Corrosion Behavior of Corrosion Resistant Alloys in Stimulation Acids

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ABSTRACT

In the oil & gas industry, selection of CRAs for downhole tubulars is generally based on resistance to corrosive species in the production environment containing CO_2, H_2S, chloride and in some case elemental sulphur. However, there are non-production environments to which these materials must also be resistant for either short term or prolonged duration; these environments include stimulation acids, brine and completion fluids. This paper reports the main results of a laboratory study performed to evaluate the corrosion and stress corrosion behaviour to the acidizing treatments of the most used CRAs for production tubing and casing.

Laboratory tests were performed to simulate both ‘active’ and ‘spent’ acids operative phases, selecting various environmental conditions.
The selected steel pipes were a low alloyed steel, martensitic, supermartensitic, duplex 22 Cr, superduplex 25 Cr and superaustenitic stainless steels (25 Cr 35 Ni).
Results obtained in the ‘active’ acid environments over the temperature range of 100-140°C, showed that the blend acids with HCl at high concentration and HCl +HF represented too much severe conditions, where preventing high general corrosion and heavy localised corrosion by inhibition package becomes very difficult, especially for duplex steel pipe, where, in some case, the specimens were completely dissolved into the solution. On the contrary, all steels pipes were successfully protected by inhibitor when organic acid solution ($HCOOH + CH_3COOH$) were used.
Furthermore, different effectiveness on corrosion protection was showed by the tested inhibitors packages: e.g. in the 90% HCl at 12% + 10 CH_3COOH acid blend.

In ‘spent’ acid environments, all steel pipes showed to be less susceptible to the localised and general corrosion attack. Moreover, no Sulphide Stress Corrosion Cracking (SSC) was observed. Only one superaustenitic stainless steel U-bend specimen showed some superficial cracks at the highest test temperature (140°C), which may have been environmentally-induced.
A sensible increment of general corrosion was observed in the test with spent mud acid when the pH was reduced from 5 to 4 and 3.5 respectively.

Keywords: Acidizing, corrosion inhibitor, HCl, HF, spent acid, corrosion resistant alloy CRA.

I. INTRODUCTION

Acidizing is frequently used as a method to stimulate the production of oil at the onset of production, during the well life or in subsequent well workover. By dissolving acid soluble components within underground rock formations, or removing material at the wellbore face, the rate of flow of oil or gas out of production wells or the rate of flow of oil-displacing fluids into injection wells may be increased.

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Acid treatments are characterised by two different steps: the first step (active acids) consists in the introduction of the acid formulation (organic and/or inorganic acids with inhibitors and additives) to stimulate the well. The end of this step occurs when the acid formulation reacts completely with the rocks, producing the spent acid. Normally, it occurs in about six hours; the second step (spent acids) represents the period (a few days) permanence of spent acid into the well.

When downhole acidising treatments are applied through stainless steel tubing, the combination of high temperature with the aggressiveness of the acid blends could be the cause of the breakdown of the passive layer. While solutions based on organic acids such as acetic and formic are relatively mild, formulations with 15 -28% hydrochloric acid (HCl) or ‘mud acids ’ (e.g. HCl-HF solutions) are particularly aggressive even in presence of corrosion inhibitors. In addition, the spent acid return can last for 7 days, causing severe localised corrosion to most of CRA tubular [1][2].

This paper presents the results of an extensive laboratory work aimed to evaluate the corrosion and stress corrosion behaviour of the most common steel pipes used for tubing and casing to the acidising treatments.
Laboratory tests were developed to simulate both active and spent acids operative phases.

T95, 13Cr, 13Cr-5Ni-2Mo, duplex 22Cr, superduplex 25Cr, superaustenitic 25-35 (110 and 125 ksi) were exposed to different fresh and spent acid blends for periods up to 7 days at three test temperature: 100, 120 and 140°C. Furthermore, two distinct types of corrosion inhibitors were utilised to protect the metal during the acid treatment.

2. EXPERIMENTAL

2.1 Materials

The steel pipes, chemical compositions and mechanical data are given in table I.
The specimens, utilised for pitting and general corrosion assessment were flat coupons (size 40X30X3). U-bend specimens (size 100X10X2) were utilised to investigate the stress corrosion susceptibility of the steels. Duplicate flat coupons and U-bend specimens for each steel pipe were used in all autoclave exposures.

Steel pipe	Lab. Mark	C %	Si %	Mn %	Ni %	Cr %	Mo %	W (%)	YS (MPa)	UTS (MPa)	El. %
T95	T95	0.23	0.28	0.5	0.1	0.9	0.6	-	747	851	30
13 Cr L80	13Cr	0.22	0.35	0.6	-	12.4	-	-	620	750	20
Super 13 Cr	S13Cr	0.02	0.22	0.4	5.9	12.1	1.9	-	745	840	21
Duplex 22 Cr	D	0.03	1.0	2.0	4.5	21.0	2.5	-	879	938	17
Superduplex 25 Cr	SD	0.03	0.3	0.5	6.73	24.9	3.1	2.1	949	998	22
Superaustenitic 25 35-110	SA-110	0.02	0.3	0.6	31.4	24.8	3.2	-	827	889	19
Superaustenitic 25 35-125	SA-125	0.02	0.31	0.59	31.5	25.8	3.19	-	986	1048	18

TABLE I - Chemical composition (wt %) and mechanical properties of the tested materials. Data obtained from the producers.

2.2 Test condition: active acid enviroments

The environmental conditions used for the autoclave testing, in term of temperatures were 100, 120 and 140 °C and the pressure was 30 bar of nitrogen.
The acid blend solutions are listed in table II.

Acid blend solution	Concentration
HCl	15 wt %
HCl	28 wt %
HCl +HF	12 wt % + 3wt %
HCl (wt 12%)+CH_3COOH	90 vol- %+ 10 vol. %
CH_3COOH +COOH	13 wt %+ 9 wt %

TABLE II - Acid blends used for the autoclave testing.

Two different kind of inhibitors, named type A and B were utilised. Composition and concentration of each inhibition system, added to the acid blend, were suggested by the service companies depending on experimental conditions (temperature, pressure, acid blend and type of steel). Inhibitors A and B are constituted by the following chemical species:

Inhibitor A:
- Inhibitor: propalgilic alcohol, dissolved in methanol
- Intensifier: potassium iodide and/or formic acid + acetic acid

Inhibitor B:
- Inhibitor: aromatic ketones+ aromatic amine-ketones
- Intensifier: Ammonium chloride and formic acid

The content of inhibitor A varied from 1 to 2% (vol./vol.), while for the inhibitor B from 0.5 to1.8 % (vol./vol.).

Specimens were degreased by acetone in ultrasonic bath, before initial weighting.
Specimens were positioned into Pyrex beaker with a holder, in order to maintain each of them separated. In the case of HCl+HF acid blend a Teflon beaker was utilised, in order to avoid reaction between HF and the container. The ratio specimens area Vs volume of solution was 6 mm/cm^2 .
Autoclave was previously filled with 1.5 litres of diathermic oil, for the transmission of heat between the autoclave and the beaker. The autoclave was filled of with pure nitrogen (99.96 %) at the pressure of 30 bar.
Heating of the autoclave until the required temperatures were reached (100, 120 and 140 °C). Such temperatures were obtained in around 10-20 minutes. The test temperatures were maintaining for 6 hours.
Specimens were cleaned with distilled water by gently brushing and degreasing by acetone in ultrasonic bath. Final weighting of the specimens for corrosion rate determination by weight loss method. Examination of the specimens by stereo-microscope, for identification of the various corrosion forms was performed at 50 X max..

2.3 Test conditions: spent acid enviroments

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Tables III and IV show the conditions used in spent acid phase for the autoclave testing. Testing exposure period was one week (168 hours).

Steel sample	Testing temperatures (°C)	CO_2 partial pressure (Bar)	H_2S partial pressure (Bar)	N_2 partial pressure (Bar)	Total pressure (Bar)
T95	100, 120, 140	13.8	0.07	16.1	30
13Cr	100, 120,140	13.8	0.1	16.1	30
S13Cr	100, 120,140	13.8	0.1	16.1	30
D	100,120, 140	13.8	0.02	16.2	30
SD	100,120, 140	13.8	0.2	16.0	30
SA-125	100, 120, 140	13.8	10	6.2	30

TABLE III- Temperatures and gas pressure used for the autoclave testing in spent acid.

Acid blend solution	Concentration
HCl	28 wt %
HCl +HF	12 wt % + 3wt %

TABLE IV - Acid blends used for the autoclave testing.

The acid blend solutions of table IV were spent utilising the following procedures:

1. 28%HCl solution:
   a. 430 g $CaCO_3$ were added to 1 l of HCl. The salt was added progressively in order to avoid an uncontrolled production of CO_2.
   b. After the addition of $CaCO_3$, the solution was maintained under stirring for 1 hour to eliminate as much as possible the presence of CO_2, which does not permit to measure a stable solution pH.
   c. The solution was cooled until reaching 23 °C. A solution pH of around 5.0 was obtained.
2. 12% HCl +3% HF solutions: three different spent acid solutions were prepared starting from 12%HCl and 3% HF acid blend: the first had a pH around 5, the second 4 and the third 3.5. A suitable amount of NaOH was added to the acid blend solution to obtain the above solutions, until reaching the established pH.

2.4 Acceptance criterion

Definition of an acceptance criterion was mutually defined on the based literature data [3-5] and the information taken directly from the field production.
In the case of active acids phase, the limit of acceptance for the general corrosion was fixed at a corrosion rate ≤ 45 mm/y (0.05 lb/ft in six hours), while for spend acids phase it was 0.5 mm/y.
Pitting corrosion acceptance criteria was the same for both phases: no pits on the surface of the exposed specimens, having a depth more than 5 μm (0.005 mm). On this regards a ranking for the pitting sizes was arranged and it is reported in table VI. Adopting such ranking, class 2 is the acceptance limit for pitting corrosion.

Ranking	Number of the pits	Diameter of the pits (mm)	Depth of the pits (mm)
0	0	0	0
1	< 25	0.005	0.005
2	> 25	0.005	0.005
3	0 - ∞	from 0.005 to 0.2	from 0.005 to 0.1
4	0 - ∞	from 0.2 to 3.2	from 0.1 to 3.0

TABLE V - Ranking classes as a function of various pit sizes.

3. RESULTS

3.1. Active acid environment

General corrosion

Results are shown in Figures 1 and 2. As a general observation, the inorganic blends 28%HCl and mud acid proved to be the most aggressive environments.
T95 steel was effectively protected by inhibitor A up to 140°C in all the tested conditions; whilst T95 steel with inhibitor B showed proneness to general corrosion at 140°C, except when organic blends were considered.

Both tested inhibitors packages A and B were able to protect 13%Cr steel towards acidic attack in 15%Cl, 90%HCl 12% +10% CH_3COOH and 13% CH_3COOH + 9% HCOOH solutions. Inhibitor A was effective even at 140°C when mud acid was utilised.

S13Cr steel showed satisfactory performances in inorganic acid, whilst in organic acid the corrosion rate was higher than the threshold limit at 140°C with inhibitor A.

D and SD steels performed poorly at 100 and 120°C when protected with inhibitor A, whatever inorganic acidic blend was adopted. Furthermore, selective ferrite attack was observed on duplex and superduplex steels coupons. Excellent protection was offered by inhibitor B at all the temperatures.

Superaustenitic steel presented excellent corrosion resistance in all acid solutions.

Pitting corrosion

Pitting corrosion evaluation is reported in Figures 3 and 4. Pitting corrosion was observed at 140 °C in almost all inorganic acid blends with some exceptions: e.g. SA in HCl solutions.
Deep pit attacks were found on duplex and superduplex stainless steels when protected by inhibitor ‘A’. On the contrary, using inhibitor ‘B’ these steels were fully protected up to 120°C except for mud acid solution.
When organic acids were tested, always satisfactory results were obtained.

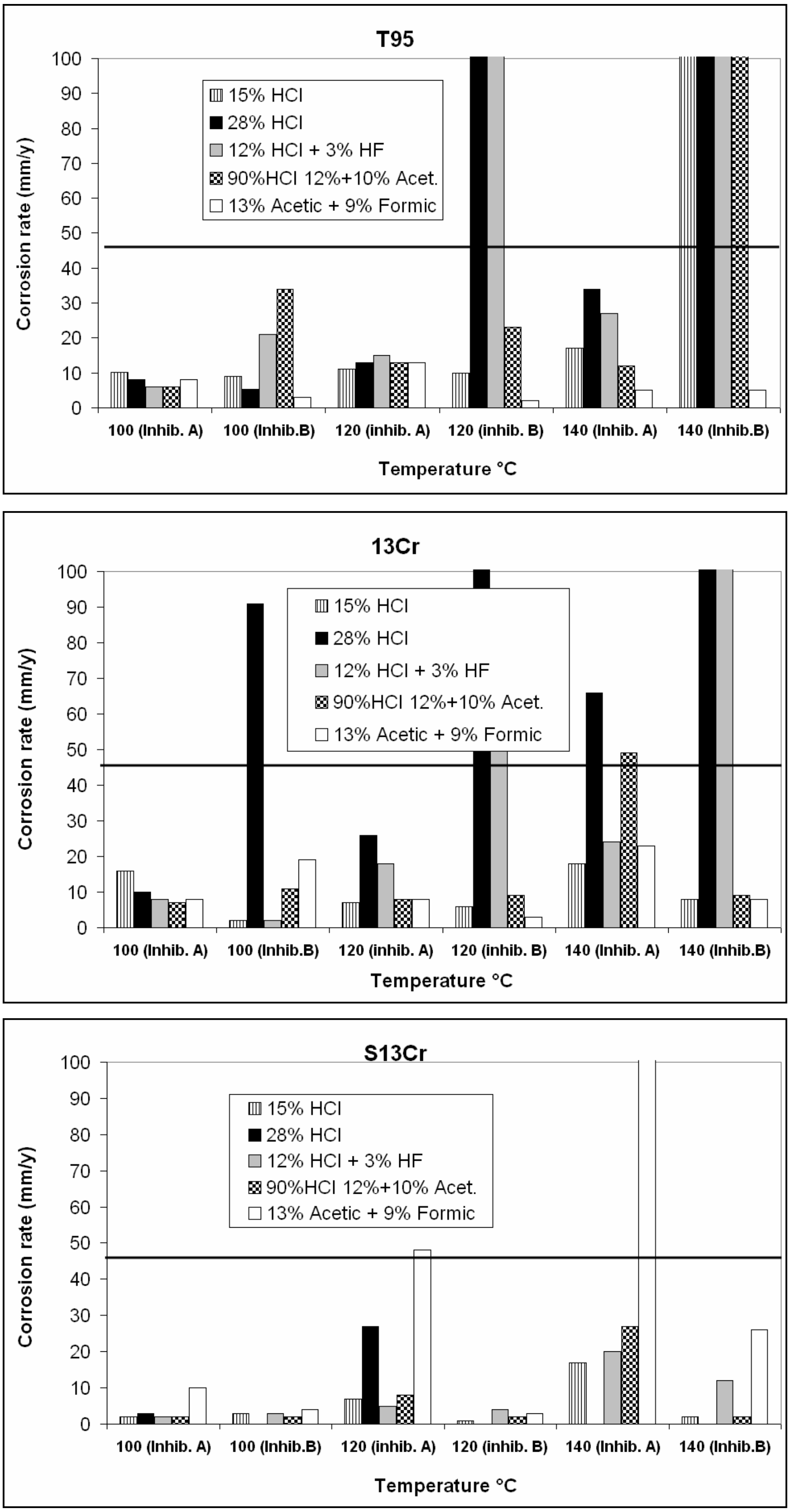

Figure 1 Corrosion rates of T95, 13Cr and S13Cr steels as a function of temperature and acid blend

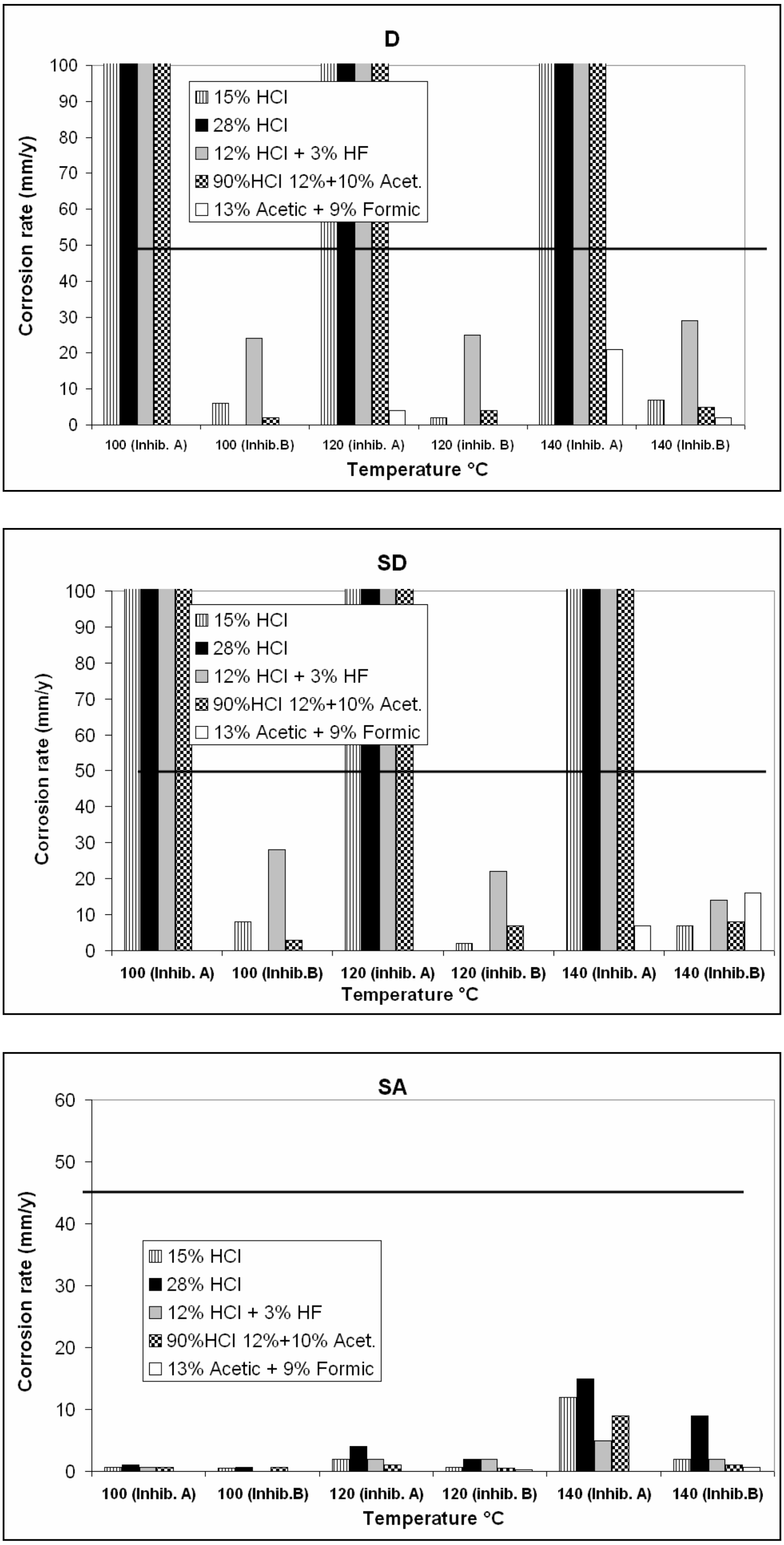

Figure 2 Corrosion rates of duplex (D), Superduplex (SD) and superaustenitic (SA) steels as a function of temperature and acid blend

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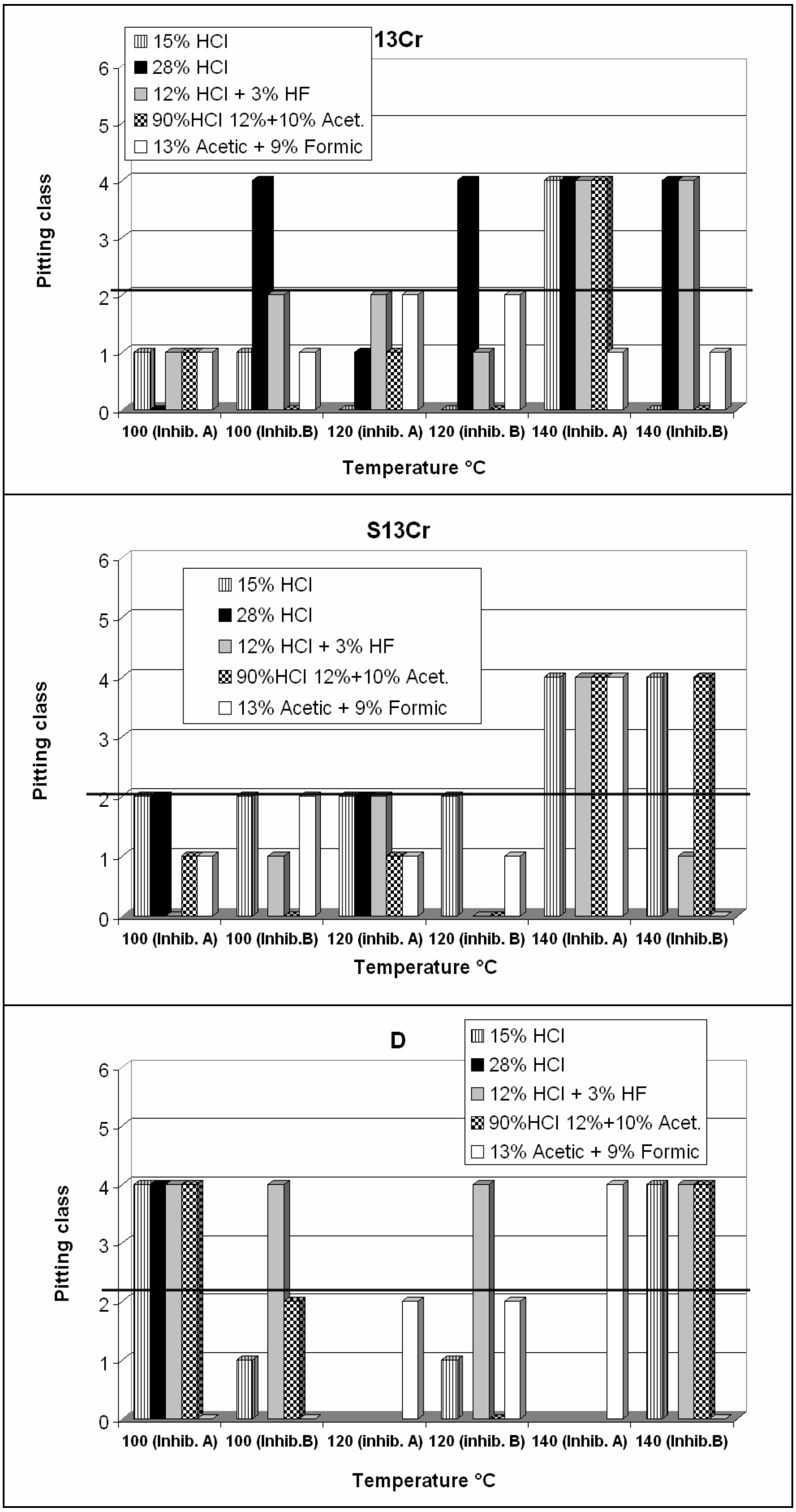

Figure 3 Pitting corrosion results of 13Cr, S13Cr and duplex (D) steels as a function of temperature and acid blend

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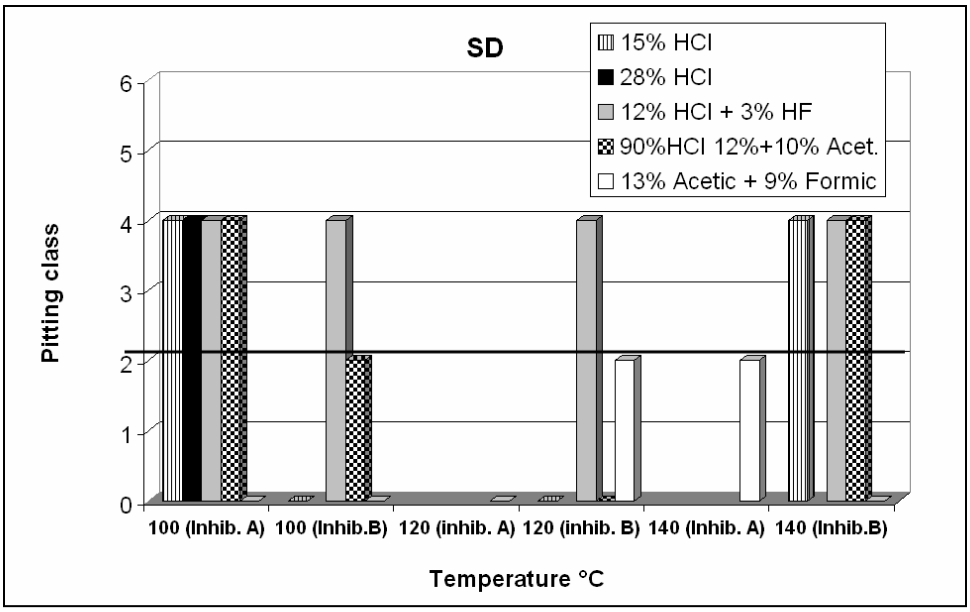

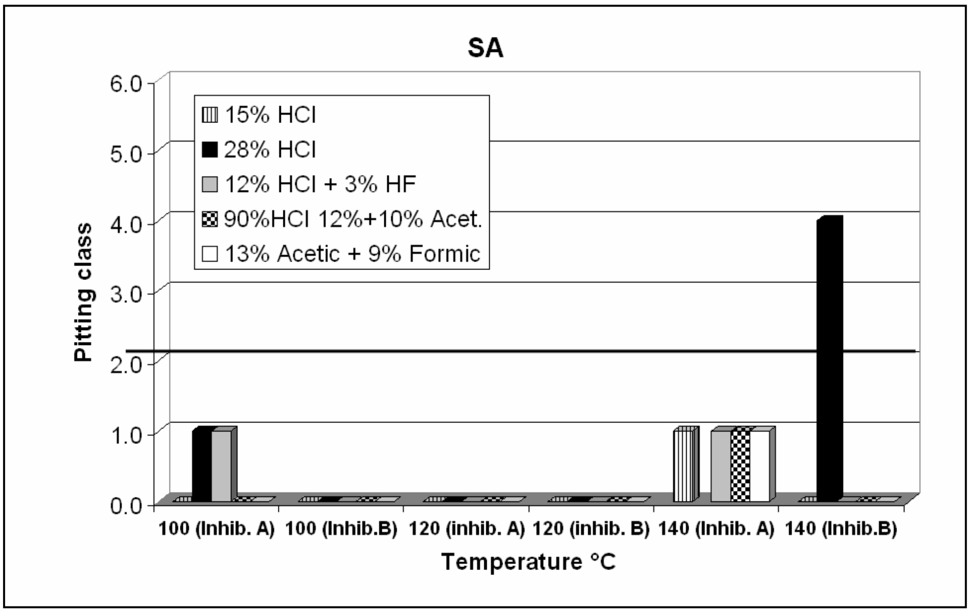

Figure 4 Pitting corrosion results of superduplex (SD) and superaustenitic (SA) steels as a function of temperature and acid blend

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3.2. Spent acid environment

3.2.1 Test with spent 28% HCl acid solution

Table VI collects the results obtained on specimens, exposed in spent 28% HCl acid solution at 100, 120 and 140 °C.

Concerning the pitting and general corrosion all steel pipes passed the test.

General corrosion attack was more pronounced in case of T95 (corrosion rates = 0.3-0.4 mm/y), even if below threshold limit, while corrosion rate values of stainless steels were in most cases lower than 0.1 mm/y.

All steel pipes were not affected by sulphide/chloride stress corrosion cracking.

Only one U-bend coupon showed some superficial cracks which may have been environmentally-induced, visible at the stereoscopic microscope at 40X magnification. These crack-like defects were oriented transverse to the principal applied stress. Examination of the external surfaces of the as-received tubulars did not identify similar crack-like defects, so they appear to have been formed as a consequence of exposure to the spent acid test environment. However, these cracks did not have the normal appearance of SCC when examined by cross-sectional metallography.

Steel	Initial pH	Final pH	Temp. °C	Corrosion rate mm/y	Rank of Pitting	SSC and / or SCC
T95	5.06	4.34	100	0.40	-	No
D	5.06	4.47	100	0.005	0	No
SD	5.06	4.16	100	< 0.001	0	No
13Cr	5.06	4.40	100	0.10	2	No
S13Cr	5.06	4.40	100	0.03	2	No
T95	5.08	4.43	120	0.23	1	No
13Cr	5.08	4.51	120	0.07	1	No
S13Cr	5.08	4.51	120	0.06	0	No
SA-125	5.13	4.73	120	< 0.001	0	No
T95	5.02	4.30	140	0.26	1	No
D	5.02	4.33	140	0.002	0	No
SD	5.02	4.50	140	0.002	0	No
13Cr	5.02	4.45	140	0.28	1	No
S13Cr	5.02	4.45	140	0.005	1	No
SA-125	5.10	4.83	140	< 0.001	0	No ?(*)

Note (*): Superficial cracks were visible on one SA-125 U-bend specimen

TABLE VI – Results of autoclave exposure of flat coupons in 28 % HCl spent acid solution

3.2.2. Test with spent 12% HCl+3% HF acid solution

3.2.2.1 Tests at pH 5

The results obtained respectively on flat coupons and U-bend specimens exposed in spent 12% HCl+3% HF acid solution at pH around 5 are collected in table VII.

Concerning pitting and general corrosion susceptibility all steel pipes passed the test.

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General corrosion was more pronounced in case of T95, S13Cr and 13Cr steels (corrosion rates = 0.2-0.4 mm/y), although within the threshold limits. The corrosion rate values of D and SD steels were lower than 0.05 mm/y. No susceptibility to SSC was observed in all steel pipes.

Steel	Initial pH	Final pH	Temp. °C	Corrosion rate mm/y	Rank of Pitting	SSC and / or SCC
T95	5.06	4.40	140	0.40	-	No
D	5.06	4.53	140	0.04	0	No
SD	5.06	4.50	140	0.03	0	No
13Cr	5.06	4.55	140	0.24	2	No
S13Cr	5.06	4.55	140	0.17	1	No
SA-110	5.02	4.50	100	< 0.001	0	No
SA-110	5.02	4.55	120	< 0.001	0	No
SA-110	5.02	4.57	140	< 0.001	0	No

TABLE VII – Results of autoclave exposure of flat coupons in 12 % HCl+ 3% HF spent acid solution at pH 5.

3.2.2.2 Tests at pH 4 and 3.5

Experimental activities was carried out with spent 12% HCl + 3% HF acid solution also at two different pH values, respectively 4.0 and 3.5, in order to study the influence of solution acidity on corrosion and stress corrosion. All tests were performed at 120 °C. Tables VIII report the results of such autoclave exposures.

Steel	Initial pH	Final pH	Temp. °C	Corrosion rate mm/y	Rank of Pitting	SSC and / or SCC
T95	4.05	4.30	120	0.47	-	No
D	4.05	4.33	120	0.05	0	No
SD	4.05	4.50	120	0.03	0	No
13Cr	4.05	4.45	120	0.31	2	No
S13Cr	4.05	4.45	120	0.06	1	No
T95	3.53	4.23	120	1.10	-	No
D	3.53	4.10	120	0.05	0	No
SD	3.53	4.12	120	0.02	0	No
13Cr	3.53	4.21	120	0.95	2	No
S13Cr	3.53	4.21	120	0.21	1	No

TABLE VIII – Results of autoclave exposure of steels in 12 % HCl+ 3% HF spent acid solution at pH 4 and 3.5, respectively.

No steel pipe was prone to SSC. In fact, no cracking was detected on T95, 13Cr, S13Cr, D and SD specimens. However, T95 and 13Cr flat coupons tested at 120 °C overcames the limit of the acceptance criteria concerning the corrosion rate (see the grey lines of table VIII).

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4. DISCUSSION

4.1. Active acid environment

Considering both general and localised corrosion susceptibility effects, some consideration can be done. The two different inhibitor packages offered different protection regards to material class. Generally speaking, the package “B”inhibitors ensure a good protection in the inorganic acid blends in terms of general and localised corrosion for D, SD, SA and S13Cr steels, with some exceptions. The protection action of the package ‘A’ inhibitors in inorganic acids shows to be effective on low alloy steel, martensitic, supermartensitic and superaustentic stainless steel while for the duplex and superduplex steels it is practically nil.

Austeno-ferritic stainless steels confirm to be more difficult to protect than austenitic and martensitic steels. This is due to the fact that in active conditions a galvanic coupling is operating between the austenitic and ferritic phases (roughly 50% in volume). The ferritic phase, which contains more chromium and less nickel than austenite, is the less noble one and is preferentially dissolved by acid mixtures [4]. Preferential attacks were found on duplex and superduplex stainless steels practically at all temperatures using inhibitor A . With inhibitor B, instead, these steels were effectively protected up to 120°C in all the tested acids with the exception of the mud acid where pitting was detected also at the lowest temperatures .

Compared to previous results [6], the actual tests suggest that duplex and superduplex stainless steels can be protected also at temperature as high as 140°C using the proper inhibitor.

Very good results were obtained in case of organic acid. No failure occurred for all tested steels at the three selected temperatures both in term of general and localised corrosion except for S13%Cr and duplex stainless steels at high T.

Based on the test results it is possible to rank the acid aggressiveness starting from the less aggressive one:
Organic acid blends < HCl 15% ≈ 90% HCl 12% + 10% CH_3COOH < HCl 12%+HF 3% < HCl 28%.

As far as HCl 28% is concerned, the aggressiveness is well known and chemical suppliers did not recommend any product on supermartensitic, duplex and superduplex since they remarked that corrosion inhibitors are not effective in this environment.

4.2 Spent acid environment

In ‘spent’ acid environments, all steel pipes showed to be less susceptible to the localised and general corrosion attack with respect to the active acid environments. Moreover, no Sulphide Stress Corrosion Cracking (SSC) occurred, probably because the duration of the tests was too short to determine cracks initiation, especially in the case of supermartensitic steels. Only one U-bend superaustenitic stainless steel specimen showed some superficial cracks at the highest test temperature (140°C), which may have been environmentally-induced.

It was noticed that general corrosion attack was more pronounced in case of low alloy steel (corrosion rates = 0.3-0.4 mm/y), even if below threshold limit, while corrosion rate values of CRAs were in most cases lower than 0.1 mm/y. Such results can be explained taking into account different mechanisms which occurred during the test: in case of low alloyed steel the

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combination of chloride content of the test solution and the presence of CO_2 determines the conditions for mass loss.
In the case of CRAs the corrosive attack of the solution was fairly weak, due to the presence of a passive film, which determines a barrier against the corrosion. Probably, at lower pH condition (less than 2) a stable rupture of passive film could be occurred.

Comparison of two spent acid solutions at pH 5 indicates that 12%HCl + 3% HF solution was more aggressive with respect to the 28% HCl. A possible reason is the presence of F^- ions in the 12%HCl + 3% HF solution, which forms very stable complexes with Fe^{+2} and Fe^{+3} , increasing strongly the kinetic of corrosion reactions. In fact, the following the semi-redox reactions take place in the 12%HCl + 3% HF solution:

1) $Fe \rightarrow Fe^{+2} + 2e^-$ and 2) $Fe^{2+} \rightarrow Fe^{3+} + e^-$

The F^-, bonding with iron ions, shifts the reaction 1 and 2 toward the products, increasing the iron dissolution.

A relevant and progressive increasing of corrosion rate was observed, expecially for T95 , 13Cr and S13Cr specimens, when the pH passed from 5 to 3.5. At pH 3.5, T95 and 13Cr steels were susceptible to general corrosion: mass losses were higher than 1 mm/y. As expected, D and SD specimens did not show any relevant variation in corrosion rate, varing the starting pH.

5. CONCLUDING REMARKS

Concerning the active acid phase, the results can be summarised as follows:

- Corrosion inhibitors A showed to be effective in the inorganic acid blends to protect the low alloy steels, superaustenitic, martensitic and supermartensitic steels, while the protection action was pratically nill on the duplex and superduplex steels.
- The corrosion inhibitors B in the inorganic acid blens ensure a good protection with the duplex, superduplex steel and superaustenitic steels, while for T95, martensitic and supermartensitic steels the performance was slightly less satisfactory especially at temperature higher than 120°C.
- The inhibitors B with mud acid 12%HCl +3% HF were ineffective in the most part of the tests at high temperature especially regards the pitting corrosion proneness.
- Concerning the organic acid blend (13% Acetic + 9% Formic), satisfactory results were obtained for the both type of inhibitors, expecially considering inhibition products B.
- Among the tested acids solutions, the organic acid blend (13%acetic + 9% formic) proves to be the less aggressive one. On the other side HCl 28% and mud acid result the most aggressive acid and difficult to protect mainly at high temperature.
- The increase of the temperature reduces the effectiveness of the inhibitors and in most part of the cases at the highest temperature (140 °C) general and/or pitting corrosion occurred. Only superaustenitic stainless steel was immune to corrosion even at 140 °C.
- Corrosion cannot be excluded in presence of HCl 28% even in presence of corrosion inhibitor; use of this acid is not recommended also at medium-low temperature.
- Concerning the corrosion inhibitor efficiency, the two different chemical packages offered a different protection in function of the material to be protected. Moreover, the necessity to develop corrosion inhibitors effective for CRAs protection in acidising environments at high temperature appeared.

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Regarding the spent acid phase, the following conclusions can be summarised:

- In spent acid environments, all steel pipes showed to be less susceptible to the localised and general corrosion, respect to active phase. Corrosion rate values were in most cases lower than 0.5 mm/y.
- No susceptibility to SSC was observed in all steel pipes; only one U-bend superaustenitic stainless steel specimen tested at 10 bar of H_2S, with 28% HCl spent acid at pH 5 showed some superficial cracks at the highest test temperature (140°C). Nevertheless, this steel passed the test with spent 13% HCl + 3% HF acid (spent mud acid) in the same test conditions.
- A sensible increment of general corrosion was observed for T95, 13Cr and S13Cr in the test with spent mud acid when the pH was reduced from 5 to 4 and 3.5 respectively.
- SMI253-125 U-bend specimens tested at 10 bar of H_2S, with 28% HCl spent acid at pH 5 and the test temperatures of 120 and 140 °C passed the test. Only one U-bend specimen showed superficial cracks at the highest temperature (140°C), visible at the stereoscopic microscopic at 40 x magnification, which may have been environmentally-induced.

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