concentrations, wt.%										
	C	B	Cr	Ni	Mn	Mo	P	S	Si	N	Cu
1	0.051	0.011	17.5	6.5	8.8	1.91	0.006	0.012	0.35	0.30	1.2
2	0.060	0.015	19.6	6.1	9.4	1.90	0.008	0.006	0.33	0.34	1.4
3	0.076	0.006	18.7	6.1	10.1	2.00	0.011	0.004	0.23	0.37	0.7

Table 2. Mechanical Properties of As Processed Steels

Specimens of Ingot Nos.	$T_{исп}$, °C	$\sigma_{0,2}$, MPa	σ_B, MPa	δ, %	ψ, %	HV_{cp}, MPa	KCU MJ/m^2
1, 13 mm sheet	20	545	835	50	70	255	2.60
	-163	840	1160	26	70	–	1.55
2, 13 mm sheet	20	515	855	40	–	190	–
3, 8 mm sheet	100	615	970	24	52	–	3.15
	20	690	925	33	62	270	2.90
	-100	1140	1405	47	83	–	2.25
	-175	1405	1775	33	84	–	1.60

* The tensile tests were at –175 °C and impact bending tests were at –196 °C.

The corrosion resistance of the alloy provided in this invention in acid media (0.5M H_2SO_4, pH = 0.44) and in seawater (3 % NaCl) was tested using different parameters (intergranular corrosion, total, pitting and crevice corrosion) was not lower and not higher compared with corrosion resistant stainless steels of the (05–12)Cr18Ni(8–10) and 06Cr18NNi(8–10) grades.

What is claimed is a

1. High strength cryogenic austenitic corrosion resistant weldable construction steel comprising carbon , chromium, nickel, manganese, molybdenum, silicon, nitrogen, aluminum, iron and impurities the latter being sulfur, phosphorus, tin, lead, bismuth and arsenic, wherein said steel further comprises copper and boron with the following element ratios, wt.%:

C: 0.05–0.07;

Cr: 18.0–20.0;

Ni: 5.0–7.0;

Mn: 9.0–11.0;

Mo: 1.8–2.2;

Si: 0.25–0.35;

N: 0.30–0.38;

Cu: 0.6–1.4;

B: 0.005-0.015;

Al: 0.015–0.035;

S: ≤ 0.0025;

P: ≤ 0.015;

Sn: ≤ 0.005;

Pb: ≤ 0.005;

Bi: ≤ 0.005;

As: ≤ 0.005;

Fe: balance.

wherein the nitrogen concentration providing, jointly with copper and boron, in the 1050 – 1300 °C temperature range for the existence of a $\gamma + Cr_2B$ structure and the combination of the claimed properties, is selected from the ratio N = 0.34-0.38% at Cu = 0.6–1.0% and B = 0.005–0.010%; N = 0.30–0.34% at Cu = 1.1–1.4% and B =0.011–0.015%.

2. Method of thermal deformation processing of the high strength cryogenic austenitic corrosion resistant weldable construction steel as per Claim 1 comprises ingot heating, ingot deformation to a workpiece in the 1240–1050 °C temperature range with a total strain rate of at least 50%, air cooling of the workpiece, straining of the resultant plate in the 1180–1080 °C temperature range with a total compression rate of at least 40% and final straining in 2-3 runs with a total compression rate of 30–80 % in the 1150–1080 °C temperature range with the finishing rolling temperature of 1050–1080 °C, followed by rapid cooling at a 20–100 °C/s rate to room temperature.

INTERNATIONAL SEARCH REPORT

International application No.

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A. CLASSIFICATION OF SUBJECT MATTER

C22C38/58 *(2006.01)*
C21D8/02 *(2006.01)*

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 38/00-38/60, C21D 8/00, 8/02

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)

ESP@cenet, PAJ, Patsearch, RUPTO

C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2016199776 A (NIPPON STEEL & SUMITOMO METAL CORP) 01.12.2016, abstract, paragraphs [0001], [0024]-[0050], claims	1
A		2
X	WO 2011/053460 A1 (ATI PROPERTIES, INC.) 05.05.2011, paragraphs [0009]-[0016], [0021]-[0034], [0055], claims	1
X	WO 2009/070345 A1 (ATI PROPERTIES, INC.) 04.06.2009, paragraphs [0008]-[0014], [0022]-[0035], [0048]-[0050], [0052], claims	1
A	RU 2392348 C2 (FEDERAL'NOE GOSUDARSTVENNOE UNITARNOE PREDPRIJATIE "TSENTRAL'NYJ NAUCHNO-ISSLEDOVATEL'SKIJ INSTITUTKONSTRUKTSIONNYKH MATERIALOV "PROMETEJ" et al.) 20.06.2010	1-2
A	RU 2545856 C2 (ROSSIYSKAYA FEDERATSIYA. OT IMENI KOTOROY VYSTUPAYET MINISTERSTVO PROMYSHLENNOSTI I TORGOVLI ROSSIYSKOY FEDERATSII) 10.04.2015	1-2

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

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Date of the actual completion of the international search	Date of mailing of the international search report
01 December 2917 (01.12.2917)	07 February 2018 (07.02.2018)

Name and mailing address of the ISA/RU:	Authorized officer
Federal Institute of Industrial Property, Berezhkovskaya nab., 30-1, Moscow, G-59, GSP-3, Russia, 125993 Facsimile No: (8-495) 531-63-18, (8-499) 243-33-37	R.Karachev Telephone No. (8-495) 531-65-15