Stress Corrosion Cracking of Duplex Stainless Steels under Evaporative Conditions

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Abstract
Duplex stainless steels with their ferritic-austenitic microstructure combine many of the beneficial properties of ferritic and austenitic stainless steels. The duplex microstructure contributes to the high mechanical strength and high resistance to stress corrosion cracking (SCC). Numerous studies have shown that duplex stainless steels have excellent resistance to SCC in chloride solutions. However, little data is available for duplex stainless steel when it comes to chloride induced SCC under evaporative conditions.

In current study, the chloride induced SCC resistance of different duplex stainless steels grades was investigated. The studied evaporative SCC test methods include drop evaporation testing, salt spray slow strain rate testing, as well as long exposure atmospheric field testing. The test results produced by the authors, as well as relevant literature data are presented, evaluated and discussed with respect to the suitability of the test methods to rank the SCC resistance of duplex stainless steels under evaporative conditions.

Keywords
Stainless Steel; Duplex, Stress Corrosion Cracking; Chlorides; Evaporation;

Introduction

Aqueous solutions with low concentrations of chlorides normally do not present any problems to the use of stainless steels. However, localised evaporation of dilute chloride solution can increase the chloride concentration and lead to pitting corrosion, or even stress corrosion cracking (SCC) under unfavourable conditions. Splash zones in marine environments, water lines, wet insulation or dripping water on hot surfaces are examples of conditions where chloride concentration could build up over time and cause corrosion.

Due to a beneficial combination of the properties from both ferritic and austenitic stainless steels, duplex stainless steels show high mechanical strength and good resistance to localised and uniform corrosion [1-5], as well as excellent resistance to chloride induced SCC [6-8]. However, much less data is available for duplex stainless steel when it comes to chloride induced SCC under evaporative conditions. The aim of current study has been to look into this subject and to discuss the suitability of various evaporative tests for ranking of the duplex grades.

Materials

Typical chemical composition, mechanical strength and pitting resistance equivalent (PRE) of the materials in this work are presented in Table 1 below.

Table 1 Typical compositions and properties of the investigated materials. The mechanical strength values refer to typical values for hot rolled plate material produced by Outokumpu.

Materials	**EN Designation**	**Typical chemical composition [wt%]**						**PRE***	**$R_{p0.2}$** [MPa]	**R_m** [MPa]
		C	**N**	**Cr**	**Ni**	**Mo**	**Others**			
Core 304L/4307	1.4307	0.02		18.1	8.1			18	260	580
Supra 316L/4404	1.4404	0.02		17.2	10.1	2.1		24	260	570
Forta LDX 2101	1.4162	0.03	0.22	21.5	1.5	0.3	5Mn Cu	26	500	700
Forta DX 2304	1.4362	0.02	0.10	23	4.8	0.3	Cu	26	450	670
Forta LDX 2404	1.4662	0.02	0.27	24	3.6	1.6	3Mn Cu	34	520	750
Forta DX 2205	1.4462	0.02	0.17	22	5.7	3.1		35	510	750
Forta SDX 2507	1.4410	0.02	0.27	25	7	4		43	580	830

* PRE = %Cr + 3.3x %Mo + 16x %N

Experimental

Test methods for evaporative conditions that are covered in this study are briefly summarised in this section.

Atmospheric field testing

The marine exposure sites for the field testing were located in Antwerp in Belgium, Tianjin in China, Lavéra in France and Dubai in United Arab Emirates. Prior to field exposure the ring welded specimen coupons (130×130 mm) were pickled. Racks with test specimens were exposed to a worst case, non-washing marine conditions. Weight loss, type of corrosion and maximum depth of corrosion attack were evaluated after the field exposure. Further details can be found in [9, 10].

Wick testing

The Wick test was developed to simulate evaporative conditions by embedding a heated stainless steel in an insulation material wetted with a dilute chloride solution. The Wick test method is stipulated in ASTM C692 [11]. The test comprises a U-bend specimen (dry ground 120 grit) that is fitted into a groove of glass fibre insulation material, which is placed in a

dilute NaCl solution (1500 pmm chlorides). The level of the solution is controlled and held stable. The specimen is electrically heated to 100°C. The test duration is 672 hours. After exposure the specimens are evaluated by visual inspection with optical light microscopy.

Chloride deposit testing in a climate chamber

This test method involves deposition of chloride (0.16 mg/mm^2) by droplets of chloride solution on selected areas of ring welded specimen. Sixteen droplets were deposited at different areas of the ring welded samples. These areas were denoted as centre, 1st ring, 2nd ring, corners, see Figure 1. Samples were exposed in a climate chamber with different combinations of salt deposit (NaCl, $MgCl_2$, $CaCl_2$), temperature (30°C, 50°C, 70°C) and relative humidity (30%. 50%, 70%) for a total of 8 week exposure time. A more exhaustive test description can be found in [9].

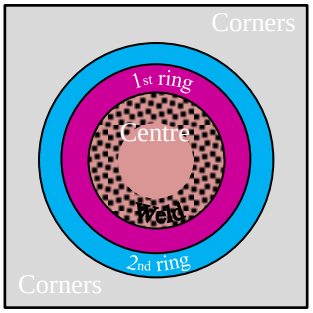

Figure 1 Overview of areas where chloride droplets were deposited.

Constant load testing with cyclic salt spray

In order to assess the SCC resistance of welded material, the constant load specimens were machined from gas tungsten arc welded (GTAW) plates. The flat specimens were of "dog bone" type. The applied load was 90% of $Rp_{0.2}$. A cyclic salt spray procedure from the automotive industry was adopted (ECC1 test by Renault). This salt spray procedure consists of a number of wet/dry and spray phases. The test was conducted at 50°C & 70°C and the total test duration was 42 days. The time to failure is recorded. The testing procedure is further detailed in [9].

Drop evaporation testing (DET)

The drop evaporation test procedure is briefly described below, detailed guidelines are stipulated in MTI Manual No.3 [12] and ISO 15324 [13]. DET specimen dimensions are detailed in Figure 2 and a schematic illustration of the test setup in Figure 3. In order to remove any residual deformation and residual stresses from the machining step, the specimens were electropolished.

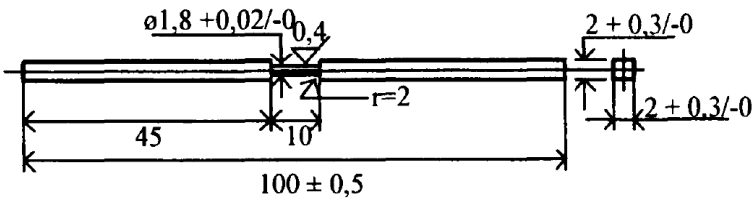

Figure 2 The drawing of a DET specimen, the dimensions are given in mm.

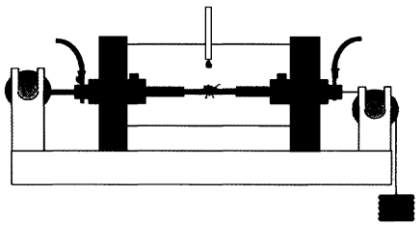

Figure 3 A schematic illustration of the DET setup.

The specimen is mounted horizontally between two fixtures. The designated uniaxial load is applied to the specimen by weights via a steel wire connected to one of the fixtures. The load is commonly selected to correspond to a stress ratio of 10 to 100% of the $Rp_{0.2}$ at 200°C. The specimen is electrically heated to an initial temperature of ≈300°C and an aerated 0.1 M NaCl solution is dripped onto the middle section of the specimen. The drop frequency is 6 drops per minute (~0.1 Hz). During a DET cycle the temperature of the specimen fluctuates from approximately 70°C to 120°C. Unless a failure occurs, the test is terminated after 500 hours. Time to failure is recorded by a timer connected to the load train.

Slow strain rate testing (SSRT) with cyclic salt spray

The cyclic salt spray tests used cylindrical slow strain rate (SSRT) specimens with a diameter of 3 mm and a 600 grit wet ground surface finish. The strain rate was $2 \cdot 10^{-7}$ s^{-1}. The specimen was electrically heated to a maximum temperature of ≈150°C. Every 20th second 1% NaCl solution was sprayed, causing the temperature to drop and then gradually increased as the solution evaporated. This test method is described in more detail in [14, 15]. An advantage of the cyclic salt spray over the dripping procedure in DET is that contact between the specimen and the solution is more even and well defined.

Results and Discussion

Apart from high mechanical strength, a recognised key property of duplex stainless steels is the high resistance to chloride induced SCC. It might seem to be relatively straightforward to find a test method to discriminate between susceptible materials (e.g. standard 304 & 316-type austenitic steels) and materials with superior SCC resistance. However, due to the intrinsic high SCC resistance, ranking of the duplex grades with respect to SCC resistance can be challenging. This is even more so for test methods designed to represent evaporative conditions. For instance, a four year atmospheric field testing at several marine sites has had little success to provoke SCC. None of the tested grades in the reported study, including duplex grades LDX 2101, DX 2304, LDX 2404 and DX 2205, showed any SCC after 1 and 4 years of exposure [9, 10]. In fact, the marine exposure conditions did not even yield SCC for the 304L and 316L materials.

Another example is the Wick test results presented in Table 2. The Wick test results clearly indicate that the duplex grades are resistant, while the standard austenitic grades fail due to SCC [8]. Thus, it seems that the evaporative conditions in the Wick and atmospheric field tests are not aggressive enough to rank the duplex grades. Still it should be noted that for DX 2205 one anomalous case of stress crack initiation was observed. The crack was reportedly trans-granular and assisted by selective dissolution of the ferrite [16]. None of the other five specimens showed any sign of cracking.

Table 2 Summary of the test results for the Wick test.

Material	Number of specimens	
	Tested	Failed due to SCC
304L	5	5
316L	4	4
LDX 2101	6	0
DX 2304	6	0
LDX 2404	7	0
DX 2205	6	1

Results of climate chamber tests are summarised in Table 3 [9]. Numerous tests at various temperatures, relative humidity (RH) and different types of salt deposits showed that 304L and 316L were prone to SCC. The primary mode of corrosion degradation for LDX 2101, DX 2304 and LDX 2404 was found to be selective dissolution of the ferrite phase. However, metallographic investigation revealed micro-cracks in all of the tested duplex specimens. The authors suggested that micro-cracks can accelerate the selective dissolution of the ferrite by providing an additional transport path for the electrolyte to the underlying ferrite grains. The proposed designation of such a mechanism was stress-assisted selective corrosion. Unexpectedly, two of the DX 2205 specimens showed evident stress corrosion cracks when tested at the most aggressive conditions (70°C, 30% RH, $MgCl_2$ & $CaCl_2$ deposits). The

tentative explanation put forward by the authors was that SCC and selective corrosion are two competing mechanisms and due to higher corrosion resistance of DX 2205 it suffered SCC when the other duplex grades corroded selectively [9].

Table 3 Average frequency of occurrence for different types of corrosion for ring welded specimens. A total of 8 weeks exposure in climate chamber with chloride deposits, test data from [9].

Material	Ring area no.1						Ring area no. 2					
	% No corrosion	% Etching	% Pitting	% Selective corrosion	% SCC	% of conditions with micro-cracks	% No corrosion	% Etching	% Pitting	% Selective corrosion	% SCC	% of conditions with micro-cracks
304L	6	6	39	-	**48**	N/A	10	19	26	-	**45**	N/A
316L	19	6	39	-	**35**	N/A	29	10	29	-	**32**	N/A
LDX 2101	6	31	3	59	**0**	31	0	53	6	41	**0**	15
DX 2304	16	29	3	52	**0**	10	26	29	0	45	**0**	3
DX 2205	25	28	13	28	**6**	9	42	19	3	35	**0**	0

The same work also used the constant load test in combination with cyclic salt spray in a climate chamber to evaluate the tendency of SCC for duplex LDX 2101, DX 2304 and DX 2205, as well as 304L and 316L as reference [9]. Welded tensile specimens were used in this study. With this approach, the authors were able to provoke SCC for 304L test specimens at both 50°C and 70°C. However, the 316L only exhibited pitting corrosion and the duplex grades mostly suffered selective ferrite dissolution with some micro-crack initiation at the HAZ in some cases.

Another candidate test method to rank duplex grades with respect to SCC resistance is the drop evaporation test (DET). For DET the minimum stress to failure is defined as the lowest stress level at which a material fails due to SCC. Figure 4 and Figure 5 show the stress to failure expressed relative to the yield stress (% of $Rp_{0.2}$ at 200°C) and in absolute terms (MPa), respectively, for selected duplex grades. As expected, the austenitic grades 304 and 316 were very sensitive to SCC, showing a very low stress to failure (<10% of $Rp_{0.2}$). Due to the much higher mechanical strength of the duplex, the difference in SCC resistance is most striking when comparing the absolute stress values in Figure 5. Despite a higher PRE the minimum stresses to failure for LDX 2404 and DX 2205 were significantly lower (20 to 30% of $Rp_{0.2}$) compared to the lower alloyed LDX 2101 and DX 2304 (50% of $Rp_{0.2}$). The highest alloyed duplex grade, SDX 2507, showed the highest stress to failure.

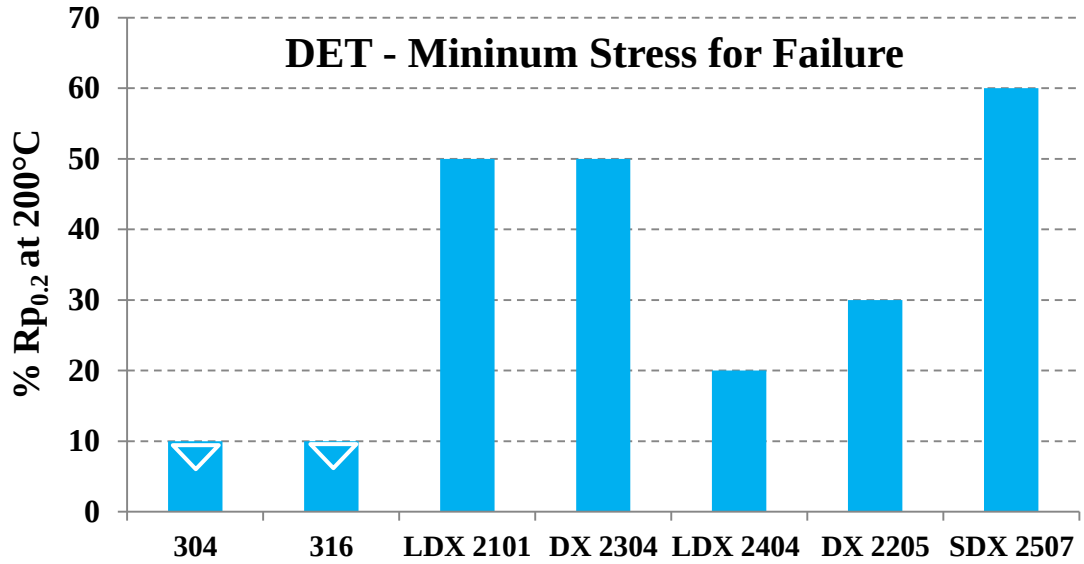

Figure 4 The minimum stress to failure of the tested stainless steels expressed in % $Rp_{0.2}$ at 200°C. At least one test specimen cracked at this load level.

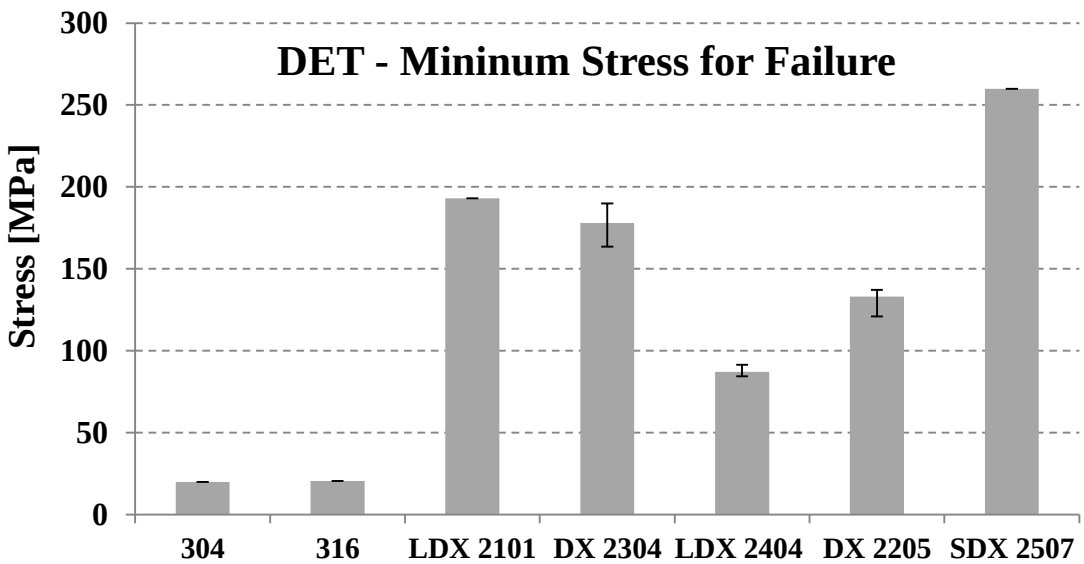

Figure 5 The minimum stress to failure of the tested stainless steels expressed in MPa, meaning that at least one test specimen cracked at this load level. The error-bars indicate the scatter due to the variation of the tensile strength between different parent materials (heats).

The lower alloyed duplex grades LDX 2101 and DX 2304 exhibited more general corrosion compared to the higher alloyed grades LDX 2404, DX 2205 and SDX 2507. In some cases rupture of LDX 2101 and DX 2304 specimens was caused by significant reduction in cross sectional area due to excessive general corrosion, as exemplified by Figure 6. The cross sectional micrograph in Figure 7 clearly shows that the ferritic phase is selectively attacked on an LDX 2101 specimen. Similar type of selective corrosion has also been seen for grade DX 2304 [17].

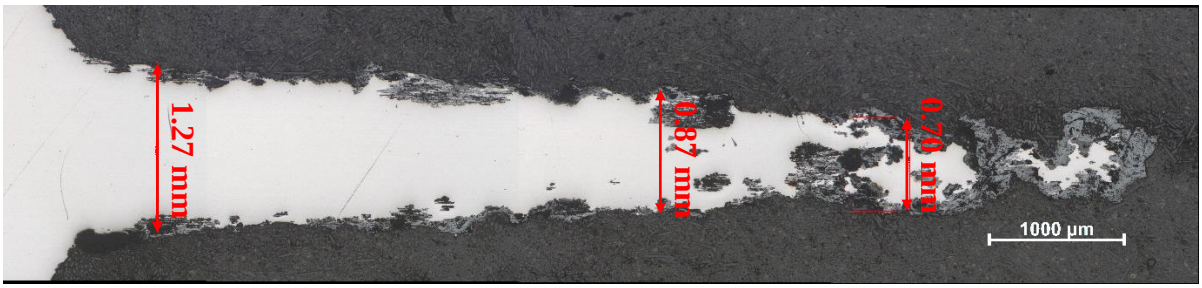

Figure 6 Optical cross sectional micrograph of a failed LDX 2101 DET specimen with general corrosion causing heavy reduction of the cross sectional area.

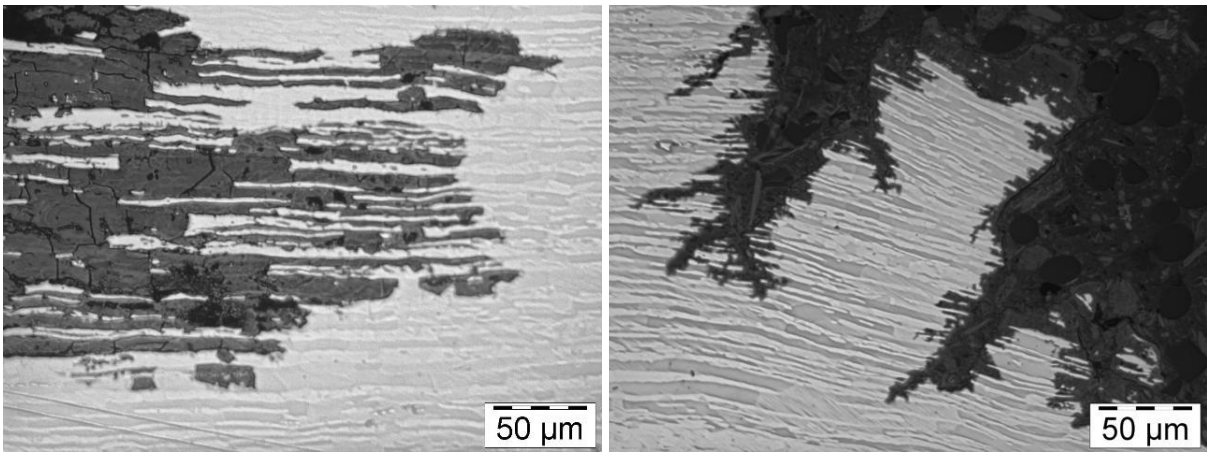

Figure 7 Optical micrographs showing selective corrosion of the ferritic phase on an LDX 2101 DET specimen.

The abundant selective dissolution seen for LDX 2101 and DX 2304 is likely to intervene with the mechanisms of the SCC, resulting in an inhibiting effect. Due to this inhibiting effect the minimum stresses to failure for LDX 2101 and DX 2304 are inflated, thus the DET results do not reflect the actual SCC resistance. On the other hand, the higher alloyed materials LDX 2404, DX 2205 and SDX 2507 have a higher resistance to pitting. The higher corrosion resistance of these grades limits selective corrosion, making the SCC the dominant corrosion mechanism. As a consequence, the drop evaporation test seems more suitable for ranking higher alloyed duplex grades, whereas for lower alloyed duplex grades the results should be evaluated with caution due to inhibiting effect of the extensive selective corrosion on SCC. For the austenitic steels 304 and 316 on the other hand, the low minimum stress to failure and relatively short times to failure lead to conclusion that SCC is the predominant corrosion mechanism, as opposed to pitting corrosion.

Looking at the times to failure for the materials, it is evident that there is some significant variation, even at the same stress level. For instance, six out of eight DX 2304 specimens tested at the minimum stress to failure (50% of $Rp_{0.2}$) failed within 118-470 test hours, but two specimens did not crack after 500 hours. There are several potential causes for the scatter, among these is the statistical nature of the SCC itself, which gives an inherent scatter of the test results. Also, the relatively small cross sectional area of the specimens make them more susceptible to material defects such as inclusions, rolled-in oxides etc. In addition, test conditions might vary slightly each time since some initial adjustments might change during the course of the test (ambient temperature, salt crust formation, fluctuating drop rate etc.).

In an excellent in-depth review of the drop evaporation test by Hitch [18], it is emphasised that stress-corrosion of stainless steel under evaporative conditions is a complex process.

Hitch also highlights some of the essential aspects that have hitherto received little to no attention in the DET related literature. The most important is the dynamic load contribution due to cyclic thermal expansion/contraction, which may play some role in crack initiation. Other factors are the importance of salt crust formation and its influence in the overall corrosion mechanism, as well as the impact of salt mixes (e.g. seawater) and its implications on concentration process, salt crust formation and control of test variables.

Some studies employing slow strain rate testing (SSRT) in combination with cyclic salt spray has also been carried out [14, 15]. An advantage of the cyclic salt spray over the dripping procedure in DET is that contact between the specimen and the solution is more even and well defined. The SCC sensitivity evaluated in 1% NaCl cyclic spray is shown in Figure 8 for DX 2304, DX 2205, SDX 2507 and the austenitic stainless steels 304, 310, and 316L.

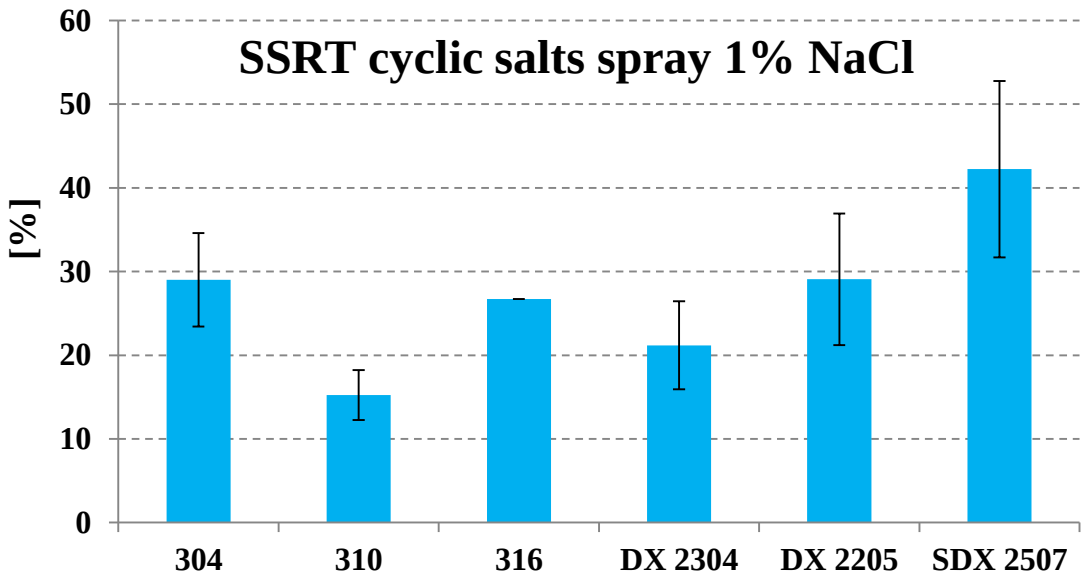

Figure 8 SCC sensitivity evaluated as ratio of failure elongation in 1% NaCl to failure elongation under inert conditions. Results from [15].

From the overall SCC sensitivity values it is apparent that all of the tested materials are sensitive to SCC under these evaporative conditions. For the duplex grades the SCC resistance seems to increase with increasing PRE. Although the difference in SCC sensitivity between DX 2304 and DX 2205 is not large, the SDX 2507 is distinctly more resistant. However, it is very surprising that there is such small difference between the austenitic grades (304, 310 and 316) and the expectedly much more SCC resistant duplex grades. This could perhaps be related to the fact that the dynamic loading in SSRT, in combination with the aggressive environment, leads to perhaps unrealistically severe test conditions [19]. The cyclic 1% NaCl salt spray causes the specimen temperature to fluctuate between 90°C and 150°C, as well as causing high chloride build-up due to evaporation [14]. These test conditions are even more aggressive than in DET, where the temperature is somewhat lower and fluctuates between 70°C and 120°C [17]. The wet-dry cycle is however much shorter – 6 seconds, compared to 20 seconds for the SSRT with cyclic salt spray. It is also worth noting from results in Figure 8 that the higher alloying levels of chromium and nickel did not make the austenitic grade 310 more resistant compared to 304 and 316, in fact quite the contrary. The cause of this is unclear and needs further elucidation.

The effect of austenite level on the SCC sensitivity has been investigated by studying a series of tie-line alloys (0% to 98% austenite) for duplex grade DX 2205 [15]. These alloys had the same individual phase compositions as for DX 2205 but different proportions of the two phases. The SCC sensitivity of the tie-line alloys in 1% NaCl cyclic salt spray SSRT is shown in Figure 9.

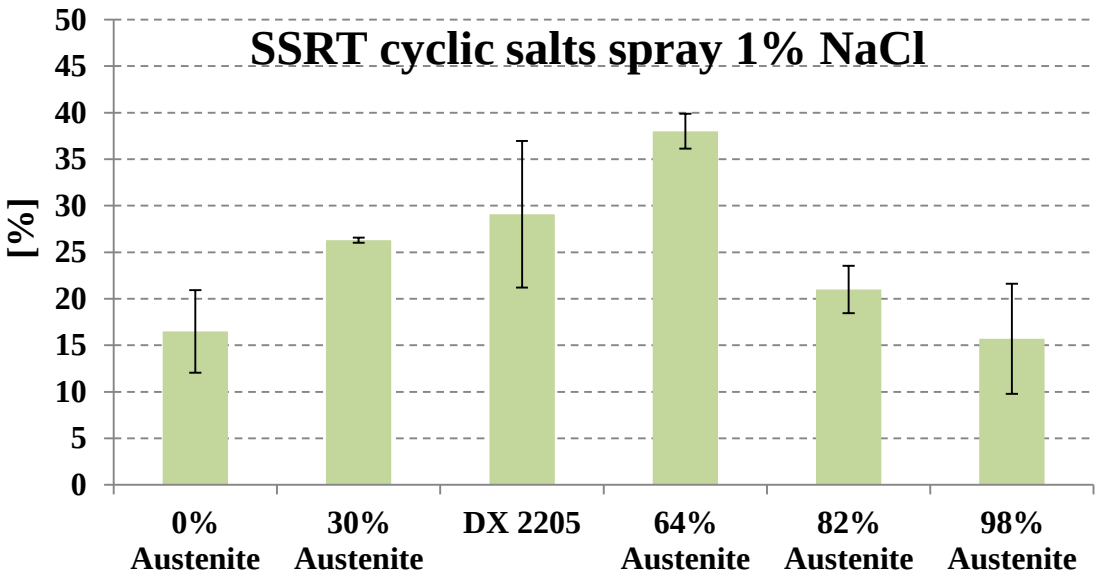

Figure 9 SCC sensitivity evaluated as ratio of failure elongation in 1% NaCl to failure elongation under inert conditions. Results from [15].

The results in Figure 9 indicate that the most extreme austenite levels (0% and 98%) are the most sensitive to SCC. The highest resistance is seen at 64% austenite, which is somewhat above the normal ≈50% level for commercially produced DX 2205 material. This clearly demonstrates a beneficial effect of duplex microstructure on the SCC resistance, compared to a single phase structure. Nonetheless, it is surprising that the single phase DX 2205 tie-line alloys have low SCC resistance, especially in the case of the ferritic alloy. Comparing to Figure 8, it seems that the SCC resistance of the austenitic DX 2205 tie-line alloy is significantly lower than the 304 and 316 austenitic grades, and on a par with 310. A metallographic investigation indicated normal microstructure for the tie-line alloys [15]. In addition, other study in boiling magnesium chloride with same austenitic alloy did not yield such poor results [20]. This indicates that the austenitic single phase alloy is more sensitive under evaporative conditions.

Considering the different evaporation test methods discussed above it is evident that choosing one test method to rank stress corrosion susceptibility of the whole range of duplex grades, from lean duplex to super duplex, is indeed very challenging. All of the investigated accelerated laboratory tests bear evidence of selective dissolution of the ferrite as being a key phenomenon complicating the evaluation of SCC resistance of duplex grades under evaporative conditions. The selective corrosion and chloride induced SCC are two competing mechanisms and when selective corrosion dominates, it seems to inhibit the SCC. This is perhaps most evident when looking at the DET results, but also for Wick test and chloride deposit tests, where a higher alloyed grade like DX 2205 shows SCC, but lower alloyed lean duplex grades show less tendency to cracking but suffer heavy selective corrosion. Figure 10 shows the apparent correlation between DET minimum stress to failure and PRE, as well as

the competing trends of selective corrosion and SCC. The cross-over point, where the SCC becomes the prevailing corrosion mechanism, appears to be around PRE 34 for LDX 2404.

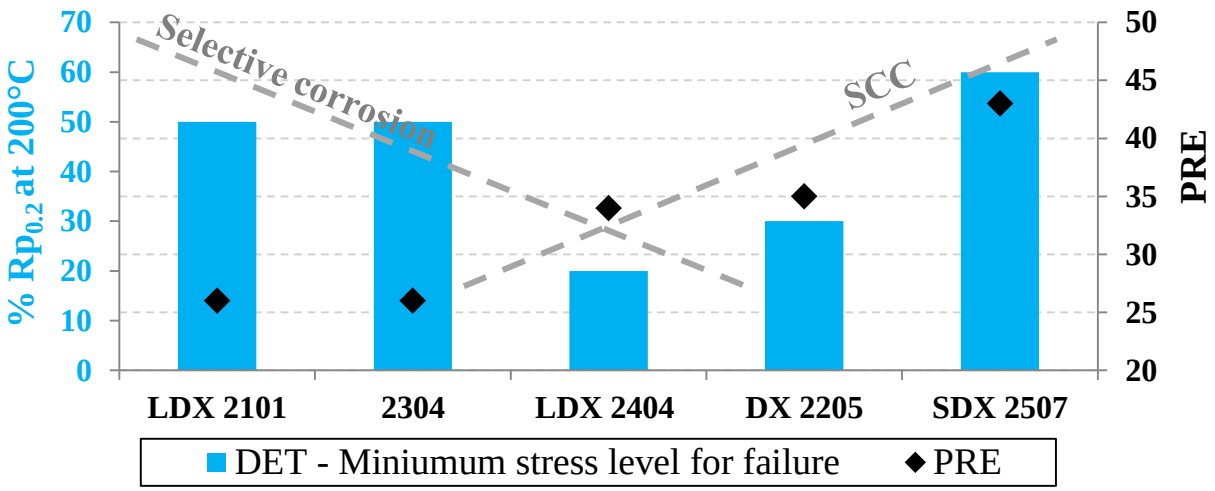

Figure 10 The PRE of the duplex grades correlated to the DET minimum stress to failure, also showing the competing trends of SCC and selective corrosion.

Worth a final comment is the potential major impact of surface preparation on the SCC. In a recent metallography study of stress corrosion cracks on DX 2205 and SDX 2507 drop evaporation test specimens, Wickström et al. [21] pointed out the importance of the surface condition. They found that stress corrosion micro-cracks clusters were formed only in the ferrite phase. Dealloying in the nanocrystalline layer (formed by surface grinding) and subsequent cracking of the dealloyed layer was considered a precursor to crack propagation [21]. This is not likely to be an issue for DET testing, for which all specimens were electropolished, but could be a contributory factor in other cases.

Conclusions

From the results presented in the current study and analysed literature data, the following conclusions can be drawn:

- Stress corrosion cracking under evaporative conditions is a complex process, which makes it very challenging to simulate such conditions in an accelerated laboratory test
- Very aggressive test conditions are necessary to induce SCC for duplex stainless steel in order to obtain a relative performance ranking
- The ferritic-austenitic microstructure makes the duplex stainless steels very resistant to chloride induced SCC, especially compared to 304 and 316-type austenitic grades
- Under evaporative conditions selective dissolution of the ferrite and SCC are two competing mechanisms. Selective corrosion can inhibit SCC for lower alloyed duplex stainless steels when it is the dominant corrosion mechanism
- Due to the crack-suppressing effect of selective corrosion, the drop evaporation test seems to be most appropriate for duplex grades with PRE $\geq$ 34. For lower alloyed duplex grades the results might be inflated and should be regarded with caution.

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