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Abstract

With increasing environmental awareness follows demands for handling of brines that remain after use in a variety of applications. For this reason, stainless steels are being used in increasingly corrosive brine environments. The risk for corrosion in brines is associated with the chloride content, temperature, pH and oxygen level. This paper aims to explore the corrosion resistance of some stainless steels in highly concentrated sodium chloride solutions.

The resistance to localized corrosion of stainless steels in 20,000, 100,000 and 200,000 ppm Cl⁻ sodium chloride solutions was investigated at 90°C. The effects of chloride content, oxygen content and solution pH were studied on test coupons with and without welds and on U-bend specimens. Evaluation was performed for pitting and crevice corrosion, as well as for stress corrosion cracking. Long-term exposures (> 1300 hours) were combined with Electrochemical measurements such as cyclic polarization curves and determination of the critical pitting temperature (CPT). Investigated materials included duplex, super duplex, as well as 6Mo and 7Mo super austenitic stainless steels, which were compared to a standard austenitic grade.

A ranking of the tested stainless steel grades according to their corrosion resistance in sodium chloride brine solutions was: UNS S31603 < UNS N08904, UNS S32205 < UNS S31254, UNS S32750 < UNS S34565 < UNS S32654. Oxygen content was found to be an important factor influencing the corrosiveness of the sodium chloride brine solutions, with aerated conditions being the most corrosive ones. Also increasing chloride content and decreasing pH increased the corrosiveness of sodium chloride brine solution.

Key words: stainless steel, corrosion, brine, sodium chloride, pitting corrosion, crevice corrosion, stress corrosion cracking.

Introduction

With growing environmental awareness and more challenging process conditions, follow demands for handling of brines that remain after processing in a variety of applications. The corrosivity of this kind of environments places high demands on materials used for brine handling. However, the availability of a wide range of stainless steel grades means that there is good chance to find suitable grades for many brine handling applications. However, the demands on stainless are increasing as brines become increasingly corrosive.

Extensive data is available for the corrosion behavior of stainless steel in seawater, but less exists for brines. Super austenitic 6Mo and 7Mo grades such as S31254 (254 SMO®) and S32654 (654 SMO®) have long been proven solutions for sea water handling. [1–4] Super duplex grades such as S32750 are also seeing increasingly utilization in seawater. [5, 6] Compared to seawater the chloride content is higher in brines, and it could well be near saturation. Also, very high chloride content will prevent formation of the biofilm which is commonly formed in seawater at temperatures below approximately 40°C. The biofilm formed on stainless steel in seawater will increase the potential, and thus also increase the risk for localized corrosion compared to e.g. a laboratory solution with the same chloride content as seawater. Chlorination is often used in order to control biological activity on stainless steel in seawater, and it will also increase the potential even further.

The risk for corrosion in brines is mainly associated with the chloride content, temperature, pH and oxygen level, and of course the alloying content of the stainless steel. This paper aims to explore the corrosion resistance of some stainless steels in highly concentrated chloride solutions. Investigations are carried out in sodium chloride solution with chloride contents up to 200,000 ppm at 90°C. The effect on the corrosivity of pH and oxygen content is investigated using both long-term exposure and Electrochemical test methods for pitting corrosion. Long-term exposures assess the effect of welds and crevices, as well as the effect of applied stress/deformation on the risk for stress corrosion cracking. Investigated materials cover a broad range of stainless steel grades such as duplex, super duplex, as well as 6Mo and 7Mo super austenitic stainless steels. The results are compared with a standard austenitic stainless steel grade.

Experimental Procedure

Materials

Materials included in the current study are presented in Table 1 with their designations, type of microstructure and typical chemical composition. Also the pitting resistance equivalent (PRE) number is included which provides a rough ranking according to their relative resistance to localized corrosion, such as pitting and crevice corrosion. As can be seen in Table 1 the tested materials

include some duplex and stainless steel grades with very similar PRE values, e.g. S32750 and S31254.

Welding of the materials was performed using welding methods, filler metals and shielding/backing gas as listed in Table 2. The welded specimens were pickled after welding in a mixed acid bath of 3.5 M HNO₃ + 3.0 M HF at 60°C. Different stainless steel materials/weldments will require different pickling times, and the pickling times were adjusted accordingly until the weld oxide/heat tint was completely removed for each weldment.

Designation		Typical chemical composition [wt %]						Micro-structure*	PRE [#]
UNS	EN	C	N	Cr	Ni	Mo	Others		
S31603	1.4432	0.02	–	16.9	10.7	2.6	–	A	28
N08904	1.4539	0.01	–	20	25	4.3	1.5 Cu	A	34
S32205	1.4462	0.02	0.17	22	5.7	3.1	–	D	35
S32750	1.4410	0.02	0.27	25	7	4	–	D	42
S31254	1.4527	0.01	0.2	20	18	6.1	Cu	A	41
S34565	1.4565	0.02	0.45	24	17	4.5	5.5 Mn	A	46
S32654	1.4652	0.01	0.5	24	22	7.3	3.5 Mn, Cu	A	54

Table 1 Tested materials

* A = austenitic microstructure, D = duplex microstructure

[#] PRE = Cr + 3.3 × %Mo + 16 × %N

UNS	Weld method	Filler metal		Shielding gas	Backing gas
		EN/ISO	AWS		
S31603	SMAW	E 19 12 3 L R	E316L-17	–	–
	GMAW	G 19 12 3L	ER316L	Ar + 8% CO ₂ + 0.03%NO	N ₂ + 10% H ₂
N08904	SMAW	E 20 25 5 Cu N L R	E385-17	–	–
	GMAW	G 20 25 5 Cu L	ER385	Ar + 2% CO ₂ + 30% He + 0.03% NO	Ar
S32205	SMAW	E 22 9 3 N L R	E2209-17	–	–
	GMAW	G 22 9 3 N L	ER2209	Ar + 2% CO ₂ + 30% He + 0.03% NO	N ₂ + 10% H ₂
S32750	GMAW	G 25 9 4 L N	ER2594	Ar + 2% CO ₂ + 30% He + 0.03% NO	N ₂ + 10% H ₂
S31254	SMAW	E Ni Cr 21 Mo Fe Nb	ENiCrMo-12	–	–
	GMAW	G Ni Cr 25 Mo 16	ERNiCrMo-13	Ar + 2% CO ₂ + 30% He + 0.03% NO	N ₂ + 10% H ₂
S34565	GMAW	G Ni Cr 25 Mo 16	ENiCrMo-13	Ar + 2% CO ₂ + 30% He + 0.03% NO	N ₂ + 10% H ₂
S32654	SMAW	E Ni Cr 25 Mo16	ERNiCrMo-13	–	–

Table 2 Welding details

Long-term test

Crevice corrosion specimens and welded specimens were of size 60 × 60 mm with a center hole with \varnothing 12 mm. The specimens were mounted on an insulated N10276 or titanium bolt and between each specimen PTFE crevice washers were placed and a torque of 1.58 Nm was applied. Each washer has 20 crevice sites, and thus there are 40 crevice sites on each specimen. A smaller specimen size of 50 × 25 mm (with a small hole for suspension with platinum wire) was used for potential measurements on grade S32654. All edge surfaces were ground to minimum 320-grit surface finish.

Stress corrosion cracking specimens were of size 127 × 13 mm and bent to U-bend specimens with a radius of 12 mm or ½ in, using a two-step procedure. The U-bends were secured using

N10276 or titanium bolts and nuts with PTFE washers insulating them from them from the test specimens. Stress corrosion specimens were ground to minimum 120-grit on all surfaces.

The test solutions, with compositions shown in Table 3, were prepared using pro analysi grade NaCl and distilled water. Adjustment of the pH was done by adding HCl (with the same chloride concentration as the test solution) for pH 4 and NaOH for pH 8. Specimens were placed in the test solution which was heated and kept at test temperature 90°C for the duration of the test, which was 56 days (> 1300 hours). Either air (using a diaphragm air pump with a capacity of 1000 cm³/min) or nitrogen gas was bubbled through the test solution throughout the duration of the test.

All specimens were weighed before and after exposure for

determination of the weight loss, and calculation of the corrosion rate in mm/year.

After the test was ended, the specimens were cleaned, weighed and examined using a microscope with 20× magnification. Any corrosion was noted and classified as pitting, crevice corrosion, stress corrosion cracking or edge attack. If crevice corrosion was observed under the crevice washers, the number of crevice corrosion sites under each washer was noted as well as the maximum depth of crevice corrosion.

Test solution	Cl ⁻ [ppm]	pH	Bubbling gas
2	100,000	4	N ₂
3	100,000	8	air
4	100,000	8	N ₂
5	20,000	4	air
6	200,000	8	air

Table 3 Sodium chloride test solutions

Critical pitting temperature

The critical pitting temperature (CPT) was determined according to ASTM G150 [7] in both the standard solution of 1 M NaCl (35,450 ppm Cl⁻) and in a NaCl solution with 100,000 ppm Cl⁻.

Specimens of size 60 × 60 mm were tested with wet ground to 320-grit surface finish. After the grinding operation the specimens were left to passivate in air for a minimum of 24 hours prior to testing.

This potentiostatic technique uses a temperature scan and a flushed port cell that eliminates the occurrence of crevice corrosion. From an initial temperature of 0°C, the test solution is heated at 1°C/min while the potential is held at 700 mV versus the standard calomel electrode (SCE). The current is monitored and the CPT is defined as the temperature at which the current density exceeds 100 µA/cm² for 60 seconds. The test area in the flushed port cell

is 10 cm². Throughout the test nitrogen gas is bubbled through the test solution. Pitting on the surface and the absence of crevice corrosion is confirmed after the test by using a microscope at 20 × magnification.

Potentiodynamic polarization measurements

Potentiodynamic polarization measurements based on ASTM G61 [8] were performed in sodium chloride solution with 100,000 ppm Cl⁻ at 90°C.

Specimens of size 30 × 30 mm were tested with wet ground 320-grit surface finish. Ground specimens were left to passivate in air for a minimum of 24 hours prior to testing which was carried out in flush port cell which eliminates the occurrence of crevice corrosion.

The specimens were held at the corrosion potential for 10 minutes before the potential sweep at a rate of 20 mV/min was started at -300 mV versus the measured corrosion potential. The breakdown potential was evaluated where the current density exceeds 100 µA/cm² for 60 seconds. The test area in the flushed port cell is 1 cm². The effect of oxygen content was evaluated by bubbling the test solution with either air or nitrogen gas.

After the test, the type of corrosion causing the breakdown of the passive film was determined by using a microscope at 20× magnification. If crevice corrosion was detected the test was deemed to be invalid and was repeated until no crevice corrosion was detected.

RESULTS

In Table 4 the results from the long-term test are summarized. For each test condition and material, it is indicated whether the material suffered crevice corrosion, pitting or stress corrosion cracking, and also if any pitting was found in the area associated with the weld (i.e. weld metal or heat affected zone) or on the edges of the tested specimens. In Figure 1 pictures of samples after long-term test in 200,000 ppm Cl⁻ are shown. Note that while there is no crevice corrosion on grade S31603 there is large attack originating at the cut edge of the specimen.



Figure 1 Samples after long-term exposure in NaCl brine solution with 200,000 ppm Cl⁻ at 90°C for 56 days with air bubbling through the solution.

UNS	100,000 ppm Cl ⁻									
	1					2				
	pH 4, aeration					pH 4, N ₂				
	C	P	W	S	E	C	P	W	S	E
S31603	x	x	x	o	x	x	o	x	o	o
N08904	x	o	x	–	x	x	o	x	–	o
S32205	x	o	o	o	x	x	o	o	o	x
S32750	–	–	–	–	–	–	–	–	–	–
S31254	x	o	o	–	x	o	o	o	–	o
S34565	–	–	–	–	–	–	–	–	–	–
S32654	x	o	o	–	o	o	o	o	–	o

UNS	100,000 ppm Cl ⁻									
	3					4				
	pH 8, aeration					pH 8, N ₂				
	C	P	W	S	E	C	P	W	S	E
S31603	x	x	o	o	x	x	x	x	o	x
N08904	x	o	x	–	x	x	o	x	–	o
S32205	x	o	x	o	x	x	o	x	o	o
S32750	o	o	o	o	o	o	o	o	o	o
S31254	x	o	o	–	o	o	o	o	–	o
S34565	o	o	o	–	o	o	o	o	–	o
S32654	o	o	o	–	o	o	o	o	–	o

UNS	20,000 ppm Cl ⁻					200,000 ppm Cl ⁻				
	5					6				
	pH 4, aeration					pH 8, aeration				
	C	P	W	S	E	C	P	W	S	E
S31603	x	x	x	o	o	o	x	x	o	x
N08904	x	o	o	–	x	x	o	x	o	o
S32205	x	o	o	o	o	(x)	o	x	o	x
S32750	–	–	–	–	–	x	o	o	o	x
S31254	x	o	o	–	o	x	o	o	o	o
S34565	–	–	–	–	–	x	o	o	–	o
S32654	o	o	o	–	o	x	o	o	o	o

Table 4 Summary of long-term test results in NaCl solutions at 90 °C

C = Crevice corrosion	o = No visible attack
P = Pitting corrosion	x = Attack
W = Pitting in weld metal / HAZ	(x) = Attack on SCC specimen
S = Stress corrosion cracking	– = Not tested
E = Attack on cut edges	

The main results were that:

- Standard austenitic grade S31603 suffers crevice corrosion or pitting in all tested environments.
- Super austenitic grade S32654 only suffers crevice corrosion in test solutions 1 and 6.
- No stress corrosion cracking was found on any material in any test solution.
- The corrosion rate was <0.05 mm/y for all tested specimens. This is to be expected since the main mode of corrosion is localized corrosion.
- The maximum depth of crevice corrosion was >1 mm for grade S31603 in test solutions 2, 3 and 5.



Figure 2 Effect of aeration and chloride content on open circuit potential for S32654 at pH 8 and 90°C.

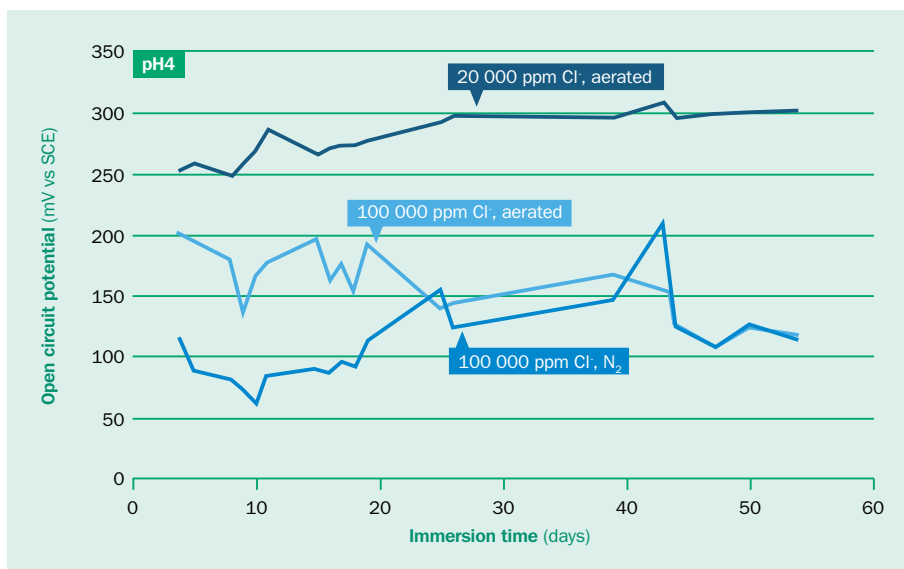


Figure 3 Effect of aeration and chloride content on open circuit potential for S32654 at pH 4 and 90°C.

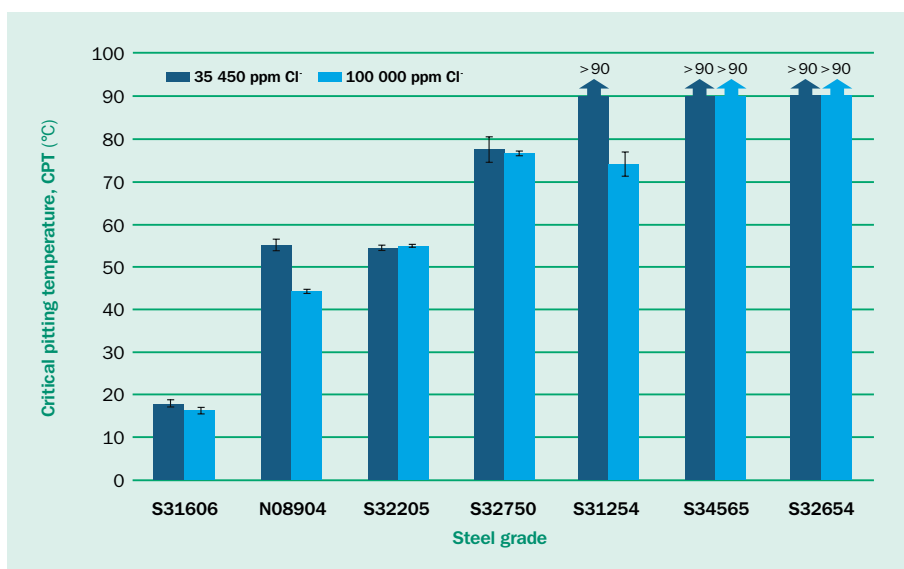


Figure 4 Critical pitting temperature (CPT) tested according to ASTM G150 in NaCl solution with 35,450 and 100,000 ppm Cl⁻.

The corrosion potentials measured on UNS 32654 specimens (without crevices) are shown in Figures 2 and 3. They show that aerated solutions have higher corrosion potentials compared to de-aerated ones. Moreover, for aerated solutions a lower chloride content increases the open circuit potential. These potential can be compared to those seen for stainless steel in natural seawater where at 30°C the potential is around +300 mV vs SCE due to biofilm formation and with chlorination it increases to around +600 mV vs SCE. [9] However, if the seawater is heated to 70°C the potential decreases dramatically to below -100 mV vs SCE. [9]

In Figure 4 the effect of the chloride content on the critical pitting temperature (CPT) measured according to ASTM G150 is shown. Measurements were performed in the standard test solutions with 35,450 ppm Cl⁻ (1 M NaCl) and 100,000 ppm Cl⁻. The results show that the chloride content has an effect on the measured CPT for some grades such as N08904 and S31254.

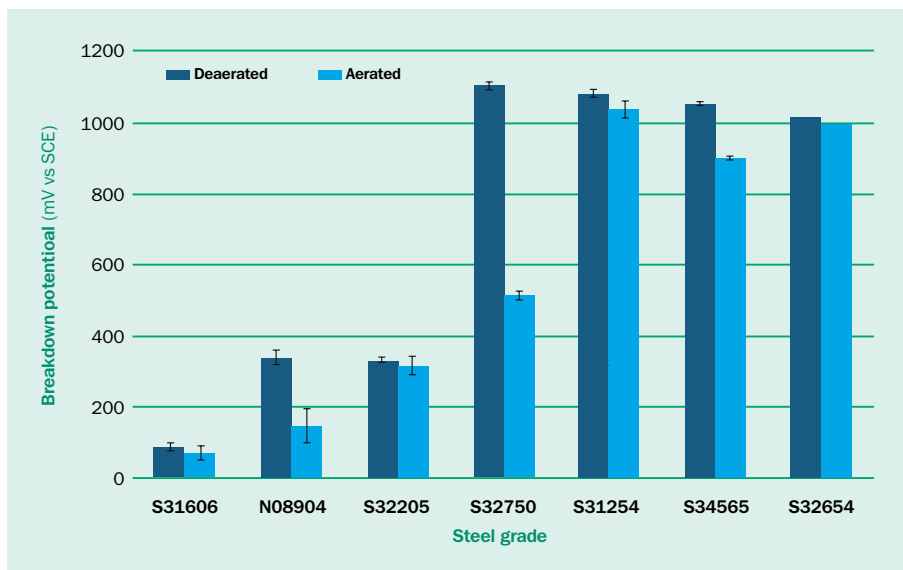


Figure 5 Breakdown potential in NaCl solution with 100,000 ppm Cl⁻ under de-aerated and aerated conditions at 90 °C.

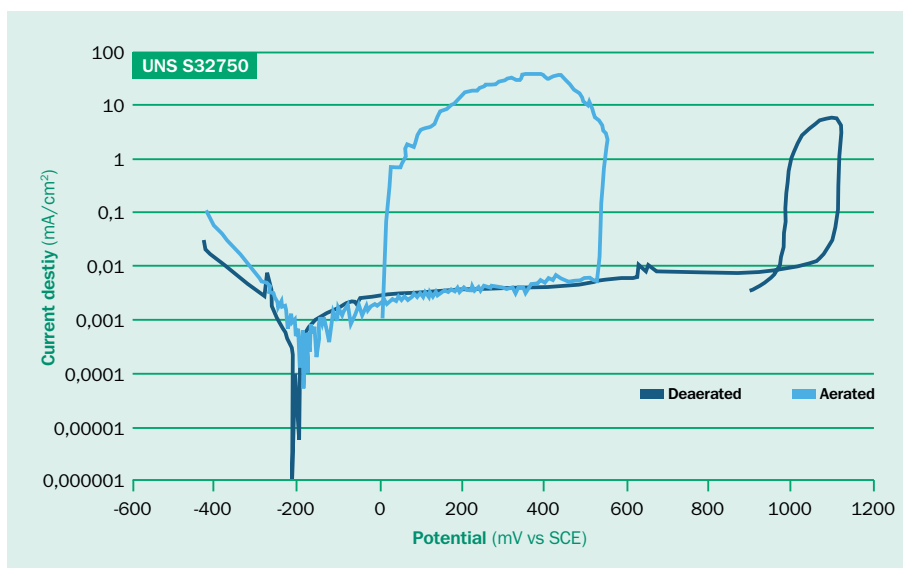


Figure 6 Cyclic polarization curves for S32750 in NaCl solution with 100,000 ppm Cl⁻ under de-aerated and aerated conditions at 90 °C.

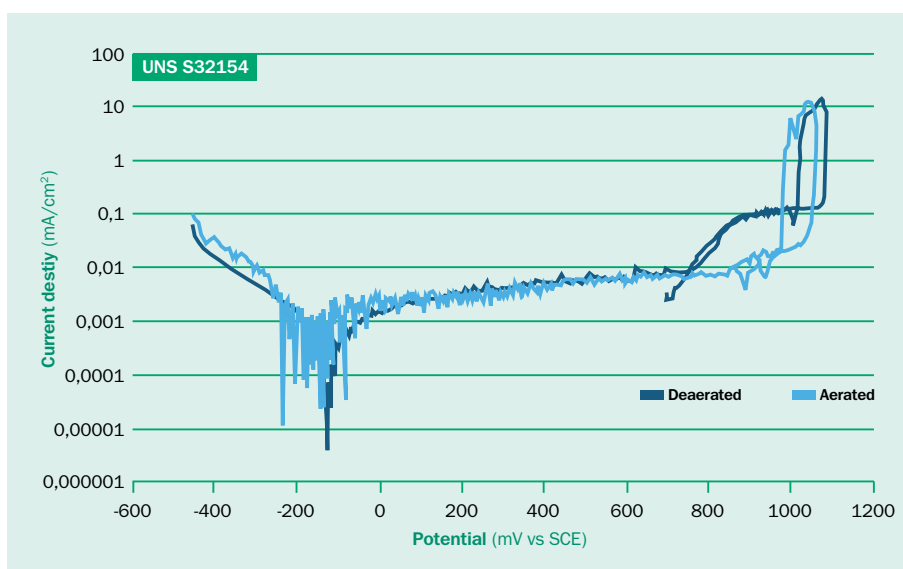


Figure 7 Cyclic polarization curves for S31254 in NaCl solution with 100,000 ppm Cl⁻ under de-aerated and aerated conditions at 90 °C.

Figure 5 shows the results of aeration and de-aeration on the breakdown potential in a test solution of 100,000 ppm Cl⁻. Also here the effect is greatest on some steel grades such as N08904 and S32750. In Figures 6 and 7 the potentiodynamic curves of S32750 and S31254 clearly show the difference in breakdown potential under aerated conditions for the two grades and with a larger hysteresis loop for S32750.

The Electrochemically tested specimens were further investigated using a microscope with 20x magnification in order to determine if pitting had occurred or not and the results can be found in Table 5. In the potentiodynamic measurements on S32750 and S31254, pitting is found on specimens with breakdown potential > 1000 mV vs SCE, i.e. in the transpassive region. These specimens do not exhibit a pronounced hysteresis loop (Figure 6), which is typical for transpassive corrosion. Similarly, observations are found for the CPT measurements on S34565 at 100,000 ppm Cl⁻ where small pits are observed on the specimens after testing even though the CPT was determined to be > 90 °C.

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UNS	ASTM G150		Potentiodynamic	
	De-aerated		100,000 ppm Cl ⁻	
	C	P	W	S
S31603	x	x	x	x
N08904	x	x	x	x
S32205	x	x	x	x
S32750	x	x	x	(x)
S31254	o	x	(x)	(x)
S34565	o	x	x	o
S32654	o	o	o	o

Table 5 Occurrence of corrosion in Electrochemical tests

o	No corrosion
x	Pitting
(x)	Pitting in the transpassive region

Discussion

Solution composition

The test solutions can roughly be ranked according to their corrosiveness based on the long-term test results in presented in Table 4. Starting with the most aggressive environment the ranking is as follows:

Test solutions 1 & 6 > test solutions 3 & 5 > test solutions 2 & 4

This points towards the importance of the oxygen content as a factor influencing the corrosiveness as the two de-aerated solutions are also the least corrosive. This is supported by the open circuit potential measurements on S32654, where de-aeration is shown to decrease the potential (see Figures 2 and 3). Also the effect of chloride content on the open circuit potential most likely reflects the solubility of oxygen, which decreases as the chloride content increases. [10]

Although pH and chloride content also have an effect, these factors seem to be of less importance than the oxygen content.

Electrochemical test methods

By comparing the Electrochemical results shown in Table 5 at 100,000 ppm Cl⁻ with the long-term tests at the same chloride concentration, it is clear that pitting occurs more frequently in the former case. This is not surprising as the potential, e.g. +700 mV vs SCE for CPT test, applied in the Electrochemical tests far exceeds what was recorded in the long term tests (c.f. Figures 2 and 3). In some cases it is also possible to see the effect of chloride content and aeration using the Electrochemical test methods.

That short term Electrochemical tests will give more conservative results for the risk for pitting than longer term immersion tests has been shown at lower chloride contents. [11] They will not give an answer if pitting will occur during longer exposure times or not but can indicate conditions where there is a risk of pitting if the potential is sufficiently high (e.g. caused by chlorination).

Stress corrosion cracking

As shown in Table 4 none of the test conditions caused cracking of grade S31603, which should be most susceptible to stress corrosion cracking. It may seem surprising that not even 200,000 ppm Cl⁻ induces cracking for this grade but earlier experience suggests that U-bend S31603 (with 2% Mo) specimens do not necessarily crack in a 25% NaCl solution (~150,000 ppm Cl⁻) at the boiling point (~105 °C). [12]

U-bend specimens are considered to be less detrimental than e.g. constant load and slow strain rate methods for SCC testing. It has been shown that constant load specimens of S31603 are susceptible to cracking in NaCl at ~180,000 ppm Cl⁻ at 100 °C. [13] However, it has also been shown that making the test conditions more aggressive by lowering the pH of 25% NaCl to 1.5 or by further increasing the chloride content to >250,000 ppm by using CaCl₂ readily cause initiation of SCC of U-bend specimens. [12, 14, 15]

In comparison, higher alloyed austenitic grade N08904, super austenitic grades S31254 and S32654, and duplex grades S32205 and S32750 all are expected to have a resistance to stress corrosion cracking that far exceeds that of S31603.

Stainless steel grades

Generally, materials that are expected based on their PRE number to have similar resistance to localized corrosion, such as N08904 and S32205, and S31254 and S32750, also behave very similarly in the current test. A rough ranking of the corrosion resistance in

S31603 < N08904, S32205 < S31254,
S32750 < S34565 < S32654

Crevice corrosion is present on grade S31603 in all tested environments, except interestingly at 200,000 ppm Cl⁻, even though all other grades suffer crevice corrosion at this chloride content. For S31603, 200,000 ppm Cl⁻ probably represents such a corrosive environment that pitting corrosion readily occurs, and once pitting has initiated the potential of the specimen decreases, thus crevice corrosion might not initiate. A recent study on the limiting conditions for pitting corrosion on S31603 stainless steel showed that the limit was <1000 ppm Cl⁻ at 80°C. [11]

In 100,000 ppm Cl⁻ at pH 4 pitting corrosion in connection with the weld occurs for N08904 under both aerated and de-aerated conditions, while no pitting is observed on S32205. This may be a result of the higher molybdenum content of N08904, which makes it more sensitive to molybdenum segregation in the weld if no heat treatment is performed after welding. Similarly to S31603 no crevice corrosion was found on the specimens with crevice former, instead edge attack was present on those specimens as well as pitting. However, crevice corrosion was detected underneath the washer on the U-bend stress corrosion specimens.

The current results indicate that crevice corrosion does not occur at 100,000 ppm Cl⁻ on S32750 at the high temperature of 90°C. Results from previous tests in 50,000 and 70,000 ppm Cl⁻ at 40°C with air saturation showed that S32750 suffered crevice corrosion at both chloride contents while no corrosion occurred on S34565 at 70,000 ppm Cl⁻. [16] This could be an effect of the higher solubility of oxygen at 40°C than at 90°C. This once again indicates the significance of oxygen content on the corrosiveness of brine solution.

Super austenitic stainless steel grades S31254, S34565 and S32654 are well-proven materials for seawater handling. [1, 3, 4, 6] In many cases S32654 can be used for substituting nickel base material or titanium. [17] The open circuit potential measured on S32654 range between +100 to +300 mV vs SCE, which can be compared to the corrosion potentials of around +300 mV measured at in natural seawater at 30°C with biofilm formation. [9] The temperature limit for crevice corrosion in seawater for these grades is in the range 30 – 50°C.

Conclusions

Corrosion testing was performed in sodium chloride solution brines at 90°C with a chloride content of 20,000, 100,000 or 200,000 ppm and pH 4 and/or 8.

S31603 < N08904, S32205 < S31254,
S32750 < S34565 < S32654

Oxygen content was found to be an important factor influencing the corrosiveness of the sodium chloride brine solutions, with the most corrosive environments also being aerated. Also increasing chloride content and decreasing pH resulted in higher corrosiveness.

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We work with our customers and partners to create long lasting solutions for the tools of modern life and the world's most critical problems: clean energy, clean water and efficient infrastructure. Because we believe in a world that lasts forever.

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