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(54) DUPLEX STAINLESS STEEL ALLOY FOR USE IN SEAWATER APPLICATIONS

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(57) ABSTRACT

The present invention relates to a stainless steel alloy, more precisely a duplex stainless steel alloy having ferritic-austenitic matrix and having high corrosion resistance in combination with good structural stability and hot-workability, in particular a duplex stainless steel having a ferritic content of 40-65% and a well-balanced composition, which gives the material corrosion properties making it more suitable for use in chloride-containing environments than what has been found possible previously. The material according to the present invention has, in view of the high alloy content thereof, extraordinarily good workability, in particular hot-workability, and should thereby be very suitable to be used for, for instance, the manufacture of bars: pipes, such as welded and weld less pipes, weld material, construction parts, such as, for instance, flanges and couplings. These objects are met according to the present invention with duplex stainless alloy, which contain (in % by weight): C more than 0 and up to max 0.03 Si up to max 0.5 Mo 0-3.0 20 Cr 24.0-30.0 Ni 4.9-10.0 Mo 3.0-5.0 N 0.28-0.5 B 0-0.0030 25 S up to max 0.010 Co 0-3.5 W 0-3.0% Cu 0-2.0 Ru 0-0.3 3 0 Al 0-0.03 Ca 0-0.010%, balance Fe together with inevitable contaminations.

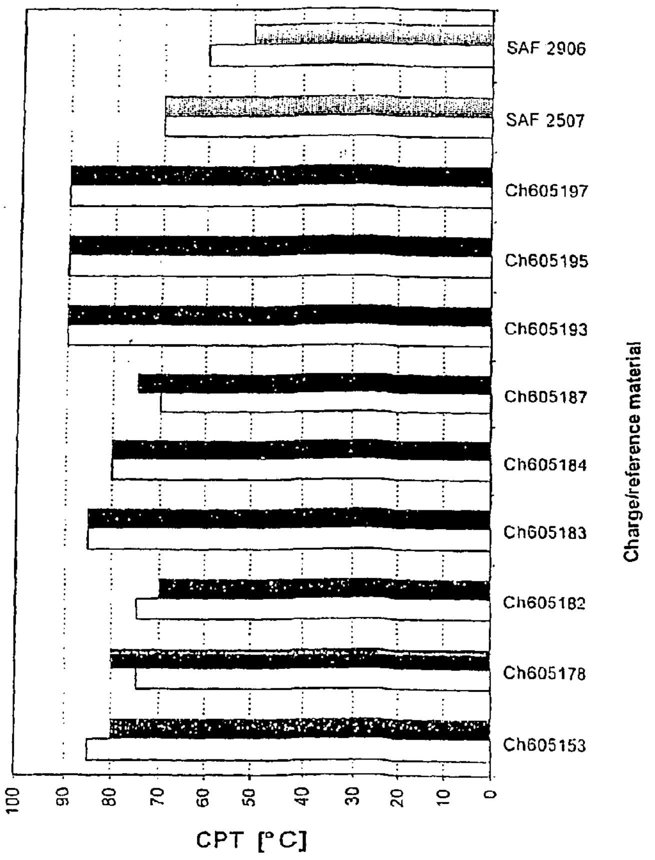


Figure 1

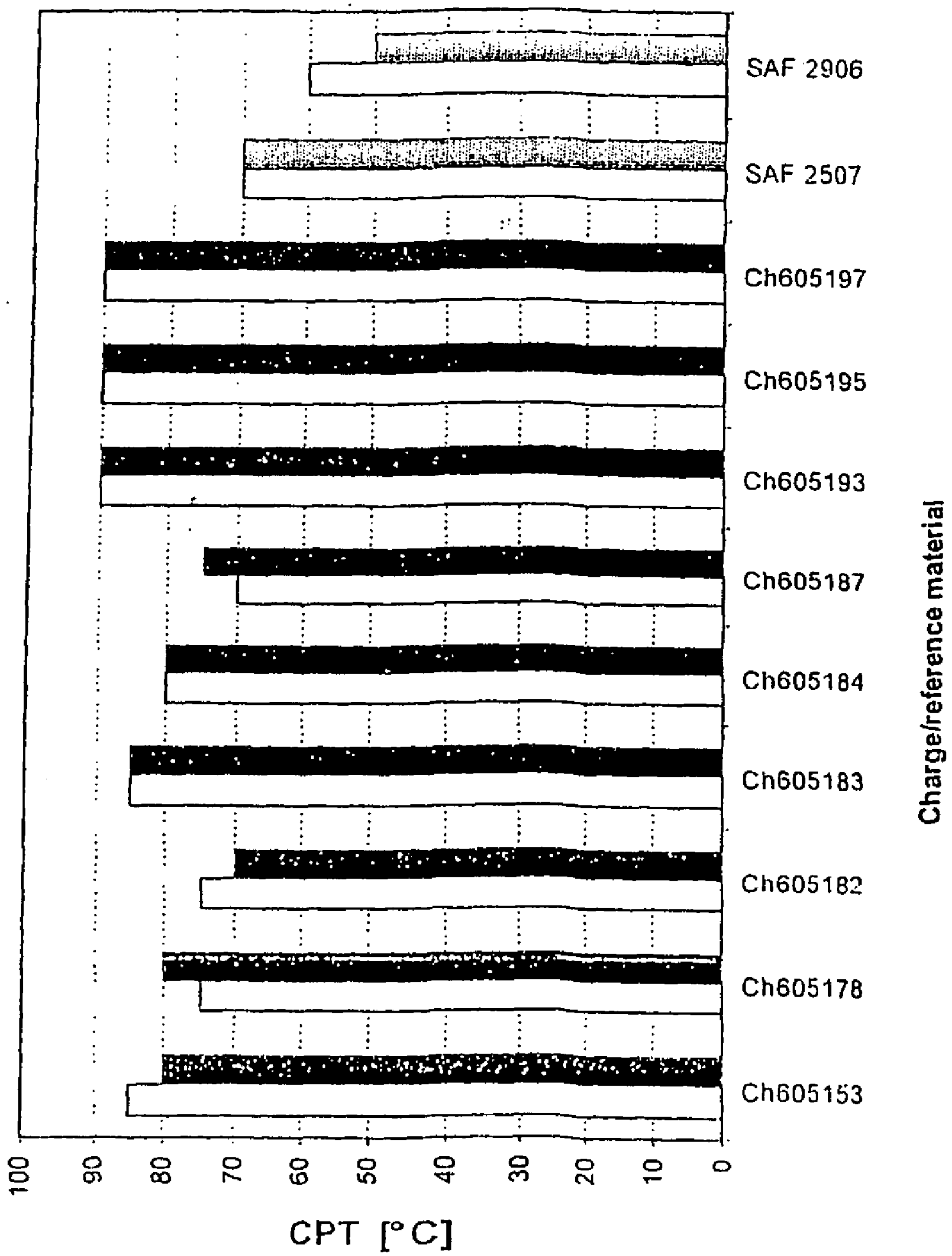

SAF 2906
SAF 2507
Ch605197
Ch605195
Ch605193
Ch605187
Ch605184
Ch605183
Ch605182
Ch605178
Ch605153
Charge/reference material
100
90
80
70
60
50
40
30
20
10
0
CPT [°C]

Figure 2

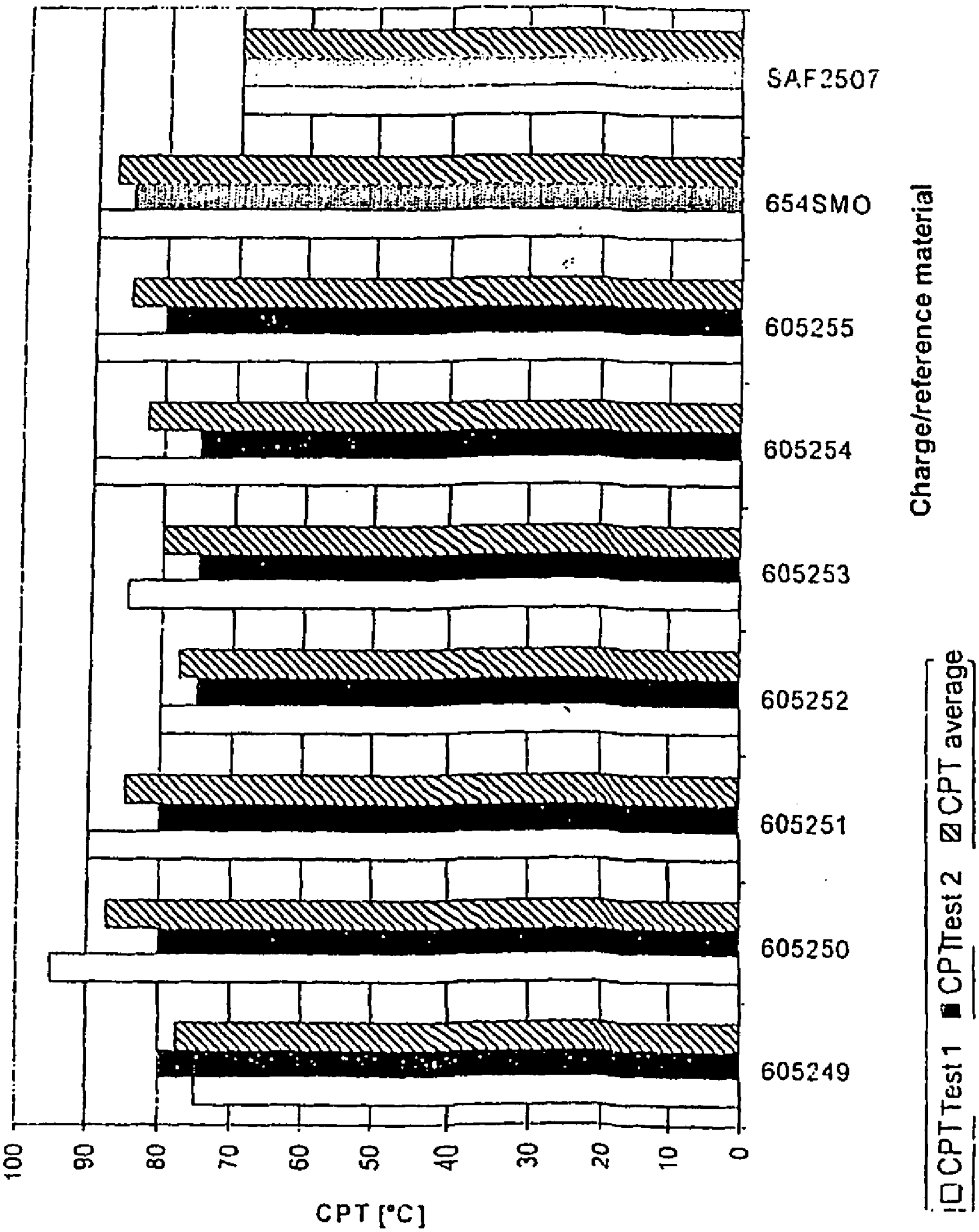
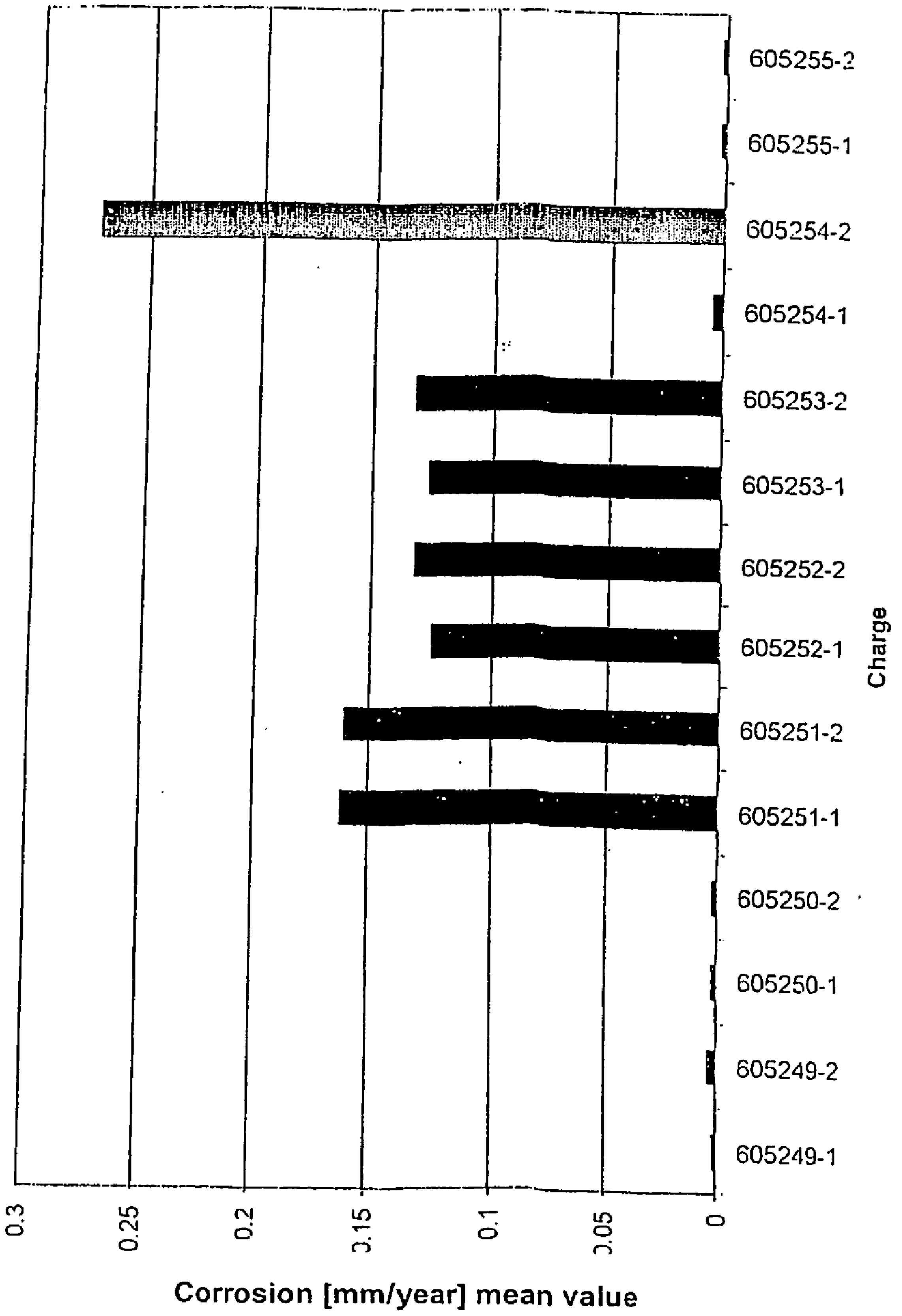

SAF2507
654SMO
605255
605254
605253
605252
605251
605250
605249
Charge/reference material
100
90
80
70
60
50
40
30
20
10
0
CPT [°C]
☐ CPT Test 1
■ CPT Test 2
▨ CPT average

Figure 3


605255-2
605255-1
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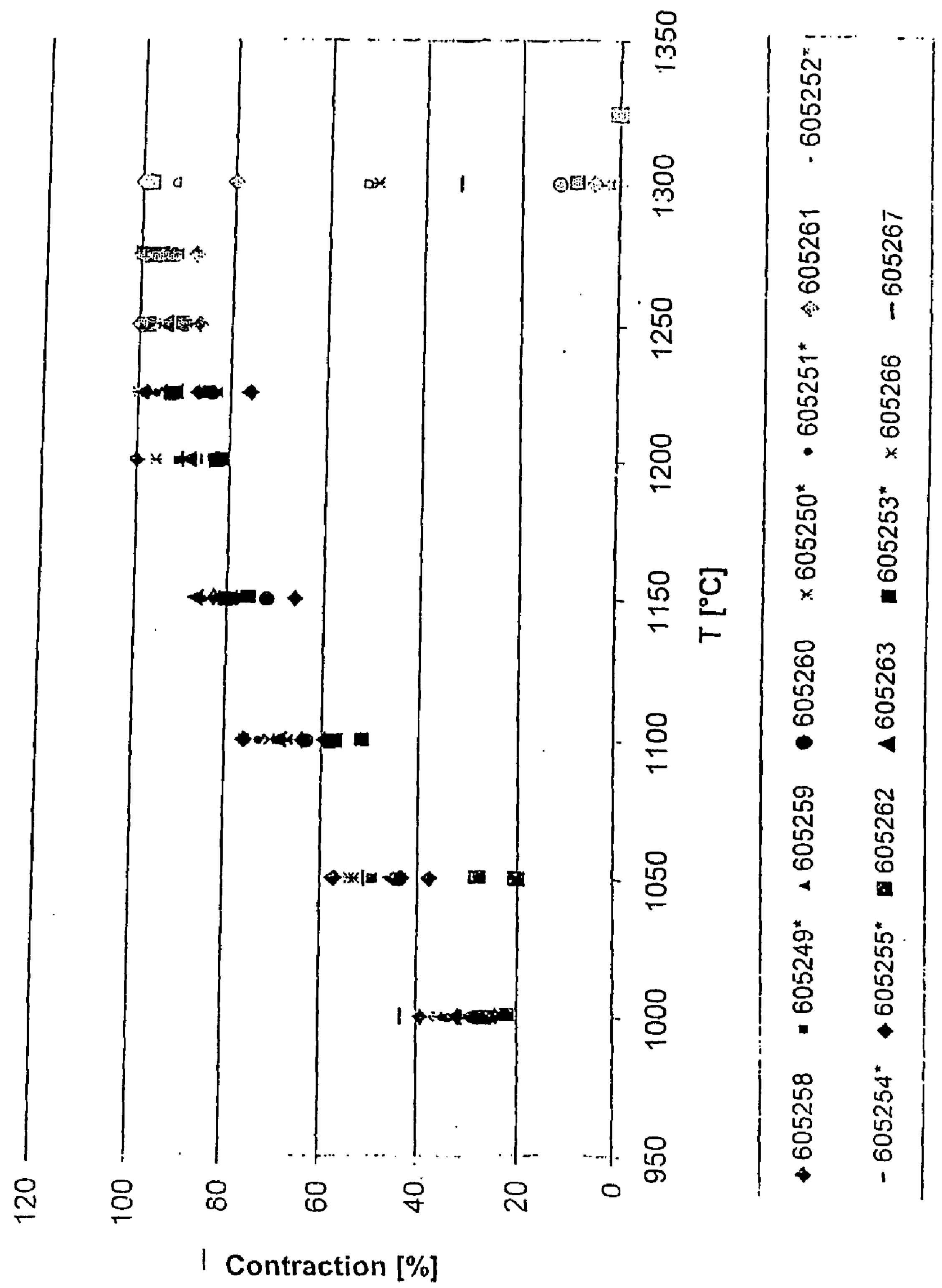
605253-2
605253-1
605252-2
605252-1
605251-2
605251-1
605250-2
605250-1
605249-2
605249-1
Charge
0.3
0.25
0.2
0.15
0.1
0.05
0
Corrosion [mm/year] mean value

Figure 4


Contraction [%]
0
20
40
60
80
100
120
T [°C]
950
1000
1050
1100
1150
1200
1250
1300
1350
605258
605249*
605259
605260
605250*
605251*
605261
605252*
605254*
605255*
605262
605263
605253*
605266
605267

DUPLEX STAINLESS STEEL ALLOY FOR USE IN SEAWATER APPLICATIONS

TECHNICAL FIELD OF THE INVENTION

[0001] The present invention relates to a stainless steel alloy, more precisely a duplex stainless steel alloy having ferritic-austenitic matrix and having high corrosion resistance in combination with good structural stability and hot workability, in particular a duplex stainless steel having a ferrite content of 40-65% by volume and a well-balanced composition that gives the material corrosion properties making it more suitable able for use in chloride-containing environments than what has previously been found possible.

BACKGROUND OF THE INVENTION

[0002] In oil production in the sea, holes are drilled down from the bottom of the sea to the oil deposit. At the bottom of the sea, a unit is installed for control of the flow of the crude oil and further transportation to the units that are to handle and refine the crude oil to useful products or semi-finished products. At the unit on the bottom of the sea there is, among other things, valves that control extraction, pressure, flow rate, etc., and couplings to pipes with possibility of injecting chemicals into the oil well. Frequently, methanol is used for injection with the object of avoiding that crude oil coagulates and causes undesired stops in the production pipes.

[0003] Valves and couplings on the unit at the bottom of the sea are controlled hydraulically and electrically from a platform, a production ship or another unit on the surface of the sea or on land. An umbilical cord pipe, a so-called umbilical, couples together the guiding unit with the units on the bottom of the sea. The part of the umbilical that lies on the bottom of the sea, for instance, between two underwater units on different extraction sites, is called static umbilical since the same only to a relatively small extent is effected by the motions of the sea. The part of the umbilical, that is situated between the bottom of the sea and the surface, is called dynamic umbilical and is effected to a large extent by motions in the water and on the surface. Examples of such motions are flows in the water, wave motions as well as motions of the platform and the production ship.

[0004] The demands that are made on the pipes in an umbilical are foremost related to corrosion and mechanical properties. The pipe material has to be resistant to corrosion in sea water, which surrounds the outer surface of the pipes. This property is what is regarded as being most important, since sea water has a very corrosive impact on stainless steel. Furthermore, the material has to have high corrosion resistance to the possible corrosive solutions that are injected in the oil well. The material has to be compatible with hydraulic liquids without contaminating the liquid. Possible contamination may affect the service function of the control unit at the bottom of the sea very negatively.

[0005] The mechanical properties of the used pipe material are very important for the application of umbilical pipes. Since the depth may be considerable on the site of the oil production, the dynamic part of the umbilical generally becomes long, and thereby heavy. The weight has to be carried by the platform or the floating production ship. In practice, there is two ways to decrease the weight of an umbilical having a given configuration. It is possible to choose a lighter material or a material having the same density but having higher tensile yield limit and ultimate tensile strength. By choosing a material having higher strength, pipes having thinner wall may be used, and thereby the total mass of the umbilical is reduced. The deeper the sea at the site of extraction, the more important the total weight per unit of length of umbilical of the material will be.

[0006] During the most recent years, when the environments in which corrosion-resistant metallic materials are used have become more heavy-duty, the requirements on the corrosion properties of the materials as well as on their mechanical properties have increased. Duplex steel alloys that were established as an alternative to hitherto used types of steel, such as, for instance, ferritic steel that previously were used in this application, nickel base alloys or other high-alloy steels, are not excepted from this development.

[0007] Furthermore, the latest development on the market for umbilical pipes implies additionally increased demands on the performance of the materials. The demands that hitherto have been made regarding strength and corrosion resistance have been able to be met by existing alloys. The new demands that are made on construction materials in the future for umbilicals mean, however, considerable exacting demands on corrosion resistance, by virtue of plants being projected in warmer waters as well as by virtue of process solutions in the umbilical will have higher temperatures. The new demands that are made may involve that the alloy must have resistance to crevice corrosion in sea water at temperatures up to 70-90° C. Today's construction materials do not meet these requirements with sufficient reliability against corrosion. It is this problem that has to be solved. However, hitherto all feasible alloys that have been evaluated, have had a weak point. An alloy of higher resistance to chloride-induced corrosion that also meets other demands such as increased strength and good structural stability, would, on the other hand, mean greater possibilities to meet the new demands made on umbilical pipes.

[0008] A recognized measure for the corrosion resistance in chloride-containing environments is the so-called Pitting Resistance Equivalent (abbreviated PRE), being defined as

$$PRE=\%\ Cr+3.3\%\ Mo+16\%\ N$$

where the percentage figures of each element refer to percentage by weight.

[0009] A higher numerical value indicates a better corrosion resistance, in particular to pitting. The principal alloying elements that affect this property are, according to the formula, Cr, Mo, N. An example of such a steel grade is seen in EP 0 220 141, which through this reference hereby is included in this description. This steel grade, having the trade mark of SAF 2507 (UNS S32750), has essentially been alloyed with high contents of Cr, Mo and N. Thus, it is developed towards this property with, above all, good corrosion resistance in chloride environments. Recently, also the elements Cu and W have turned out to be efficient alloying additives for additional optimization of the corrosion properties of the steel in chloride environments. The element W has, on that occasion, been used as substitution for a part of Mo, as for instance in the commercial alloys DP3W (UNS S39274) or Zeron100, which contain 2.0% and 0.7% of W, respectively. The latter also contains 0.7% of Cu with the purpose of increasing the alloy's corrosion resistance in acid environments.

[0010] Addition of tungsten led to a further development of the measure for the corrosion resistance, and thereby the PRE formula to the PREW formula, which also elucidates the relation between the impact of Mo and W on the corrosion resistance of the alloy:

$$\text{PREW}=\%\ \text{Cr}+3.3(\%\ \text{Mo}+0.5\%\ \text{W})+16\%\ \text{N},$$

as described, for instance, in EP 0 545 753, which relates to a duplex stainless alloy having generally improved corrosion properties.

[0011] The above-described steel grades have a PRE number, irrespective of method of calculation, which is above 40 but the PRE number is limited upwards to about 43 since higher values mean that the alloys obtain inferior structural stability. A higher degree of alloying increases the risk of precipitation of internietallic phase, and therefore the level of alloying in duplex steel is regarded as limited to achieve PRE values around a maximum of about 43, irrespective of method of calculation.

[0012] Of the alloys having good corrosion resistance in chloride environments, SAF 2906 should also be mentioned, the composition of which is seen in EP 0 708 845. This alloy, which is characterized by higher contents of Cr and N in comparison with, for instance, SAF 2507, has turned out to be especially suitable for use in environments where the resistance to intercrystalline corrosion and corrosion in ammonium carbamate is of importance, but it has also a high corrosion resistance in chloride-containing environments.

[0013] The alloy has a corrosion resistance in chloride environment corresponding to the alloy UNS S32750, but simultaneously a higher yield point in tension $Rp_{0.2}$. This makes that this alloy has advantages in comparison with UNS S32750 as umbilical material, since lower weight of the umbilical can be obtained. The corrosion resistance gives, however, no improvements in comparison with UNS S32750, which means considerable limitations in umbilical pipes that are exposed to higher temperatures in future plants.

[0014] The alloy 19D (UNS S32001) is a duplex alloy characterized by the composition 19.5-21.5% of Cr, 0.05-0.17% of N and max 0.6% of Mo. This alloy has a PRE number of about 22, and therefore the alloy is unsuitable in sea-water applications such as umbilicals. Accordingly, in order to achieve a sufficient corrosion resistance in this alloy, a cathode protection has to be applied in the form of a zinc layer on the outer surface of the umbilical pipe. If the zinc layer is consumed or if a greater surface becomes damaged, the corrosion protection is, however, ruined and a fast corrosion process may occur, which means expensive repairs and down periods.

[0015] A problem with the above-described alloys, all having high PRE numbers, is the appearance of hard and brittle intermetallic precipitations in the steel, such as, for instance, sigma phase, especially after heat treatment, such as, for instance, upon welding during later working. This results in a harder material having worse workability and finally a deteriorated corrosion resistance.

[0016] Another group of alloys having good corrosion resistance is austenitic steels, with PRE numbers of up to 55 having been made possible by the addition of high contents of Cr, Mo and NJ combined with high contents of Ni. Said alloys should work very well to the new tougher corrosion conditions in umbilicals. The disadvantage of the same alloys is that they have considerably lower yield point in tension than duplex steel and are, furthermore, considerably more expensive to manufacture, foremost by virtue of their high percentage of Ni, which is an expensive alloying material. Examples of austenites having good resistance in chloride environment are UNS S32654 having a PRE number of about 55, and UNS S34565 having a PRE number of about 45. These have, however, too low a strength and high a cost in order to be a realistic alternative for umbilical pipes.

[0017] In order to additionally improve, among other things, the pitting resistance of duplex stainless steel, an increase of the PRE number is required in both the ferrite phase and the austenite phase without, because of this, jeopardizing the structural stability or the workability of the material. If the composition in the two phases is not equivalent in respect of the active alloying components, one of the phases becomes more susceptible to pitting and crevice corrosion. Thus, the more corrosion-susceptible phase controls the resistance of the alloy, while the structural stability is controlled by the highest alloyed phase.

[0018] The demands that may be made on an alloy that shall meet the requirements in the future for umbilical pipes, can be summarized in table 1, with examples of the best various alternative alloys existing on the market in the present situation being included. It is clear that all existing alloys on at least one point do not meet the new stiffer demands that are made on umbilical pipes.

TABLE 1

Property	Demands = alloy according to the invention	UNS S32750	UNS S32906	UNS S32654	UNS S32001
PRE	Min 46	42.5	42	55	22
Yield point in tension $Rp_{0.2}$ (N/mm^2)	720	550	650	430	450
Pitting CPT in ° C.	>90° C.	50	50	>95	<20
Crevice corrosion CCT in ° C.	60° C.	35	35	60	<20
Structural stability	Max 0.5% sigma phase	OK	OK	OK	OK
Manufacture	Weldable by means of conventional technique	OK	OK	OK	OK

SUMMARY OF THE INVENTION

[0019] Therefore, it is an object of the present invention to provide a duplex stainless steel alloy, which has high corrosion resistance in combination with improved mechanical properties and simultaneously having good structural stability and that is most suitable for use in environments where a high resistance is required to general corrosion and local corrosion, such as, for instance, in chloride-containing environments.

[0020] It is an additional object of the present invention to provide a duplex stainless steel alloy having a Critical Pitting Corrosion Temperature (henceforth abbreviated CPT) value greater than 90° C., preferably greater than 95°

C. and a Critical Crevice Corrosion Temperature (henceforth abbreviated CCT) value of at least 60° C. in 6% $FeCl_3$.

[0021] It is an additional object of the present invention to provide an alloy having an impact resistance of at least 100 J at room temperature and a yield point in tension $Rp_{0.2}$ of at least 720 N/mm^2 and an elongation upon tensile testing of at least 25% at room temperature.

[0022] The material according to the present invention has, in view of the high alloy content thereof, extraordinarily good workability, in particular hot-workability, and should thereby be very suitable to be used for, for instance, the manufacture of bars, pipes, such as welded and seamless pipes, weld material, construction parts such as, for instance, flanges and couplings.

[0023] These objects are met according to the present invention by means of duplex stainless steel alloys, which contain (in % by weight)

C	more than 0 up to max 0.03%
Si	up to max 0.5%
Mn	0–3.0%
Cr	24.0–30.0%
Ni	4.9–10.0%
Mo	3.0–5.0%
N	0.28–0.5%
B	0–0.0030%
S	up to max 0.010%
Co	0–3.5%
W	0–3.0%
Cu	0–2.0%
Ru	0–0.3%
Al	0–0.03%
Ca	0–0.010%

balance Fe together with inevitable contaminations.

BRIEF DESCRIPTION OF THE FIGURES

[0024] FIG. **1** shows CPT values from test of the experimental charges in the modified ASTM G48C test in the “Green Death” solution in comparison with the duplex steels SAF 2507, SAF 2906.

[0025] FIG. **2** shows CPT values produced by means of the modified ASTM G48C test in “Green Death” solution for the experimental charges in comparison with the duplex steel SAF 2507 as well as SAF 2906.

[0026] FIG. **3** shows the mean value of the corrosion in mm/year in 2% HCl at the temperature of 75° C.

[0027] FIG. **4** shows the results from hot ductility test for most of the charges.

DETAILED DESCRIPTION OF THE INVENTION

[0028] A systematic development work has surprisingly shown that by a well balanced combination of the elements Cr, Mo, Ni, N, Mn and Co, an optimum distribution of the elements in the ferrite and in the austenite can be obtained, which enables a very corrosion resistant material having only a negligible quantity of sigma phase in the material. The material also gets good workability, which enables the extrusion to weldless pipes. In order to obtain the combination of high corrosion resistance in connection with good structural stability, a very narrow combination of the alloying elements in the material is required. Therefore, the alloy according to the invention contains (in % by weight):

C	more than 0 up to max 0.03%
Si	up to max 0.5%
Mn	0–3.0%
Cr	24.0–30.0%
Ni	4.9–10.0%
Mo	3.0–5.0%
N	0.28–0.5%
B	0–0.0030%
S	up to max 0.010%
Co	0–3.5%
W	0–3.0%
Cu	0–2.0%
Ru	0–0.3%
Al	0–0.03%
Ca	0–0.010%

balance Fe together with normally occurring contaminations and additives, the ferrite content being 40-65% by volume.

[0029] The impact of the alloying elements is described in the following:

[0030] Carbon (C) has limited solubility in both ferrite and austenite. The limited solubility means a risk of precipitation of chromium carbides and therefore the content should not exceed 0.03% by weight, preferably not exceed 0.02% by weight.

[0031] Silicon (Si) is utilized as deoxidizer in the steel production and increases the flowability in production and upon welding. However, too high contents of Si lead to precipitation of undesired intermetallic phase, and therefore the content should be limited to max 0.5% by weight, preferably max 0.3% by weight.

[0032] Manganese (Mn) is added in order to increase the solubility of N in the material. However, it has turned out that Mn only has a limited impact on the solubility of N in the alloy type in question. Instead, there are other elements having higher impact on the solubility. Furthermore, Mn may in combination with high sulphur contents give rise to the formation of manganese sulphides, which work as initiation spots for pitting. Therefore, the content of Mn should be limited to between 0-3.0% by weight, preferably 0.5-1.2% by weight.

[0033] Chromium (Cr) is a very active element in order to improve the resistance to the majority of corrosion types. Furthermore, a high chromium content means that a very good solubility of N is obtained in the material. Thus, it is desirable to hold the content of Cr as high as possible in order to improve the corrosion resistance. For very good values of the corrosion resistance, the chromium content should be at least 24.0% by weight, preferably 27.0-29.0% by weight. However, high contents of Cr increases the risk of intermetallic precipitations, and therefore the chromium content has to be limited upwards to max 30.0% by weight.

[0034] Nickel (Ni) is used as austenite-stabilizing element and is added in suitable contents so that the desired ferrite content is attained. In order to achieve the desired relation between the austenitic and the ferritic phase of between 40-65% by volume of ferrite, an addition of between 4.9-

10.0% by weight of nickel is required, preferably 4.9-9.0% by weight, in particular 6.0-9.0% by weight.

[0035] Molybdenum (Mo) is an active element that improves the corrosion resistance in chloride environments as well as preferably in reducing acids. Too high a content of Mo, in combination with the contents of Cr being high, means that the risk of intermetallic precipitations increases. The content of in the present invention should be in the interial of 3.0-5.0% by weight, preferably 3.6-4.9% by weight, in particular 4.4-4.9% by weight.

[0036] Nitrogen (N) is a very active element that increases the corrosion resistance, the structural stability as well as the strength of the material. Furthermore, a high content of N improves the reformation of austenite after welding, which gives good properties of welded joints. In order to achieve a good effect from N, at least 0.28% by weight of N should be added. At high contents of N, the risk of precipitation of chromium nitrides increases, especially when the chromium content simultaneously is high. Furthermore, a high content of N means that the risk of porosity increases by virtue of the solubility of N in the charge being exceeded. The content of N should, for these reasons, be limited to max 0.5% by weight, preferably is >0.35-0.45% by weight of N added.

[0037] Too high a chromium as well as a nitrogen content result in the precipitation of Cr_2N, which is to be avoided since it deteriorates the properties of the material, especially upon heat treatment, for instance welding.

[0038] Boron (B) is added in order to increase the hot workability of the material. At too high a boron content, the weldability and the corrosion resistance may be deteriorated. Therefore, the boron content should be greater than 0 and up to 0.0030% by weight.

[0039] Sulphur (S) affects the corrosion resistance negatively by forming easily soluble sulphides. Furthermore, the hot workability is deteriorated, and therefore the sulphur content is limited to max 0.010% by weight.

[0040] Cobalt (Co) is added foremost in order to improve the structural stability as well as the corrosion resistance. Co is an austenite stabilizer. In order to have an effect, at least 0.5% by weight, preferably at least 1.0% by weight should be added. Since cobalt is a relatively expensive element, the cobalt addition is therefore limited to max 3.5% by a weight.

[0041] Tungsten increases the resistance to pitting and crevice corrosion. But addition of too high contents of tungsten in combination with the contents of Cr and contents of Mo being high, means that the risk of intermetallic precipitations increases. The content of W in the present invention should be in the interval of 0-3.0% by weight, preferably between 0-1.8% by weight.

[0042] Copper is added in order to improve the corrosion resistance in acid environments such as sulphuric acid. Cu also affects the structural stability. However, high contents of Cu means that the solid solubility is exceeded. Therefore, the content of Cu is limited to max 2.0% by weight, preferably between 0.1 and 1.5% by weight.

[0043] Ruthenium (Ru) is added in order to increase the corrosion resistance. Ruthenium is a very expensive element, and therefore the content is limited to max 0.3% by weight, preferably greater than 0 and up to 0.1% by weight.

[0044] Aluminum (Al) as well as Calcium (Ca) are utilized as deoxidizers in the steel production. The content of Al should be limited to max 0.03% by weight in order to limit nitride formation. Ca has a favourable effect on the hot ductility but the content of Ca should, however, be limited to 0.010% by weight in order to avoid undesired quantity of cinder.

[0045] The ferrite content is important in order to obtain good mechanical properties and corrosion properties as well as good weldability. From a corrosion and a weldability point of view, it is desirable having a ferrite content of between 40-65% in order to obtain good properties. Furthermore, high ferrite contents means that the low-temperature impact resistance as well as the resistance to hydrogen embrittlement risk being deteriorated. Therefore, the ferrite content is 40-65% by volume, preferably 42-60% by volume, in particular 45-55% by volume.

DESCRIPTION OF PREFERRED EMBODIMENT EXAMPLES

[0046] In the examples below, the composition of a number of experimental charges is given, which illustrate the impact of various alloying elements on the properties. Charge 605182 represents a reference composition and is accordingly not included in the field of this invention. Neither should other charges be regarded as limiting the invention but only states examples of charges that illustrate the invention according to the claims.

[0047] Given PRE numbers or values always relate to values calculated according to the PREW formula, even if not explicitly stated.

EXAMPLE 1

[0048] Experimental charges according to this example were produced by laboratory casting of 170 kg of ingot that was hot-forged into round bar. The same was hot extruded into bar (round bar as well as flat bar), where test material was sampled from round bar. Furthermore, flat bar was annealed before cold rolling took place, and then additional test material was sampled. The process may, from a material technology point of view, be regarded as representative for the manufacture on a larger scale, for instance for the manufacture of seamless pipes by means of the extrusion method followed by cold rolling. Table 2 shows composition of experimental charges of the first batch.

TABLE 2

Charge	Mn	Cr	Ni	Mo	W	Co	V	La	Ti	N
605193	1.03	27.90	8.80	4.00	0.01	0.02	0.04	0.01	0.01	0.36
605195	0.97	27.90	9.80	4.00	0.01	0.97	0.55	0.01	0.35	0.48
605197	1.07	28.40	8.00	4.00	1.00	1.01	0.04	0.01	0.01	0.44
605178	0.91	27.94	7.26	4.01	0.99	0.10	0.07	0.01	0.03	0.44

TABLE 2-continued

Charge	Mn	Cr	Ni	Mo	W	Co	V	La	Ti	N
605183	1.02	28.71	6.49	4.03	0.01	1.00	0.04	0.01	0.04	0.28
605184	0.99	28.09	7.83	4.01	0.01	0.03	0.54	0.01	0.01	0.44
605187	2.94	27.74	4.93	3.98	0.01	0.98	0.06	0.01	0.01	0.44
605153	2.78	27.85	6.93	4.03	1.01	0.02	0.06	0.02	0.01	0.34
605182	0.17	23.48	7.88	5.75	0.01	0.05	0.04	0.01	0.10	0.26

[0049] With the purpose of examining the structural stability, samples from each charge were annealed at 900-1150° C. with steps of 50° C. and were quenched in air and water, respectively. At the lowest temperatures, intermetallic phase was formed. The lowest temperature where the amount of intermetallic phase became negligibly small, was determined by means of studies in light-optical microscope. New samples from the respective charge were then annealed at said temperature during five minutes, and then the samples were cooled down by the constant cooling rate of −140° C./min to room temperature. The area fraction of sigma phase in the materials was then determined by means of digital image processing of images recorded by mean of back-scattered electrons in scanning electron microscope. The results are seen in Table 3.

[0050] T_{max} sigma is calculated by means of Thermo-Calc (T-C version N the thermodynamic database of steel TCFE99) based on guiding values of all stated elements in the different variants. T_{max} sigma is the resolution temperature of the sigma phase, with high resolution temperature indicating lower structural stability.

TABLE 3

Charge	Heat treatment	Quantity of σ [% by vol.]	Tmax σ
605193	1100° C., 5 min	7.5%	1016
605195	1150° C., 5 min	32%	1047
605197	1100° C., 5 min	18%	1061
605178	1100° C., 5 min	14%	1038
605183	1050° C., 5 min	0.4%	997
605184	1100° C., 5 min	0.4%	999
605187	1050° C., 5 min	0.3%	962
605153	1100° C., 5 min	3.5%	1032
605182	1100° C., 5 min	2.0%	1028

[0051] The object of this investigation is to be able to rank materials in respect of the structural stability, i.e. this is not the actual content of sigma phase in the test pieces that have been heat treated and quenched before, for instance, corrosion test. It is evident that T_{max} sigma that has been calculated by means of Thermo-calc does not directly corresponds with measured quantity of sigma phase, but in this investigation it is, however, clear that the experimental charges having the lowest calculated T_{max} sigma contain the lowest quantity of sigma phase.

[0052] The pitting properties of all charges have been tested for ranking in the so-called "Green Dealth" solution that consists of 1% $FeCl_3$, 1% $CuCl_2$, 11% H_2SO_4, 12% HCl. The test procedure corresponds to the pitting testing according to ASTM G48C, but is carried out in the more aggressive "Green Death" solution. Furthermore, some charges have been tested according to ASTM G48C (2 experiments per charge). Also electrochemical testing in 3% NaCl (6 experiments per charge) has been carried out. The results in the form of critical pitting temperature (CPT) from all experiments are seen in Table 4, such as the PREW number (Cr+3.3(Mo+0.5W)+16 N) of the total alloy composition as well as of austenite and ferrite. The indexing alpha relates to ferrite and gamma relates to austenite.

TABLE 4

Charge	PRE α	PRE γ	PRE γ/PRE α	PRE	CPT ° C. Modified ASTM G48C Green Death	CPT ° C. ASTM G48C 6% $FeCl_3$	CPT ° C. 3% NaCl (600 mv SCE
605193	51.3	49.0	0.9552	46.9	90/90		64
605195	51.5	48.9	0.9495	48.7	90/90		95
605197	53.3	53.7	1.0075	50.3	90/90	>95	>95
605178	50.7	52.5	1.0355	49.8	75/80		94
605183	48.9	48.9	1.0000	46.5	85/85	90	93
605184	48.9	51.7	1.0573	48.3	80/80		72
605187	48.0	54.4	1.1333	48.0	70/75		77
605153	49.6	51.9	1.0464	48.3	80/85	85	90
605182	54.4	46.2	0.8493	46.6	75/70	85	62
SAF2507	39.4	42.4	1.0761	41.1	70/70	80	95
SAF2906	39.6	46.4	1.1717	41.0	60/50	75	75

[0053] It is recognized that there is a linear relation between the lowest PRE value in the austenite or the ferrite and the CPT value in duplex steels, but the results in Table 4 show that the PRE number not solely explains the CPT value.

[0054] It is clear from these results that all test materials have better CPT in the modified ASTM G48C than SAF 2507 and SAF 2906. Test charge 605 183 alloyed with cobalt shows good structural stability at controlled cooling rate (−140° C./min), in spite of it containing high contents of chromium as well as molybdenum, has better results than SAF 2507 as well as SAF 2906. In this investigation, it is seen that a high PRE not solely explains the CPT values, but the ratio of PRE austenite/PRE ferrite is of utmost importance for the properties of higher alloyed duplex steels, and a very narrow and accurate levelling between the alloying elements is required in order to obtain this optimal ratio, which is between 0.9-1.15; preferably 0.9-1.05 and simultaneously obtain PRE values above 46. The ratio of PRE austenite/PRE ferrite versus CPT in the modified ASTM G48C test for the experimental charges are accounted for in Table 4.

[0055] The strength at room temperature (RT), 100° C. and 200° C. and the impact resistance at room temperature (RT) have been determined for all charges and are shown as mean value of three experiments.

[0056] Tensile test pieces (DR5C50) were produced from extruded bars Ø20 mm, which were heat treated at temperatures according to Table 2 for 20 min followed by cooling down in either air or water (605 195, 605 197, 605 184). The results of the investigation are presented in Tables 5 and 6. The results of the tensile strength investigation show that the contents of chromium, nitrogen and tungsten strongly affect the tensile strength in the material. All charges except 605 153 meet the requirement on a 25% elongation upon tensile testing at room temperature (RT).

TABLE 5

Charge	Temperatur	$R_{p0.2}$ (MPa)	$R_{p0.1}$ (MPa)	R_m (MPa)	A5 (%)	Z (%)
605193	RT	652	791	916	29.7	38
	100° C.	513	646	818	30.4	36
	200° C.	511	583	756	29.8	36
605195	RT	671	773	910	38.0	66
	100° C.	563	637	825	39.3	68
	200° C.	504	563	769	38.1	64
605197	RT	701	799	939	38.4	66
	100° C.	564	652	844	40.7	69
	200° C.	502	577	802	35.0	65
605178	RT	712	828	925	27.0	37
	100° C.	596	677	829	31.9	45
	200° C.	535	608	763	27.1	36
605183	RT	677	775	882	32.4	67
	100° C.	560	642	788	33.0	59
	200° C.	499	578	737	29.9	52
605184	RT	702	793	915	32.5	60
	100° C.	569	657	821	34.5	61
	200° C.	526	581	774	31.6	56
605187	RT	679	777	893	35.7	61
	100° C.	513	628	799	38.9	64
	200° C.	505	558	743	35.8	58
605153	RT	715	845	917	20.7	24
	100° C.	572	692	817	29.3	27
	200° C.	532	611	749	23.7	31
605182	RT	627	754	903	28.4	43
	100° C.	493	621	802	31.8	42

[0057]

TABLE 6

Charge	Annealing [° C./min]	Cooling down	Impact resistance [J]	Annealing [° C./min]	Cooling down	Impact resistance [J]
605193	1100/20	Air	35	1100/20	Water	242
605195	1150/20	Water	223			
605197	1100/20	Water	254	1130/20	Water	259
605178	1100/20	Air	62	1100/20	Water	234
605183	1050/20	Air	79	1050/20	Water	244
605184	1100/20	Water	81	1100/20	Air	78
605187	1050/20	Air	51	1100/20	Water	95
605153	1100/20	Air	50	1100/20	Water	246
605182	1100/20	Air	22	1100/20	Water	324

[0058] This investigation shows very clearly that water quenching naturally is required in order to obtain the best structure and accordingly good impact resistance values. The requirement is 100 J upon testing at room temperature and this do all charges manage except charge 605 184 and 605 187, where, however, the last-mentioned one is very near the requirement.

[0059] Table 7 shows results from Tungsten Inert Gas remelting test (henceforth abbreviated TIG), with the charges 605 193, 605 183, 605 184 as well as 605 253 having a stable structure in the heat affected zone (henceforth abbreviated HAZ). The Ti-containing charges have TiN in HAZ.

TABLE 7

Charge	Precipitations Protective gas Ar (99.99%)
605193	HAZ: OK
605195	HAZ: Large amounts of TiN and a phase
605197	HAZ: Small amounts of Cr_2N in δ grains, however not much
605178	HAZ: Cr_2N in δ grains, otherwise OK
605183	HAZ: OK
605184	HAZ: OK
605187	HAZ: Cr_2N fairly close to the melting boundary, no precipitations farther out
605153	HAZ: OK
605182	HAZ: TiN as well as decorated grain boundaries δ/δ.

EXAMPLE 2

[0060] In the example below, the composition is given of an additional number of experimental charges manufactured with the intention of finding the optimal composition. Said charges are modified, based on the properties of the charges having good structural stability as well as high corrosion resistance, from the results that were shown in Example 1. All charges in Table 8 are comprised of the composition according to the present invention, with charges 1-8 being included in a statistical experimental plan, while charges e to n are additional experimental alloys within the scope of this invention.

[0061] A number of experimental charges were produced by casting a 270 kg casting, which was hot-forged into round bar. This was extruded to bar, from which test materials were sampled. Then the bar was annealed before cold rolling of flat bar took place and then additional test materials were sampled. Table 8 shows the composition of the same experimental charges.

TABLE 8

	Charge	Mn	Cr	Ni	Mo	W	Co	Cu	Ru	B	N
1	605258	1.1	29.0	6.5	4.23		1.5			0.0018	0.46
2	605249	1.0	28.8	7.0	4.23		1.5			0.0026	0.38
3	605259	1.1	29.0	6.8	4.23		0.6			0.0019	0.45
4	605260	1.1	27.5	5.9	4.22		1.5			0.0020	0.44
5	605250	1.1	28.8	7.6	4.24		0.6			0.0019	0.40
6	605251	1.0	28.1	6.5	4.24		1.5			0.0021	0.38
7	605261	1.0	27.8	6.1	4.22		0.6			0.0021	0.43
8	605252	1.1	28.4	6.9	4.23		0.5			0.0018	0.37
e	605254	1.1	26.9	6.5	4.8		1.0			0.0021	0.38
f	605255	1.0	28.6	6.5	4.0		3.0			0.0020	0.31
g	605262	2.7	27.6	6.9	3.9	1.0	1.0			0.0019	0.36
h	605263	1.0	28.7	6.6	4.0	1.0	1.0			0.0020	0.40
i	605253	1.0	28.8	7.0	4.16		1.5			0.0019	0.37
j	605266	1.1	30.0	7.1	4.02					0.0018	0.38
k	605269	1.0	28.5	7.0	3.97	1.0	1.0			0.0020	0.45
l	605268	1.1	28.2	6.6	4.0	1.0	1.0	1.0		0.0021	0.43
m	605270	1.0	28.8	7.0	4.2		1.5		0.1	0.0021	0.41
n	605267	1.1	29.3	6.5	4.23			1.5		0.0019	0.38

[0062] The distribution of alloying elements in the ferrite and austenite phase was examined by means of micro probe analysis, the result is seen in Table 9.

TABLE 9

Charge	Phase	Cr	Mn	Ni	Mo	W	Co	Cu	N
605258	Ferrit	29.8	1.3	4.8	5.0		1.4		0.11
	Austenit	28.3	1.4	7.3	3.4		1.5		0.60
605249	Ferrit	29.8	1.1	5.4	5.1		1.3		0.10
	Austenite	27.3	1.2	7.9	3.3		1.6		0.53
605259	Ferrite	29.7	1.3	5.3	5.3		0.5		0.10
	Austenite	28.1	1.4	7.8	3.3		0.58		0.59
605260	Ferrite	28.4	1.3	4.4	5.0		1.4		0.08
	Austenite	26.5	1.4	6.3	3.6		1.5		0.54
605250	Ferrite	30.1	1.3	5.6	5.1		0.46		0.07
	Austenite	27.3	1.4	8.8	3.4		0.53		0.52
605251	Ferrite	29.6	1.2	5.0	5.2		1.3		0.08
	Austenite	26.9	1.3	7.6	3.5		1.5		0.53
605261	Ferrite	28.0	1.2	4.5	4.9		0.45		0.07
	Austenite	26.5	1.4	6.9	3.3		0.56		0.56
605252	Ferrite	29.6	1.3	5.3	5.2		0.42		0.09
	Austenite	27.1	1.4	8.2	3.3		0.51		0.48
605254	Ferrite	28.1	1.3	4.9	5.8		0.89		0.08
	Austenite	26.0	1.4	7.6	3.8		1.0		0.48
605255	Ferrite	30.1	1.3	5.0	4.7		2.7		0.08
	Austenite	27.0	1.3	7.7	3.0		3.3		0.45
605262	Ferrite	28.8	3.0	5.3	4.8	1.4	0.9		0.08
	Austenite	26.3	3.2	8.1	3.0	0.85	1.1		0.46
605263	Ferrite	29.7	1.3	5.1	5.1	1.3	0.91		0.07
	Austenite	27.8	1.4	7.7	3.2	0.79	1.1		0.51
605253	Ferrite	30.2	1.3	5.4	5.0		1.3		0.09
	Austenite	27.5	1.4	8.4	3.1		1.5		0.48
605266	Ferrite	31.0	1.4	5.7	4.8				0.09
	Austenite	29.0	1.5	8.4	3.1				0.52
605269	Ferrite	28.7	1.3	5.2	5.1	1.4	0.9		0.11
	Austenite	26.6	1.4	7.8	3.2	0.87	1.1		0.52
605268	Ferrite	29.1	1.3	5.0	4.7	1.3	0.91	0.84	0.12
	Austenite	26.7	1.4	7.5	3.2	0.97	1.0	1.2	0.51
605270	Ferrite	30.2	1.2	5.3	5.0		1.3		0.11
	Austenite	27.7	1.3	8.0	3.2		1.4		0.47
605267	Ferrite	30.1	1.3	5.1	4.9			1.3	0.08
	Austenite	27.8	1.4	7.6	3.1			1.8	0.46

[0063] The pitting properties of all charges have been tested in the "Green Death" solution (1% $FeCl_3$, 1% $CuCl_2$, 11% H_2SO_4, 1.2% HCl) for ranking. The test procedure is the same as pitting testing according to ASTM G48C, but the testing is carried out in a more aggressive solution than 6% $FeCl_3$, the so-called "Green Death" solution. Also general corrosion test in 2% HCl (2 experiments per charge) has been carried out for ranking before dew point testing. The results from all experiments are seen in Table 10, FIG. **2** and FIG. **3**. All tested charges perform better than SAF 2507 in the Green Death solution. All charges are within the identified interval of 0.9-1.15; preferably 0.9-1.05 as regards the ratio PRE austenite/PRE ferrite at the same time as PRE in both austenite and ferrite is higher than 44 and for most of the charges also substantially higher than 44. Some of the charges even reach the limit total PRE 50. It is very interesting to note that charge 605 251, alloyed with 1.5% by weight of cobalt, performs almost equivalent to charge 605 250, alloyed with 0.6% by weight of cobalt, in "Green Death" solution, in spite of the lower chromium content in charge 605 251. It is particularly surprisingly and interesting when charge 605 251 has a PRE number of approx. 48, which is higher than any commercial super duplex alloy today at the same time as the T_{max} sigma value below 1010° C. indicates a good structural stability based on the values in Table 2 in example 1.

[0064] In Table 10, also the PREW number (% Cr+3.3%(Mo+0.5% W)+16% N) is given for the total alloy composition and PRE in austenite as well as ferrite (rounded) based on phase composition being measured by means of micro probe. The ferrite content is measured after heat treatment at 1100° C. followed by water quenching.

TABLE 10

Charge	α content	PREW Total	PRE α	PRE γ	PREγ/ PREα	CPT ° C. the Green Death
605258	48.2	50.3	48.1	49.1	1.021	65/70
605249	59.8	48.9	48.3	46.6	0.967	75/80
605259	49.2	50.2	48.8	48.4	0.991	75/75
605260	53.4	48.5	46.1	47.0	1.019	75/80
605250	53.6	49.2	48.1	46.8	0.974	95/80
605251	54.2	48.2	48.1	46.9	0.976	90/80
605261	50.8	48.6	45.2	46.3	1.024	80/70
605252	56.6	48.2	48.2	45.6	0.946	80/75
605254	53.2	48.8	48.5	46.2	0.953	90/75
605255	57.4	46.9	46.9	44.1	0.940	90/80
605262	57.2	47.9	48.3	45.0	0.931	70/85
605263	53.6	49.7	49.8	47.8	0.959	80/75
605253	52.6	48.4	48.2	45.4	0.942	85/75
605266	62.6	49.4	48.3	47.6	0.986	70/65
605269	52.8	50.5	49.6	46.9	0.945	80/90
605268	52.0	49.9	48.7	47.0	0.965	85/75
605270	57.0	49.2	48.5	45.7	0.944	80/85
605267	59.8	49.3	47.6	45.4	0.953	60/65

TABLE 10-continued

Charge	CPT Average	CCT Average	RP0.12 RT	Rm RT	A RT	Z RT
605258	84	68	725	929	40	73
605249	74	78	706	922	38	74
605259	90	85	722	928	39	73
605260	93	70	709	917	40	73
605250	89	83	698	923	38	75
605251	95	65	700	909	37	74
605261	93	78	718	918	40	73
605252	87	70	704	909	38	74
605254	93	80	695	909	39	73
605255	84	65	698	896	37	74
605262	80	83	721	919	36	75
605263	83	75	731	924	37	73
605253	96	75	707	908	38	73
605266	63	78	742	916	34	71
605269	95	90	732	932	39	73
605268	75	85	708	926	38	73
605270	95	80	711	916	38	74
605267	58	73	759	943	34	71

[0065] In order to more closely examine the structural stability, the samples were annealed for 20 min at 1080° C., 1100° C. and 1150° C., and then they were quenched in water.

[0066] The temperature where the amount of intermetallic phase became negligibly small was determined by means of investigations in light-optical microscope. A comparison of the structure of the charges after annealing at 1080° C. followed by water quenching indicates which of the charges that are more inclined to contain undesired sigma phase. The results are seen in Table 11. Structural control shows that the charges 605 249, 605 251, 605 252, 605 253, 605 254, 605 255, 605 259, 605 260, 605 266 as well as 605 267 are free from undesired sigma phase. Furthermore, charge 605 249, alloyed with 1.5% by weight of cobalt, is free from sigma phase, while charge 605 250, alloyed with 0.6% by weight of cobalt, contains a little sigma phase. Both charges are alloyed with high percentage of chromium, almost 29.0% by weight, as well as molybdenum content of almost 4.25% by weight. When the compositions of the charges 605 249, 605 250, 605 251 and 605 252 are compared considering the sigma phase content, it is very clear that the composition interval for the optimal material in respect of, in this case, structural stability, is very narrow. Furthermore, it is evident that charge 605 268 contains only occasional sigma phase in comparison with charge 605 263, which contains much sigma phase. What essentially separates these charges, is addition of copper to charge 605 268. In charge 605 266 as well as 605 267, the sigma phase is free in spite of high chromium content, the later charge is alloyed with copper. Furthermore, the charges 605 262 and 605 263, having the addition of 1.0% by weight of tungsten, have a structure with much sigma phase, while it is interesting to note that charge 605 269, also having 1.0% by weight of tungsten but of a higher nitrogen content than 605 262 and 605 263, has a considerably smaller quantity of sigma phase. Thus, a very well-adjusted balance between the various alloying elements is required at these high alloy contents for, e.g., chromium and molybdenum, in order to obtain good structural properties.

[0067] Table 12 shows the results from the light optical investigation after annealing at 1080° C., 20 min, followed by water quenching. The amount of sigma phase is indicated by means of values from 1 to 5, with 1 representing that no sigma phase has been detected upon the investigation, while 5 representing that a very high percentage of sigma phase has been detected upon the investigation.

TABLE 12

Charge	Sigma phase	Cr	Mo	W	Co	Cu	N	Ru
605249	1	28.8	4.23		1.5		0.38	
605250	2	28.8	4.24		0.6		0.40	
605251	1	28.1	4.24		1.5		0.38	
605252	1	28.4	4.23		0.5		0.37	
605253	1	28.8	4.16		1.5		0.37	
605254	1	26.9	4.80		1.0		0.38	
605255	1	28.6	4.04		3.0		0.31	
605258	2	29.0	4.23		1.5		0.46	
605259	1	29.0	4.23		0.6		0.45	
605260	1	27.5	4.22		1.5		0.44	
605261	2	27.8	4.22		0.6		0.43	
605262	4	27.6	3.93	1.0	1.0		0.36	
605263	5	28.7	3.96	1.0	1.0		0.40	
605266	1	30.0	4.02				0.38	
605267	1	29.3	4.23			1.5	0.38	
605268	2	28.2	3.98	1.0	1.0	1.0	0.43	
605269	3	28.5	3.97	1.0	1.0		0.45	
605270	3	28.8	4.19		1.5		0.41	0.1

[0068] Table 13, results are shown from impact resistance testing of some of the charges. The results are very good, which indicates a fine structure after annealing at 1100° C. followed by water quenching and the requirement of 100 J is met by a large margin by all tested charges.

TABLE 13

Charge	Annealing [° C./min]	Cooling down	Impact resistance [J]	Impact resistance [J]	Impact resistance [J]
605249	1100/20	Water	>300	>300	>300
605250	1100/20	Water	>300	>300	>300
605251	1100/20	Water	>300	>300	>300
605252	1100/20	Water	>300	>300	>300
605253	1100/20	Water	258	267	257
605254	1100/20	Water	>300	>300	>300
605255	1100/20	Water	>300	>300	>300

[0069] FIG. **4** shows the results from hot ductility test of most of the charges. A good workability is naturally crucial in order to be able to manufacture the material into product shapes such as bars, pipes, such as welded and seamless pipes, thread, weld material, construction parts such as, for instance, flanges and couplings. The charges 605 249, 605 250, 605 251, 605 252, 605 255, 605 266 as well as 605 267, most having a nitrogen content of around 0.38% by weight, have somewhat better hot ductility values.

[0070] The strain controlled fatigue properties give information about how much, and how many times, a material may be elongated, before strain controlled fatigue cracks arise in the material. Since umbilical pipes are welded together into long lengths, are reeled on drums before the are twisted into the umbilical, it is not unusual that a number of operations occurs where certain plastic deformation arises before the umbilical starts function. The strain controlled fatigue data that has been established emphasize that the risk of rupture as a consequence of strain controlled fatigue in an umbilical pipe borders on zero.

SUMMARY

[0071] The demands that are made on umbilical pipes in the future and that are met by an optimised alloy according to above, is that PRE of min 46 in the alloy combined with the fact that PRE in austenite or ferrite exceeds 45 is required in order to obtain sufficiently good pitting and crevice corrosion properties. Thus, it is required that:

CPT in 6% $FeCl_3$>90° C.

CCT in 6% $FeCl_3$>60° C.

[0072] The strength that is required for being able to substantially reduce the weight of an umbilical is:

Yield point in tension $Rp_{0.2}$ min 720 N/mm^2

[0073] In order to be able to manufacture umbilical pipes and in order to guarantee that pitting and crevice corrosion resistance as well as mechanical properties being preserved, the following is required regarding the structural stability:

[0074] The alloy shall be weldable by means of conventional welding methods

[0075] Maximally 0.5% sigma phase in the structure

[0076] Maximum resolution temperature of sigma phase is 1010° C.

[0077] The material according to the present invention has, in view of the high alloys content thereof, extraordinarily good workability, in particular hot-workability, and should thereby be very suitable to be used for, for instance, the manufacture of bars, pipes, such as welded and weldless pipes, weld material, construction parts, such as, for instance, flanges and couplings.

1. Ferrite-austenitic duplex stainless steel alloy having the following composition (in % by weight):

C	more than 0 and up to max 0.03%
Si	up to max 0.5%
Mn	0–3.0%
Cr	24.0–30.0%
Ni	4.9–10.0%
Mo	3.0–5.0%
N	0.28–0.5%
B	0–0.0030%
S	up to max 0.010%
Co	0–3.5%
W	0–3.0%
Cu	0–2.0%
Ru	0–0.3%
Al	0–0.03%
Ca	0–0.010%

as well as balance Fe together with normally occurring contaminations and additives, the ferrite content being 40-65% by volume and the relation PRE=% Cr+3.3% Mo+16% N exceeding 46 for the total composition of the alloy, as well as that PRE in austenite and ferrite phase exceeds 45 as well as that the yield point in tension $Rp_{0.2}$ of the alloy exceeds 720 N/mm^2, as well as that CPT>90° C. as well as CCT≧60° C.

2. Alloy according to claim 1, wherein the chromium content is between 26.5 and 29.0% by weight.

3. Alloy according to claim 1, wherein the manganese content is between 0.5 and 1.2% by weight.

4. Alloy according to claim 1, wherein the nickel content is between 5.0 and 8.0% by weight.

5. Alloy according to claim 1, wherein the molybdenum content is between 3.6% and 4.9% by weight.

6. Alloy according to claim 1, wherein the nitrogen content is between 0.35 and 0.45% by weight.

7. Alloy according to claim 1, wherein the ruthenium content is between 0 and 0.3% by weight.

8. Alloy according to claim 1, wherein the cobalt content is between 0.5 and 3.5% by weight.

9. Alloy according to claim 1, wherein the copper content is between b **0.5** and 2.0% by weight.

10. Alloy according to claim 1, wherein the ferrite content is between 42 and 60% by volume.

11. Alloy according to claim 1, wherein the total PRE or PREW value of the alloy exceeds 46, wherein PRE=% Cr+3.3% Mo+16 N and PREW=% Cr+3.3 (% Mo+0.5% W)+16 N, wherein % relates to % by weight.

12. Alloy according to claim 11, wherein the PRE or PREW value of both the ferrite and austenite phase is greater than 45 and the PRE or PREW value of the total alloy composition is greater than 46.

13. Use of an alloy according to claim 1 as umbilical cord pipe in chloride-containing environments.

14. Use of an alloy according to claim 1 for the manufacture of bars, pipes, weld material, and construction parts.

15. Alloy according to claim 7, wherein the ruthenium content is greater than 0 and up to 0.1% by weight.

16. Alloy according to claim 8, wherein the cobalt content is between 1.0 and 3.0% by weight.

17. Alloy according to claim 9, wherein the copper content is between 1.0 and 1.5% by weight.

18. Alloy according to claim 10, wherein the ferrite content is between 45 and 55% by volume.

19. Use of an alloy according to claim 13, wherein said chloride-containing environments comprise sea-water environments.

20. Use of an alloy according to claim 14, wherein said pipes comprise welded and weldless pipes, and said construction parts comprise flanges and couplings.

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