The Influence of ppb Levels of Chloride Impurities on the Stain-Induced Corrosion Cracking and Corrosion Fatigue Crack Growth Behavior of Low-Alloy Steels under Simulated Boiling Water Reactor Conditions

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Abstract

The effect of chloride on the strain-induced corrosion cracking (SICC) and corrosion fatigue (CF) crack growth behaviour in low-alloy reactor pressure vessel steels was evaluated under simulated boiling water reactor normal water chemistry conditions by slow rising load and cyclic constant load amplitude tests with air fatigue pre-cracked fracture mechanics specimens. Chloride in the ppb level range increased the SICC initiation susceptibility, but had almost no effect on the subsequent SICC and CF crack growth. A strong effect of chloride addition of 100 ppb on CF crack growth was observed at intermediate corrosion potentials and very low loading frequencies only.

Key words: Low-alloy steel, strain-induced corrosion cracking, corrosion fatigue, chloride, reactor conditions

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0 Abbreviations

AL	Action level of EPRI water chemistry guidelines
ASTM	American Society of Testing and Materials Standards
ASTM E399	Test method for plane-strain fracture toughness of metallic materials
BWR	Boiling water reactor
CF	Corrosion fatigue
CL	Constant load
<i>COD_{LL}</i>	Crack opening displacement at load line
C(T)	Compact tension specimen
DCPD	Direct current potential drop crack length measurement method
<i>DH</i>	Dissolved hydrogen (content)
<i>DO</i>	Dissolved oxygen (content)
DSA	Dynamic strain ageing
EAC	Environmentally-assisted cracking
<i>ECP</i>	(Free) electrochemical corrosion potential
ENSI	Swiss Nuclear Safety Inspectorate, Switzerland
EPRI	Electric Power Research Institute
F&A	Ford & Andresen (EAC model)
FRAD	Film rupture anodic dissolution crack growth mechanism
GE GR	General Electric Global Research
HAZ	Weld heat-affected zone
HWC	Hydrogen water chemistry
LWR	Light water reactor
NWC	Normal water chemistry
ppb	Parts per billion by weight, 1 ppb = 1 µg/kg
ppm	Parts per million by weight, 1 ppm = 1 mg/kg
PSI	Paul Scherrer Institut, Switzerland
PWHT	Post-weld heat treatment
PWR	Pressurized water reactor
Q	Quenched
R	Load ratio
RPV	Reactor pressure vessel
SCC	Stress corrosion cracking
SEM	Scanning electron microscope
SICC	Strain-induced corrosion cracking
SHE	Standard hydrogen electrode
SR	Stress-relieved
SRL	Slow rising load test
T	Tempered or temperature
YS	Yield stress

1 Introduction

Carbon and low-alloy steels and their associated weld filler metals are widely used for pressure vessels and piping in both the primary and secondary coolant circuits of light water reactors (LWR), as well as in service water systems [1]. The main reasons for their use are their combination of relatively low cost, good mechanical strength and toughness properties in thick sections (hardenability) and good weldability, as well as their good stress corrosion cracking (SCC) resistance in primary coolant environments [1]. The pressure-boundary components, in particular in the primary circuit such as the reactor pressure vessel (RPV), are very critical systems with regard to plant safety and lifetime (extension).

The formation and growth of cracks by environmentally-assisted cracking (EAC) is a potential ageing and degradation mechanism for these components [1-3] and an ongoing research topic since more than three decades [1-31]. The different basic types of EAC are summarized and explained in Table 1 [1]. The operating experience of carbon and low-alloy steel primary pressure-boundary components in LWRs is very good worldwide [1-3]. However, isolated instances of EAC have occurred, particularly in boiling water reactor (BWR) service, most often in piping and more rarely in the RPV itself [1-3]. Oxidizing conditions (usually dissolved oxygen) and relevant plastic straining were always involved. Most cracking incidents in pressurized water reactors (PWR) involved aerated feedwater under specific start-up operating conditions (no application of nitrogen blankets in condensate storage tanks) or at special locations (e.g., locations with air present during shut-down), where the normally low contents of oxygen during operation cannot be assumed upon start-up. All these cases were attributed either to strain-induced corrosion cracking (SICC) or very low-frequency/very low-cycle corrosion fatigue (CF) during plant transients like start-up/shut-down or hot stand-by and can be rationalized by the mechanistic [1, 2, 4, 8, 27, 28] and experimental background knowledge on SCC [9, 13, 17, 18, 21-27] and SICC / CF [5-12, 14-16, 19, 20, 27-30]. Both service experience [1-3] and appropriate laboratory experiments [1-31] confirm the high resistance of carbon and low-alloy steels to SCC under static loading conditions in high-purity water (representative of steady-state power operation) and clearly reveal that slow, positive (tensile) straining, with associated (localized) plastic yielding and sufficiently oxidizing conditions are usually necessary for EAC initiation in high-temperature water.

Table 1: Basic types of EAC in carbon and low-alloy steels in high-temperature water [1].

Mechanism	Environmentally-assisted cracking (EAC)		
	SCC <u>S</u> tr <u>e</u> ss <u>c</u> or <u>r</u> u <u>s</u> i <u>o</u> n <u>c</u> rack <u>i</u> ng	SICC <u>S</u> tr <u>a</u> in- <u>i</u> nduced <u>c</u> or <u>r</u> u <u>s</u> i <u>o</u> n <u>c</u> rack <u>i</u> ng	CF <u>C</u> or <u>r</u> u <u>s</u> i <u>o</u> n <u>f</u> atigue
Type of loading	Static	Slow monotonically rising or very low-cycle	Cyclic: low-cycle, high-cycle
LWR operation condition	Transient-free, steady- state power operation	Start-up/shut-down, thermal stratification	Thermal fatigue, thermal stratification, ...

LWRs operate with high-purity water and during stationary BWR operation and the concentrations of harmful anionic impurities for SCC in the reactor coolant like sulphate and chloride are usually less than 1 ppb (ppb: parts per billion by weight, 1 ppb = 1 µg/kg). Variations in water chemistry may periodically (e.g., conductivity/impurity and oxygen/*ECP* transients during start-up/shut-down) or occasionally (ion exchanger resin intrusions or condenser leakages during steady-state power operation) occur in nuclear

power plants. Some of these transients are also related to temperature and pressure changes (e.g., start-up/shut-down) and thus mechanical load changes with an increased SICC concern. Nowadays the extent (magnitude, period) and frequency of such water chemistry variations are strongly minimized, e.g., by following the current BWR Water Chemistry Guidelines (Table 2) of the Electric Power Research Institute (EPRI) [32]. Nevertheless, the possible effect of short-term water chemistry transients on the transition EAC crack growth behaviour during, and in particular after a transient under subsequent steady-state power operation is of great interest for safety assessments. In the past, the focus was placed on the effect of water chemistry transients on SCC in stainless steels [33-37] and these investigations clearly revealed that low concentration levels of sulphate and chloride (> 5 to 10 ppb) can result in significant acceleration of SCC crack growth in BWR normal water chemistry (NWC) environment.

Table 2: EPRI Water Chemistry Guideline recommendations for the reactor water during stationary BWR power operation for normal water chemistry (NWC) conditions [32].

Control parameter	Median value	Action levels (AL)		
		1	2	3
Conductivity ($\mu\text{S/cm}$)	0.095	> 0.3	> 1.0	> 5.0
Chloride (ppb)	0.3	> 5	> 20	> 100
Sulphate (ppb)	1.0	> 5	> 20	> 100
Allowed time for reduction of parameters below AL (h)	-	96	24	0
Mitigation action, if values are still above AL	-	Programme & schedule for corrective measures	Orderly unit shut-down	Prompt unit shut-down

In recent investigations by Paul Scherrer Institut (PSI) [23-25] and General Electric Global Research (GE GR) [26], a tremendous effect of small amounts of chloride in the ppb concentration range was observed on the SCC growth behaviour of low-alloy RPV steels in simulated, highly oxidizing BWR NWC environment. In this environment, even small amounts of chloride (≥ 3 ppb) may result in acceleration of the SCC crack growth by one to three orders of magnitude after rather short incubation periods of a few hours [25]. The accelerated, fast SCC crack growth rates in the range of 10^{-9} m/s to 10^{-8} m/s hereby approached the high-sulphur SCC crack growth curve of the Ford & Andresen model [27, 28]. At high corrosion potentials ($ECP \geq \sim +100$ mV_{SHE}), fast SCC crack growth occurred down to low stress intensity factors of 30 and 20 MPa $\cdot\text{m}^{1/2}$ for 5 and 10 ppb of chloride, respectively. Tests with temporary chloride transients revealed possible long-term effects with accelerated SCC rates above the low-sulphur SCC crack growth curve of the Ford & Andresen model after prolonged (≥ 200 h) and severe (≥ 15 ppb) chloride transients [25]. In reducing BWR hydrogen water chemistry (HWC) environment, on the other hand, a much higher chloride tolerance was observed, where even 100 ppb of chloride were not sufficient to induce SCC below 60 MPa $\cdot\text{m}^{1/2}$. The main reason for the strong “anomalous” effect of chloride on SCC crack growth in simulated BWR/NWC is the very low SCC crack growth susceptibility at K_I -values < 60 MPa $\cdot\text{m}^{1/2}$ and the difficulty to sustain plastic crack-tip straining under these conditions [25].

To evaluate the effect of chloride on SICC and CF crack growth behaviour in BWR NWC environment, slow rising load and cyclic constant load amplitude tests were performed with air fatigue pre-cracked fracture mechanics specimens in oxygenated high-temperature water with various chloride concentration levels ranging from 5 to 100 ppb. The SICC and CF behaviour in chloride-free high-temperature water and the applied experimental procedure is described in detail in Ref. [29] and [30], respectively. This paper summarizes the results of the chloride test and gives a tentative mechanistic interpretation. Related PSI investigations on the effect of chloride on EAC initiation from smooth surfaces are discussed in a different paper [31].

2 Materials and Experimental Procedure

2.1 Materials and Specimens

Two low- and two high-sulphur low-alloy nuclear RPV steels were investigated (Tables 3 and 4) [29, 30]. The materials investigated are characteristic for RPVs of Western light water reactors. All materials were quenched and tempered. Some steels were post-weld heat-treated or stress relieved. The RPV steels had a granular, bainitic (alloy A, C, E) or a mixed bainitic/ferritic-pearlitic structure (alloy B) with an average former austenitic grain size of 10 to 20 μm . The spatial distribution and morphology of the MnS inclusions was fairly homogeneous and similar in alloys A to C covering the range from small spherical to large (up to a few 100 μm) elongated inclusions. Alloy E revealed distinct banded sulphur segregation zones with large clusters of MnS inclusions.

25 mm thick 1T C(T) compact tension specimens according to ASTM E399 were used for most experiments. Slow rising load tests with chloride addition were performed with 12.5 mm thick 0.5 C(T) specimens. The specimens were manufactured from the forged ingots or hot-rolled steel plates mainly in T-L or L-T orientation. The specimens were pre-cracked to an initial crack length to ligament ratio a_0/W of ~ 0.4 by fatigue in air at room temperature, using a load ratio R of 0.1. The maximal K_I at the final load step was $\leq 15 \text{ MPa}\cdot\text{m}^{1/2}$.

Table 3: Overview on investigated low-alloy RPV steels (Q = quenched, T = tempered, SR = stress relieved, PWHT = post-weld heat treated, YS = yield stress, DSA = dynamic strain ageing).

Materials		S [wt.%]	Heat treatment	Microstructure	YS _{288 °C} [MPa]	DSA- susceptibility
20 MnMoNi 5 5	A	0.004	Q+T	Bainitic	418	high
SA 508 Cl. 2	B	0.004	Q+T	Bainitic/ferritic- pearlitic	396	high
SA 533 B Cl. 1	C	0.018	N, Q+T, SR	Bainitic	412	medium
20 MnMoNi 5 5	E	0.015	Q+T, PWHT	Bainitic	438	medium

Table 4: Chemical composition in wt.% of investigated RPV steels.

Material		C	Si	Mn	P	S	Cr	Mo	Ni	V	Al	Cu
20 MnMoNi 5 5	A	0.21	0.25	1.26	0.004	0.004	0.15	0.5	0.77	0.008	0.013	0.06
SA 508 Cl. 2	B	0.21	0.27	0.69	0.005	0.004	0.38	0.63	0.78	0.006	0.015	0.16
SA 533 B Cl. 1	C	0.25	0.24	1.42	0.006	0.018	0.12	0.54	0.62	0.007	0.03	0.15
20 MnMoNi 5 5	E	0.26	0.32	1.44	0.016	0.015	0.15	0.61	0.63	0.02	0.029	0.17

2.2 Experimental Procedure

The SICC and CF tests were performed in 101 stainless steel autoclaves with integrated electromechanical loading systems, which were attached to sophisticated refreshing high-temperature water loops [30]. During the experiments all important mechanical (load, pull rod stroke) and environmental parameters (dissolved oxygen (DO), dissolved hydrogen (DH), conductivity (κ), temperature (T), pressure, flow, etc.) were recorded continuously. The ECP of the specimens and the redox potential (platinum probe) were monitored by use of a $\text{Cu}/\text{Cu}_2\text{O}/\text{ZrO}_2$ -membrane reference electrode. Ionic impurities of the water (inlet and outlet) were analysed by Inductive Coupled Plasma – Atomic Emission Spectroscopy and Ion Chromatography.

Two air fatigue pre-cracked C(T) specimens were simultaneously tested in a “daisy chain”. The crack advance was continuously monitored using the reversed direct current potential drop (DCPD) method

with a resolution limit of about 1 μm . Crack length was calculated by the Johnson equation and linearly corrected by post-test fractography [29, 30]. The error of DCPD in case of even crack growth was usually smaller than $< 5\%$. The stress intensity factor K_I values were calculated according to ASTM E 399 by the measured load and the actual corrected crack length a .

The determination of the crack initiation point and the stress intensity factor $K_{I,i}$ at the onset of SICC in the slow rising load tests is described in Figure 1 [28]. The SICC initiation point in the slow rising load tests was determined at the point, where the potential drop vs. COD_{LL} curve started to deviate from the linear behavior and roughly corresponds to a small mean SICC crack advance over the specimen thickness of a few μm . This point was derived graphically at high zoom after performing a linear regression fit for the linear part and a polynomial fit for the following part of the potential drop vs. COD_{LL} curve. The corresponding potential drop was assigned to the initial crack length a_0 determined by post-test fractography and used to calculate the final crack length by the Johnson equation. The initial crack length a_0 and load at the point of initiation P_i was used to calculate the stress intensity factor $K_{I,i}$ at SICC initiation according to ASTM E 399. In previous PSI investigations, the crack mouth opening displacement $CMOD_{LL}$ was measured by internal clip gauges (MTS 632.10F-01) [29]. In the current tests, the pull rod stroke corrected by the measured elastic compliance of the load train and compliance equation of ASTM E1820-13 for C(T) specimens was considered to be load line crack opening displacement COD_{LL} of the specimen.

After the tests, all specimens were broken open at liquid nitrogen temperature for detailed fractographical analysis in the scanning electron microscope (SEM). Examples of the typical fracture surface appearance for SICC, CF and SCC from PSI tests and a detailed summary of metallo- and fractographical observations are given in Section 7 in Ref. [27].

Figure 1: Schematic of DCPD signal (potential ΔU or resistance ΔR change vs. crack opening displacement at load line COD_{LL}) during slow rising load tests. The transition from region II to region III corresponds to the point of SICC initiation. The stress intensity factor $K_{I,i}$ is calculated by the corresponding load P_i and initial pre-crack length a_0 .

The BWR NWC conditions were simulated with high-purity water (inlet conductivity $\kappa_{25^\circ\text{C}} = 0.055 \mu\text{S/cm}$) at a temperature of 274 or 288 $^\circ\text{C}$ under low-flow conditions (4 to 5 autoclave exchanges per hour) with a local fluid velocity of some few mm/s. The ECP (DO content) was varied

between $-100 \text{ mV}_{\text{SHE}}$ (0.05 ppm DO , ppm: parts per million by weight, $1 \text{ ppm} = 1 \text{ mg/kg}$) and $+150 \text{ mV}_{\text{SHE}}$ (8 ppm DO). A DO content of 0.4 ppm ($\text{ECP} = +50 \text{ mV}_{\text{SHE}}$, redox potential $= +250 \text{ mV}_{\text{SHE}}$) represents the total oxidant concentration in the reactor water during BWR steady-state power operation in a realistic way. The increased DO value of 8 ppm ($\text{ECP} = +150 \text{ mV}_{\text{SHE}}$, redox potential $= +290 \text{ mV}_{\text{SHE}}$) was applied to achieve a realistic ECP for a surface crack penetrating the stainless steel cladding on the RPV wall. Depending on DO level and loop system, the outlet conductivity varied between $0.06 \mu\text{S/cm}$ (0.05 to 0.4 ppm DO) and 0.07 to $0.09 \mu\text{S/cm}$ (2 to 8 ppm DO). The slightly higher outlet conductivity was mainly caused by chromate release from the corrosion of the stainless steel pipings and autoclave.

A simplified schematic of the applied test procedure in the tests with continuous chloride addition is shown in Figure 2. One day after the heating-up of the autoclaves, 5 to 100 ppb of chloride, in terms of NaCl or HCl , were added to the high-purity water for the whole remaining test duration. The chloride addition was controlled by the conductivity in the inlet water and periodically analysed by ion chromatography. An excellent correlation between calculated theoretical and measured chloride values was observed. Since the observed EAC behaviour for a given chloride content was identical in line with high-temperature water crevice chemistry models, the nature of the added chloride species is not further specified in the following Sections (at 288°C , both conductivity and pH are dominated by the (increased) dissociation of water and are similar for high-purity water and water with NaCl and HCl in the ppb concentration range).

Figure 2: Simplified schematic of SICC and CF tests with continuous chloride addition.

The specimens were then in-situ pre-oxidized for one week at a small constant pre-load with a stress intensity factor K_I between 2 and $18 \text{ MPa}\cdot\text{m}^{1/2}$. A fully developed mature chloride-enriched occluded crack crevice chemistry prevails after the pre-oxidation period. The loading of the specimens in slow rising load tests was performed by using a constant rate of the pull rod stroke up to a final $K_{I,\text{max}}$ of ~ 50 and $\sim 70 \text{ MPa}\cdot\text{m}^{1/2}$ for 0.5 and 1T C(T) specimens, which roughly corresponds to the limit of small scale yielding. The loading in the chloride tests was done in the loading rate range that revealed the highest SICC susceptibility in previous investigations ($d\text{COD}_{\text{LL}}/dt \sim 2$ to $4 \cdot 10^{-6} \text{ mm/s}$) [29]. This resulted in

constant crack opening displacement rates $dCOD_{LL}/dt$ until the onset of SICC (and a constant dK_I/dt and load rate dP/dt under small scale yielding conditions also). For the same $dCOD_{LL}/dt$ values, the resulting dK_I/dt values before the onset of SICC were roughly 70 % lower in the 1T C(T) specimens, but this difference had little impact on the SICC initiation and crack growth behaviour as seen in Figure 3. SICC crack growth rates were comparable in 0.5T and 1T C(T) specimens for given loading rates up to at least $\sim 50 \text{ MPa} \cdot \text{m}^{1/2}$.

The cyclic loading in CF tests was performed under load control. Constant load amplitude loading with a positive saw tooth (slow loading, fast unloading) and a load ratio of 0.3 or 0.7 was applied. The loading frequency was either kept constant or stepwise decreased from $2 \cdot 10^{-2}$ down to $3 \cdot 10^{-6}$ Hz. In most cases the $K_{I,max}$ values were well below the ASTM E647 limit. Time-based crack growth rates da/dt were calculated by dividing the cycle-based crack growth rates $\Delta a/\Delta N$ by the applied rise time Δt_R . The corrosion fatigue behaviour was analysed in the time domain by plotting the time-based crack growth rates in high-temperature water $da/dt_{CF} = \Delta a/\Delta N_{CF} / \Delta t_R$ versus the corresponding time-based crack growth rate in air $da/dt_{Air} = \Delta a/\Delta N_{Air} / \Delta t_R$ under otherwise identical conditions [30]. The corresponding air crack growth rates were calculated according to Eason et al. [31]. In very few cases, the corrosion fatigue tests started in high-purity water and a chloride transient was then applied later on during the test.

3 Results and Discussion

3.1 Strain-Induced Corrosion Cracking

SICC behaviour in chloride-free, oxygenated high-temperature water [29]:

Figure 3 exemplarily shows the effect of loading rate on SICC initiation and subsequent crack growth in the high-sulphur RPV steel C in chloride-free, oxygenated high-temperature water with 8 ppm DO and 65 ppb SO_4^{2-} [29]. The SICC susceptibility increased with decreasing loading rate with a maximum in susceptibility (minimum in $K_{I,i}$ values) at a low crack opening displacement rate of 2 to $4 \cdot 10^{-6}$ mm/s. The subsequent SICC crack growth rates were constant during the whole rising load phase after a short transition period following SICC initiation. The SICC crack growth rate depended strongly on the applied loading rate, but not on the actual value of K_I , at least up to a K_I of 60–70 $\text{MPa} \cdot \text{m}^{1/2}$ for 1T C(T) specimens. Here, a simple power law relationship with an exponent of 0.8 was found between the SICC crack growth rate and $dCOD_{LL}/dt$ and dK_I/dt . Thus, both SICC initiation and growth were strongly dependent on the applied loading rate, but with opposite tendency.

Figure 3: Opposite effect of loading rate on SICC initiation and crack growth from incipient cracks in chloride-free oxygenated high-temperature water in the high-sulphur RPV steel C [29]. A maximum in SICC initiation susceptibility (minimum in $K_{I,i}$) was observed at very slow loading rates of 2 to 4 · 10⁻⁶ mm/s

As shown in Figure 4, the experimental observations from slow rising load tests suggest that SICC from incipient cracks is essentially governed by the crack-tip strain rate and the concentration of sulphur-anions in the crack-tip electrolyte. The SICC initiation and subsequent crack growth are crucially dependent on simultaneously maintaining a slow, positive crack-tip strain rate below an upper threshold and critical sulphur anion content in the crack-tip electrolyte. If these two conjoint requirements are not met, no SICC is observed in slow rising load tests, otherwise the SICC crack growth rates only depend on the crack-tip strain rate and applied loading rate and are identical for low- and high sulphur steels or high-purity or sulphate containing water. The critical sulphur anion threshold in the crack-tip electrolyte is synergistically controlled by material (sulphur content) and environmental parameters (*ECP* and sulphate concentration). The sulphur anion content in the crack-tip environment increases with increasing steel sulphur content, concentration of sulphur-anions in the bulk environment outside the crack, crack growth rate (exposure of new fresh dissolvable MnS-inclusions by the growing crack) and corrosion potential/*DO* content (anion enrichment in the crack enclave by migration). The crack-tip strain rate increases with increasing applied loading rate and resulting crack growth rate, as well as with yield stress and susceptibility to DSA [1, 23, 27]. Suitable combinations of different system parameters may help to exceed the critical crack-tip sulphur-anion concentration, provided that there is at least one source of sulphur (MnS inclusions or sulphur anion in the bulk environment). [29]

Figure 4: Effect of sulphate concentration and steel sulphur content on SICC crack growth in oxygenated high-temperature water. The similar crack growth rates for the various conditions indicate the existence of a critical sulphur anion concentration in crack-tip electrolyte for SICC [29]. (Dashed region: scatter range of SICC crack growth rates in the loading rate region for the chloride tests. If no SICC occurred (semi-open symbols), an apparent SICC growth rate is plotted by dividing the DCPD resolution limit by the total rise time).

Effect of chloride on SICC:

The effect of chloride on SICC was investigated with the low- and high sulphur steel A and C and 12.5 mm thick 0.5T C(T) specimens in slow rising load tests in oxygenated high-temperature water with a DO of 2 or 0.2 ppm in the loading rate range of maximum SICC susceptibility at 288 °C ($\sim 3 \cdot 10^{-6}$ mm/s). The chloride content was hereby varied between 5 and 50 ppb. The results of these tests are summarized in Figures 5 and 6. In high-purity water with a DO of 2 ppm, no SICC was observed in both materials up to $K_{I,i}$ -values of at least 50 MPa·m^{1/2}. With a chloride content of 5 ppb, SICC initiation occurred in the high-sulphur steel C at a $K_{I,i}$ -value of ~ 38 MPa·m^{1/2}, but not in the simultaneously tested low-sulphur steel A. At 50 ppb of chloride SICC was observed at $K_{I,i}$ values of ~ 18 and 22 MPa·m^{1/2} in the high- and low-sulphur steels C and A, respectively. The SICC initiation susceptibility thus increased with increasing chloride content. The critical chloride level for SICC seemed to be somewhat lower in the high-sulphur steel C. At a lower DO of 0.2 ppm, no SICC was observed in both materials, demonstrating that the chloride tolerance is strongly increasing with decreasing DO and ECP. Under these highly oxidizing conditions, the effect of chloride on SICC initiation seemed to be more pronounced than that of sulphate. In the low-sulphur steel, some few hundreds of ppb of sulphate were necessary to induce SICC under these conditions [29], whereas 50 ppb (or less) were sufficient in case of chloride.

Figure 5: Effect of chloride on the SICC initiation in oxygenated high-temperature water (if no SICC occurred (open symbols), the $K_{I,max}$ at the final maximum load is plotted). The SICC susceptibility increased with increasing chloride concentration. The critical threshold chloride concentration for SICC increased with decreasing sulphur content and ECP .

The effect of chloride on the subsequent SICC crack growth is summarized in Figure 6. In this figure, the typical SICC crack growth rate range from slow rising load tests in chloride-free, oxygenated high-temperature water for these loading conditions is shown as a shaded band. The resulting SICC crack growth rates after initiation were identical for all chloride contents and the low- and high-sulphur steels and in the same range as in chloride-free high-temperature water. Chloride thus has a strong effect on SICC initiation, but very little impact on the subsequent SICC growth rates, in contrast to SCC crack growth under constant load [25]. A very similar behaviour on the effect of chloride on SICC initiation and subsequent crack growth was observed in slow strain rate tests with smooth specimens at PSI [31].

Figure 6: Effect of chloride on SICC crack growth in oxygenated high-temperature water (if no SICC occurred (open symbols), an apparent SICC growth rate is plotted by dividing the DCPD resolution limit by the total rise time). Similar SICC crack growth rates were observed in high-purity water and in presence of chloride.

3.2 Corrosion Fatigue

CF behaviour in chloride-free, oxygenated high-temperature water [30]:

The CF behaviour of low-alloy RPV steels in chloride-free oxygenated high-temperature water is exemplarily shown by time-domain plots in Figures 7 and 8 [30]. Under BWR NWC conditions, environmental acceleration of fatigue crack growth was observed in all materials at loading frequencies below 0.1 to 1 Hz and air fatigue crack growth rates below 10^{-6} m/s. Above these upper limits, the crack growth rate is dominated by pure mechanical fatigue (which is much faster than the environmental contributions under these conditions) and does not depend on environment. Below these upper critical frequency and critical air crack growth rate, the environmental acceleration of fatigue crack growth was comparable for all materials and increasing with decreasing loading frequency down to the lowest tested loading frequencies under highly oxidizing conditions (Figure 7). Depending on ECP , the environmental acceleration dropped to moderate values or even completely disappeared again at very low loading frequencies or ΔK -values. As shown in Figure 8, this transition from strong (factor of 10 to 100 or even higher) to minor (factor of ≤ 5) environmental acceleration, which appeared as critical air fatigue crack growth rates $da/dt_{Air,crit} = f(ECP)$ in the time-based form or as critical frequencies $\nu_{crit} = f(\Delta K, R, ECP)$ and ΔK -thresholds $\Delta K_{CF} = f(\nu, R, ECP)$ in the cycle-based form, was shifted to lower values with increasing ECP (or DO contents). Under highly oxidizing BWR NWC conditions ($ECP > 0$ mV_{SHE}) fast CF crack growth could be sustained down to very low frequencies of $3 \cdot 10^{-6}$ Hz and small ΔK values of $2 \text{ MPa} \cdot \text{m}^{1/2}$.

Figure 7: Corrosion fatigue crack growth in the time-domain as a function of material and loading parameters for highly oxidizing BWR NWC conditions ($ECP \geq +50$ mV_{SHE}).

Figure 8: CF crack growth in the time-domain as a function of ECP for the high-sulphur RPV steel C [30].

The CF tests thus also confirmed the previously mentioned crack-tip strain rate - sulphur-anion conjoint requirement for EAC. A critical high-sulphur anion content has to be maintained in the crack-tip electrolyte to sustain fast CF crack growth rates (with strong environmental acceleration of fatigue crack growth), otherwise the crack growth rates quickly drop down to low rates (with minor environmental acceleration). When this conjoint requirement is satisfied, the CF crack growth rates are identical for low- and high-sulphur steels and different environments for given loading conditions and only dependent on crack-tip strain rate. The crack-tip strain rate and the sulphur anion concentration in the crack-tip electrolyte are controlled by a set of synergistic and interrelated corrosion system parameters: a high *ECP/DO* content (sulphur anion enrichment/retention in the crack enclave by migration), a high sulphur anion concentration in the bulk environment, a high steel sulphur content (dissolution of MnS inclusions), or a sufficiently high crack growth rate/crack-tip strain rate/loading frequency (exposure of new, dissolvable MnS inclusions by the growing crack) favour the formation of a crack-tip environment rich in sulphur anions and therefore a high CF crack growth rate. The crack-tip strain rate generally increases with increasing loading frequency/rate, load level, crack growth rate, yield stress and susceptibility to DSA [1, 27].

Effect of chloride on CF behaviour:

The effect of chloride on CF was evaluated at an *ECP* of -100, 0 and $\geq +50$ mV_{SHE}. For the two lower *ECP*s, 100 ppb of chloride was used. At the high *ECP*, the chloride concentration levels were varied between 10 and 50 ppb. In Figure 9, these three cases are compared with the conditions that resulted in fast SCC under static load [23, 25].

Figure 9: Comparison of CF test conditions (cases A, B and C) with system conditions that resulted in fast SCC in RPV steels under constant load [23]. The Action Level limits of the EPRI BWR/NWC water chemistry guidelines as well as typical stationary power operation conditions for HWC and NWC are also shown [32].

Case A: $ECP = -100 \text{ mV}_{SHE}$ & 100 ppb of chloride: Under these conditions, no SCC was observed under static load below K_I values of $\sim 60 \text{ MPa}\cdot\text{m}^{1/2}$ and only moderate chloride enrichment in the crack-tip environment is expected. In high-purity water, the transition from fast to slow CF crack growth was observed at an air crack growth rate of around 10^{-10} m/s . The corresponding effect of 100 ppb of chloride on CF is shown in Figure 10. Additionally, the low- and high-sulphur CF crack growth curves of the Ford & Andresen model [28] and the PSI NWC CF mean curve [30] are shown for comparison reasons. The same CF crack growth rates were observed over the whole tested loading frequency range as in high-purity water and also the transition from high- to low CF growth rates was not affected by the chloride addition, although a slight shift to lower crack-tip strain rates or loading frequencies might be anticipated. The chloride enrichment in the crack crevice by migration at this moderately high ECP probably was not high enough to cause such a shift of the transition region.

Figure 10: No effect of 100 ppb of chloride on CF crack growth at an ECP of -100 mV_{SHE} .

Case B: $ECP = 0 \text{ mV}_{SHE}$ & 100 ppb of chloride: Under these conditions, fast SCC was observed under static load below K_I values of $\sim 60 \text{ MPa}\cdot\text{m}^{1/2}$ and some significant chloride enrichment in the crack-tip environment is expected. In high-purity water, the transition from fast to slow corrosion fatigue crack growth was observed at an air crack growth rate of around $5 \cdot 10^{-11} \text{ m/s}$. The corresponding effect of 100 ppb of chloride on corrosion fatigue is shown in Figure 11. Above the transition, the same CF crack growth rates were observed in high-purity water and water with chloride addition. The chloride addition shifted the transition to lower air crack growth rates and loading frequencies and significantly accelerated CF rates at the very low loading frequencies by a factor of 10 to 100. At very low loading frequencies the CF crack growth rates were in the same range as the SCC crack growth rates at similar K_I values and environmental conditions, indicating a strong or dominating SCC contribution.

Figure 11: Effect of 100 ppb of chloride on CF crack growth at an ECP of 0 mV_{SHE} . An accelerating effect of chloride on CF crack growth was observed at low loading frequencies (low air fatigue crack growth rates) only. The open triangles correspond to CF rates after the addition of 100 ppb chloride, when cessation of CF crack growth was observed in high-purity water (solid triangles) in tests with decreasing loading frequency (see also Figure 12).

In some CF tests in high-purity water with stepwise decrease of loading frequency, 100 ppb of chloride was added to the water in the transition region after the CF crack growth rates dropped to low-sulphur rates with minor environmental acceleration. Such an example is shown in Figure 12. Here, in high-purity water low-sulphur CF crack growth rates with minor environmental acceleration by a factor of 2 prevailed prior to the addition of 100 ppb of chloride. Three hours after the addition of chloride, the CF crack growth rates increased and reached fast, high-sulphur CF crack growth rates with strong environmental acceleration by a factor of 250 within a few hours. This also confirms that a chloride-enriched mature occluded crack crevice chemistry is established within a few hours after chloride addition.

Figure 12: CF crack growth behaviour during a 100 ppb chloride transient at an ECP of 0 mV_{SHE}.

Case C: $ECP \geq +50$ mV_{SHE} & 10 to 50 ppb of chloride: Under these conditions, very fast SCC was observed under static load below K_I values of ~ 60 MPa $\cdot$ m^{1/2} and very strong chloride enrichment in the crack-tip environment is expected. In high-purity water, fast high-sulphur CF crack growth rates were sustained down to the lowest tested loading frequencies of $3 \cdot 10^{-6}$ Hz. The corresponding effect of 10 to 50 ppb of chloride on CF is shown in Figure 13. As expected, the chloride addition resulted in the same CF crack growth rates as in high-purity water. Since fast CF crack growth is sustained down to the lowest loading frequencies, only minor effects of chloride were expected. In the loading frequency range of $3 \cdot 10^{-6}$ to 10^{-5} Hz, maximum CF crack growth rates of $5 \cdot 10^{-10}$ to $2 \cdot 10^{-9}$ m/s were observed in high-purity water that are only slightly lower than the typical SCC rates with chloride in the K_I -range from 20 to 50 MPa $\cdot$ m^{1/2} of $5 \cdot 10^{-10}$ to $2 \cdot 10^{-9}$ m/s (see shaded region in Figure 13). Some slight acceleration of CF crack at these low loading frequencies might thus be possible, but no additional testing with chloride was done in this region.

Figure 13: No significant effect of chloride on CF crack growth at high $ECPs \geq +50$ mV_{SHE}.

The observed effect of chloride on CF crack growth under BWR NWC condition is summarized in Figure 14. Under cyclic load, little effect of moderate chloride additions (≤ 100 ppb) was observed on CF crack growth at high $ECP \geq +50$ mV_{SHE} and low $ECP \leq -100$ mV_{SHE}, since fast high-sulphur CF crack growth prevails down to the lowest loading frequencies in high-purity water in the former case or due to the absence of SCC in the latter case, respectively. A strong effect of chloride was observed at intermediate ECP s (-100 mV_{SHE} $< ECP < +50$ mV_{SHE}) at or below the transition region from low- to high-sulphur CF crack growth rates, where slow low-sulphur CF crack growth would prevail in high-purity water, but some SCC crack growth may still occur in presence of chloride. In contrast to SCC under static load, moderate chloride additions (≤ 100 ppb) usually have little effect on SICC and CF crack growth under BWR/NWC conditions over a wide range of system conditions.

Under reducing BWR HWC conditions, where slow, low-sulphur CF crack growth rates prevail over a larger frequency range, chloride in principle could result in significant acceleration of CF crack growth, but due to the absence of any significant chloride enrichment in the crack crevice, chloride in the range of several hundreds of ppb or probably even a few ppm are required to cause such shifts. Furthermore, acidic pH-shifts in the crack-tip electrolyte in HWC environment are only produced with HCl. At low chloride levels, ECP reduction by changing from NWC to HWC clearly reduced the CF crack growth rates from fast high-sulphur to slow low-sulphur CF crack growth rates demonstrating again the significantly higher chloride tolerance at low ECP s (Figure 15).

Figure 14: Schematic summary of chloride effects on CF crack growth at different ECP s.

Figure 15: Significant reduction of CF crack growth rates in high-temperature water with 20 ppb of chloride by changing from BWR NWC (high *ECP*) to HWC (low *ECP*) conditions.

3.3 Comparison with SCC and mechanistic interpretation

As discussed in detail in [25], a tremendous effect of chloride in the ppb level range on SCC crack growth was observed in highly oxidizing BWR NWC environment. Such pronounced effects were not observed for SICC and CF crack growth. A significant chloride effect, on the other hand, was observed on SICC initiation from incipient cracks and smooth surfaces [31] and, in principal, although not investigated, may also be expected for CF initiation from smooth surfaces under certain mild conditions. The observed EAC crack growth behaviour with chloride can be rationalized by the film rupture/anodic dissolution (FRAD) model [39] of Ford & Andresen [28] and crack crevice chemistry models [40-42] and is discussed for the SCC case in detail in [25].

The main reason for the strong “anomalous” effect of chloride on SCC crack growth in RPV steels NWC environment is the very low SCC crack growth susceptibility and crack growth rates ($< 1 \cdot 10^{-11}$ m/s) at K_I -values < 60 MPa·m^{1/2} in high-purity or sulphate containing oxygenated high-temperature water [25]. Under these conditions with an aggressive sulphur anion enriched occluded crack crevice chemistry, fast high-sulphur SCC crack growth rates of the Ford & Andresen model [28] in the range of $6 \cdot 10^{-9}$ m/s (20 MPa·m^{1/2}) to $3 \cdot 10^{-8}$ m/s (60 MPa·m^{1/2}) should prevail. But in reality, the initially fast high-sulphur EAC crack growth rates in the range of 10^{-9} to 10^{-7} m/s cannot be sustained after switching from cyclic/slow rising load to constant load and cessation of SCC crack growth occurs within a few hundred hours with crack arrest or very slow SCC crack growth rates $< 10^{-11}$ m/s (Figures 16 and 17) [29]. Furthermore, the decay of crack growth at the higher K_I -values often follows a reciprocal time law (Figure 17) indicating that crack growth and crack-tip strain rate could be controlled by low-temperature creep [23, 25]. Sustained SCC crack growth at K_I -values < 60 MPa·m^{1/2} (if it occurred at all) was usually very localized (some few isolated locations with very limited crack growth, often around MnS-inclusions, which were intersected by the crack front) and could usually only be detected by post-test fractography with some very few exceptions [23, 25]. Small amounts of chloride here help to overcome the sustained crack-tip strain rate and cracking problems and thus seem to have a tremendous effect. The different

behaviour with chloride is illustrated in Figure 18 for a chloride content of 20 ppb and a test with a change from cyclic to constant load. Here, fast SCC with a rate of $5.6 \cdot 10^{-9}$ m/s was observed at a K_I value of $34 \text{ MPa} \cdot \text{m}^{1/2}$ after switching from cyclic to constant load. This rate is more than 500 times higher than in high-purity water. The CF rate, on the other hand was in the upper range of the scatter band of the high-purity water data for these loading and *ECP* conditions.

Figure 16: Cessation of SCC after switching from cyclic to constant load in oxygenated high-temperature water with very low levels of chloride below the critical chloride threshold at K_I -values below $60 \text{ MPa} \cdot \text{m}^{1/2}$ (see also Figure 9). The same behaviour is observed in chloride-free environments.

Figure 17: Decay of SCC crack growth rate according to a reciprocal time law at a high K_I value of $73 \text{ MPa} \cdot \text{m}^{1/2}$ after switching from slow rising to constant load, indicating low-temperature creep controlled crack-tip strain rate and SCC crack growth [23, 25].

Figure 18: Sustained fast SCC after switching from cyclic to constant load in oxygenated high-temperature water with a chloride content above the threshold concentration for SCC.

Under conditions, where sustained (high-sulphur) EAC (e.g., under cyclic or slow rising load) or SCC crack growth prevail (e.g., at high K_I -values > 70 to $80 \text{ MPa}\cdot\text{m}^{1/2}$, in weld heat-affected zone (HAZ) materials with high hardness or in steels with high DSA susceptibility at intermediate temperatures, [23-25]), chloride has no or only a very minor accelerating effect on crack growth. Here a sustained crack-tip strain rate is assured by the external cyclic and slow rising load or facilitated by the very high load level and distinct low-temperature creep, steeper strain gradient at the crack-tip with higher yield stress (weld HAZ) and higher work hardening rate and localization of plastic deformation by DSA [23, 25].

The different behaviour is schematically summarized in Figure 19 and a potential tentative mechanistic explanation is outlined in Figures 20 and 21.

Figure 19: Schematic overview on EAC behaviour in high-purity and chloride containing oxygenated high-temperature water. (C_{CT} = chloride content in crack-tip crevice environment).

Figure 20: Overview on film rupture/anodic dissolution model according to Ford & Andresen [28] with major control parameters for crack growth. It is stressed that crack growth rate depends on crack-tip strain rate and crack crevice chemistry and vice versa, which may result in significant feedback effects.

Figure 21: Potential effects of chloride on EAC in film rupture/anodic dissolution model.

In chloride-free NWC environment at K_I -values $< 60 \text{ MPa}\cdot\text{m}^{1/2}$, the SCC crack advance by anodic dissolution under constant load between film rupture and full repassivation is only marginally too small to induce sufficient straining for a new rupture and the crack-tip strain rate and crack growth rate are thus controlled by the declining low-temperature creep solely (Figure 17). The slight acidification of the crack crevice environment due to potential-gradient driven chloride enrichment in the crack crevice results in a higher metal dissolution rate after rupture of the protecting oxide film and in a further delay of the repassivation of the metal. This results in a moderately larger SCC crack growth increment due to anodic dissolution of the metal. The less protecting, thinner oxide film [43] with smaller film rupture strain due to chloride adsorption decreases the necessary crack advance to induce sufficient straining for a new film rupture. Above a certain critical crack-tip chloride content threshold, and in combination with the positive feedback effects due to the mutual interrelation between EAC crack growth rate and crack-tip strain rate and crack crevice chemistry, this results in an acceleration of SCC crack growth that may be fast enough to finally establish a sustained dynamic equilibrium between crack-tip straining and crack growth with high-sulphur SCC crack growth under static load. Above the critical crack-tip environment chloride concentration, a further increase in the bulk chloride concentration does not further accelerate SCC crack growth. At higher *ECPs*, less chloride in the bulk environment is necessary to exceed the critical crack-tip environment concentration due to the stronger chloride enrichment in the crack crevice (Figure 9). At lower stress intensity factors, the crack-tip strain accumulation due to low-temperature creep is lower and a somewhat higher critical crack-tip chloride concentration is necessary to establish fast high-sulphur SCC crack growth.

The crack-tip strain rate hypothesis is also confirmed by tests with a high hardness (350 HV5) weld heat-affected zone material in high-purity oxygenated high-temperature water, where the same SCC growth rates were observed as in tests with chloride with base metal (Figure 22), but no acceleration of CF occurred with respect to experiments with chloride and base metal (Figure 23). The much higher yield stress results in a smaller plastic zone size and higher crack-tip strain and thus in a steeper strain gradient at the crack-tip. This ends up in a higher crack-tip strain rate with crack growth (rate) [44-46]. Similarly, a high susceptibility to DSA can result in fast SCC in high-purity oxygenated high-temperature water, since DSA results in a higher crack-tip strain/strain rate than for loading outside the DSA temperature-strain rate range, or than in a material which is not susceptible to DSA [1, 23]. The localisation of plastic deformation and increase in planar deformation from DSA furthermore facilitates the mechanical rupture of the oxide film [1, 23, 27].

Figure 22: Similar SCC crack growth rates in high strength weld HAZ material in high-purity water as in moderate strength base metal in chloride containing water [25].

Figure 23: Similar CF growth rates in high strength weld HAZ material in high-purity water as in moderate strength base metal in high-purity or chloride containing high-temperature water.

4 Conclusions

The effect of chloride on the SICC and CF crack growth behaviour in low-alloy RPV steels was evaluated under simulated BWR NWC conditions by slow rising load and cyclic constant load amplitude tests with air fatigue pre-cracked fracture mechanics specimens.

Chloride addition in the ppb level range in slow rising load tests in highly oxidizing, oxygenated high-temperature water, remarkably increased the SICC initiation susceptibility, but had almost no effect on the subsequent SICC crack growth in the loading rate range of maximum SICC susceptibility ($dCOD_{LL}/dt \sim 2$ to $4 \cdot 10^{-6}$ mm/s). The critical threshold concentration for chloride effects on SICC initiation increased with decreasing *ECP* and steel sulphur content.

Under cyclic load, little effect of moderate chloride additions (≤ 100 ppb) was observed on CF crack growth at high $ECP \geq +50$ mV_{SHE} as well as at low $ECP \leq -100$ mV_{SHE}, since fast high-sulphur CF crack growth prevails down to the lowest loading frequencies in high-purity water in the former case or due to the absence of SCC in the latter case, respectively. A strong effect of chloride was observed at intermediate *ECP*s (-100 mV_{SHE} $< ECP < +50$ mV_{SHE}) at or below the transition region from low- to high-sulphur CF growth rates, where slow low-sulphur CF crack growth would prevail in high-purity water, but some SCC may still occur in presence of chloride. In contrast to SCC under static load, moderate chloride additions (≤ 100 ppb) usually have little effect on SICC and CF crack growth under BWR NWC conditions over a wide range of system conditions.

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