

The Underestimated Role of the Oxygen on RCS Components Failures

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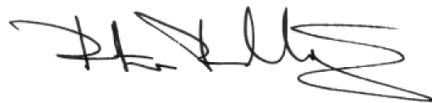


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A handwritten signature in black ink, appearing to read 'P. Rudling', with a stylized flourish at the end.

Mr Peter Rudling, President of ANT International

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1 Summary

Historically, the presence of oxygen in the RCS of PWRs has been banished mainly because of the risk of corrosion for stainless steels. To suppress oxygen into the RCS, scavengers, such as hydrogen, are added to the primary water. As a consequence, because of the hydrogen injection, oxygen is not monitored in some plants¹ when the reactor is in operation. In operation, the expected oxygen content in the RCS of EDF PWRs is less than 10 ppb², with an upper limit of 100 ppb, providing the temperature is higher than 120°C (there is no limit if the RCS temperature is lower than 120°C). In plants monitoring oxygen in the RCS, the oxygen content is generally in the order of one to a few ppb. Thus, we should consider there is virtually almost no oxygen in the RCS. This is true providing we are in free-flowing conditions. Unfortunately, the RCS contains many flow-restricted or occluded areas where oxygen can settle and so induce a corrosion risk for the concerned component. This trapped oxygen can induce some corrosion. This report is about failures due to the presence of this “hidden” oxygen. A deleterious effect of oxygen has been observed on canopy seals, omega seals, CEDMs, baffle bolts, dead legs, valve drains, pressurizer heaters...

¹ EDF plants do not monitor the oxygen concentration in the RCS since Belleville tests (see section 4) have shown that this concentration is very low. A side benefit was a radiation dose reduction for chemists.

² The relevant expected value is less than 5 ppb in the EPRI Primary Water guidelines.

2 Introduction

Because it is known that oxygen concentration has a significant influence on SCC behaviour of many materials, there is a concern that the presence of oxygen in the RCS may increase the likelihood of SCC of RCS structural materials.

Some of the main effects of oxygen on SCC of structural materials used in the RCS include the following:

- Oxygen leads to SCC of sensitized stainless steels, including sensitized heat affected zones at welds. This type of SCC has been a major problem in boiling water reactors for more than 25 years;
- Oxygen leads to SCC of sensitized Alloy 600 if reduced sulphur species are present. Crack growth rates are fastest at relatively low temperatures, of about 100°C or less. This phenomenon was the cause of the large scale primary side cracking of the sensitized tubes in the Three Mile Island 1 steam generators that experienced attack during a long shutdown in 1981. It probably also was involved in the cracking and intergranular attack observed in the Zorita control rod drive mechanisms which were sensitized Alloy 600 and are believed to have suffered attack as a result of resin ingress which made sulphur species available;
- Oxygen at the levels seen in normal BWR reactor coolant leads to or aggravates SCC of some high strength alloys such as A286 and X750. For example, the stress level at which SCC occurs in 200 ppb BWR water is about 60% of yield, while stresses over 100% of yield are required for SCC in deaerated PWR environments, despite the higher temperatures in PWRs;
- Tests from the 1960s show that oxygen at high concentrations leads to intergranular attack and SCC of crevice areas in Alloy 600. This was demonstrated in tests performed by the International Nickel Company that found that intergranular attack/stress corrosion cracking occurred in crevices formed by double U-bends in 316°C water with oxygen at 5% or more in the autoclave cover gas at the start of the test. Tests by AECL at about that same time showed that crevices and cracks grew in Alloy 600 in 288°C water with 10 to 100 ppb oxygen. These tests show that, if sufficient oxygen is present at high temperature, IGA/SCC occurs of Alloy 600 in crevices areas, apparently due to acidification in the crevice caused by the oxygen. The AECL tests, where growth of IGA/SCC occurred at 10 to 100 ppb oxygen concentration, showed that high concentrations of oxygen are not necessary;
- Slow strain rate tests in the 1980s showed that PWSCC of Alloy 600 at 350°C occurred for electrochemical potentials below a potential at about the reversible hydrogen potential, and at potentials about 400 mV above this potential;
- Measurements of ECP in hydrogenated water indicate that a few ppb of oxygen, less than 5 ppb, increase the potential of corrosion resistant materials by several hundred millivolts, despite the presence of hydrogen;
- A generally observed phenomena is that oxygen or high ECP driven attack (hereafter called oxidant driven attack) is that it is much less likely to occur in regions of high flow velocity than in regions of low velocity. This is attributed to the following: (1) oxidant driven attack involves accumulation of impurities in crevices that lead to acidification in the crevice, (2) the accumulation of impurities is caused by the voltage difference provided by the oxidant, and (3) high flow velocities tend to cause turbulence that removes accumulated impurities from crevices, such that the locally aggressive conditions needed for oxidant driven attack to occur do not develop.

In summary, plant experience and laboratory tests indicate that (1) oxygen at the typical BWR level of 200 ppb clearly increases the susceptibility to SCC of some structural materials, (2) laboratory tests indicate that oxygen in the range of 10 to 100 ppb causes IGA/SCC of Alloy 600, (3) laboratory tests indicate that oxygen levels as low as about 5 ppb or a little less can raise the ECP into a range where SCC of Alloy 600 may occur, and (4) laboratory tests and experience indicate that oxidant driven attack is more likely in low flow areas than in high flow velocity areas.

3 The deleterious action of oxygen on stainless steels

3.1 Intergranular corrosion of stainless steels

3.1.1 Background

Historically, oxygen wash banished from the RCS because of the risk of intergranular corrosion of stainless steels.

The intergranular corrosion is a localized corrosion with a preferential attack of grain boundaries of sensitized stainless steel. The origin of the mechanism is a chromium depletion of the grain boundaries which induces a loss of corrosion resistance when the material is exposed to a harsh environment.

The intergranular corrosion of stainless steels is a two steps process. The first step is the material sensitization and the second step is the intergranular corrosion per say, when a harsh environment is present.

3.1.2 First step: stainless steel sensitization

Some types of stainless steels can be sensitized when they experience an intergranular precipitation of type Cr_{23}C_6 chromium carbides as shown on Figure 3-1. On this figure, the precipitation appears as dark grey zones.

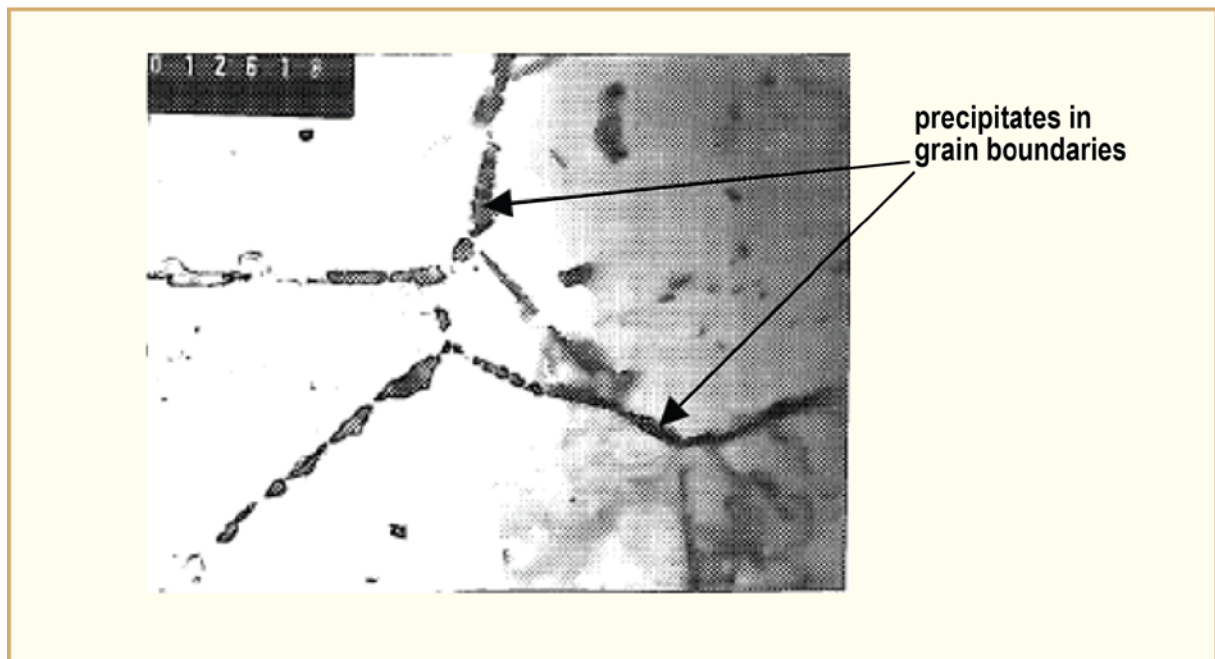
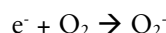
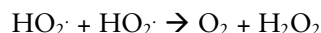
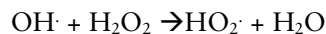


Figure 3-1: View of intergranular Cr_{23}C_6 carbides (transmission electronic microscope image) ([Cattant, 2014]).

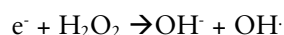
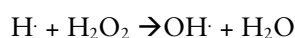
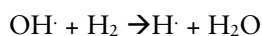
This precipitation occurs when the dissolved carbon content is superior to the carbon solubility at the given temperature. This is typical from mill annealed stainless steel operating at a lower temperature. As the carbon solubility decreases with the temperature, the oversaturation generates a progressive precipitation of chromium carbides in the grain boundaries. Because the diffusion of carbon is faster than the diffusion of chromium, the precipitation generates a chromium depleted zone around the chromium carbides (Figure 3-2). Should the chromium content in the depleted zone decreases to less than 12%, then this zone becomes susceptible to corrosion when in contact with an aggressive environment.

4 Behaviour of oxygen into the RCS

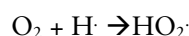
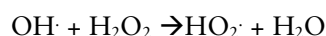
In normal operation, because of the radiolysis phenomenon, oxygen and hydrogen cannot co-exist downstream the core. Water is decomposed by radiolysis which generates H_2O_2 and H_2 molecules along with e^- , H^\cdot and OH^\cdot radicals. These species interact with each other and form O_2 and O_2^\cdot according to the following reactions;



With such reactions, the environment tends to turn oxidizing, thus hydrogen is added to change the medium to reducing conditions because of the following reactions:



The oxidizing species H_2O_2 and O_2 are consumed via the reactions:



These reactions show that even if no oxygen is present at the beginning into the RCS, the core will generate oxidizing species such as: HO_2^\cdot , O_2 , H_2O_2 and O_2^\cdot . Adding oxygen to the RCS will favor the radiolytic decomposition to: HO_2^\cdot , O_2 , H_2O_2 and O_2^\cdot , whereas adding hydrogen will suppress the radiolytic decomposition. Figure 4-1 from the reference [Solomon, 1978] shows that to minimize the production of O_2 and HO_2^\cdot oxidizing species in the core, hydrogen concentration can be as low as 10 cc/kg; unfortunately, H_2O_2 is not considered in this Figure 4-1.

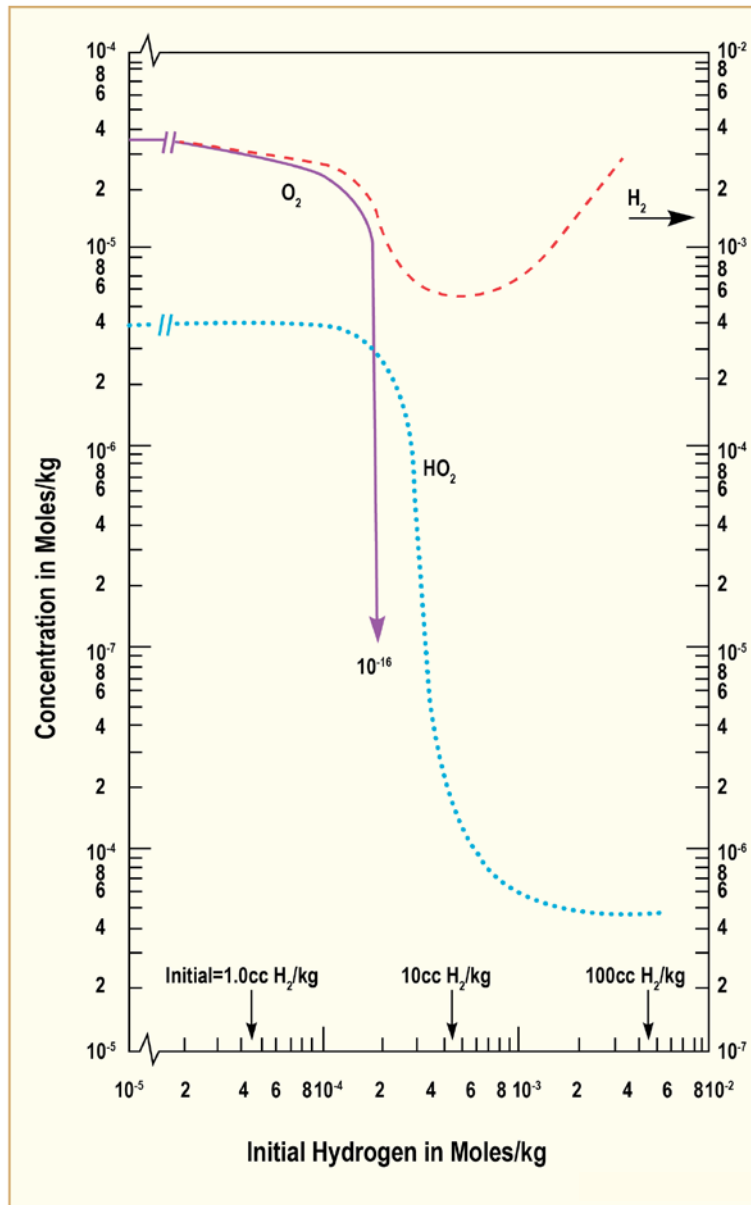


Figure 4-1: Steady state concentration of O_2 and HO_2 versus initial H_2 ([Solomon, 1978]).

However, more recent studies show that the minimum hydrogen content to prevent the significant formation of oxidizing species is lower than that, i.e.: 7 cc/kg or even less. In order to determine the hydrogen content in the RCS below which the effects of water radiolysis appear, a series of tests have been performed at Belleville (EDF 4 loop PWRs) in 1992 and 1993 ([Brun et al., 1994]).

During the first test, the hydrogen concentration has been reduced to 5 cc/kg in the RCS and a slow rise in hydrogen in the primary water and in the gas phase of the CVCS has been observed (Figure 4-2). One hypothesis is that this rise could result from some desorption phenomenon.

5 Sources of oxygen ingress into the RCS

There are many ways for the oxygen to enter the RCS. The major one is during a refuelling outage, when the operator breaks the RCS. But there are several others, such as:

- Addition of reactor make-up water, concentrated boric acid and/or blended (recycled and fresh) boric acid via the CVCS charging line induces an oxygen ingress;
- Load follow also results in oxygen ingress. Boric acid is added to reduce the power load, and the RCS is diluted with make-up water to return to 100% power;
- Oxygen ingress can occur at power. A number of stations have fully oxygenated reactor make-up water tanks. In VVERs a large fraction of the make-up enters the primary circuit via the main coolant pump seals. In these stations, which only have stainless steel in the RCS, it has been reported that the oxides adjacent to the RCPs are primarily hematite, rather than the normal spinels grown under reducing conditions;
- Oxygen ingress can increase at power towards end-of-cycle. For PWRs without a BTRS, increasingly larger volumes of reactor make-up water are added at end-of-cycle to dilute the boron concentration, carrying with it the risk of significant oxygen ingress into the RCS;
- Oxygen can also enter the RCS during boration under hot shutdown conditions. Under these conditions part or all of the CVCS charging and seal injection flows can be oxygenated;
- Oxygen ingress can occur when aligning the RHRS trains at less than 177°C. The water contained in the RHRS can sometimes be fully aerated;
- Oxygen ingress can also happen when adding blended boric acid during cooldown to accommodate changes in density and on collapsing the pressurizer bubble;
- Oxygen can enter the pressurizer during shutdown. Typically, boric acid and blended boric acid can be added to the RCS via the auxiliary spray line, thus local oxygenated conditions can be formed in the pressurizer;
- Regarding the pressurizer again, there is another possibility for oxygen to be present. Normally the RCS is fully oxygenated before start-up and the oxygen concentration is reduced by adding hydrazine, via the CVCS, before the temperature can be raised above 120°C (<100 ppb oxygen). If the sprays are not fully opened to maximize the flow rate through the pressurizer, the latter could remain static and oxygenated up to higher temperatures than normally permitted.

Thus, oxygen can be present in the RCS:

- In outage, because the RCS is open to the atmosphere. Given the oxygen enters as a gas, it can fill all drained locations;
- During start-up when the temperature is less than 120°C;
- In operation in flow restricted or occluded areas. In some locations, the oxygen results from air being dissolved in water during start-up;
- During shutdown, when H₂O₂ is injected and/or when the RHRS is connected to the RCS.

So, whatever the operating mode (from 1 to 5), there are many locations in the RCS where oxygen can be “hidden”. This “hidden” oxygen can be responsible for components and materials failures; some examples of such degradations are presented in the next chapter.

6 Components and materials failures induced by the presence of oxygen in the RCS

6.1 Canopy seals

Many PWRs worldwide, including in France, USA, Japan, Belgium and Sweden, have suffered from canopy seals leaks.

Reactor vessel head penetrations in most of the pressurized water reactors consist of nickel alloy tubes which are inserted into openings in the head and secured by J-Groove welds. The upper ends of these tubes are welded to 300 series stainless steel flanges equipped with threads. CRDM housings are screwed onto these threads. Due to the fact that the American Society of Mechanical Engineers Boiler and Pressure Vessel Code does not generally permit the use of a threaded joint as the sole means for retaining pressure and leakage in these components, the threaded joint, which is designed to provide structural integrity (forms the pressure boundary), is supplemented by a seal weld, which is intended to retain leakage past the threads.

While canopy seals leaks do not constitute a significant risk of a loss of coolant accident because the threaded joint maintains the connection's structural integrity, leakage through the seal weld and surrounding base metal poses some risk of boric acid corrosion to adjacent components. In addition, boric acid "cooking" for months on a head is very difficult to clean afterwards, it's much easier to clean when the boric acid is still fresh.

Figure 6-1 shows a typical canopy seal design. The seal weld is a TIG weld with a meltable ring typically made of AISI 308L. In EDF units, the canopy seals temperature in operation is 195°C.

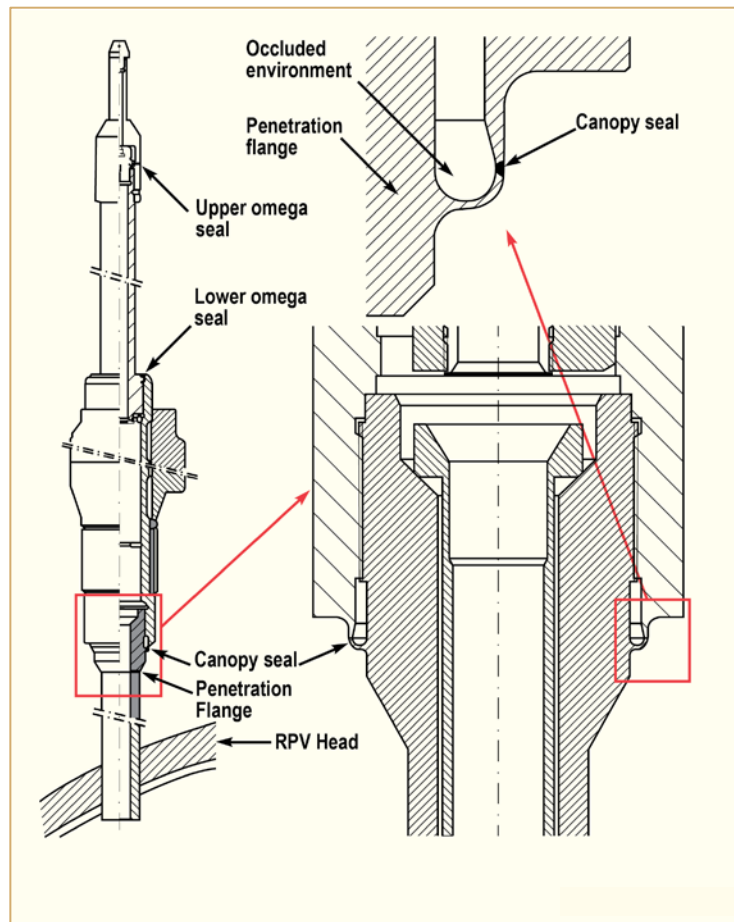


Figure 6-1: Left: sketch of a CRDM. Right: details of the canopy seal zone ([Cattant, 2014]).

As mentioned, many leaks have been observed worldwide (Figure 6-2).



Figure 6-2: View of canopy seals leaks; left: Comanche Peak ([Baker, 2002]), right: Ringhals 4 ([Efsing et al, 2006]).

At start-up, air is trapped in the CRDM housings. Some housings are self-venting³; thus, the air is rapidly eliminated. Some CRDMs have a vent screw at the top; however, this vent is not often used because venting can impact the start-up critical path and there is a major risk of spraying primary water on the head when using such vent. Some housings cannot be vented at all.

The reference [NRC, 1991], states that air bubble trapped in the unvented housing of Fort Calhoun 1, could result in an oxygen concentration at start-up between 300 to 1,300 ppm in the stagnant water in the upper housing. Estimates of decay of the oxygen concentration in the housing based solely on diffusion showed that the high levels of oxygen would remain for several months. Flow in the housing due to natural circulation, which would accelerate the decay of oxygen concentration, was restricted by installed baffles.

The reference [Alley et al, 2013], referring to a CRDM leak at Palisades, reports that analyses performed as part of the failure analysis indicated that the oxygen content in the spare housings (204°C) was also between 300 and 1,300 ppm. Additional analyses indicated that this level of oxygen would not change substantially during an 18 month refuelling cycle but would begin to drop slowly after that time due to natural circulation and diffusion. Conversely, the oxygen level in active CRDM housings (vented) should drop to levels consistent with bulk primary water, i.e., 5 ppb in a relatively short time following a refuelling outage.

The reference [Efsing et al, 2006] indicates that it will take 2 to 3 weeks for the air entrapped in the CRDMs to dissolve in the water.

To know more about the environment in contact with canopy seals, some grab samples have been taken, after drilling of the seal during an outage. The Table 6-1 gathers the results of the chemical analyses.

³ For example, the active CEDMs of Fort Calhoun 1 have a self-venting seal design feature that allows air venting during RCS fill and CEDM operation.

Table 6-1: Results of the chemical analyses of canopy seals grab samples ([Cattant, 2014]).

Plant	Penetration #	B (ppm)	Li (ppm)	Cl (ppm)	F (ppm)	SO ₄ (ppm)
Specification		0 to 2500	0.4 to 2.2/3.5	< 0.15, however < 0.05 expected		
EDF 1 3 loops	T47	970	1.4	0.03	0.02	1.09
	T53	990	1.4	0.04	0.02	1.12
EDF 2 3 loops	T47	430	1.4	0.03	0.02	0.89
	T49	920	3.8	0.02	0.02	0.39
EDF 3 3 loops	T47	460	1.9	0.09	0.11	0.85
	T49	880	2.0	0.05	0.02	1.03
EDF 4 4 loops	T71	1,000	2.5	0.09	0.02	3.1
	T72	890	2.3	0.09	0.02	3.1
EDF 5 4 loops	T70	2,240	1.6	0.05	0.02	2.15
	T71	1,050	1.9	0.29	0.10	7.9
	T72	1,510	2.6	0.31	0.06	2.5
EDF 6 4 loops	T72	670	1.2	0.02	0.02	0.05
EDF 7 3 loops	TC51	440	1.0	0.11	0.06	2.30
EDF 8 3 loops ⁴	T47	830	2.4	0.04	0.03	2.15
USA 1	-	-	-	0.356	<0.053	0.679
USA 2	-	-	-	<0.1	<0.09	0.31
USA 3	-	-	-	<0.1	<0.09	0.87
USA 4	-	-	-	0.27	<0.09	0.81

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The fluorides contents always meet the technical specification. The chlorides contents meet the specification 14 times out of the 18 analyses; however, half of the times, the content is superior to the maximum expected figure. Except for one analysis, the sulphate contents always exceed the specification upper limit. These high sulphates concentrations could be related to the presence of oxygen. As a matter of fact, the reference [Sala et al, 1993] mentions that in reducing conditions, sulphates can be reduced to sulphur. So, the presence of large quantities of sulphates in the canopy seal environment is consistent with an oxidizing environment rather than with a reducing environment.

Thus, chlorides pollution may exist but is not generic, in other words, although chlorides are definitively involved in many canopy seals SCC, all canopy seals cracks may not be related to chlorides induced SCC.

All the canopy seals cracks are from SCC. They are ID initiated, transgranular and branched. They propagate into the base metal of the penetration flange or of the CRDM housing and into the weld (Figure 6-3). The crack faces exhibit brittle features similar to pseudo-cleavage: steps, fans and rivers (Figure 6-4).

Canopy SCC can initiate at the bottom of corrosion pits (Figure 6-5).

⁴ New (replacement) head.

Canopy cracks can also initiate on welding defects like TIG tack weld remnants or repaired zones and on lack of fusion.

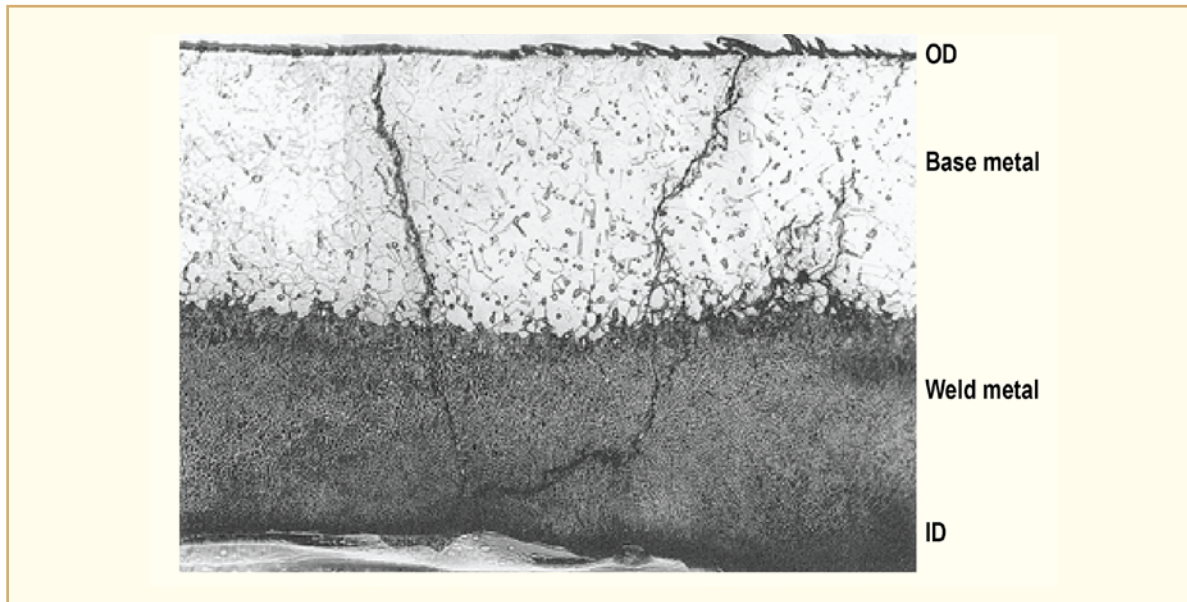


Figure 6-3: Canopy seals. SCC cracks propagating into the base metal from the penetration flange and into the weld (x34) ([Cattant, 2014]).

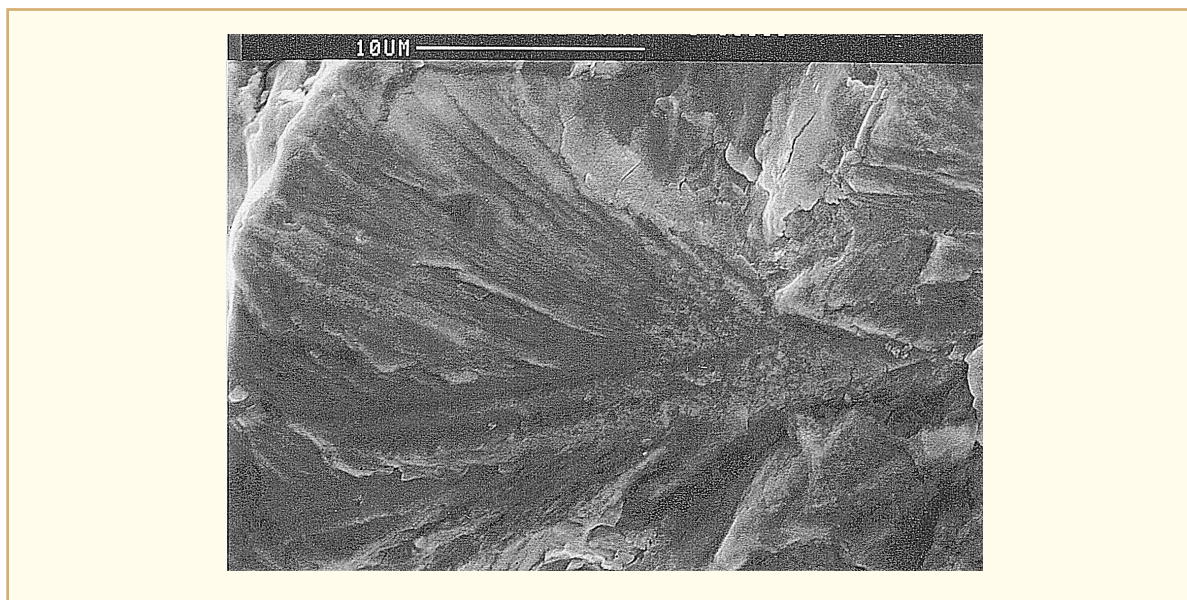


Figure 6-4: SEM view of canopy seal SCC ([Cattant, 2014]).

7 Conclusion

In a PWR, there are multiple ways for oxygen to enter the RCS:

- At refuelling outage, when the operator breaks the RCS.
- Addition of reactor make-up water, concentrated boric acid and/or blended boric acid;
- Load follow;
- Boration under hot shutdown conditions. Under these conditions part or all of the CVCS charging and seal injection flows can be oxygenated;
- Alignment of the RHRS trains at less than 180°C. The water contained in the RHRS can sometimes be fully aerated;
- Addition of blended boric acid during cooldown to accommodate changes in density and on collapsing the pressurizer bubble.

Some of the oxygen injected or trapped in the RCS can stay for weeks or even months at the same location (canopy seals, omega seals, baffle bolts, barrel bolts, dead legs...).

Oxygen can induce general corrosion, pitting and/or cracking of stainless steels, especially for sensitized stainless steel. A good many of the RCS components are made of stainless steels, which makes oxygen presence undesirable.

Field experience shows that many components made of stainless steel have experienced general corrosion, pitting and/or stress corrosion cracking because of the assumed or proved presence of oxygen in the environment in contact with the material. This report presents several examples of failures due to oxygen pollution or presence.

Unfortunately, oxygen presence is very often unavoidable in many RCS locations; thus, there are very little means to fight against this oxygen pollution. Moreover, the synergistic effect of oxygen with chlorides, even at very low concentrations, makes oxygen even more aggressive for stainless steels.

Amongst the limited solutions to minimize oxygen presence or to limit oxygen induced failures, are:

- Use qualified oxygen scavengers, not only when the environment temperature exceeds 120°C, but at lower temperature at some locations. Of course, oxygen scavengers, such as hydrazine, have a lower efficiency at low temperature but corrosion rate is also slower at low temperature, allowing more time for scavengers to work. Note that the RCS hydrogen concentrations maintained by PWR operators worldwide exceed by far the minimum hydrogen concentration required to suppress radiolysis consequences.
- Vent or flush when possible. Some CRDMs can be vented, some dead legs can be flushed with low oxygen water.
- Fill the RCS under vacuum or fill the RCS with deaerated water whenever possible (already done by a Japanese Utility).
- Use very low carbon stainless steels to prevent any intergranular corrosion sensitization at welding or during some heat treatments.
- Educate the maintenance personnel regarding work cleanliness, a good after work cleaning is paramount to mitigate failures due to an oxygen/chlorides synergistic effect.

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Nomenclature

cc/kg	cubic centimetre per kilogram
dpa	displacement per atom
mV	millivolt
pH	Hydrogen potential
ppb	parts per billion
ppm	parts per million

List of Abbreviations

AECL	Atomic Energy of Canada Limited
AISI	American Iron and Steel Institute
ASME	American Society of Mechanical Engineers
ATEM	Analytical Transmission Electron Microscopy
BTRS	Boron Thermal Regeneration System
BWR	Boiling Water Reactor
CEDM	Control Element Drive Mechanism
CRDM	Control Rod Drive Mechanism
CVCS	Chemistry and Volume Control System
DE	Destructive Examination
ECP	Electrochemical Corrosion Potential
EDF	Electricité de France
EDS	Energy Dispersive Spectroscopy
HPSIS	High Pressure Safety Injection System
IASCC	Irradiation Assisted Stress Corrosion Cracking
IGA	InterGranular Attack
IGSCC	InterGranular Stress Corrosion Cracking
LWR	Light Water Reactor
NDE	NonDestructive Examination
NPP	Nuclear Power Plant
NRC	Nuclear Regulatory Commission
PT	Penetrant Test
PWR	Pressurized Water Reactor
PWSCC	Primary Water Stress Corrosion Cracking
RCP	Reactor Coolant (main) Pump
RCS	Reactor Cooling System
RHR	Residual Heat Removal
RHRS	Residual Heat Removal System
RWST	Refuelling Water Storage Tank
SCC	Stress Corrosion Cracking
SEM	Scanning Electron Microscope
SIS	Safety Injection System
SS	Stainless Steel
SSRT	Slow Strain Rate Testing
TEM	Transmission Electron Microscopy/Microscope
TGSCC	TransGranular Stress Corrosion Cracking
TIG	Tungsten Inert Gas
VVER	Voda-Vodyanoi Energetichesky Reaktor

Unit conversion

TEMPERATURE		
$^{\circ}\text{C} + 273.15 = \text{K}$	$^{\circ}\text{C} \times 1.8 + 32 = ^{\circ}\text{F}$	
T(K)	T($^{\circ}\text{C}$)	T($^{\circ}\text{F}$)
273	0	32
289	16	61
298	25	77
373	100	212
473	200	392
573	300	572
633	360	680
673	400	752
773	500	932
783	510	950
793	520	968
823	550	1022
833	560	1040
873	600	1112
878	605	1121
893	620	1148
923	650	1202
973	700	1292
1023	750	1382
1053	780	1436
1073	800	1472
1136	863	1585
1143	870	1598
1173	900	1652
1273	1000	1832
1343	1070	1958
1478	1204	2200

Radioactivity	
1 Sv	= 100 Rem
1 Ci	= 3.7×10^{10} Bq = 37 GBq
1 Bq	= 1 s^{-1}

MASS	
kg	lbs
0.454	1
1	2.20

DISTANCE	
x (μm)	x (mils)
0.6	0.02
1	0.04
5	0.20
10	0.39
20	0.79
25	0.98
25.4	1.00
100	3.94

PRESSURE		
bar	MPa	psi
1	0.1	14
10	1	142
70	7	995
70.4	7.04	1000
100	10	1421
130	13	1847
155	15.5	2203
704	70.4	10000
1000	100	14211

STRESS INTENSITY FACTOR	
MPa $\sqrt{\text{m}}$	ksi $\sqrt{\text{inch}}$
0.91	1
1	1.10