MATERIALS AND CHEMISTRY FOR A SUPERCRITICAL WATER-COOLED REACTOR - PROGRESS AND CHALLENGES

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Abstract

The identification of suitable materials, and an appropriate water chemistry regime to minimize their degradation, are key challenges in the development of a Supercritical Water-cooled Reactor. In recent years, a significant body of research has been carried out to characterize the behavior of materials under conditions expected in an SCWR. With the increased amount of data available, it is now possible to evaluate the experimental uncertainties and to assess the role of key variables. It is also becoming possible to more accurately identify the mechanisms through which the SCWR environment (e.g., water chemistry) will interact with materials leading to degradation. In this paper, the interface between materials and chemistry is examined in the context of the role of dissolved oxygen in the corrosion of candidate alloys. Some suggestions for future work are given.

1. Introduction

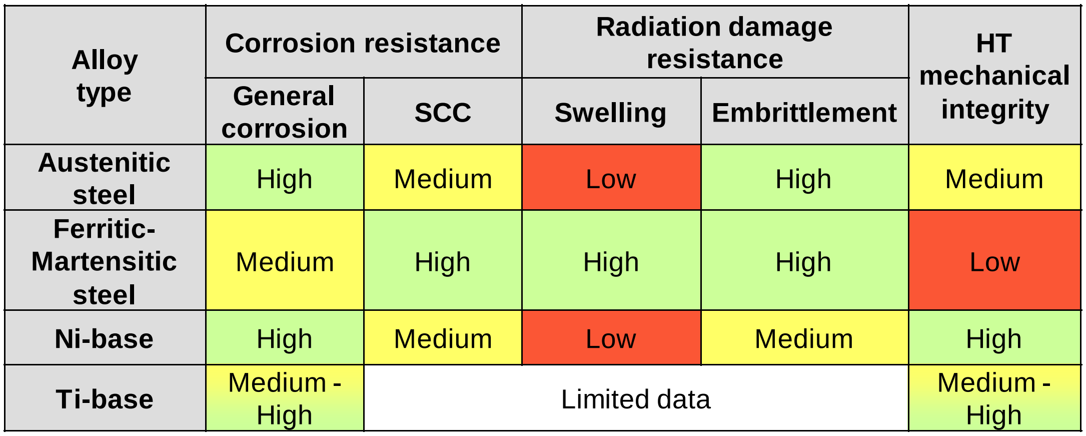
At its inception several years ago, the Generation IV International Forum Supercritical Water-cooled Reactor Materials and Chemistry Project Management Board identified two major challenges that needed to be overcome to ensure the safe and reliable performance of an SCWR [1]:

1. There were insufficient data available for any single alloy to unequivocally ensure its performance in an SCWR, especially for alloys to be used for in-core components.
2. The current understanding of SCW chemistry was inadequate to specify a chemistry control strategy, as the result of the large changes in physical and chemical properties of water through the critical point, coupled with the as yet poorly understood effects of water radiolysis.

Since those words were written, a significant amount of research and development work to address these two issues has been performed. While it would be premature to state that the issues have now been resolved, our understanding of the behavior of materials under SCWR conditions, and of the water chemistry issues that must be addressed, is advancing rapidly. Table 1 summarizes both the classes of materials that have been tested and their performance with respect to the key parameters of corrosion resistance (general and stress corrosion cracking), radiation damage resistance (swelling and embrittlement) and high temperature mechanical integrity [2]. It is clear from Table 1 that no existing material meets all the requirements.

As an example of the progress that has been made, in an extensive materials screening program, Japanese researchers have examined a number of candidate materials (both commercial and modified materials) for their usefulness in an SCWR. Testing to characterize the high temperature tensile strength, creep, He embrittlement, general corrosion, stress corrosion cracking, and effects of neutron irradiation (with post-irradiation examination) were performed. The result was the selection of the modified stainless steel SUS310S + Zr as the candidate fuel cladding for a thermal spectrum SCWR, because of its high temperature strength, low creep, general corrosion resistance, and good irradiation resistance. [3, 4, 5]

Table 1: Summary of Damage Types Relevant to SCWR Candidate Material Classes (adapted from [2]). SCC- Stress Corrosion Cracking; HT – High Temperature



Alloy type	Corrosion resistance		Radiation damage resistance		HT mechanical integrity
	General corrosion	SCC	Swelling	Embrittlement	
Austenitic steel	High	Medium	Low	High	Medium
Ferritic-Martensitic steel	Medium	High	High	High	Low
Ni-base	High	Medium	Low	Medium	High
Ti-base	Medium - High	Limited data			Medium - High

In 2011, Canada, the European Union, and Japan have all reached, or are reaching, the end of initial phases in their materials and chemistry development programs. It is therefore of value to look back on the work that has been performed to date, with the aim of developing a better understanding of the work that will need to be carried out on materials and chemistry in order to build a prototype. Obviously, such a review would be beyond the scope of a conference paper, so the discussion will be limited to the interface between water chemistry and materials, and in particular, the effect of oxygen on general corrosion in SCW.

2. Corrosion

Initial alloy selection for testing for the SCWR was guided by existing data from supercritical and ultrasupercritical fossil-fired power plants, supercritical water oxidation (SCWO) systems, and from programs developing advanced materials for other reactor systems including fusion. There are a number of excellent summaries of the work that has been carried out in support of materials development for supercritical and ultrasupercritical fossil-fired power plants, including a recent paper by Wright and Dooley [6]. While extensive testing was carried out in support of the development of SCWO processes, the chemistry conditions were typically not of direct relevance to an SCWR, being very acidic with high concentrations of aggressive species such as chloride ion. Therefore, although knowledge gained from current reactor designs, modern boiler technologies and research in support of SCWO has provided valuable insights that have aided in the identification of key parameters, there are still significant gaps in our knowledge about the properties of the materials under proposed SCWR operating conditions.

Tests in support of SCWR development have been performed at temperatures ranging from below the critical temperature up to 650°C. The water chemistry has typically been low conductivity "pure" water with nominal dissolved oxygen concentrations ranging from <10 ppb to 8 ppm. Coupons have been examined after SCW exposures ranging from 24 to 3000 hours in length. After exposure to SCW, test specimens have been characterized using techniques ranging from weight change to surface analytical methods such as Scanning Electron Microscopy, Transmission Electron Microscopy and Scanning Auger Microscopy. Tests have been performed in static autoclaves, capsules, and loops; each type of test facility has advantages and disadvantages.

The result of these programs has been the generation of a large amount of data; the SCW materials database being developed in Canada currently contains general corrosion data for over 90 alloys [7]. Ultimately, the rationale for these measurements is to collect the data necessary to ensure that the various SCWR components, for example, the fuel cladding, will meet the design requirements (e.g., corrosion allowance). However, critical assessments of these data are scarce and review articles quickly become outdated because of the rapid pace of experimental work. In additional, there are few data sets in which the experimental conditions (temperature, water density, water chemistry, test duration, flow) are the same, or at least similar enough to allow meaningful comparison.

Figure 1 shows the results of an extensive series of tests of the corrosion of 304 SS in SCW as a function of exposure time, SCW density, water chemistry and surface finish, performed at Chalk River Laboratories. The purpose of these tests was to elucidate the relative contributions of the various factors that contribute to corrosion in SCW, in order to better target the experimental work on material selection and testing. A second goal was to assess the reproducibility of the tests. The conclusion of this work was that the relative contributions were (in order of decreasing importance):

$$\text{Temperature} \approx \text{Surface Finish} > \text{Water Chemistry} > \text{Density}$$

It was found that the major factor affecting the reproducibility of the tests was the coupon preparation method (e.g., polishing). The effect of surface cold work of austenitic stainless steels has long been known to improve the corrosion resistance of these materials. This is believed to result from the rapid build-up of a protective Cr-rich oxide because of enhanced Cr diffusion to the alloy surface via the multiple diffusion pathways created by the local deformation.

While data from the SCWO literature suggested that a maximum in the corrosion rate occurs at temperatures near the critical temperature, such a maximum is not observed in Figure 1 (or in other testing for the SCWR). The rationale for occurrence of such a maximum was the balance between the increasing corrosion rate with temperature and the decreasing SCW density and degree of water dissociation above the critical point [8].

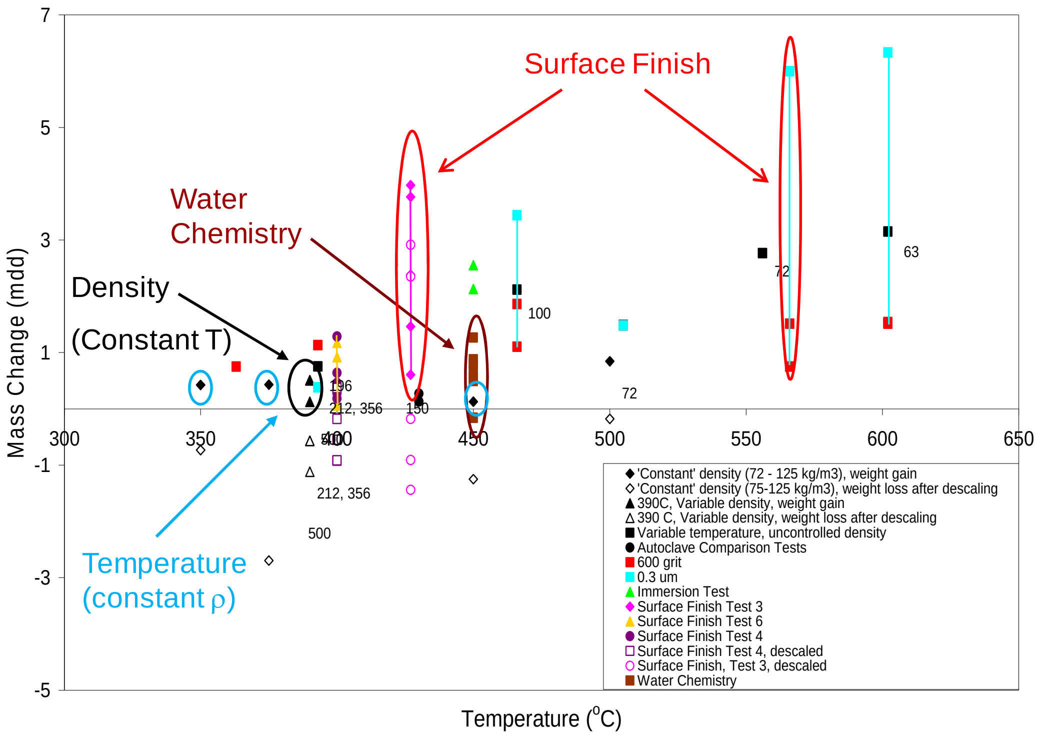


Figure 1: Weight change versus temperature for 304 SS in supercritical water in a static autoclave. Filled symbols - weight change before descaling, open symbols - weight change after descaling. The highlighted data are from selected tests in which the indicated variable was studied.

The testing reported in Figure 1 also showed little effect of dissolved oxygen concentration (water chemistry in the Figure); however, the tests were performed in static autoclaves, so that the dissolved oxygen concentration could not be controlled. Therefore, a review of the SCWR corrosion literature was performed to better understand the oxygen effect on corrosion in SCW.

2.1 The Nature of the Oxidizing Species

It is clear, upon careful examination of the SCWR corrosion literature, that the nature of the oxidizing species involved in the corrosion process has not been clearly defined. In fossil-fired SCW plants, it is generally assumed that the oxidant is water, as the concentration of oxygen in the steam is typically low [6]. The dissociation of water on the surface yields:

$$H_2O(g, ads) = 2H^+ + O^{2-}$$

or

$$H_2O(g, ads) = H^+ + OH^-$$

and the resulting O^{2-} and/or OH^- act as the oxidants.

In an SCWR, corrosion in the core and downstream piping is complicated by the radiolytic breakdown of water [9]. As in existing water-cooled reactors, the formation of oxidizing products, such as $\cdot OH$, H_2O_2, O_2, and $HO_2\cdot/O_2-\cdot$ by water radiolysis will be additional source of oxidizing species in the SCWR coolant. Current understanding of water radiolysis in SCW is limited due to a lack of experimental data, and it is not yet possible to predict the concentration of dissolved oxygen expected to be formed in an SCWR core. It is also unclear whether the strategy of adding hydrogen to the coolant to suppress the net radiolytic formation of oxidizing species, as is current practice in pressurized light and heavy water reactors, will work in an SCWR core [10]. As a result of these uncertainties, it is conservative to test over a range of concentrations of dissolved oxygen (and peroxide), and to elucidate the role of oxygen in the corrosion of materials in SCW. In this context, it is important to note that oxygen (and other gases) is completely miscible with SCW, and as a result, the oxidizing power of the coolant near the end of the core and into the downstream piping in an SCWR could be very high.

There are three potential sources of oxygen in an SCWR core and downstream piping:

a) The feedtrain, if O_2 is added as part of the chemistry control regime
b) Thermal decomposition of H_2O
c) Water radiolysis

If oxygen is added as part of the chemistry control regime, it is expected that the amount of residual oxygen at the core inlet will be low due to reactions with the feedtrain piping. The feedtrain chemistry can likely be adjusted to ensure an acceptable inlet oxygen concentration. The thermal decomposition of water is calculated to produce less than 5 ppb of oxygen at 625°C. Therefore, the major source of oxygen in the coolant in-core is expected to be water radiolysis.

A truly systematic experimental study of the effects of dissolved oxygen concentration in SCW over a wide range of temperatures and oxygen concentrations has yet to be performed. Some studies at low pressures relevant to the fossil-fired power plant industry have been performed to examine the effect of oxygen in steam. Studies have also been performed in support of the development of SCWO process, although the chemistry conditions (acid concentration and concentrations of aggressive ions such as chloride) are considerably higher than those expected in an SCWR, except perhaps in crevice regions. In the following section, a detailed review of the existing literature is presented.

2.1.1 Review of the Experimental Data

Figure 2 shows weight change data for HCM12A in a test system with flowing refreshed water [11]. The weight gain at 500°C decreases slightly (~13%) as the dissolved oxygen concentration increases from 10 and 300 ppb; it is unclear from the paper whether this change is larger than the experimental uncertainty. At a dissolved oxygen concentration of 2000 ppb, the weight gain is about 50% higher than that measured between 10 and 300 ppb. Figure 3 compares weight gain data at a number of dissolved oxygen concentrations for T91 and T92, as reported by a number of different groups. Within the estimated experimental uncertainty, and despite the slight differences in alloy composition, the data from the different groups appear to be the same, although in the test with 8000 ppb dissolved oxygen (500 h duration), the rate of weight gain appears to be slightly lower than in the other tests. It should be noted that the 8000 ppb data are from tests performed in a static autoclave; the corrosion rate in the static autoclave may become lower at longer exposure times because the test solution will become saturated with dissolved corrosion products, limiting their further release into the solution.

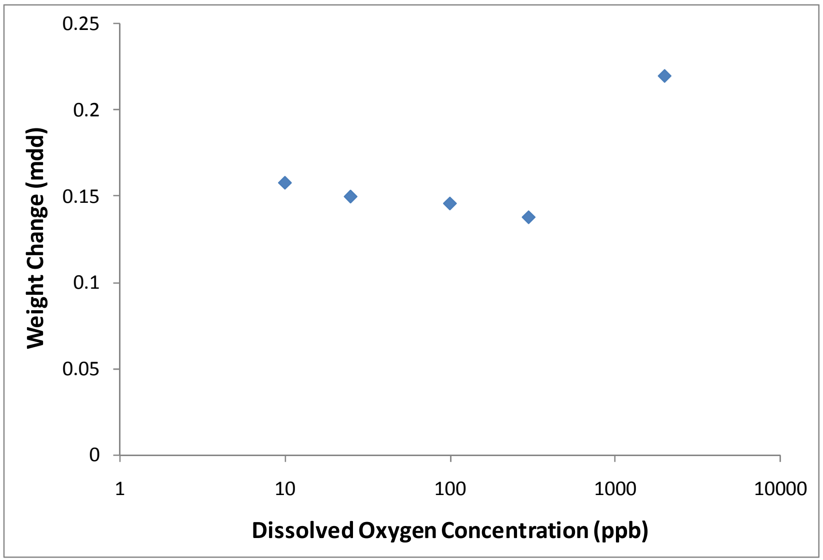


Figure 2: Weight change data in milligrams per decimeter^2 per day (mdd) for HCM12A in a test system with flowing refreshed water [11].

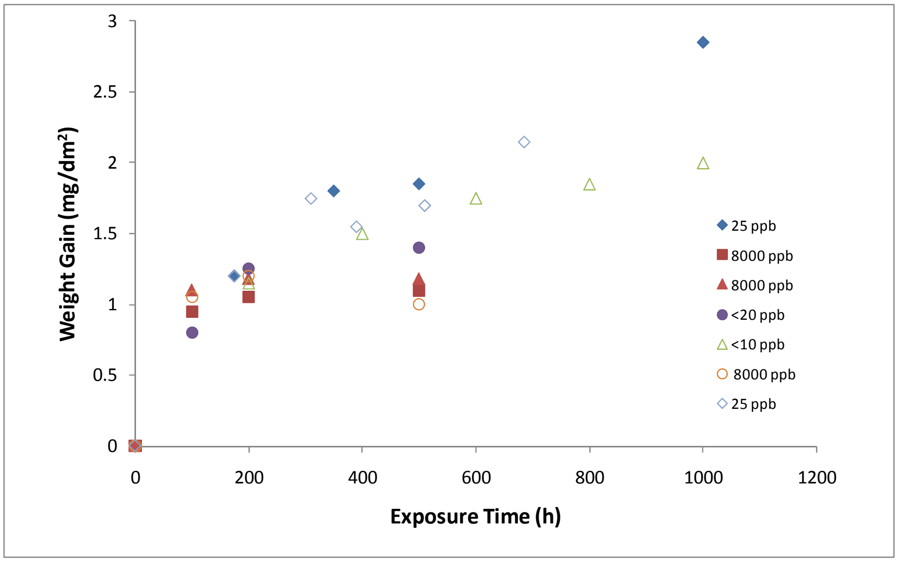


Figure 3. Weight gain as a function of time for T91 (closed symbols) and T92 (open symbols) exposed to SCW at 500°C and varying concentrations of dissolved oxygen (DO). Compositions of T91 and T92 are: Fe: Bal, C: 0.09, Mn: 0.38, Ni: 0.10, Cr: 9.38, Mo: 0.911, Cu: 0.08, V: 0.19, Al: 0.032, Nb: 0.080, and Fe: Bal, C: 0.07, Mn: 0.45, Cr: 9, Mo: 0.5, V: 0.2, Nb: 0.05, W: 1.8, N: 0.06, respectively. Data are from the following sources: 25 ppb – [12]; 8000 ppb – [13]; <20 ppb – [14]; <10 ppb – [15].

The usual materials in the feedtrain of most fossil-fired power plants are carbon or low alloy steels. The ability to use these in high temperature water takes advantage of the fact that, in high temperature water and steam, corrosion results in the formation of protective oxide layers; at temperatures greater than 200°C, a dense, uniform magnetite film is formed spontaneously on these steels. Below 200°C, the surfaces of these steels in contact with water remain active; however, corrosion or erosion-corrosion in the low temperature region, and corrosion product transport from there into the high temperature region, can be reduced by appropriate chemical measures ("feedwater treatments" [6]). It has been shown that the addition of molecular oxygen to flowing high purity water raises the free corrosion potential of the steel by several hundred millivolts [6]. The increase is especially significant in the temperature range below 150°C. The result is the formation of a double oxide surface layer consisting of magnetite covered by a layer with a high hematite (or other iron(III) oxide/hydroxide solid phase) content; these layers have very low corrosion product release rates into the coolant.

This dependence of oxidation on dissolved oxygen concentration is exploited in fossil-fired power plants. Combined water chemistry control in these plants involves addition of small concentrations of oxygen to the feedwater to enhance the formation of lower solubility oxidized iron species (e.g., hematite) between the magnetite grains, thus reducing the oxidation rate, perhaps by reducing the diffusion of oxygen through the multi-phase film. It has been suggested that a similar argument applies to the corrosion of F/M steels in SCW. However, it is not clear if such a mechanism is relevant in SCW, especially at temperatures above 500°C, where the corrosion process appears to be analogous to gas phase oxidation. Oxidation in gaseous environments is dominated by molecular processes and the diffusion rates of cations and/or anions through the corrosion film are the rate determining steps. Studies of ferritic-martensitic (F/M) alloys for use in SCWRs do not always show the presence of significant quantities of hematite on these surfaces at the concentrations of dissolved oxygen typically used in the feedwater (10 to 300 ppb). Indeed, electron back-scatter diffraction studies of oxide layers formed on F/M steels have found a two-layer film with an outer magnetite oxide on surfaces exposed to low concentrations of dissolved oxygen, and a three layered oxide film with a thin outer hematite layer on surfaces exposed to high concentrations of dissolved oxygen [16].

Figure 4a presents data for the corrosion of 316 and 316L stainless steels exposed to SCW over the temperature range 270 - 550°C with different concentrations of dissolved oxygen, from several different research groups [11, 17, 19]. Test durations for the data in Figure 4 were typically 500 h or higher; tests with shorter durations showed higher corrosion rates, as expected for corrosion processes that follow a parabolic behavior. It can be seen that the data from the different laboratories are in excellent agreement. Also included in Figure 4a is a data point for 316 stainless steel measured in SCW containing 2 wt.% H_2O_2 under similar conditions [18]. In spite of the high concentration of oxidant, the corrosion rate measured in this study was not significantly higher than that measured in deoxygenated water. Figure 4b shows that, above about 450°C, the data from Figure 4a at all dissolved oxygen concentrations are well-described by a single Arrhenius fit, suggesting that, under these conditions, the effect of dissolved oxygen on the corrosion rate of these materials is small. Below 450°C, the corrosion rate passes through a shallow minimum around the critical point; this is in contrast to the reports in the SCWO literature of a maximum in the corrosion rate at temperatures near the critical point.

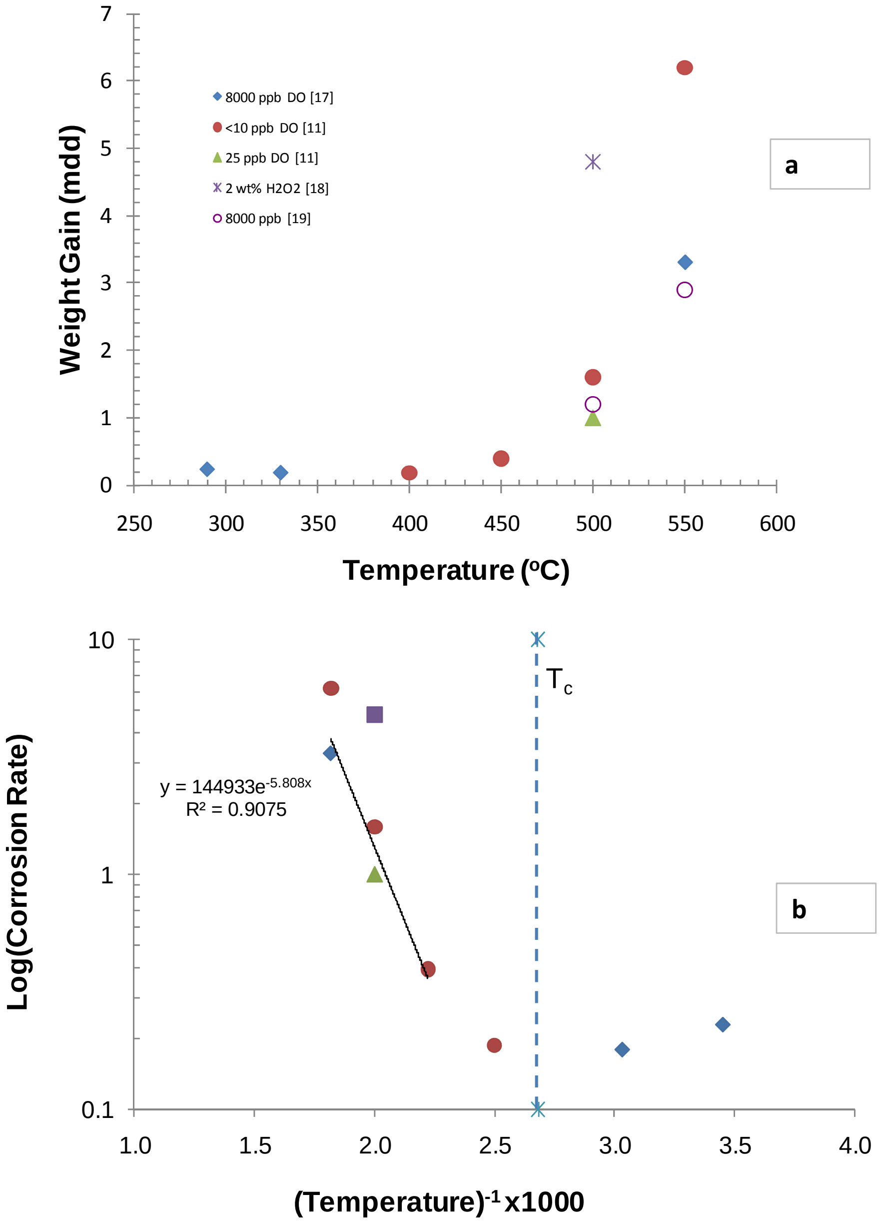


Figure 4. a) Weight gain rate as a function of temperature for 316 and 316L exposed to SCW containing various concentrations of dissolved oxygen. The source references are listed in the legend. Also included is a data point measured in 2 wt.% H_2O_2 under similar conditions. b) Arrhenius plot (log[corrosion rate]) vs 1/T) for the data in a). The vertical dashed line denotes the critical temperature; the line through the data points is a least squares fit to the data above 450°C.

Studies of material corrosion reported in the SCWO literature show that high concentrations of oxidants such as oxygen or hydrogen peroxide lead to very rapid corrosion of materials. It is interesting to note, however, that at high oxygen concentrations the corrosion rate of 316SS in 0.01 mol/kg H_2SO_4 solutions at 400°C, 25 MPa was lower at 800 ppm than at 8 ppm and 3 ppb dissolved oxygen concentration (Figure 5) [20]. This was attributed to the enhanced stability provided by the enhanced concentration of iron in the corrosion film. It is evident that more work is needed to understand the effect of oxygen concentration on corrosion of austenitic steels in SCW.

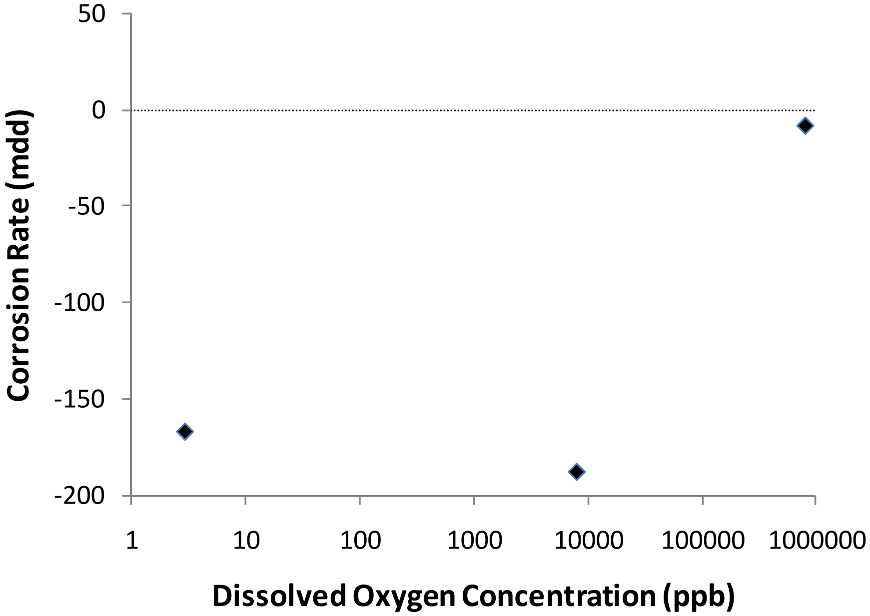


Figure 5: Corrosion rate of 316SS in 0.01 mol/kg H_2SO_4 solutions at 400°C, 25 MPa at 800 ppm, 8 ppm, and 3 ppb dissolved oxygen concentration (adapted from [21]).

The detrimental effect of dissolved oxygen on those alloys that depend on the formation of a Cr-rich protective oxide for their corrosion resistance occurs because, at high concentrations, oxygen (or other oxidants) will increase the solubility of the protective oxide. Figure 6a and 6b show the calculated Pourbaix diagrams for Fe and Cr in SCW at 400°C[1] [21]. Note that over a large range of potential, soluble Cr^{VI} species are more stable than the Cr^{III} species in the oxide, and therefore any Cr-rich oxide film will have a tendency to dissolve and be carried away by the coolant. Alternatively, one can consider that Cr^{III} species in the protective oxide film become volatile by the formation of $CrO_2(OH)_2(g)$ [6]; in SCW the two processes are equivalent. In corrosion tests of five alloys in SCW [22], coupons of AL6XN showed a pair of bands in the Raman spectra not observed in spectra from other materials (Figure 6c), attributed to the presence of Cr^{VI}-oxygen stretching modes [23,24]. The presence of more soluble Cr^{VI} species in the film on the AL6XN coupons were consistent with the observation that this alloy had the highest weight change of the five alloys studied.

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[1] Based on extrapolation of thermodynamic data from lower temperatures.

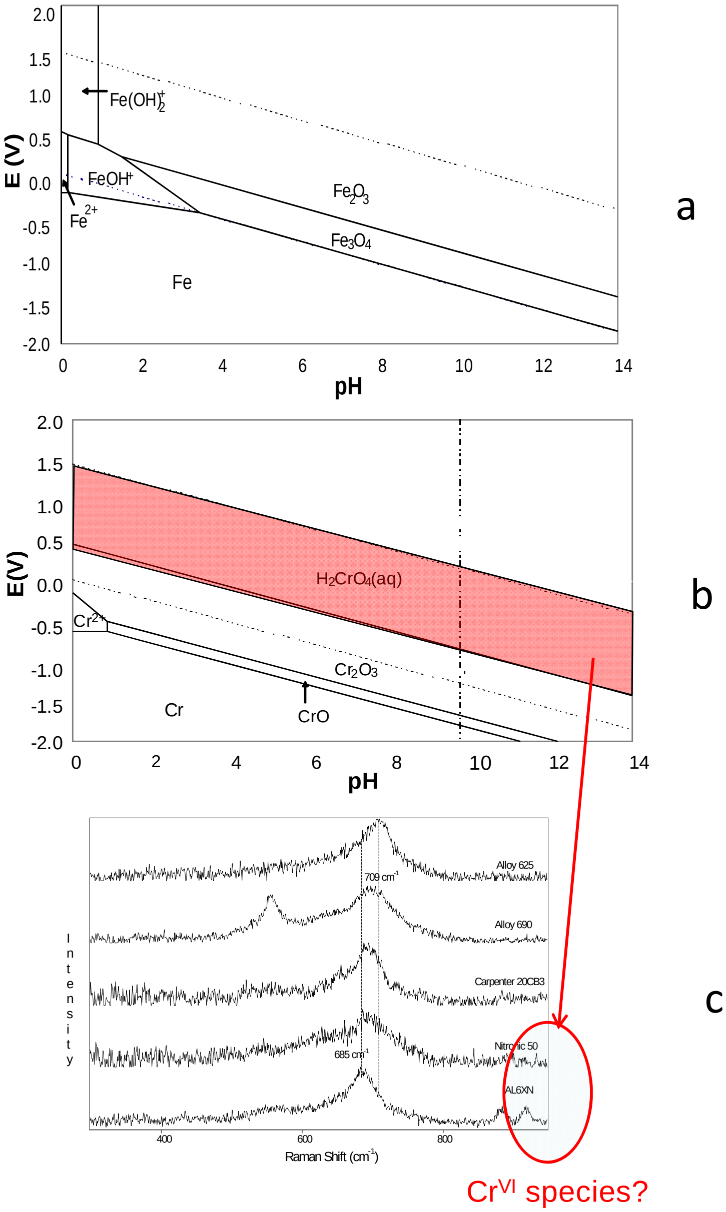

Figure 6: Calculated Pourbaix diagrams for Fe a) and Cr b) in SCW at 400°C (from [22]). The Raman spectra obtained from coupons of various alloys exposed to SCW at 450°C, 25 MPa are shown in c) [22]. From top to bottom the alloys are Alloy 625, Alloy 690, Carpenter 20CB3, Nitronic 50 and AL6XN.

3. Summary

A significant body of literature now exists on the corrosion of iron and nickel-base alloys in SCW under conditions relevant to an SCWR, and new data are being generated at a rapid pace. While much work remains to be done, it seems likely that suitable materials can be found for in-core components that could be used in a prototype SCWR that would operate with a core outlet temperature of less than 550°C.

As we move forward with longer-term and in-reactor testing, including fuel qualification testing, it is prudent to take a careful look at this body of data, assess the reliability, and identify areas where additional work is needed. From the data reviewed in this paper, it is clear that the corrosion data measured in different laboratories under similar test conditions are in reasonably good agreement; the proposed round robin testing to be carried out under the GIF Materials and Chemistry Project will help quantify the uncertainties.

In an SCWR, corrosion in the core and downstream piping is complicated by the radiolytic breakdown of water, leading to the formation of oxidizing products such as $\cdot OH$, H_2O_2, O_2, and $HO_2\cdot/O_2-\cdot$. In the absence of a predictive model of water radiolysis in an SCWR core, it is not possible to predict the concentration of oxidizing species that will form. As a result, it is conservative to test over a range of concentrations of dissolved oxygen (and peroxide) that bound the expected values. This is challenging experimentally, as oxygen is completely miscible in SCW; the typical practice of using air-saturated water at room temperature as a source of 'high-oxygen'(~8 ppm), while simple experimentally, may not give conditions that bound those that could be encountered in an SCWR core, or in piping immediately downstream of the core.

To reduce the required conservatism in the testing, it is therefore important to:

- Develop a predictive model of water radiolysis in SCW, and
- Elucidate the role of oxygen (and other potential oxidants) in the corrosion of materials in an SCWR.

While the role of dissolved oxygen concentration on the corrosion rate of iron-base alloys in SCW remains unclear, the data reviewed in this paper suggest that the effect may not be large. It is recommended that a systematic study of the effect of dissolved oxygen be carried out.

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