# The Role of Surface Texture on the Corrosion Fatigue Behavior of High Alloyed Stainless Steel Exposed to Saline Aquifer Water Environment

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**Abstract:** Corrosion fatigue specimen with different surfaces (technical surfaces after machining and polished surfaces) of high alloyed martensitic stainless steel X46Cr13 (1.4043) and duplex stainless steel X2CrNiMoN22-3-2 (1.4462) were compared at load amplitudes from 175 MPa to 325 MPa in the geothermal brine of the Northern German Basin at 98 °C. Surface corrosion layers and pits reveal carbonate corrosion products on the surface such as FeCO<sub>3</sub> and FeOOH as the main precipitation phases with no dependence on the original surface roughness. At high stress amplitudes above 275 MPa technical surfaces (P50% at  $\sigma_a$  300 MPa=5×10<sup>5</sup>) resulted in more cycles to failure than polished (P50% at  $\sigma_a$  300 MPa=1.5×10<sup>5</sup>). The greater slope coefficient for technical surfaces k = 19.006 compared to polished surfaces k=8.78 demonstrates earlier failure at given stress amplitude  $\sigma_a$ .

Key words: corrosion, CCS, carbon storage, aquifer, heat treatment, high alloyed steel.

## 1. Introduction

During the injection of CO<sub>2</sub> into deep geological saline aquifer reservoirs (carbon capture and storage CCS) and in geothermal energy production as found in the Northern German Basin [1], the CO<sub>2</sub> is dissolved in the aquifer water building a highly corrosive environment easily causing the failure of pipe steeks and components [2]. A FeCO<sub>3</sub> corrosion layer (siderite) grows on the alloy surface as a result of the anodic iron dissolution of the pipe steel [2], [3]. Still, there is very little knowledge on the corrosion behavior of steeks in supercritical CO<sub>2</sub> [4] or in CO<sub>2</sub>-saturated saline aquifer waters [5].

1.4043 is a well-known martensitic stainless steel with 13% Cr and 0.46% C that is widely used in industry. Duplex stainless steel (DSS) X2CrNiMoN22-5-3 (1.4462, UNS S31803) is well known in industry for its excellent general corrosion resistance [6] and good resistance to stress corrosion cracking [7]. Because of its additional high strength this alloy is the most widely produced DSS today [8] and most promising in CCS environment and for future geothermal applications. The corrosion and corrosion fatigue behaviour has been reported earlier [3], [9], but not for CCS or geothermal environments.

Different authors show a direct dependence of the mechanical and corrosion behavior of various steel qualities on the surface roughness resulting from machining processes [10]-[17]. In general, the corrosion resistance increases with decreasing valley of depth on the surface for carbon steel [10], austenitic stainless steel [11], [12] after shot peeing [13] and ferritic stainless steel when  $R_a$  exceeded 0.5 µm [14].

For 316L austenitic stainless steel Zhang *et al.* [11] show that the machined surface with a high level of tensile residual stress is highly susceptible to SCC initiation. Crack number and density appeared a significant increase when the residual stress (190 MPa=76% of the yield strength [15]) surpassed the critical stress value, but on the other hand cracking is absent on the electropolished surface where the machined affected layer is removed and the residual stress is compressive [16]. Pitting corrosion resistance of the passive film formed on 2205 duplex stainless steel decreased with the increasing of strain in borate buffer solution with chloride ions after immersion test into borate buffer solution with chloride ions [17]. The corrosion rate of low carbon steel is proportional to the surface roughness parameter  $R_a$  for laminar and turbulent flow conditions and increases with increasing surface roughness [10]. However, the effect of relative humidity outweighs that of initial surface roughness [12].

Many authors correlate smooth surfaces and compressive surface stress with enhanced fatigue behavior in air [18], but also with corrosion resistance resulting in enhanced corrosion fatigue behavior [18]-[20]. Sanjurjo *et al.* [18] state that the surface finish of AISI 2205 stainless duplex steel was the critical factor for the enhancement of the fatigue behavior. 90% of the total fatigue improvement are due to the elimination of surface stress raisers and the improvement of the product roughness and only 10% is due to the compressive residual stress layer induced by shot peening. However, residual stress was correlated anti-proportional to the corrosion resistance. Smooth surfaces, deeper plastic deformations good corrosion resistance resulted in good fatigue performance under corrosive environment for AA5083 [19] and ASTM A533B [20].

The influence on environmental factors, e.g. the composition of surrounding media and alloy, temperature, CO<sub>2</sub> partial pressure, flow conditions, contaminations and formation of protective scales have been reported by various authors [3], [21]. In this study the dependence of the corrosion fatigue behavior and endurance limit of 1.4462 and 1.4043 on the surface treatment was investigated in geothermal environment at 100 °C.

## 2. Experimental Procedure

#### 2.1. Materials

Corrosion fatigue tests were conducted with high alloyed steel 1.4043 (X46Cr13, AISI 420C) (Table 1) and duplex steel 1.4462 (X2 CrNiMoN 22-5-3, AISI A182 F51 (329LN)) (Table 2) manufactured by means of continuous casting followed by an appropriate tempering and water quenching allowing for an equilibrium of ferrite and austenite phase. This microstructure has empirically proven to avoid intermetallic phases guaranteeing optimal mechanical performance [8]. In order to confirm the material's chemical composition, samples were analyzed via spark emission spectrometry SPEKTROLAB M and by Electron Probe Microanalyzer JXA8900-RLn.

Table 1. Chemical Composition of 1.4043 (X46Cr13, AISI 420C), (in Mass Percent)

Elements	С	Si	Mn	Р	S	Cr	Мо	Ni	Со	Fe	
acc standard <sup>a</sup>	0.42-0.5	<1.00	≤ 1.00	≤ 0.045	≤ 0.03	12.5 - 14.5				0.20 - 0.45	
analysed $^{\rm b}$	0.46	0.25	0.45	0.018	0.003	13,39	0.03	0.13	0.03	85.4	

a) elements as specified according to DIN EN 10088-3 in %

b) spark emission spectrometry

Table 2. Chemical Composition of 1.4462 X2 CrNiMoN 22 5 3 (UNS S31803) (in Mass Percent)

phases	С	Si	Mn	Р	S	Cr	Мо	Ni	N
$\alpha \& \gamma^{**}$	0.023	0.48	1.83	0.024	0.008	22.53	2.92	5.64	0.146
α*	0.02	0.55	1.59	-	-	24.31	3.62	3.81	0.07
γ*	0.03	0.47	1.99	-	-	20.69	2.17	6.54	0.28

The PRE-Nummer PREN = %Cr + 3.3% Mo + 16% N [8] is 30.8-38.07, yield strength 672 MPa and tensile

strength 864 MPa in air. The high strength of 1.4462 results from fabrication process, microstructure and additional alloying elements, in particular nitrogen and [22]. Fig. 1 depicts the alloy's microstructure obtained via etching prior to exposure and fatigue testing.



Fig. 1. Microstructure of 1.4462 test specimens prior to exposure; left: 1.4542 after heat treatment, middle (a) transversal and right (b) longitudinal sections respecting the casting direction [8]. Etching: Beraha II.

To simulate in-situ geothermal condition the geothermal aquifer water of the Northern German Basin were synthesized in a strictly orderly way to avoid precipitation of salts and carbonates (Table 3).

Table 3. Chemical Composition of the Northern German Basin and Stuttgart Formation Electrolyte [1]	
According to Northern German Basin [8]	

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	NaCl	KCl	$CaCl_2 \times 2H_2O$	$MgCl_2 \times 6H_2O$	NH <sub>4</sub> Cl	ZnCl <sub>2</sub>	SrCl <sup>2+</sup> 6H <sub>2</sub> O	PbCl <sub>2</sub>	$Na_2SO_4$	pH value
g/L	98.22	5.93	207.24	4.18	0.59	0.33	4.72	0.30	0.07	5.4 - 6

## 2.2. Corrosion Fatigue Tests

The shape and dimensions of hourglass specimens (Fig. 2) were selected in accordance to the standard DIN EN ISO 11782-1 and to the recommendations of the FKM Research Issue [23]. The critical cross-section is 12.5 mm in diameter; the total contact surface area did not undergo 25 cm<sup>2</sup> in order to minimize the influence of irregularities [24].

The specimens were manufactured by means of precision turning without an additional surface finish in order to replicate the technological conditions. The machining of the surfaces was set to Rz=4. For comparison Polishing was done by first grinding with SiC-Paper from 180  $\mu$ m to 1200  $\mu$ m under water and then finishing with diamond paste 6  $\mu$ m, 3  $\mu$ m and 1  $\mu$ m. Considering the fact that surface finish quality may influence corrosion fatigue the verification of the surface roughness was accomplished via optical surface scanner FRT MicroProf 300 prior to corrosion fatigue experiments. The surface roughness was examined within the critical cross-section of the specimen. The scanner was operated in the 2D measuring mode, with the measurement length of 3 mm and an interval of 2.1  $\mu$ m between the measuring points. The surface roughness of the specimens varied from 2.4  $\mu$ m to 5.3  $\mu$ m for 1.4462, resulting in a mean arithmetic value of 3.2  $\mu$ m. Due to the rather heterogeneous fine machined surfaces these specimens are comparable with prefabricated parts. Surface roughnesses of polished specimens varied from 0.58  $\mu$ m to 2.01  $\mu$ m with a mean of 1.59  $\mu$ m.

The corrosion chamber is fixed directly onto the specimen leaving the resonant testing machine unaffected (Fig. 2) [8], [24]. It is equipped with measurement technique to obtain electrochemical data, temperature, the pH and the electrochemical potential during the mechanical tests using a special shock resistant silver-silver chloride electrode.

The duplex steel 1.4462 with an endurance limit in air of 485 MPa is examined in dynamic stress-strain

tests at 98 °C under CCS conditions of the synthetically produced aquifer solution according to the Northern German Basin without flowing CO<sub>2</sub> [8], [24]. Therefore a resonant testing machine (sinusoidal dynamic test loads, R=-1; resonant frequency ~ 30 Hz) has been used to test 9 specimens between 280 MPa and 390 MPa.



Fig. 2. Schematic set-up of operating corrosion chamber for in-situ corrosion fatigue testing [8], [24]. (clamping socket (1), union nut (2), corrosion chamber (3), clamping plate (4)).

#### 2.3. Analysis

X-ray diffraction was carried out in a URD-6 (Seifert-FPM) with CoK $\alpha$ -radiation with an automatic slit adjustment, step 0.03 and count 5 sec and AUTOQUAN (B) by Seifert FPM was used for phase analysis. For gravimetric measurement descaling of the samples was performed by exposure to 37% HCl for 24 hours and mass gain was analyzed according to DIN 50 905 part 1-4. To characterize surface corrosion SEM/EDX was performed using: Leo Gemini 1530 VP, acceleration voltage 15 kV. Non-descaled parts of the samples were embedded in a cold resin (Epoxicure, Buehler), cut and polished first with SiC-Paper from 180  $\mu$ m to 1200  $\mu$ m under water and then finished with diamond paste 6  $\mu$ m, 3  $\mu$ m and 1  $\mu$ m. The measurement of the layer thicknesses and residual pipe wall thickness as well as microstructure analysis were performed via light and electron microscopy techniques using the semi-automatic analyzing program Analysis Docu ax-4 by Aquinto.

## 3. Results and Discussion

The influence of surface roughness of the fatigue behavior is demonstrated in Fig. 3. Here the surface roughness of specimens of 1.4043 (X46Cr13) is shown as a function of the proportion of cycles to failure (P50 to P).



Fig. 3. Comparison of differently machined surfaces of 1.4043 (X46Cr13) in corrosive media [1] at 60 °C after machine turning (technical surface).

The higher the number on the x-axis the higher is the 50% probability of survival at a given stress amplitude. The numbers in the graph exhibit the stress amplitudes in N/mm<sup>2</sup>. Surface roughness of as-machined specimens varied between 3.6 and 4.7  $\mu$ m. As the main result there is no significant

dependence of the surface roughness after fine machining on the fatigue behavior. Although there are 2 specimens with very high P50 cycles to failure (orange square) there are as many specimens with similar surface roughness revealing higher or lower numbers of cycles to failure (blue square).

In general the endurance limit for specimen of duplex stainless steel 1.4462 (X2CrNiMoN22-5-3) cycled in air is higher by a factor of 2.5 (485 MPa with regard to a fatigue limit of 10<sup>7</sup> cycles ( $P_f$  = 50%, purely alternating push/pull load) compared to specimen cycled in saline aquifer water. The line of regression of specimens with technical surfaces has greater negative slope (steeper) (k=8.78) than that of specimen with polished surfaces (k=19.006). Comparing slope coefficients k reveals that lines of regression with small number of k decline greater than those with large numbers of k (Fig. 4).



Fig. 4. S-N curve of 1.4462 X2CrNiMoN22-5-3 in air and in corrosive media Northern German Basin at 98 °C after machine turning (technical surface) and polishing (polished surface).

However, in the corrosive environment of the Northern German Basin specimen with technical surfaces (average surface roughness  $3.2\mu$ m) perform better at stress amplitudes higher than 270 MPa than polished (average surface roughness  $1.59 \mu$ m). At stress amplitudes S<sub>a</sub> below 270 MPa polished surfaces reveal much higher numbers of cycles to failure (Fig. 4).

Although Sanjuro *et al* [18] clearly state, that the surface finishing accompanied with the removal of surface stress raisers contributes mostly to the enhanced corrosion fatigue behavior (whereas residual stress only accounts for 10%), one possible explanation of this surface dependent fatigue behavior is that at stress amplitudes above 275 MPa the surfaces of turned specimen (technical surface) underwent microstructural hardening processes during machining due to plastic deformation resulting in residual stress. That is: turning implements compressive stress surpassing the increased surface roughness.

Furthermore, the supercritical crack growth mechanism may change after exceeding stress amplitudes above 275 MPa. At stress amplitudes greater 275 MPa the push-pull load will lead to overall tensile stress in the specimen surface resulting in micro cracks (Fig. 5). Further cycles alternately open and close the crack exposing the crack flanks to the highly corrosive brine. Due to a low solubility limit in aqueous solutions (pKsp = 10.54 at 25  $\circ$  C [21], [25] siderite will form as a result from the transient phase precipitation of Fe(OH)<sub>2</sub> [25] and segregate. Further mechanical stress and the strain mismatch of corrosion layer and base

metal causes crack growth until failure. Because in general higher strength results in higher endurance limits, the higher surface strength due to compressive stress resulting from the machining process results in a higher number of cycles to fatigue for technical surfaces.

At 275 MPa the regression lines overlap indicating that at 275 MPa stress amplitude the surface condition has little to no influence on the corrosion fatigue behavior. Below 275 MPa the crack growth is not initiated by micro cracks but via pit corrosion. Initial pits first grow in dimension and after reaching a critical size and are then followed by the formation of a fatigue crack (Fig. 5) due to a notch effect [8] resulting in early failure. Therefore it is very likely that corrosion prior to micro cracking is primarily cause for fatigue failure under given experimental conditions. Because here pitting is reason for crack initiation and not micro cracks, a polished surface is preferable because it provides better resistance to corrosive attack and higher numbers of cycles to failure.



Fig. 5. Micro sections of major crack initiation (left) and major crack termination zone(right). 1D13X (380 MPa; 0.36·10<sup>6</sup> cycles).

For small stress amplitudes the surface roughness may be neglected. Pitting is the initiating process whereas at high stress amplitudes the formation of micro cracks is reason for crack initiation, propagation perpendicular to the laminar duplex structure and final failure. Note, there is no influence of the surface condition on the microstructure of the crack region and on the crack propagation; therefore there is no obvious dependence on the mechanism of failure.

At high stress amplitudes good corrosion fatigue performance will be achieved with technical surfaces. Below 275 MPa the surface roughness of engineering materials in geothermal and CCS environment should be kept as small as technically and economically possible to perform at high stress amplitudes.

## 4. Conclusion

Corrosion fatigue specimen with different surfaces (technical surfaces after machining and polished surfaces) martensititic stainless steel X46Cr13 (1.4043, AISI 420C) of duplex stainless steel X2CrNiMoN22-3-2 (1.4462) were compared at load amplitudes from 175 MPa to 325 MPa in the geothermal brine of the Northern German Basin at 98 °C.

The surface roughness (Rz=~3.2 for turned surfaces and Rz=~1.59 for polished surfaces) has large impact. For duplex stainless steel 1.4462 the equilibrium at 98 °C in the saline water of the Northern German Basin was 275 MPa. At high stress amplitudes technical surfaces are preferable to achieve high numbers of cycles to failure. At high stress amplitudes above 275 MPa technical surfaces (P50% at  $S_a$  300 MPa=5×10<sup>5</sup>) resulted in more cycles to failure than polished (P50% at  $S_a$  300 MPa=1.5×10<sup>5</sup>). The greater slope coefficient

for technical surfaces k = 19.006 compared to polished surfaces k=8.78 demonstrate earlier failure at given stress amplitude  $S_a$ . Rather low scatter ranges (technical surface: TN=1:1.35, polished surface: TN=1.1.95) indicate no change in failure mechanism. Still, it may be assumed that at low stress pitting is the initiating crack growth process whereas at high stress amplitudes the formation of micro cracks is reason for crack propagation and failure. Residual compressive stress enhances fatigue life at high stress amplitudes

With no regard to the corrosion mechanism, whether corrosion leads to crack initiation or micro cracking leads to enhanced corrosion, at stress amplitudes >275 MPa or very small stress amplitudes, good corrosion fatigue performance will be achieved with technical surfaces in CCS environment.

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