Stress corrosion cracking of SCWR candidate alloys: A review of published results

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Abstract

Research on the development of supercritical water-cooled reactors (SCWRs) has generated a large pool of corrosion and stress-corrosion data in the open literature [1]. These data complement other aspects of reported materials properties such as creep and irradiation damage as well as microstructural degradation under various exposure/testing conditions. Unlike mechanical performance, the stress corrosion cracking (SCC) susceptibility of an alloy in a given environment can be affected by many factors including alloy composition and microstructure (including the degree of cold-working), chemistry of the environment, and the mechanical loading condition including the rate of loading (the so-called strain-rate). For in-core materials, the amount of neutron damage to which the alloy is subject also plays a key role in its SCC susceptibility. A summary is provided in this paper of the key findings from a survey of SCC test results published since the 1950s.

1. Introduction on SCWR and material selection

A large number of researchers from the Generation IV International Forum (GIF) community are currently working on various types of supercritical water-cooled reactors (SCWR). Both pressure vessel and pressure tube SCWR concepts are being explored. In Canada, the CANDU-SCWR is seen as a logical evolution of current CANDU[1] designs. The operating temperatures and pressures in the proposed CANDU-SCWR will be significantly higher (625°C and 25 MPa at the point of coolant exit) than those of existing water-cooled reactors and other proposed pressure-vessel SCWRs. These aggressive conditions render most commercial alloys unsuitable for use in-core and even some out-of-core components. For example, Zr-based alloys, currently used for fuel-cladding and CANDU pressure tubes, show excessive corrosion and creep at SCWR temperatures. While the insulated fuel channel concept [2] is expected to solve this problem for the pressure tube, the current Zr-based cladding alloy will not likely be suitable, except possibly at the entrance to the fuel-channel where the temperature is not yet over the critical transition point.

In selecting candidate in-core materials, the effects of the corrosion products released from the materials into the coolant must also be considered. The release and transport of corrosion products from the surfaces of system components and their subsequent deposition has been a serious concern for current water-cooled nuclear power plants. Very little is known about how

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corrosion products may behave in an SCWR; data from fossil-fired SCW power plants suggests a significant risk of deposition of corrosion products released from out-of-core surfaces onto fuel cladding surfaces in-core, even when materials with low general corrosion rates are used [3]. Surface alloying or coating [4] is an approach that can be employed to minimize the effects of corrosion by deposition of a highly corrosion-resistant metal layer on a creep and SCC-resistant substrate.

Materials research for the SCWR has been going on for many decades, starting in the early 1950s [5, 6], not too long after the Chicago-pile was commissioned. The past decade saw a rapid increase in SCWR materials-related technical publications. It is interesting to note that some of the highest test temperatures reported for SCW corrosion tests were those reported by Boyd and Pray [5], at the 12th Annual NACE Conference in 1956. They studied the corrosion and stress-corrosion cracking (SCC) behavior of twelve Ni-Cr-Fe alloys (410, 302, 347, 309, 310, 17-4PH, 17-7PH, A-286, Inconel X, Hastelloy F, X, AMS5616) at 427, 538 and 732^{o}C.

In the course of establishing a SCWR corrosion database [7], a joint effort among NRCan's Material Technology Laboratory (MTL), AECL, the University of Alberta and the University of New Brunswick, some important observations were made in terms of data and knowledge gaps in the corrosion or SCC tests reported in the literature. At the time of writing, over 500 corrosion and 37 SCC data sets have been collected, covering over 100 different alloys and a variety of test temperatures and pressures. These data span the time period from 1957 [6] to December 2010. Representative observations of the data gaps in terms of stress-corrosion and general corrosion are presented and discussed in this paper.

2. Stress-corrosion cracking tests on candidate SCWR alloys

The fuel cladding material, as well as alloys for other components, must be resistant to SCC, which is a well know failure mechanism for many metals and alloys in various environments. In boiling water reactor (BWR) type oxidizing hot-water conditions, sensitized austenitic stainless steels are known to suffer cracking [8]. Many factors affect the SCC susceptibility of an alloy in a given environment; some of the key factors are alloy composition and microstructure (including the degree of cold-working), chemistry of the environment, and the mechanical loading condition including the rate of loading (the so-called strain-rate). For in-core materials, the amount of neutron damage to which the alloy is subject also plays a key role in its SCC susceptibility. Irradiation can accelerate the degradation process in alloys; it affects, for example, segregations of elements to grain boundaries, void-formation in the microstructure and hardening. The irradiation assisted stress corrosion cracking (IASCC) phenomenon has been a generic problem in light-water reactors for many austenitic and nickel-base alloys.

Some candidate SCWR alloys have been tested for SCC in SCW, mostly using un-irradiated materials, although some tests have been performed on pre-irradiated samples. The SCC test conditions for the various alloys tested are summarized Table 1. The general finding is that the 3XX series stainless steels such as 304 and 316 are prone to SCC, as are many Ni-based alloys such as Alloy 600, 625 and 690. On the other hand, many ferritic materials such as T/P91 and F82H show good immunity to SCC.

Table 1. Summary of SCC tests surveyed in the open literature

Record #	Alloy	Test T (C)	Test Pressure (Mpa)	[Oxygen,ppb]	Test method
SCC-001	304	500	30	200	tensile bar
SCC-001	F/M steels, austenitic SS, Ni,Zr,Ti-base alloys	from 400 to 732	25	<10ppb-8ppm	CERT
SCC-002	F/M steels, austenitic SS, Ni,Zr,Ti-base alloys	from 250 to 732	25-60	<10ppb-8ppm	CERT
SCC-003	316L,316LGBE,690,690GBE	500	24	<10ppb	tensile
SCC-004	T91, HCM12A, HT-9, weld T91, weld HCM12A	400-600	25	100, 300 appb	tensile bar
SCC-005	316L, D9, 690 and 800H	400-500	24	<10ppb	CERT
SCC-006	304, 316, 690	<600	<30	<10ppb	CERT
SCC-007	304, 316, 690,JPCA	385-550	23.4-27.6	<10ppb	CERT, CGR
SCC-008	316	288-500	from 10 to 25	<10ppb-2ppm	Compact tesion
SCC-09	304,316,625,690	400-550	25.5	<10ppb	CERT
SCC-010	A718, A690	400, 600	25	<10ppb, 8ppm	CERT
SCC-011	304,316,600	290-550	25	8ppm	SSRT
SCC-012	304,316,625,690	500, 550	25.5	<10ppb, 8ppm	CERT
SCC-013	ODS steels	510, 288	25, 7.8	8-10ppm	SSRT
SCC-015	625	<=500	<37	mol fraction7×10^{-2}	Pressure tubes
SCC-016	316	400	25, 60	8, 800ppm	SSRT
SCC-017	316,625,C-276,MC-alloys	400	25	8ppm	SSRT
SCC-018	800H, HT-9	370-600	25	2ppm	tensile bar
SCC-019	Ni-based and Fe-based alloys	400-500	22-25		SSRT, CL
SCC-020	316L, 690	400-500	25	<10ppb	CERT
SCC-022	T91, T92, T122, 625, 690, 800H, MA956	370-600	25	<10ppb	U-bend sample
SCC-023	316, 625, HC276, MC alloy, MAT21	400	25		SSRT
SCC-024	Good modeling paper				
SCC-025	F82H	290-550	23.5	0.2ppm	round bar specimen
SCC-026	HT-9, T91, HCM12A	400-500	25	<10,100, 300 ppb	CERT
SCC-027	304L, 310S, 316	max T: 620	maxi pressure 30	8 ppm	SSRT
SCC-028	Ferritic/martensitic steels and autenitic alloys				
SCC-029	316, 316L	360, 400	25, 30, 40, 60	8 ppm and 800 ppm	SSRT
SCC-030	625, 617	600	26.7		19.0 mm thick plate
SCC-031	316L	550	25	5, 200 and 900	SSRT
SCC-032	304, 316L	500	25	8 ppm	SSRT
SCC-033	316L, 690, 625, 718		25.5	<10 ppb	CERT
SCC-034	304,310,316	500, 650	>30 Mpa	8 ppm	capsule

3. The effect of strain rate on SCC

In certain metal/environment systems where the metal sustains general passivity, it is known that there is a strain rate dependence in SCC testing [9]. To control the strain rate in a SCC test, two methods are widely used: constant extension rate tensile (CERT) and slow strain-rate tensile (SSRT) tests.

For water in the subcritical regime, Enjo *et al* [10] reported that in SSRT, the intergranular SCC (IGSCC) susceptibility of solution-treated 304 increased with decreasing strain rate independent of the solution treatment temperature, within the tested strain rate range of $8.35\text{x}10^{-7}$ s^{-1} to $4\text{x}10^{-5}$ s^{-1}. The SCC susceptibility was assessed using the reduction-in-area parameter, i.e., a relative comparison of the changes in the cross-section area of the ruptured sample. Nobuo *et al.* [11] tested austenitic steel (304 SS) and Inconel 600 using SSRT in high temperature water (360^{o}C), at an applied strain rate ranging from 1×10^{-7} s^{-1} to 1×10^{-5} s^{-1}, and found that the fraction of transgranular facets in the brittle fracture increases with increasing strain rate.

In SCW, Novotny, *et al* [12] reported the strain rate effect in their work on 316 L for a strain rate range of $1\times10^{-7}\ s^{-1}$ to $6.7\times10^{-7}\ s^{-1}$ with the oxygen concentration controlled at 5, 200 and 900 ppb in various tests conducted at 550°C. The results revealed that the combined effect of the strain rate and oxygen level correlated well with the fraction of SCC area found on the fracture surface, in that the high-strain rate/low oxygen condition produced less SCC in the sample and the low strain rate/high oxygen condition produced the most, up to 15% SCC areas on the fracture. In these tests, the strain rate did not significantly affect the values of maximum stress.

The effects of strain rate on SCC of austenitic stainless steels under BWR and PWR water chemistry conditions have been well studied. The role of bulk or crack tip plasticity is generally understood from the viewpoint of a slip-dissolution/oxidation process that governs the crack formation and growth. While an in-depth discussion of the mechanistic aspects of the strain rate effects is beyond the scope of this paper, it suffices to state that it is a critically important factor in SCC of all metal/environment systems. When the applied straining is applied at a rate that is above a critical strain rate, protective film formation cannot occur fast enough to repair the bare metal site exposed by mechanical straining, and the test sample fails by ductile rupture. If the strain rate is below a certain threshold value, film formation will rapidly repair the ruptured film, reducing the amount of metal oxidation taking place at crack tip, and thus the cracking is reduced or avoided. The latter explains why certain alloys do not exhibit SCC under static loading condition whereas cracking is readily produced under identical environmental conditions in a dynamic loading test such as SSRT.

It is generally recognized that dissolution or oxidation process in SCW conditions will be drastically different from those occurring in the subcritical water regime, because of large differences in both thermodynamic and kinetic properties of the water and of the relevant compounds forming the corrosion product layer. Advances in understanding of passivity of various metals and alloys in SCW will facilitate greatly our study of stress-corrosion cracking, which, as a subject of scientific research, is still in the early stage of raw data generation/collection. Only a limited number (less than 50) of papers have been published to date.

4. SCC mode in various candidate alloys

Different types of Cr-Ni-Fe alloys have shown distinctly different SCC properties in the screening SCC tests reported by various workers. For example, for austenitic and Ni-based steels, significant IGSCC occurred in Alloy 625 and 304 SS, whereas Alloy 690 and 316L SS showed both transgranular (TG) and intergranular (IG) cracking [13]. For F/M steels, Hwang, *et al* [14] performed SCC tests using F/M steels at 500, 550 and 600°C, 30 MPa in SCW, and no SCC was observed on the fractured surface of the T91 steel at these temperatures. High Cr ODS steel with Al addition also showed better performance at 561 K in SCW using the SSRT method [2].

Table 2 summarizes the 'predominant' mode of cracking found in some of the alloys that have been found to be susceptible to SCC in SCW. It should be pointed that for cracking in a given stainless or Ni-based alloy, transition from intergranular mode to transgranular mode is expected to be possible in the course of the crack growth process, as in the well-known case of cracking of 304 SS in BWR chemistry.

Table 2. Mode of cracking of select alloys tested in SCW conditions

Alloys	Alloy type	TGSCC	IGSCC
304	Austenitic	x	x
310	Austenitic	o	x
310+Zr	Austenitic	o	o
310+T+Nb	Austenitic	o	o
316	Austenitic	x	x
321	Austenitic	o	o
600	Austenitic		x
625	Austenitic		x
690	Austenitic	x	x
718	Austenitic		x
800H	Austenitic		o
T91	Ferritic	o	o
HT9	Ferritic		x
F82H	Ferritic	o	o
C276	Austenitic	o	o

(x: cracked in SCC tests; O: no cracking in the tests reported)

5. Metallurgical effects

Alloy composition has a great influence on its resistance to corrosion or SCC in SCW. Although Ni-base alloys and austenitic stainless steels are generally known to be prone to SCC, individual alloys within this group have varied resistance to cracking. For example, 310SS tends to show less SCC susceptibility than 304SS owing to the high Ni and Cr levels in the former. Since 310SS and 316SS have desirable high-temperature creep properties and relatively high general corrosion resistance, further improvement in SCC resistance would make them suitable candidates for use as in-core components. As Cr-carbide formation in these alloys is the leading cause of Cr depletion leading to susceptible SCC paths in the microstructure, reduction of overall carbon level or other means of avoiding MC-type of carbide should lead to improved SCC resistance. This is the rationale for the development of Zr-modified 310SS (the Japanese H2 alloy) and Ti-Nb modified 310SS (known as T6F alloy in Japan) [15] as well as the low carbon 316L SS. SSRT tests at a strain rate of 10^{-7} s^{-1} at 550^{o}C so far, did not produce visible cracking in these modified alloys. [15].

For other alloys, grain boundary engineering (GBE) and surface modification have also been shown to mitigate SCC. Allen and Was [16] tested H800 and HT9 using GBE and surface modification methods. Result showed that the alloys improved by these two techniques showed good performance in terms of SCC or spallation. Similar experiments using grain-boundary-engineered 316L and Alloy 690 were also performed [17], and the results showed the effectiveness in reducing cracking propensity at low strain levels.

6. Water chemistry effect on SCC

Fujisawa, *et al* [18] reported that the concentration of NaOH would increase the susceptibility to SCC of Hastelloy C-276 or other Ni-based alloys; when the concentration of NaOH was increased to 0.01 mol/ L, the failure mode was almost fully intergranular. They also investigated the effect of reducing atmospheres on SCC by increasing the hydrogen gas partial pressure, and showed that the susceptibility to SCC of these alloys decreased with the addition of hydrogen [19]. Bosch, *et al* [20] investigated the effect of chloride on SCC using Ni- and Fe-based alloys and found that chloride additions lead to crack initiation and propagation, even during the first few hours of constant load tests at 400°C and 460°C. It should be noted, however, that the water chemistry conditions in these tests are not representative of those expected in an SCWR, except perhaps in crevices.

The concentration of oxygen in SCW, which ise a -product of water radiolysis, has been studied for a number of austenitic stainless steels and Ni-based alloys. The general finding is that the SCC susceptibility is higher when higher levels of oxygen are present in the test water. Ampornrat, *et al* [21] tested F/M steels in SCW containing different concentrations of oxygen and the results showed that both crack density and maximum crack depth increased in the more oxidizing environment.

7. Effects of temperature and pressure on SCC

Teysseyre and Was[13] reported that SCC severity is highly temperature-dependent. The crack growth rate increased nonlinearly with temperature and the dependence can be described by Arrhenius behavior.

The pressure (density) of SCW can affect the SCC mode. Above the critical point, the water density can be varied continuously at constant temperature by varying the pressure. Watanabe et al [22] performed SSRT tests on 316 SS in SCW at a temperature of 400°C and a strain rate of $2.78x10^{-6}$ s^{-1}. The 316 alloy exhibited intergranular cracking at high density (P > 35 MPa) but the cracking changed to a transgranular mode at lower water density (pressures of 25 MPa and 30 MPa).

8. Irradiation Effects

Teysseyre *et al.* [23] showed that irradiation up to 7 dpa at 400°C and 500°C resulted in increased SCC in SCW for 316L or Alloy 690 in comparison with the unirradiated case; the effects were more pronounced with increasing temperature. Zhou, *et al* [24] reported similar results for 316L, D9, Alloy 690 and Alloy 800H. Results also showed that severe cracking occurred in these materials when irradiated.

9. Comments on the SCC test methods

It should be pointed out most SCC tests have been conducted using the SSRT and constant extension rate tests (CERT). Tests using pressurized capsules, as well as tests using U-bend samples, have also been reported by a few groups. Different test techniques can sometimes produce very different results regarding the SCC susceptibility of an alloy. For example, in a study in supercritical water containing hydrogen peroxide (up to 10% by wt), Alloy 625 was shown to be sensitive to SCC in slow strain rate tension both at 400°C and 500°C under a pressure of 25 MPa [16]. Constant load tests did not show any significant amount of cracking when the applied constant stress was at 100% and 140% of the yield strength. This shows the role played by the dynamic plasticity at the metal surface in the SCC initiation and propagation process.

In a SSRT or CERT test, test samples are strained to failure and then the presence or absence of stress corrosion cracks is confirmed by post-mortem examination. The technique is very severe in comparison with real-life stressing conditions, as the cracks are usually developed after the yield point of the alloy has been exceeded, and in fact, they could have initiated anywhere between the yield and the UTS point of the alloy on its stress-strain curve. SSRT or CERT tests are commonly carried out for materials susceptibility studies. However, in terms of data generation for alloy qualification or confirmation of code requirement, these tests have limited value.

A general observation of this literature survey is that very few experimenters have used the same test conditions (temperature, pressure, strain rate, water chemistry), so inter-laboratory comparisons are difficult to make. The study on SCC of 304SS is a good example. As seen in Table 1, this alloy has been extensively tested by many groups but each using a different set of test conditions or sample surface conditions. Whereas most tests showed that SCC can be readily produced in SSRT or CERT tests, there is however one reported result of no cracking [15]. This discrepancy points to the need for round-robin testing under well-controlled conditions.

10. Concluding remarks

As the surface temperature of the fuel cladding in the proposed CANDU-SCWR can reach as high as 850°C, corrosion and stress corrosion data are required at the relevant temperatures. A fundamental challenge is that, with the increase in temperature, an increasing number of precipitates will start to form in the microstructure, which could have strong effects on long-term SCC properties. The highest temperature for which corrosion and stress-corrosion testing in SCW has been carried out in known to date is 732°C, in the work carried out at Battelle in the mid-1950s using static pressure-capsules. The longest test duration was also from this work, with testing lasting for about 130 days. The synergistic effects of evolving alloy microstructure at higher temperature and long durations on SCC, and on corrosion as well, are largely unknown.

The test environment is another area where significant gaps exist. Most of the data produced so far are for pure water in the supercritical state; the effects of various coolant additives, which may be necessary in order to control the water chemistry, are also unknown.

Surface finishing and the degree of cold-working of the test alloys are known to affect the SCC susceptibility of austenitic stainless steels in subcritical BWR and PWR environments. It is reasonable to anticipate that these metallurgical factors are also important in SCC under SCW condition. However, related reports are very limited at the current time.

A general finding is that, even for the well-tested alloys such as 316SS and 304SS, very few experimenters have used the same test conditions (temperature, pressure, strain rate, water chemistry), so inter-laboratory comparisons are difficult to make. Round-robin testing under well-controlled test conditions appears to be essential in resolving some of the discrepancies reported in the literature.

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