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(54) Title: USE OF A FERRITIC STEEL IN UREA MANUFACTURING

(57) Abstract: Use of a ferritic stainless steel for the manufacture of a component of a high- pressure urea synthesis section of a urea plant, said component being exposed, in use, to contact with an ammonium carbamate-containing stream, wherein Cr is between 22% and 25%, Ni is not more than 0.5%, the weight percentages of Ti, Nb, nitrogen and carbon in the steel satisfy the following condition: $(4(N + C) + 0.2) \leq (Ti + Nb) \leq 1.0$.



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Use of a ferritic steel in urea manufacturing

DESCRIPTION

Field of the invention

5 The present invention refers to the field of materials for the manufacturing of the equipment of urea synthesis plants, particularly in the high-pressure urea synthesis section.

Prior Art

10 Urea is produced industrially by reacting ammonia and carbon dioxide at high temperature and high pressure. The reaction involves basically the formation of ammonium carbamate and its dehydration to form urea. The production of urea is known to be a challenge in terms of resistance to corrosion of the equipment because of the combination of highly corrosive substances (particularly the ammonium carbamate), high temperature and pressure.

15 Most of the urea production capacity currently installed use the so-called stripping process. In a stripping process, the synthesis solution leaving the reactor containing unreacted ammonia and carbon dioxide, mostly in the form of ammonium carbamate, is sent to a stripper where it is heated still at a high pressure which may be substantially the same pressure of the reactor.

20 During the stripping process, the ammonium carbamate decomposes into ammonia and carbon dioxide in the liquid phase and part of the liberated ammonia and carbon dioxide passes from the liquid phase to the gas phase. The stripping process therefore produces an aqueous solution of urea with a reduced content of unconverted carbamate and a gas phase containing the unconverted ammonia and carbon dioxide removed from the liquid phase. The liquid phase is
25 normally sent to one or more stages of further recovery at a lower pressure; the gas phase is condensed at high pressure and recycled to the reactor.

The stripping process may be promoted by adding a gaseous stripping agent which may be carbon dioxide or ammonia. In absence of added stripping agent, the process is termed self-stripping.

5 The stripper is typically a shell-and-tube apparatus where the reaction effluent flows through the tubes, e.g., with a falling-film flow, and the tube bundle is externally heated by hot steam. The condenser is also, in most cases, a shell-and-tube apparatus. The reactor is typically a vertical pressure vessel with a suitable set of perforated trays.

10 The reactor, the stripper and the condenser are part of a high-pressure synthesis section also termed synthesis loop. The synthesis section may also include additional equipment such as a scrubber for the gases vented from the reactor. These pieces of equipment operate typically at a pressure around 150 bar or more and a high temperature around or above 200 °C. The operating conditions, in combination with the presence of the aggressive ammonium carbamate, are
15 very demanding for the materials. In general, a target corrosion rate should be not greater than around 0.1 mm/y to provide an acceptable service life of components, for example 15 or 20 years.

A known provision is to introduce an oxygen-containing stream for passivation. For example, passivation air is introduced at the bottom of the stripper. However,
20 the introduction of air affects the overall efficiency of the synthesis loop (particularly of the condensation) and introduces a potential explosion hazard. Materials adapted to resist corrosion without the addition, or with a lower amount, of O₂ would be preferable.

25 The equipment of the synthesis section that need protection from corrosion can be categorized, in most cases, into pressure vessels, internals of vessels, shell-and-tube heat exchangers and piping. The pressure vessels (e.g., vessel of the urea synthesis reactor) are typically made of carbon steel and protected by a corrosion-resistant internal lining 4 to 10 mm thick, usually 5 to 7 mm. The corrosion resistant material therefore must be available in sheets of at least 4 mm
30 thickness, must be weldable for coupling with the material of the vessel and the

adjacent sheet of lining, and must be sufficiently ductile for adaptation to the shape of the vessel. The internals of vessels include, notably, the perforated trays usually provided in the urea reactor.

Shell-and-tube (S&T) heat exchangers are normally found in the stripper and high-pressure condenser. In such case, at least one side of the heat exchanger (shell side or tube side) is directly in contact with a highly aggressive ammonium-carbamate containing solution. A corrosion-resistant material is required primarily for one or more of the following: the tubes, an overlay of the tube sheet, the welding between tubes and tube sheet. The tube sheet is typically a large steel plate made of carbon steel protected by a corrosion resistant overlay. The tubes are welded to such overlay. S&T heat exchangers include typically two tube sheets at opposite ends of straight tubes, or a single tube sheet in case of U-tubes.

The tubes of the stripper are among the most critical components because they operate under high temperature (possibly above 200 °C) and high concentration of carbamate.

The use of high-grade materials for large components like tubes and/or plates for lining introduces a relevant cost.

In relation to piping, a clear requirement is that the corrosion-resistant material must be easily weldable, e.g., for welding of pipe sections, flanges, etc.

Many corrosion-resistant materials have been proposed over the years including titanium; super austenitic steel such as 25/22/2 (UNS: S31050); full zirconium tubes or bimetallic titanium-zirconium (Ti-Zr) tubes. However, no one was completely satisfactory and some were very expensive. The bimetallic construction, in addition to being expensive, is practical only for the manufacturing of tubes and not suitable for sheets or lining. In recent years, duplex stainless steels emerged as a preferred material for the construction of the urea HP synthesis equipment, notably for HP stripper. Duplex steels are distinguished by a two-phase structure showing both ferrite and austenite.

Examples of high-performance duplex steels include UNS S32906 and UNS S32808. More recently, the use of ferritic steel was proposed.

Ferritic steels are of considerable interest because certain ferritic steels have been found to perform equal to or better than duplex steels at a significantly less cost. However, there are problems in connection with the use of ferritic steels in the urea plants, including: ferritic steels may be difficult to weld due to precipitation of carbides; a limited ductility makes the material less suitable for lining, risk of selective corrosion of welded joints with different material even if typically suitable for urea service such as UNS32906.

WO 2021/006729 A1 discloses a ferritic steel including a certain amount of niobium (Nb) and a maximum of 0.005% of carbon to reduce precipitation of carbides which facilitates welding. However, this steel is still expensive and difficult to produce due to the very stringent limit of maximum content of carbon.

JP 2018 168415 A and EP 3 153 599 A1 belong to the known art.

Description of the invention

The invention aims to provide a ferritic steel suitable for manufacturing components of urea plants exposed to contact with ammonium carbamate containing streams, particularly in the high-pressure urea synthesis section (high-pressure synthesis loop). The invention aims to provide a ferritic steel for said use which is adapted, among others, for manufacturing of internal lining of any of: pressure vessels, tubes, and tube sheet overlay of shell-and-tubes heat exchangers, and/or piping. The invention also aims to a ferritic steel with good properties of weldability and having a competitive cost.

The aims are reached with the use of a ferritic steel according to the claims.

The invention addresses the manufacturing of urea equipment in contact with ammonium-carbamate containing streams. Examples of such streams are the reactor effluent, the stripper effluent, a recycle stream obtained in the high-pressure condenser and recycled to the reactor. For the manufacturing of such

equipment, the invention teaches the use of a ferritic steel according to the claims.

Among others, said ferritic steel has a low content of nickel, being max. 0.5 wt%, and a content of titanium and niobium that meets the equation $(4(N + C) + 0.2) \leq (Ti + Nb) \leq 1.0$. Here, the symbols of the elements are used to denote the content in weight percent (wt%) in the steel.

The applicant has found that ferritic steels with Ti and Nb meeting the above equation, in combination with Cr being in the range of 22.0 to 25.0 wt%, show a reduced precipitation of chromium carbides during the welding, thus being suitable for making components like internal linings, shell-and-tube equipment and piping. The low content of nickel has the additional advantage of reducing cost.

Preferred features are described in the dependent claims. They include the following.

The wt% of Ti and Nb satisfy preferably the condition $0.2 \leq (Ti + Nb) \leq 0.5$ and more preferably, in addition to the above, the condition $0.5 \leq Ti / Nb \leq 2.0$ and, preferably, the condition $0.7 \leq Ti / Nb \leq 1.0$.

Preferably the wt% of both Ti and Nb is not null, preferably at least 0.01 or at least 0.05.

A preferred ferritic steel for the use of the present invention has the composition, expressed in weight percentage (wt%, or briefly %) of each component with respect to the overall weight of the ferritic steel:

$C \leq 0.015\%$

$Si \leq 1\%$

$Mn \leq 1\%$

$P \leq 0.05\%$

$S \leq 0.005\%$

$22.0\% \leq \text{Cr} \leq 25.0\%$

$\text{N} \leq 0.04\%$, preferably $\leq 0.02\%$

$\text{Ni} \leq 0.5\%$

$\text{Cu} \leq 0.5\%$

5 $\text{Mo} \leq 0.5\%$

$\text{Al} \leq 0.05\%$

$\text{Ti} \leq 0.25\%$

$\text{Nb} \leq 0.25\%$.

10 Preferably, in all the above embodiments, the steel contains at least 23.0% chromium, preferably at least 24.0%.

Even more preferably, said ferritic steel comprises or consists of:

C	from 0.001 wt% to 0.015 wt%; preferably from 0.002 wt% to 0.014 wt%; more preferably from 0.003 wt% to 0.013 wt%; even more preferably 0.011 wt%;
Cr	from 22.0 wt% to 25.0 wt%; preferably from 23.0 wt% to 24.8 wt%; more preferably from 23.5 wt% to 24.5 wt%; even more preferably 24.07 wt%;
Ni	from 0.05 wt% to 0.5 wt%; preferably from 0.1 wt% to 0.4 wt%; more preferably from 0.2 wt% to 0.3 wt%; even more preferably 0.24 wt%;
Mn	from 0.08 wt% to 0.4 wt%; preferably from 0.1 wt% to 0.35 wt%; more preferably from 0.15 wt% to 0.25 wt%; even more preferably 0.2 wt%;

Mo	from 0.005 wt% to 0.5 wt%; preferably from 0.01 wt% to 0.2 wt%; more preferably from 0.02 wt% to 0.1 wt%; even more preferably 0.04 wt%;
Ti	from 0.05 wt% to 0.4 wt%; preferably from 0.1 wt% to 0.35 wt%; more preferably from 0.15 wt% to 0.25 wt%; even more preferably 0.2 wt%;
Nb	from 0.05 wt% to 0.45 wt%; preferably from 0.1 wt% to 0.4 wt%; more preferably from 0.15 wt% to 0.3 wt%; even more preferably 0.23 wt%;
Cu	≤ 0.5 wt%; preferably ≤ 0.25 wt%; more preferably ≤ 0.1 wt%; even more preferably absent;
Al	≤ 0.5 wt%; preferably ≤ 0.25 wt%; more preferably ≤ 0.1 wt%; even more preferably absent;
P	from 0.001 wt% to 0.05 wt%; preferably from 0.005 wt% to 0.04 wt%; more preferably from 0.008 wt% to 0.03 wt%; even more preferably 0.026 wt%;
Si	from 0.05 wt% to 1.0 wt%; preferably from 0.1 wt% to 0.7 wt%; more preferably from 0.2 wt% to 0.5 wt%; even more preferably 0.35 wt%;
N	from 0.005 wt% to 0.04 wt%; preferably from 0.007 wt% to 0.03 wt%; more preferably from 0.01 wt% to 0.02 wt%; even more preferably 0.017 wt%;
Fe	balance.

The compositions are given in wt% of relevant alloy elements, unless otherwise specified in the present description. The balance is predominantly Fe and may include unavoidable impurities and/or processability elements if required.

5 A ferritic steel suitable for the use of the present invention is EN 1.4613, also designated X2CrTi24. This steel is commercially available with thickness up to 6 mm and is particularly suitable for making internal lining of vessels.

The ferritic steel, according to the invention, may be used preferably for making any of: a tube sheet, tubes of a tube bundle; an internal lining of a pressure vessel, a (e.g., perforated) tray of a pressure vessel; a pipe; a weld; an internal
10 component of any of a reactor, a stripper, a condenser, a scrubber located in the high-pressure urea synthesis section of the urea plant. In case of internal components of a reactor, a preferred use is the manufacturing of internal plates of the reactor.

A particularly preferred use is the making of a corrosion-resistant overlay of a
15 tube sheet, and the overlay process is performed by explosion cladding. Tests performed by the applicant have shown that the ferritic steel as above identified is well adapted for this use.

Another preferred use of the ferritic steel of the invention is for making an autogenous weld of any of: said tube sheet overlay, said tubes of the tube bundle;
20 said internal lining of a pressure vessel, said pipe; said internal component of the reactor, the stripper, the condenser, the scrubber located in the high-pressure urea synthesis section.

In the present description, "autogenous" means a welding wherein the filler material is either supplied by melting the base material, or such filler material is
25 provided by a filler rod of identical composition.

Preferably, the ferritic steel operates in absence of an addition of oxygen (O₂) or of an oxygen-containing gas for passivation.

An aspect of the invention is also an equipment of a high-pressure urea synthesis section wherein the equipment includes at least one component which is contact with an ammonium-carbamate containing stream during the use. Such component is made as previously described, i.e., expressed in other terms, said use of ferritic stainless steel is made for the manufacture of said at least one component.

The equipment is for example any of: a reactor, a stripper, a condenser, a scrubber of the high-pressure synthesis section, a pipe. According to a further embodiment, the equipment is a heat exchanger of the high-pressure urea synthesis section; preferably the tubes, the lining and/or the tubesheet overlay of such heat exchanger is/are made as described above. Hence, according to such embodiment, said use of ferritic stainless steel is made for the manufacture of said tubes, lining and/or tubesheet overlay of said heat exchanger.

Test data

Welding tests were performed under the following conditions which simulate the environment of a urea reactor.

N/C = 3.2 (Nitrogen/Carbon ratio)

H/C = 0.8 (Hydrogen/Carbon ratio)

T = 210 °C

P = 240 bar

The ferritic steel was a steel according to EN 1.4613 with the following composition:

	wt%
C	0.011
Cr	24.07
Ni	0.24
Mn	0.2

Mo	0.04
Ti + Nb	0.43
Ti	0.2
Nb	0.23
Cu	-
Al	-
P	0.026
Si	0.35
N	0.017

The reference material was a duplex stainless steel according to UNS32906. A corrosion rate was determined based on weight loss. The presence of localized corrosion was also checked.

5 Test 1: *1.4613 base material as plate*

Material	Corrosion rate [mm/y]
EN 1.4613	0.03
Ref.	0.20

Test 2: *1.4613 autogenous welding of 2 plates*

Material	Corrosion rate [mm/y]
EN 1.4613	0.03
Ref.	0.20

No local corrosion was detected, neither at the plate nor at the welding area.

CLAIMS

5

- 1) Use of a ferritic stainless steel for the manufacture of a component of a high-pressure urea synthesis section of a urea plant, said component being exposed, in use, to contact with an ammonium carbamate-containing stream, wherein said ferritic stainless steel has the following composition in weight percentages (wt%):

C	from 0.001 wt% to 0.015 wt%;
Cr	from 22.0 wt% to 25.0 wt%;
Ni	from 0.05 wt% to 0.5 wt%;
Mn	from 0.08 wt% to 0.4 wt%;
Mo	from 0.005 wt% to 0.5 wt%;
Ti	from 0.05 wt% to 0.4 wt%;
Nb	from 0.05 wt% to 0.45 wt%;
Cu	≤ 0.5 wt%;
Al	≤ 0.5 wt%;
P	from 0.001 wt% to 0.05 wt%;
Si	from 0.05 wt% to 1.0 wt%;
N	from 0.005 wt% to 0.04 wt%;
balance	Fe and unavoidable impurities

and wherein the weight percentages of Ti, Nb, nitrogen and carbon satisfy the following condition:

$$(4 (N + C) + 0.2) \leq (Ti + Nb) \leq 1.0.$$

- 2) The use of claim 1, wherein:

in the ferritic steel, the wt% of Ti and Nb satisfy the condition:

$$0.2 \leq (Ti + Nb) \leq 0.5.$$

- 5 3) The use of claim 2, wherein:

in the ferritic steel, the wt% of Ti and Nb satisfy the additional condition:

$$0.5 \leq Ti / Nb \leq 2.0.$$

- 4) The use of any of the previous claims, wherein the steel contains at least 23.0 wt% chromium, preferably at least 24.0 wt%.

- 10 5) The use of claim 1, wherein said ferritic steel comprises or consists of:

C	from 0.002 wt% to 0.014 wt%; preferably from 0.003 wt% to 0.013 wt%;
Cr	from 23.0 wt% to 24.8 wt%; preferably from 23.5 wt% to 24.5 wt%;
Ni	from 0.1 wt% to 0.4 wt%; preferably from 0.2 wt% to 0.3 wt%;
Mn	from 0.1 wt% to 0.35 wt%; preferably from 0.15 wt% to 0.25 wt%;
Mo	from 0.01 wt% to 0.2 wt%; preferably from 0.02 wt% to 0.1 wt%;
Ti	from 0.1 wt% to 0.35 wt%; preferably from 0.15 wt% to 0.25 wt%;

Nb	from 0.1 wt% to 0.4 wt%; preferably from 0.15 wt% to 0.3 wt%;
Cu	≤ 0.25 wt%; preferably ≤ 0.1 wt%;
Al	≤ 0.25 wt%; preferably ≤ 0.1 wt%;
P	from 0.005 wt% to 0.04 wt%; preferably from 0.008 wt% to 0.03 wt%;
Si	from 0.1 wt% to 0.7 wt%; preferably from 0.2 wt% to 0.5 wt%;
N	from 0.007 wt% to 0.03 wt%; preferably from 0.01 wt% to 0.02 wt%;
balance	Fe and unavoidable impurities.

- 6) The use according to the previous claim, wherein said ferritic steel comprises or consists of:

C	0.011 wt%;
Cr	24.07 wt%;
Ni	0.24 wt%;
Mn	0.2 wt%;
Mo	0.04 wt%;
Ti	0.2 wt%;
Nb	0.23 wt%;
Cu	absent;
Al	absent;

P	0.026 wt%;
Si	0.35 wt%;
N	0.017 wt%;
balance	Fe and unavoidable impurities

- 7) The use according to any of the previous claims, wherein said component exposed to ammonium carbamate includes any of: a tube sheet overlay, tubes of a tube bundle; an internal lining of a pressure vessel, a tray of a pressure vessel; a pipe; a weld or an autogenous weld; an internal component of any of a reactor, a stripper, a condenser, a scrubber located in the high-pressure urea synthesis section of the urea plant.
- 8) The use of claim 7, wherein the ferritic steel is used for making a corrosion-resistant overlay of a tube sheet, and the overlay process is performed by explosion cladding.
- 9) The use of claim 7 or 8, wherein the ferritic steel is used for making an autogenous weld of any of: said tube sheet overlay, said tubes of the tube bundle; said internal lining of a pressure vessel, said pipe; said internal component of the reactor, the stripper, the condenser, the scrubber located in the high-pressure urea synthesis section.
- 10) The use of any of the previous claims, wherein the steel operates in absence of an addition of oxygen (O₂) or of an oxygen-containing gas for passivation.
- 11) An equipment of a high-pressure urea synthesis section wherein the equipment includes at least one component which is contact with an ammonium-carbamate containing stream during the use, and wherein use of a ferritic stainless steel according to any of claims 1 to 10 is made for the manufacture of said at least one component.

- 12) The equipment according to claim 11, wherein the equipment is any of: a reactor, a stripper, a condenser, a scrubber of the high-pressure urea synthesis section, a pipe.
- 5 13) The equipment according to claim 11 or 12, wherein the equipment is a heat exchanger of the high-pressure urea synthesis section.
- 14) The equipment according to the claim 13, wherein use of a ferritic stainless steel according to any of claims 1 to 10 is made for the manufacture of tubes, lining and/or tubesheet overlay of said heat exchanger.
- 10 15) The equipment according to any of claims 11-14 having no addition of O₂ or of a gas containing O₂ for passivation.

INTERNATIONAL SEARCH REPORT

International application No
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A. CLASSIFICATION OF SUBJECT MATTER		
INV.	C22C38/44	C22C38/48
	C22C38/00	C22C38/50
		C22C38/02
		C22C38/04
ADD.	B01J19/02	
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 B01J		
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) EPO-Internal, WPI Data		
C. DOCUMENTS CONSIDERED TO BE RELEVANT		
Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	JP 2018 168415 A (NIPPON STEEL & SUMIKIN SST) 1 November 2018 (2018-11-01) the whole document -----	1 - 15
X	EP 3 153 599 A1 (JFE STEEL CORP [JP]) 12 April 2017 (2017-04-12) claims 1-3 paragraphs [0006] - [0041] table 1 -----	1 - 15
A	WO 2021/006729 A1 (STAMICARBON [NL]) 14 January 2021 (2021-01-14) claims 1-17 table 1 pages 4-27 ----- - / - -	1 - 15
☒ Further documents are listed in the continuation of Box C. ☒ See patent family annex.		
* Special categories of cited documents : "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 "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 "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 "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 "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 "&" document member of the same patent family		
Date of the actual completion of the international search 17 March 2025		Date of mailing of the international search report 27/03/2025
Name and mailing address of the ISA/ European Patent Office, P.B. 5818 Patentlaan 2 NL - 2280 HV Rijswijk Tel. (+31-70) 340-2040, Fax: (+31-70) 340-3016		Authorized officer Vlassi, Eleni

INTERNATIONAL SEARCH REPORT

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C(Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT		
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INTERNATIONAL SEARCH REPORT

Information on patent family members

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