Environmental Degradation of Materials during Wet Storage of Spent Nuclear Fuels

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Wet storage is the predominant mode of storage of spent nuclear fuels. Due to legislation and other constraints, many countries do not reprocess spent fuels and have to store these for extended periods in spent fuel storage pools (SFSPs). Although the water chemistry of the pool is benign, certain factors such as stagnancy of water, crevices, and galvanic contacts between various materials of the fuel clad and the lining of the pools can result in unexpected localized corrosion. In this study, the susceptibility to localized corrosion of aluminum-1S (Al-1S), Zircaloy-2, and type 304 stainless steel (SS) has been assessed using accelerated tests with crevice bent beam (CBB) assemblies. The pool water constituents have been analyzed and electrochemical potentials (ECPs) measured in water samples drawn from different locations of the pool. The ECP has also been measured *in situ,* **in the pools. It has been demonstrated that under conditions of crevice and galvanic contact, aluminum clad fuels from research reactors are prone to localized corrosion even in the benign environments of a SFSP. The ECP experiments indicate the importance of surface condition of the material and irradiation on degradation of various materials due to corrosion.**

1. Introduction

The nuclear fuel is removed from the power and research reactors after a certain level of burnup and stored for a long duration.[1,2] The predominant mode of storage is wet storage in spent fuel storage pools (SFSPs). The SFSPs use demineralized light water and generally employ a water purification system to keep the specific conductivity of water less than 1 to 5 μ S/cm. The materials handled in SFSPs are the spent fuels with clad of various alloys of zirconium, aluminum, and magnesium. The CANDU type pressurized heavy water reactors (PHWRs) use Zircaloy-2 (Westinghouse Electric Company, Pittsburgh, PA) as a fuel clad, and a number of research reactors use pure aluminum, aluminum alloys, magnesium, and zirconium alloys as a fuel clad.[1,3,4] The spent fuels are stored on racks made of either aluminum and its alloys or stainless steel (SS). The pool walls and bottom are made of concrete and in some cases lined with type 304/ 304 L stainless steel.

Some countries must store the spent fuels for long periods either due to legislation or other constraints.[5] This calls for evaluation of the corrosion degradation aspects during extended wet storage (up to 100 years). The uniform corrosion rates of the fuelclad materials are reported^[6–10] to be too low to be of concern even during extended storage. Localized corrosion, particularly crevice corrosion, pitting corrosion, and stress corrosion cracking (SCC), is of utmost concern during extended storage.[11] The

galvanic coupling of two materials may also accelerate the rate of localized corrosion. Stainless steels and Zircaloys are known to be susceptible to SCC.^[12,13] During wet storage, the environmental parameters (C1– content and temperature) do not favor SCC. It was shown^[14] in studies with stressed SS that, in demineralized water with chloride concentration up to $10⁵$ ppm and $pH > 2$, pitting or SCC does not occur below 60 °C. However, there are many reported cases describing SCC of SS at temperatures less than 60 $^{\circ}$ C.^[15] Many of these are suspected to be caused by species other than chloride ions (sulfides, metabolic products of bacteria, *etc.*). On the other hand, Zircaloys undergo^[13] pellet-clad interaction-stress corrosion cracking (PCI-SCC) during use in the reactors. Therefore, it is possible that many Zircaloy-clad spent fuels enter SFSPs with incipient cracks. It is a matter of interest to establish if these SCC cracks propagate during wet storage to cause breaching of the clad. High purity aluminum-1S (Al-1S) is not known to be prone to SCC. The susceptibility of these materials to SCC under crevice and galvanic contact conditions is not known in SFSP environments.

In the present study, susceptibility to crevice and pitting corrosion and SCC is investigated for Al-1S, Zircaloy-2, and type 304 SS under accelerated conditions of galvanic contact and crevice geometry. The typical chemical composition of these materials is given in Table 1. Electrochemical potentials (ECPs) or the open circuit potentials are measured in SFSP environment for these materials to evaluate the effect of radiation on corrosion. Available relevant data from literature are also analyzed to assess the susceptibility of these materials to corrosion degradation during extended storage.

2. Experimental

2.1 Crevice, Pitting, and SCC Tests

The accelerated, localized corrosion tests were carried out by crevice bent beam (CBB) assemblies. These assemblies provide

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Table 1 Typical chemical composition of materials studied

Chemical composition (wt.%)											
Material	Fe	Cr	Ni		Mn	Si	Al	Mg	Zr	Sn	
304 SS	70	18	8	0.08(a)	2.0(a)	1.0(a)	\cdot \cdot \cdot	\cdots	\cdots	\cdots	
304L SS	70	18	8	0.03(a)	2.0(a)	1.0(a)	\cdot \cdot \cdot	\cdots	\cdots	\cdots	
$AI-1S$	0.35	\cdots	\cdot \cdot \cdot	\cdots	0.03	0.25	99.5	\cdot \cdot \cdot	\cdot \cdot \cdot	\cdot \cdot \cdot	
Zircaloy-2	0.15	0.10	0.05	\cdots	\cdots	\cdots	\cdot \cdot \cdot	\cdots	98	1.5	
$\sqrt{2}$											

(a) Denotes maximum values

crevices between surfaces of the sample and the CBB assembly. In addition, the corrosion process is accelerated for Al-1S samples due to its galvanic contact with SS assemblies. The bend of the samples in the assemblies provides for tensile stresses. The CBB assemblies^[16] were fabricated from type 304 L SS plates, and the details are given in Fig. 1. Samples, $110 \times 15 \times 1.5$ mm, were fabricated from the following:

- (a) Al-1S—as received and mechanically polished on emery paper, grit 120;
- (b) Al-1S—as in (a) and autoclaved in water at 150 \degree C for 15 days, prior to test;
- (c) Zircaloy-2—as received;
- (d) Zircaloy-2—as in (c) and autoclaved in steam at 400 °C for 3 days, prior to test;
- (e) Type 304 SS—as received and mechanically polished on emery paper, grit 120; and
- (f) Type 304 SS—as received and sensitized at 675 °C for 1 h and mechanically polished with grit 120 emery paper.

These samples were fixed in individual CBB assemblies and immersed close to the bottom of two SFSPs. The radiation level at the site of immersion of the samples is 1000 R/h (Roentgen per hour) maximum. At the Trombay SFSP, periodic inspection of these assemblies was carried out every 3 months, up to a total period of 1 year. At the Tarapur SFSP, inspection was carried out after a gap of 1 year. The parameters examined were surface appearance, pH in the crevices, and corrosion mapping. The CBB assemblies with fitted-in samples are shown in Fig. 2, at the time of removal from SFSP.

2.2 Evaluation of Pool Characteristics

In order to correlate the corrosion behavior of various materials, the following parameters were evaluated for the SFSPs: pH, Cl[−] ion concentration, specific conductivity, dissolved Fe, Cr and Ni concentrations. Water samples were collected from various locations of the pools, to assess the effect of corners and depth. In this paper, pool P refers to a pool situated at Trombay, where the spent fuel from research reactors (*i.e.,* metallic U fuel clad with Al-1S) is stored. Pool T is situated at Tarapur, where the spent fuel from PHWRs $(i.e., UO₂$ fuel clad in Zircaloy-2) is stored.

2.3 ECP Measurements

The ECP was measured in the freely corroding (or open circuit) conditions, and water samples were kept in "open to air" condition. The ECP measurements were carried out (a) in water samples collected from various locations of the SFSPs and

Fig. 1 Sketch of the CBB assembly used for crevice, pitting, and SCC experiments^[16]

(b) *in situ,* in the SFSP-at Trombay. The water samples were collected from the bottom, middle, and near top locations at the center and corners of the pools at Trombay and Tarapur. The materials used for ECP measurement were type 304 SS [(1) annealed and (2) sensitized], type 304L SS [(1) annealed and (2) sensitized], Zircaloy-2 (as received), and Al-1S (as received). These samples were polished on grit 120 emery papers. The ECP was measured by immersing the samples and the saturated calomel electrode (SCE) in the water samples and connecting these to a high impedance voltmeter. The ECP values were recorded immediately upon immersion and then after 5 min of immersion.

The *in-situ* ECP measurements were taken in the Trombay pool using a special setup, as shown in Fig. 3. In this setup, the sample of size $40 \times 20 \times 1.5$ mm and the SCE are fixed to the SS pipe, so that the distance between the sample and the calomel electrode is 25 mm. The samples used were type 304 SS, Al-1S, and Zircaloy-2 in the as-received condition. No surface preparation was carried out for these samples. The lead wires from the sample and the calomel electrode were long and connected outside the pool to the high impedance voltmeter. The contact points on the sample and the calomel electrode were insulated using lacquer and polytetrafluoroethylene tape, respectively. The ECP was measured when the sample and the calomel electrode setup was immersed about 1 m below the top water level (*i.e.,* away from the stored spent fuel) and then again at about 1 m from the bottom of the pool (*i.e.,* near the stored fuel).

Fig. 2 CBB assemblies after exposure in the spent fuel storage pool

Fig. 3 Schematic diagram of the setup used for *in-situ* ECP measurements

3. Results

3.1 Crevice, Pitting, and SCC Tests

Following are the observations/results of the tests carried out with CBB assemblies in two SPSPs.

- The pH of the water in the crevices generated between the sample and the CBB assembly decreased gradually from around 5.5 to around 2 to 3 in less than 6 months for all the assemblies.
- The Al samples (both the as received and the autoclaved) in the CBB assemblies were found to be corroded after 6 months of exposure, in both the pools.
- After 6 months of immersion, the as-received sample showed more crevice corrosion compared to the autoclaved sample. The extent of corrosion in the two samples was comparable after 1 year of exposure.
- The Al samples (in both the surface conditions) showed pitting attack on the cross-sectional surface of the samples. The pits were decorated with whitish products on the surfaces and the pitting attack occurred in both the pools. However, the number of pits was less (<5) on the cross-sectional surfaces.
- Corrosion mapping carried out on corroded Al samples indicated that more than 60% of the crevice surfaces had undergone attack. After 1 year of exposure, the overall corrosion rate was measured to be 122 and 112 μ m/yr for the polished and autoclaved samples, respectively. The maximum depth of corrosion damage was measured in the metallographic examination to be 0.62 and 0.44 mm, respectively.
- There was no evidence of any localized attack on any of the SS or Zircaloy samples.
- There was no evidence of SCC on any sample.

The appearance of these corroded samples after 6 months exposure in SFSP is shown in Fig. 4.

3.2 Evaluation of Pool Characteristics

The results of the chemical analysis of water samples from the two SFSPs are summarized in Table 2. The temperature of both pools is ambient, generally around 28 ± 1 °C.

3.3 ECP Measurements

The ECP values measured in water samples collected from various locations of both pools are given in Table 3. The values mentioned are those measured immediately after immersion of the samples. The ECP values measured after 5 min of immersion are also shown in the same table. The error in the measured potential due to the use of long lead wires during the *in-situ* measurements was found to be less than 5 mV. The measured *in-situ* ECP values are shown in Table 4.

4. Discussion

The corrosion aspects of materials have been studied in two SFSPs having broadly similar (Table 2) and benign water chemistries. However, both the pools show some variation in specific conductivity of water at some locations, possibly due to nonuniform mixing of water in some pockets of the pools. Although the pool water is demineralized water, the measured pH

Fig. 4 Surface appearance of (a) type 304 stainless steel, (b) Zircaloy-2, (c) Al-1S (oxidized), and (d) Al-1S (polished) samples after exposure with CBB assemblies in spent fuel storage pool for 6 months

is 5 to 6. This is due to the fact that the pools are open to air and absorb CO_2 , forming $HCO^{-3}[17]$ in water to lower the pH.

4.1 Crevice Corrosion

The occurrence of severe crevice corrosion of aluminum in the short test duration is attributed to its galvanic contact with SS assemblies and lowering of pH within the crevices. It is reported^[2] that dissolution of the aluminum oxide protective films can occur in solutions with pH <4 and >9. Galvanic corrosion accelerated the rate of crevice corrosion for Al samples. On the other hand, for up to 1 year of exposure of CBB assemblies, no Zircaloy or SS samples showed any evidence of crevice/pitting corrosion. This shows that even in the benign environments of the SFSPs, existing crevices (between stored spent fuels and the Al/SS racks or between two racks) could pose problems only for the aluminum clad.

Another interesting result is for the two surface conditions of aluminum. While the as-polished aluminum samples have only very thin air formed oxide film (2 to 3 nm),^[18,19] the autoclaved Al sample used in the study had about 20 μ m oxide film to begin with. After 6 months of exposure of the CBB assemblies, the extent of crevice attack on the polished sample of aluminum was much greater compared to that on the autoclaved sample, as is shown in Fig. 4. This is due to much easier penetration of the thin, air-formed oxide film on the polished samples. In fact, the lowering of pH and concentration of Cl[−] ions within crevices of the autoclaved sample would be much slower due to a lower uniform corrosion rate for the sample with a thick oxide film. However, once the pH has lowered and Cl[−] ion concentration is increased, the dissolution of the oxide film takes place and further attack would be comparable to that on an as-polished sample. That is the reason for the comparable extent of corrosion after 1 year of exposure for aluminum samples with different surface conditions used in this study. As the aluminum-clad fuel remains in research reactors for 1 to 2 years at temperatures greater than 60 to 70 °C, boehmite is expected to be present on

the surfaces[18,19,20] and thickness should be in the range of 20 to 35μ m.^[21] In the present experiment, oxidation was experienced at 150 °C during the autoclave treatment, which would have resulted in a similar nature of the film and comparable thickness of the oxide (20 μ m), simulating the oxide film present on spent fuels. These results from the CBB assembly experiments therefore indicate that under crevice and galvanic contact conditions, aluminum clad (having oxide films) would initially resist localized corrosion, but once the crevice corrosion is initiated, the subsequent corrosion would be equally fast, as on polished or scratched surfaces of aluminum.

4.2 Pitting Corrosion

The CBB assemblies also resulted in pitting corrosion on the aluminum samples, after an immersion for just 6 months. While galvanic contact with SS assemblies had helped in accelerating the rate of attack, the accumulation of chloride ions in the vicinity of crevices also resulted in increased probability of pit initiation. These pits were decorated with whitish deposits in the form of a ring around the pit opening. Such deposits around pits are reported on stored aluminum-clad fuels in pools with aggressive water chemistries.[22,23] A parameter based on water chemistry, pitting resistance index, is used to predict the time to develop a (1 mm) pit.[24] However, this study shows that water chemistry in the crevices could be more aggressive than that of the pool, and it would determine the susceptibility to pitting. Nonuniform mixing or stagnation of the pool water may also result in a higher conductivity in certain pockets of the pool. In addition to the water chemistry parameters, the following factors are reported to promote pitting of aluminum alloys: $[25-27]$ high conductivity pool water (180 μ S/cm), aggressive ions (>20 ppm Cl[−]), sludge having concentrated Fe⁺⁺⁺ and Cl[−] impurities, scratches/imperfections in protective oxide film, and stagnancy. The edge effect may also have caused enhanced pitting in these samples. It should be noted that these results are for pure aluminum and other aluminum alloys used as fuel clad were not investigated in this study.

4.3 Stress Corrosion Cracking

No SCC was observed in any of the samples in the CBB assemblies. In an operating crevice region, pH is low (2 to 3) and chloride ions are concentrated. Tensile stresses are also present in CBB assemblies. Therefore, the possible parameter, which had prevented SCC to occur for a sensitized SS sample, should be the threshold temperature and pH needed to initiate SCC. It is reported^[14] that SCC occurs only above 60 $^{\circ}$ C under such conditions for SS. However, if such conditions prevail over a long term (years), then a threshold on temperature may not remain valid. This remains to be established. It is also reported^[28] that much lower pH (<1) is required for SCC to occur in SS at ambient temperatures.

Zircaloys and aluminum are not known to be susceptible to chloride-induced SCC. On the other hand, Zircaloy-clad spent fuels may enter SFSPs with incipient SCC cracks on the internal surfaces, initiated due to interaction between the uranium fuel and its clad (PCI) during reactor operation. The behavior of such spent fuels with initiated cracks in SFSPs is not clear. The fis-

Table 2 Pool characteristics of SFSPs

	Pool (Tarapur)		Pool (Trombay)			
Parameter	Minimum	Maximum	Minimum	Maximum		
pH	5.4 (center-top)	5.7 (SE corner-bottom)	5.8 (middle-center)	5.9 (corner-bottom)		
$Cl- concentration (ppm)$	0.43 (center-top)	2.40 (center-middle)	0.20 (SW corner-bottom)	3.23 (NE corner-bottom)		
Specific conductivity $(\mu S/cm)$	3.43 (SW corner-bottom)	13.25 (center-middle)	2.51 (SW corner-bottom)	13.49 (NE corner-bottom)		
Activity (dps/5 mL)	$<$ 50 (center-top)	$<$ 50 (center-bottom)	$<$ 50 (center-top)	620 (north corner-middle)		
Fe concentration (ppm)	0.20	0.26	0.17 (SW corner-bottom)	0.35 (center-bottom)		
Cr concentration (ppm)	2.7	2.7	2.5 (SW corner-bottom)	3.8 (center-bottom)		
Ni concentration (ppm)	0.04	0.04	0.03 (SW corner-bottom)	0.07 (center-bottom)		
SE: southeast, SW: southwest, and NE: Northeast						

Table 3 Electrochemical potential of various materials in water samples collected from different locations of SFSPs [in mV (SCE)]

ECP values in brackets show potentials 5 min after immersion. Other values are measurements taken immediately after immersion. P—Trombay Pool, and T—Tarapur Pool

sion product iodine is considered responsible for PCI-SCC. Iodine is associated with Cs as CsI and as such there should not be any possibility of propagation of the PCI-SCC during wet storage. However, lab studies have shown that CsI dissociates under gamma irradiation (at 109 R/h for a few hours) and causes cracking at 300 to 400 $^{\circ}$ C.^[29] Such data at ambient temperatures are not available. The spent fuels emit substantial gamma fields in the storage pools, of the order of $10⁶$ R/h upon discharge. It decreases exponentially with time to levels of 10^3 to 10^2 R/h after several years.^[30] The dissociation of CsI during storage appears unlikely. In addition, it has been shown^[31] that dry storage of spent fuel is possible at clad temperatures of up to 400 °C without SCC causing any breach of clad. A model (SCCIG developed for EPRI) applied for dry storage conditions had shown[31] that Zircaloy-clad fuels can be stored safely for 1000 years. As

conditions during wet storage are much less aggressive than during dry storage (creep/thermal expansion and related factors contributing to stresses would not be present), the results of this model for dry storage remain valid for wet storage also. Therefore, SCC cracks of Zircaloy clad are not expected to propagate during wet storage.

4.4 Electrochemical Potential

For determining the susceptibility to corrosion, a number of factors may be contributing, *e.g.,* water chemistry (pH, temperature, water constituents, radiolysis, *etc.*), material characteristics (heat treatment, microstructure, surface oxide, and cold work), and stresses (residual stresses/tensile stresses, *etc.*). The overall effect of all of these factors is reflected by the freely corroding potential or ECP. It represents the overall contribution from various ionic species present in water, some of these causing reduction and others, oxidation reactions.[32] Radiolysis of water is also taken care of as it changes concentration and type (reducing/oxidizing) of species in water.[32,33] Some studies have correlated the ECP with uniform corrosion rates and evaluated the effect of various water chemistries on the ECP,^[34,35] whereas in other studies, ECP is used to predict the susceptibility to crevice/pitting corrosion or SCC.[36,37] However, the potential of materials in low conductivity solutions is not steady. These values shift toward nobler (or positive) values with time of immersion. This trend is due to a reaction of the ionic species with materials, and particularly due to the formation of oxide films on the surfaces of polished samples. As oxide films form, further reaction of ionic species with the material becomes less and a few of the electrochemical reactions may not take place. This is supported by the results of Howell and Zapp,^[26] who showed that with a lowering of the conductivity of water, the ECP values moved in a nobler direction.

This trend of the shift of ECP with oxide films is further substantiated by nobler values of ECP (Table 4) measured *in situ* in the SFSPs. The difference in these ECP values is basically due to the use of unpolished (as received) samples for the *in-situ* measurements compared to the use of polished samples for the ECP measurements in water samples collected from SFSPs. All the ECP values reported in Table 3 are when the water samples are kept open to air. There are not many reported ECP values in similar environments, but the ECP values of Zapp and Howell^[26] show a range of +20 to −230 mV (SCE) for polished aluminum 1100 alloy, in water samples of various conductivities. However, they had bubbled air through water during the experiments. This aids in surface oxide formation and results in nobler values compared to that for the samples immersed in water kept open to air. As reported by Zapp and Howell for aluminum alloys and also from the results of the present study, there is wide variation of ECP values for all the materials in water of high purity and a number of parameters influence the ECP. The ECP values keep shifting with time for days. Because an equilibrium between the ionic species and the material is not obtained during an initial phase of immersion in SFSPs, short-term immersion tests would not provide accurate data about uniform corrosion rates. Second, the uniform corrosion rates of all three materials of interest are very low in SFSPs with water of low specific conductivity; therefore, any measurable change can be obtained only in a longterm immersion test. Hence, any quantitative measurement for uniform corrosion (by weight change) should be done by immersing coupons for periods in the range 1 to 10 years. Precautions should be taken to ensure the absence of any other form of corrosion during this period by avoiding crevices, galvanic contact, *etc*. Laboratory measurements can be carried out by measuring corroded ion concentration in water as reported for SS.[38,39] Results applicable to spent fuels can be obtained only if materials with carefully grown surface oxides, similar in structure and thickness to that on the stored spent fuels, are used in the tests.

Radiolysis of water results in an increase of radical species, *e.g.,* e-aq., H, and OH, by β/γ irradiation and molecular species, *e.g.,* H_2O_2 , HO_2 , H_2 *etc.,* by neutron irradiation.^[40] Table 4 indicates more active (or negative) ECP values for aluminum and

Zircaloy samples when measured at locations close to the stored spent fuels. This is due to the influence of irradiation. Since in a SFSP the extent of neutron irradiation is negligible, more active ECP values are mainly due to β/γ irradiation. The more active ECP values could be due to the concentration of reducing species near the stored spent fuels due to β/γ irradiation. This would cause a slightly aggressive water chemistry in the regions around the stored spent fuels and a higher rate of corrosion. Laboratory studies for SS had shown a threefold increase in uniform corrosion rates due to irradiation.^[9,38,39] However, this enhanced rate of corrosion (∼10 µm/year) is also too small to cause any appreciable thinning in the SS lining of the pool during its long service life.

5. Conclusions

The susceptibility to corrosion of the materials of different fuel clad (pure aluminum and Zircaloy-2) and the lining of SFSP (type 304 SS) has been evaluated in accelerated tests. The following conclusions can be drawn from the present study.

- Pure aluminum-clad research reactor spent fuels are susceptible to crevice and pitting corrosion under conditions of galvanic coupling and crevice geometry. In contrast, Zircaloy-2 and type 304/304 L stainless steels are resistant. The corrosion behavior of aluminum alloys used as fuel clad was not evaluated in this study.
- The existing crevices (*e.g.*, between aluminum-clad fuel and storage racks, between two racks, *etc.*) can operate even in low conductivity water of SFSPs. This would be accelerated by galvanic contacts (*e.g.,* with SS racks).
- Stress corrosion cracking (of Zircaloy-2 clad or of SS lining of the pool) induced by chlorides in the water of the pools is not a probability during wet storage.
- The ECP values indicate a change in ionic concentration around the stored spent fuels, which shift the ECP to more active values. Therefore, the uniform corrosion rates are expected to be higher in the presence of radiation, *e.g.,* for Al and Zircaloy clad, compared to those measured in the absence of radiation. These effects are not significant, as other coupon studies in SFSP had indicated very low uniform corrosion rates.
- The ECP values show a wide variation with surface condition of materials, water chemistry parameters, and time of immersion. Therefore, experiments to determine uniform corrosion rates by immersion in the pool should be long-term tests, as differences in corrosion rates would creep in as was indicated by the variation in ECPs for short-term tests.

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