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TESTING VAPOR SPACE AND LIQUID-AIR INTERFACE CORROSION IN SIMULATED ENVIRONMENTS OF HANFORD DOUBLE-SHELLED TANKS

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LIST OF ACRONYMS

CPP	Cyclic Potentiodynamic Polarization
PNNL	Pacific Northwest National Laboratory
SRNL	Savannah River National Laboratory

1.0 EXECUTIVE SUMMARY

Electrochemical coupon testing were performed on 6 Hanford tank solution simulants and corresponding condensate simulants to evaluate the susceptibility of vapor space and liquid/air interface corrosion. Additionally, partial-immersion coupon testing were performed on the 6 tank solution simulants to compliment the accelerated electrochemical testing. Overall, the testing suggests that the SY-102 high nitrate solution is the most aggressive of the six solution simulants evaluated. Alternatively, the most passive solution, based on both electrochemical testing and coupon testing, was AY-102 solution. The presence of ammonium nitrate in the simulants at the lowest concentration tested (0.001 M) had no significant effect. At higher concentrations (0.5 M), ammonium nitrate appears to deter localized corrosion, suggesting a beneficial effect of the presence of the ammonium ion. The results of this research suggest that there is a threshold concentration of ammonium ions leading to inhibition of corrosion, thereby suggesting the need for further experimentation to identify the threshold.

2.0 INTRODUCTION

Localized corrosion in the form of pitting in the vapor space and at the liquid/air interface of tank walls is an ongoing challenge in maintaining the structural integrity of the liquid waste tanks at the Savannah River Site and Hanford Site. The interaction between corrosive and inhibitor species in the bulk liquid waste and the vapor phase constituents at the stagnant liquid/air interface influence the amount of corrosion that occurs at this location. Similarly, the presence of corrosive or inhibitor species in condensates/supernates on the tank wall above the liquid level, and their interaction with the vapor phase constituents as the liquid evaporates from the tank wall influence the degree of corrosion that occurs in the vapor space. To minimize pitting corrosion, effort is underway to gain an understanding of the pitting response in various simulated waste solutions. Electrochemical testing has been used as an accelerated tool in the investigation of pitting corrosion. [1]

In response to recommendations made by the expert panel workshop on double-shelled tank (DST) vapor space corrosion participants [2], modeling and experimental studies have been performed on DST supernatants and condensates to predict the changes in chemical composition during evaporation [3, 4]. Six Hanford site tank solutions have been targeted for the study: AY-101 (Segment 3), AY-101 (Segment 8), AN-102, AY-102, SY-102 (high chloride), and SY-102 (high nitrate). Results of the modeling and initial experimental studies have indicated a need for further corrosion sensitivity testing of the remaining four simulants not tested at SRNL in FY10 [3,4] and the evaporated condensates of all six original solutions modeled and evaluated by PNNL [3]. Additionally, the effect of ammonium nitrate on the solution pH and subsequent corrosion behavior need to be explored further with the six Hanford solutions of interest [5,6]. Liquid-air interface testing would aid in understanding the effect of the solutions on liquid-air interface corrosion, complementing the parallel experimentation targeted toward vapor space corrosion.

3.0 EXPERIMENTAL

3.1 EFFORT 1: ELECTROCHEMICAL TESTING OF CONDENSATE SOLUTION SIMULANTS

Cyclic potentiodynamic polarization testing of ASTM A537 carbon steel, a steel similar to what was used in the construction of the Hanford tanks, was performed in duplicate using six Hanford solution simulants and corresponding condensate simulants. Sample surfaces were ground with 600 grit SiC paper prior to cyclic potentiodynamic polarization testing. Electrochemical cyclic potentiodynamic polarization (CPP) testing was performed on 1.00 inch diameter ASTM A537 carbon steel, attached to an electrical lead and centered in mounting compound.

Testing solutions were prepared based on chemistries reported by PNNL modeled and measured experimentally in FY10. Primarily, experimental values cited in the PNNL report were used to make the solutions. Modeling values were used only when experimental values were unavailable. Species amounts targeted in the simulant preparation are listed in Appendix A. Solutions were prepared the day prior to electrochemical testing and were stirred overnight. Solution appearance was noted prior to testing. The pH for each solution was measured before electrochemical testing. All solutions having an opaque appearance were electrochemically tested directly after stirring. During electrochemical testing, the solutions were not stirred. After testing, the undissolved solids were permitted to settle out of solution and a sample of the remaining liquid was chemically analyzed to determine the true aqueous chemistry that was used during electrochemical testing. Results of the analysis are provided in Appendix B.

All electrochemical testing was performed in duplicate at 40 ºC in stagnant solution with air in the vapor space above the solution. A carbon rod counter electrode and a saturated Ag/AgCl reference electrode connected to a Luggin bridge were used in the cyclic potentiodynamic polarization testing. The potential was increased at a rate of 0.5 mV/s up to a current density of 1.0×10^{-4} A/cm^2. Runs were terminated when the potential on the reverse scan equaled the original open circuit potential. The electrochemical results were analyzed using the protocol described in RPP-RPT-50092, Rev. 1. Optical images documenting the sample surface appearance were taken of each sample after CPP testing.

3.2 EFFORT 2: EFFECT OF AMMONIA ON CORROSION RESPONSE

In addition to the solution simulants described in the PNNL report [3], ammonium-containing simulants were also prepared using 0.001 and 0.5 M ammonium nitrate. Target composition details are listed in Appendix A. Parameters used in the CPP testing performed using the ammonium- containing solutions simulants were identical to those used in Effort 1 on solutions lacking ammonium nitrate. Analysis of the electrochemical results followed the protocol described in RPP-RPT-50092, Rev. 1. Optical images documenting the sample surface appearance were taken of each sample after CPP testing.

3.3 EFFORT 3: LIQUID/AIR INTERFACE TESTING ON HANFORD SOLUTIONS

Partial immersion testing of A537 coupons was started using tank simulant solutions (0% evaporated) containing no ammonium nitrate. Photographs were taken on a weekly basis. Investigation of liquid/air interface corrosion with an accelerated testing protocol was originally planned. Due to delays in the development of the testing protocol, this task was not initiated.

4.0 RESULTS

4.1 ELECTROCHEMCAL TESTING OF CONDENSATE SOLUTION SIMULANTS

4.1.1 Testing Solution Analysis

Prior to electrochemical testing, solution pH was measured and the appearance of the solutions was documented, see Table 1. Several solutions tested had an opaque appearance, suggesting undissolved solids remained suspended in several of the solutions.

Table 1 Solution appearance and pH prior to electrochemical testing of solutions containing no added ammonium nitrate.

Solution	pH	Appearance
0% evaporated AN-102	9.74	Opaque white with white solids
0% evaporated AY-101 (Segment 3)	9.12	Clear
0% evaporated AY-101 (Segment 8)	9.38	Opaque white
0% evaporated AY-102	9.47	Clear
0% evaporated SY-102 (high chloride)	10.06	Opaque white with white solids
0% evaporated SY-102 (high nitrate)	9.00	Clear
36% evaporated AN-102	9.77	Yellow tint with white solids
34% evaporated AY-101 (Segment 3)	9.43	Clear
27% evaporated AY-101 (Segment 8)	9.39	Clear with white solids
33% evaporated AY-102	9.61	Clear
25% evaporated SY-102 (high chloride)	9.68	Opaque white
33% evaporated SY-102 (high nitrate)	9.14	Clear with white solids
55% evaporated AN-102	9.72	Opaque white with white solids
76% evaporated AY-101 (Segment 3)	9.39	Clear with white solids
68% evaporated AY-101 (Segment 8)	9.51	Yellow tint with white solids
77% evaporated AY-102	9.50	Yellow tint with white solids
63% evaporated SY-102 (high chloride)	9.54	Yellow tint with white solids
78% evaporated SY-102 (high nitrate)	--	--

Solution analysis was preformed after electrochemical testing. Results are provided in Appendix B and a summary of significant component concentrations is reported in Table 2 and 3. The results indicate that the solutions tested were not always equal to the target solutions listed in Appendix A.

Table 2 Summary of solution concentrations post electrochemical testing in solutions containing no ammonium ions yet had undissolved solids. * denotes the solution was clear, therefore, post analysis was not performed.

Solution	Nitrate (M)	Nitrite (M)	Chloride (M)	Sulfate (M)
0% evaporated AN-102	6.64	3.17	0.15	0.27
0% evaporated AY-101 (Segment 3)	*	*	*	*
0% evaporated AY-101 (Segment 8)	1.41	0.83	0.08	0.03
0% evaporated AY-102	*	*	*	*
0% evaporated SY-102 (high chloride)	4.14	2.63	0.22	0.03
0% evaporated SY-102 (high nitrate)	*	*	*	*
36% evaporated AN-102	9.03	5.52	0.25	0.29
34% evaporated AY-101 (Segment 3)	*	*	*	*
27% evaporated AY-101 (Segment 8)	2.11	13.45	0.16	0.05
33% evaporated AY-102	*	*	*	*
25% evaporated SY-102 (high chloride)	9.00	7.35	0.58	0.07
33% evaporated SY-102 (high nitrate)	4.92	0.13	0.01	0.07
55% evaporated AN-102	5.85	3.74	0.17	0.09
76% evaporated AY-101 (Segment 3)	3.66	0.61	0.05	0.06
68% evaporated AY-101 (Segment 8)	3.29	3.54	0.28	0.10
77% evaporated AY-102	0.04	1.42	0.04	0.03
63% evaporated SY-102 (high chloride)	7.14	5.87	0.48	0.02
78% evaporated SY-102 (high nitrate)	--	--	--	--

Table 3 Summary of solution concentrations post electrochemical testing in solutions containing 0.001 M ammonium ions yet had undissolved solids. * denotes the solution was clear, therefore, post analysis was not performed.

Solution	Nitrate (M)	Nitrite (M)	Chloride (M)	Sulfate (M)
0% evaporated AN-102	3.19	0.00	0.09	0.15
0% evaporated AY-101 (Segment 3)	*	*	*	*
0% evaporated AY-101 (Segment 8)	1.59	0.92	0.09	0.03
0% evaporated AY-102	*	*	*	*
0% evaporated SY-102 (high chloride)	1.77	1.12	0.14	0.02
0% evaporated SY-102 (high nitrate)	*	*	*	*
36% evaporated AN-102	3.64	2.16	0.10	0.14
34% evaporated AY-101 (Segment 3)	*	*	*	*
27% evaporated AY-101 (Segment 8)	2.16	1.39	0.16	0.05
33% evaporated AY-102	*	*	*	*
25% evaporated SY-102 (high chloride)	3.02	2.52	0.25	0.04
33% evaporated SY-102 (high nitrate)	4.98	0.13	0.01	0.07
55% evaporated AN-102	4.55	2.74	0.13	0.07
76% evaporated AY-101 (Segment 3)	3.48	0.57	0.05	0.06
68% evaporated AY-101 (Segment 8)	3.29	3.59	0.26	0.11
77% evaporated AY-102	0.04	1.43	0.00	0.03
63% evaporated SY-102 (high chloride)	5.32	4.35	0.37	0.01
78% evaporated SY-102 (high nitrate)	--	--	--	--

4.1.2 Electrochemical Analysis and Corresponding Optical Images

The CPP curves and corresponding optical images for all testing solutions evaluated are given in Appendix C and D. Table 2 below summarizes the results of the electrochemical testing based on the 5 category system outlined in RPP-RPT-50092, Rev. 1. The category system is as follows:

Category 1: Negative hysteresis.

Category 2: Positive hysteresis, but with pitting and protection potentials well above the zero current potential.
Category 3: Positive hysteresis with a noble pitting potential, but with the protection potential relatively near the zero current potential.
Category 4: Positive hysteresis with the protection potential lower than then zero current potential
Category 5: Spontaneous pitting at the zero current potential so that the current increases rapidly upon polarization to potentials above the zero current potential.

If an electrochemical curve did not fit one of these categories, it was assigned to the closest category that the curve resembled, and a "-UC" was added to denote "unclear".

A descriptive summary of corresponding optical results is also provided.

Table 4 Summary of electrochemical and optical results for solutions containing no added ammonium nitrate. Tests were run in duplicate. To demonstrate experimental consistency between similar tests, electrochemical and optical results for the duplicate tests are recorded separately.

Contains No Ammonium Nitrate				
Solution	**Electrochemical Results**		**Optical Results**	
	Test 1	**Test 2**	**Test 1**	**Test 2**
0% evaporated AN-102	3-UC	1	Large pits	Few pits
0% evaporated AY-101 (segment 3)	4-UC	1	Pits	Few pits
0% evaporated AY-101 (segment 8)	3	1	Few pits	No pits
0% evaporated AY-102	1	1	No pits	No pits
0% evaporated SY-102 (high chloride)	1	1-UC	Pits	Pits
0% evaporated SY-102 (high nitrate)	4	4-UC	Lots of pits	Pits
36% evaporated AN-102	2-UC	1	Few pits	No pits
34% evaporated AY-101 (segment 3)	4	4	Pits	Pits
27% evaporated AY-101 (segment 8)	4	3	Lots of pits	Lots of pits
33% evaporated AY-102	1	1	No pits	No pits
25% evaporated SY-102 (high chloride)	2	1	Small pits	No pits
33% evaporated SY-102 (high nitrate)	4	4	Lots of pits	Lots of pits
55% evaporated AN-102	1	1-UC	Minor pits	Minor pits
76% evaporated AY-101 (segment 3)	4	4-UC	Lots of pits	Few pits

68% evaporated AY-101 (segment 8)	2-UC	1	Few pits	No pits
77% evaporated AY-102	1	1	No pits	No pits
63% evaporated SY-102 (high chloride)	1	3-UC	No pits	Few pits
78% evaporated SY-102 (high nitrate)	High viscosity solution. Did not run CPP.			

4.2 EFFECT OF AMMONIUM IONON CORROSION RESPONSE

4.2.1 Testing Solution Analysis

Table 5 Solution appearance and pH prior to electrochemical testing of solutions containing 0.001 M ammonium nitrate.

Solution	pH	Appearance
0% evaporated AN-102	10.04	Clear with white solids
0% evaporated AY-101 (segment 3)	9.48	Clear
0% evaporated AY-101 (segment 8)	9.28	Opaque with white solids
0% evaporated AY-102	9.24	Clear
0% evaporated SY-102 (high chloride)	10.70	Opaque white with solids
0% evaporated SY-102 (high nitrate)	9.04	Clear
36% evaporated AN-102	9.66	Yellow tint with white solids
34% evaporated AY-101 (segment 3)	9.27	Clear
27% evaporated AY-101 (segment 8)	9.43	Opaque with white solids
33% evaporated AY-102	9.39	Clear
25% evaporated SY-102 (high chloride)	10.44	Opaque white
33% evaporated SY-102 (high nitrate)	9.15	Clear
55% evaporated AN-102	9.46	Clear with white solids
76% evaporated AY-101 (segment 3)	9.52	Clear
68% evaporated AY-101 (segment 8)	9.58	Clear with white solids
77% evaporated AY-102	9.58	Yellow tint with white solids
63% evaporated SY-102 (high chloride)	9.48	Viscous opaque white solution
78% evaporated SY-102 (high nitrate)	--	--

Table 6 Solution appearance and pH prior to electrochemical testing of solutions containing 0.5 M ammonium nitrate.

Solution	pH	Appearance
0% evaporated AY-101 (segment 8)	9.09	Clear with white solids
27% evaporated AY-101 (segment 8)	9.32	Clear with white solids
68% evaporated AY-101 (segment 8)	9.42	Yellow tint with white solids

4.2.2 Electrochemical Analysis and Corresponding Optical Images

Tables 5 and 6 summarize the results of the electrochemical testing of the ammonium ion -containing testing solutions based on the 5 category system outlined in RPP-RPT-50092, Rev. 1. The category system was detailed earlier in Section 4.1.2 of this document.

Table 7 Summary of electrochemical and optical results of solutions containing 0.001 M ammonium nitrate. **Tests were run in duplicate. To document experimental consistency between similar tests, electrochemical and optical results for the duplicate tests are recorded separately.**

Contains 0.001 M Ammonium Nitrate				
Solution	**Electrochemical Results**		**Optical Results**	
	Test 1	**Test 2**	**Test 1**	**Test 2**
0% evaporated AN-102	4	4	Large pits	Large pits
0% evaporated AY-101 (segment 3)	3-UC	2-UC	Large pits	Large pits
0% evaporated AY-101 (segment 8)	1	3-UC	No pits	Few pits
0% evaporated AY-102	1	1	No pits	No pits
0% evaporated SY-102 (high chloride)	4	4-UC	Several pits	Few pits
0% evaporated SY-102 (high nitrate)	4	4	Lots of pits	Lots of pits
36% evaporated AN-102	1-UC	1	Few pits	No pits
34% evaporated AY-101 (segment 3)	4	4	Large pits	Large pits
27% evaporated AY-101 (segment 8)	4	4	Lots of pits	Lots of pits
33% evaporated AY-102	1	1	No pits	No pits
25% evaporated SY-102 (high chloride)	1	1	Small pits	Small pits
33% evaporated SY-102 (high nitrate)	4	4	Large pits	Large pits
55% evaporated AN-102	1	1-UC	No pits	No pits
76% evaporated AY-101 (segment 3)	4	3-UC	Small pits	Few pits
68% evaporated AY-101 (segment 8)	4-UC	3-UC	Few pits	Few pits
77% evaporated AY-102	1	1	No pits	No pits
63% evaporated SY-102 (high chloride)	1	1-UC	Small pits	Small pits
78% evaporated SY-102 (high nitrate)	High viscosity solution. Did not run CPP.			

Table 8 Summary of electrochemical and optical results for solutions containing 0.5 M ammonium nitrate. **Tests were run in duplicate. To document experimental consistency between similar tests, electrochemical and optical results for the duplicate tests are recorded separately.**

Contains 0.5 M Ammonium Nitrate				
Solution	**Electrochemical Results**		**Optical Results**	
	Test 1	**Test 2**	**Test 1**	**Test 2**
0% evaporated AY-101 (segment 8)	1	1	Few pits	Few pits
27% evaporated AY-101 (segment 8)	1	2	Few pits	Few pits
68% evaporated AY-101 (segment 8)	1	2	Few pits	Few pits

4.3 EFFORT 3: LIQUID/AIR INTERFACE TESTING ON HANFORD SOLUTIONS

Partial immersion testing images are shown in APPENDIX E.

The summary of the testing to date is listed in Table 7. The key for the table is as follows:

N = None
I = Corrosion near interface
V = Corrosion in vapor space, distant from interface
S = Corrosion in solution below interface
+ = Significant corrosion
- = Minor corrosion

Table 9 Initial results of partial immersion testing.

Solution	**Week 1**	**Week 2**	**Week 3**	**Week 4**
0% evaporated AN-102	N	I/-	I/-	I/-
0% evaporated AY-101 (segment 3)	I/+	I/+	I/+	I/+
0% evaporated AY-101 (segment 8)	I/-	I/-	I/-	I/V/-
0% evaporated AY-102	N	N	V/-	V/-
0% evaporated SY-102 (high chloride)	I/V/-	I/V/-	I/V/-	V/+
0% evaporated SY-102 (high nitrate)	I/V/+	I/V/S/+	I/V/S/+	I/V/S/+

There are no available results at this time using an accelerated test procedure.

5.0 DISCUSSION

While solutions were prepared utilizing species concentrations in the PNNL report, several of the solutions did not result in 100% of the solids dissolving into solution. The pHs of the solutions were adjusted using specific ratios of bicarbonate and carbonate. Most likely due to bicarbonate and/or carbonate not dissolving into solution completely, the resulting solution pH was frequently lower than the target pH. At pH 10, minor changes in the ratio of bicarbonate to carbonate can create large swings in pH.

The electrochemical results suggest that susceptibility of corrosion strongly depends on the solution chemistry. However, there were discrepancies between the electrochemical responses and the optical results, Tables 2, 5, and 6. Several cases occurred in which the electrochemical test resulted in a negative hysteresis. Based on electrochemical theory, a negative hysteresis should result in no pitting. In tests where pits occurred on samples that electrochemically resulted in negative hysteresis, the pits were small and the repassivation occurred at a potential significantly larger than OCP. Therefore, the pits resulting in conjunction with negative electrochemical hysteresis are not of concern.

In electrochemical testing of solution simulants containing no ammonium nitrate, AY-102 is a solution simulant where pitting susceptibility is low at all levels of evaporation tested. In solutions AN-102 and SY-102 high chloride and nitrate, as the solution evaporates, pitting susceptibility decreases. Minor pits occurred. Additional testing could determine whether the pits resulted from inclusions within the A537 carbon steel. Pitting clearly occurs in AY-101 segment 3 and segment 8 solutions. In segment 3, pitting susceptibility increases with evaporation. Segment 8 does not appear to have a pitting trend versus evaporation percentage as the solution representative of 27% evaporation resulted in the highest pitting activity. Furthermore, pitting susceptibility appears to increase as the solution evaporates. The highest degree of pitting susceptibility was observed in the SY-102 high nitrate solution.

The addition of 0.001 M ammonium nitrate did not significantly change the electrochemical response of the samples. These tests, in essence, can be considered duplicates of the solutions containing no ammonium nitrate. However, when 0.5 M ammonium nitrate was added to the AY-101 segment 8 solution simulants, the extent of pitting was drastically reduced. This result suggests further studies are needed to evaluate the effect of ammonium nitrate concentration on corrosion, particularly for solutions AY-101 segment 3, AY-101 segment 8, and SY-102 high nitrate, which demonstrated significant corrosion.

Partial immersion testing resulted in varying degrees of corrosion. The SY-102 high nitrate solutions resulted in a significant amount of corrosion in the solution space, interface, and vapor space areas of the sample. AN-102 and AY-102 resulted in the smallest corrosion activity. Solutions AY-101 segment 3, AY-101 segment 8 and SY-102 high nitrate are the three solutions that resulted in the most visible corrosion at the liquid air interface. These results are in agreement with the electrochemical testing results.

6.0 CONCLUSIONS

Electrochemical and coupon testing utilizing solutions prepared with the aim to target six Hanford tank solution simulants and condensate simulants chemistries. Both the electrochemical testing and coupon testing suggest that SY-102 high nitrate is the most aggressive solution of the six. Alternatively, the most passive solution, based on both electrochemical testing and coupon testing, was AY-102 solution. Experimentation using solution simulants containing ammonium ions at low concentrations (0.001 M), no significant effect was found. At higher concentrations (0.5 M), the increased ammonium ion concentration appears to deter localized corrosion, suggesting a beneficial effect of the presence of the ammonium ion.

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