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For a Tuff Repository

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CORROSION TEST PLAN TO GUIDE CANISTER MATERIAL SELECTION AND DESIGN FOR A TUFF REPOSITORY.

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ABSTRACT

Corrosion rates and the mode of corrosion attack form a most important basis for selection of canister materials and design of a nuclear waste package. Type 304L stainless steel was selected as the reference material for canister fabrication because of its generally excellent corrosion resistance in water, steam and air. However, 304L may be susceptible to localized and stress-assisted forms of corrosion under certain conditions. Alternative alloys are also investigated; these alloys were chosen because of their improved resistance to these forms of corrosion. The fabrication and welding processes, as well as the glass pouring operation for defense and commercial high-level wastes, may influence the susceptibility of the canister to localized and stress forms of corrosion.

INTRODUCTION

Lawrence Livermore National Laboratory (LLNL) is responsible for high-level nuclear waste package development as part of the Nevada Nuclear Waste Storage Investigations (NNWSI) Project. This project is part of the Department of Energy's Civilian Radioactive Waste Management (CRWM) Program. The waste package effort at LLNL is developing multibarriered packages for safe, permanent disposal in the proposed Yucca Mountain tuff repository.

The primary containment of the high-level waste forms is provided by a metallic barrier, variously referred to as a canister, overpack, or container. This barrier is expected to perform for a period of up to 1000 years and provide "substantially complete" containment of the waste form. In addition, this container is expected to protect the repository from contamination during the operational period, including waste handling activities associated with emplacement and possible retrieval for a period of up to fifty years after emplacement. The purpose of this paper is discussion of selection of candidate metal barrier materials to meet these containment objectives. These candidate materials are now being evaluated for their corrosion and oxidation performance under tuff repository relevant conditions. In this paper we shall refer to the first metal container surrounding the waste form (clad spent fuel rods or reprocessed borosilicate glass) as the canister. An optional, second metal container surrounding the canister is termed an overpack.

ENVIRONMENTAL CONSIDERATIONS

Previous papers in this session have concentrated on characterizing the geochemical environment and modification of this environment after emplacement of the nuclear waste packages. Location of the repository in a horizon above the static water level should reduce the corrosivity of the environment because of minimal water content and eliminate any hydrostatic stress component on the metal containment barrier.

Emplacement of the thermally hot waste packages will vaporize the pore water and any incoming vadose water, so that the dominant

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environment in the repository will be steam/air mixture. The boiling point at the repository elevation is 95°C. Representative canister surface time-temperature profiles for the different waste packages are shown in Figure 1. These curves indicate that the canister surface temperatures of the spent fuel (SF) and reprocessed commercial high level waste packages (CHLW) will remain above the water boiling point for hundreds of years before resaturation of the environment can occur. The lower thermal output of defense high level waste (DHLW) packages results in considerably lower canister surface temperatures on this package. The environment around this package may therefore attain resaturation approximately 50 years after emplacement. Ironically, the lower temperature DHLW canister may present more significant corrosion problems because of the longer time it may contact an aqueous environment.

The composition of J-13 well water has been previously presented (1). This well water is believed representative of the vadose water associated with the Topopah Spring member in which the repository would be located. The water is oxidizing (5.7 ppm dissolved oxygen; 5.6 ppm nitrate) and would be corrosive toward carbon steel. The low concentrations of halide ions (7.5 ppm chloride; 2.2 ppm fluoride) suggest that the water should not be aggressive toward stainless steels and other alloys which depend on passive film formation for corrosion protection. The relatively high concentration of bicarbonate (122 ppm) indicates pH buffering effects to keep the water near neutral. The absence of heavy metal ions is beneficial, as these ions tend to pit otherwise corrosion-resistant alloys, such as stainless steels.

RADIATION EFFECTS

The interaction of gamma radiation produced by decay of fission products in the waste form, with the water and vapor outside the metal containment barrier may produce a significant effect on the corrosion behavior of the metal. Intense gamma radiation will produce $e^{-aq.}$, H, H₂, OH, H₂O₂, O₂, HO₂, O₂, H⁺, and OH⁻. In addition, gaseous and ionic species dissolved in water or steam may be oxidized or reduced. A major concern is oxidation of nitrogen to its oxides which can react with water to form nitric acid. Because of a higher inventory of radionuclides, CHLW and SF waste packages are expected to produce the highest initial radiation fluxes. The gamma radiation flux drops with time. For example, the gamma flux 100 years after emplacement of a CHLW package is about 10% of the original flux.

Radiolysis products formed in the steam/air mixture surrounding the packages will possibly enhance the oxidation rate of the canister surface. Because stainless steels are proposed as canister materials, this modest increase in oxidation rate will likely only stabilize the usually protective, passive film on these alloys. By the time aerated liquid water can contact the canister surface, the reduced gamma flux should lower concentrations of radiolysis-produced species, except for those which are stable and accumulate. These species are usually not corrosive to stainless steels, except when the stainless steel is in the sensitized condition.

Past studies of the corrosion of austenitic stainless steels in irradiated moist air have revealed little or no measurable general corrosion. Effects on stress corrosion behavior were not studied. For example, Primak and Fuchs (2) studied the behavior of several metals exposed to ionizing radiation in the presence of humid and dry air, oxygen, nitrogen, and water vapor. Metals subject to nitric acid

corrosion were attacked, while stainless steel was uncorroded. Makepeace (3) performed general corrosion tests on several metals, including AISI types 316L, 316, 304, and 347 stainless steels, in wet air at room temperature for 6 months in a gamma radiation field of 1×10^5 to 6×10^6 rads/hr. Weight loss measurements indicated general corrosion rates for the stainless steels to be less than $0.1 \mu\text{m/yr}$.

DESIGN AND FABRICATION CONSIDERATIONS

Many uncertainties directly influence the design of the waste package. Among these uncertainties are: (1) whether the waste packages will be vertically or horizontally emplaced in the repository; (2) whether one or two metal barriers (canister with or without overpack) will be needed for the reprocessed borosilicate glass waste forms (DHLW and CHLW); (3) whether packing material (e.g., backfilled crushed tuff) will be needed around spent fuel waste packages; and (4) the final selection of materials for fabricating the metal barriers. Therefore, several alternative designs must be considered along with a reference design for each kind of waste package. The reference designs employ a 304L single metal barrier (canister) for each waste package. The thickness of the canister is 9.8 mm. The other dimensions of the reference waste packages (right circular cylinders) are given in Table 1. As the BWR and PWR fuel elements are of different dimensions, separate designs are maintained. The time-temperature profiles in Figure 1 were calculated from the conditions given in Table 1. More complete discussions of the conceptual designs of waste packages in a tuff repository are available (4,5).

Figure 1. Comparative Canister Surface Temperature-Time Profiles for Different Waste Packages in a Tuff Repository (Vertical Emplacement, 50 kW/acre Areal Loading, Dimensions Given in Table 1)

TABLE 1. Dimensions and Power Outputs for Reference Waste Package

Waste Package	Canister** Outer Diameter (cm)	Waste Length (cm)	Canister Length (cm)	10 yr old Power Load (kW)
CHLW	32.0	264	300	2.21
DHLW	61.0	231	300	0.42
S.F.-BWR	57.0	413.7	450*	3.42
S.F.-PWR	50.0	410.6	450*	3.30

* length includes pintle length of 16.5 cm.

** assumed canister wall thickness is 9.8 ± 0.3 mm.

Choices exist for fabricating the metal containers. The DHLW waste package is the most developed to date (6) and serves as a prototype for the others. The cylinder may be fabricated from rolled and welded plate or may be fabricated from seamless pipe. End caps are welded onto the cylinder; the top cap has an opening through which the molten glass can be poured (DHLW and CHLW packages). After completion of the pouring operation, the canister is sealed by upset resistance welding of a plug in this opening. For spent fuel packages, the metal canister consists simply of a cylinder body with welded end caps. The fabricating, welding, and glass pouring operations may influence the metal selection for the canister as these operations affect residual stress in the canister, and the higher temperature operations may cause microstructural changes in the stainless steel with a resulting "sensitized" microstructure developing at the grain boundaries. This results from formation of chromium carbide precipitates in the grain boundaries.

REFERENCE AND ALTERNATIVE MATERIALS FOR CANISTERS

Type 304L stainless steel serves as the reference construction material for canisters. This material is expected to have excellent general corrosion resistance in air and steam at temperatures in the range 95-300°C and in non-saline, near-neutral pH, oxic waters below 95°C. Water from the J-13 well has these characteristics. A conservative estimate of the wastage of 304L during the 1000-year containment period shows a loss of 0.1 cm from a 1-cm thick canister wall. This estimate is based on "high values" of uniform corrosion and oxidation rates in water, steam, and air (7) and assumes linear oxide growth kinetics. The 0.1 cm "corrosion allowance" is considerably smaller than the nominal 1 cm-thick canister wall.

The limiting use conditions of 304L stainless steel are rarely general corrosion wastage, but rather occur by much more rapid penetration via localized or stress-assisted forms of corrosion. The experimental test plan is, therefore, largely aimed at resolving the likelihood of these forms of corrosion occurring during the containment period. For purposes of organization, the localized/stress forms of corrosion can be placed into two groups: (1) corrosion forms favored by a sensitized microstructure. Such a microstructure can develop during the fabrication and welding of the canister, the glass pouring operation (DHLW and CHLW waste packages) or at relatively low temperatures during storage in the repository at canister surface temperatures in excess of,

say, 150°C (CHLW and SF waste packages). The sensitized microstructure may lead to intergranular corrosion (IG) or intergranular stress corrosion cracking (IGSCC) because of the expected oxidizing nature of the aqueous environment, should it come in contact with the canister surface. (2) Corrosion forms favored by concentration of the different chemical species in J-13 water. Fractures in the host rock above the repository could admit episodic surges of water. Contact of the water with the hot canister surface would concentrate electrolytic species by evaporation of water. The chloride-ion concentration is of paramount concern with regard to resistance of stainless steels to localized and stress-assisted forms of corrosion. The other ions present in J-13 water may favor or retard these kinds of corrosion attack. Radiolysis of the aqueous environment can cause chemical changes in the water and the dissolved species which, in turn, influence the form and rate of corrosion. Pitting attack, crevice attack, and transgranular stress corrosion cracking (TGSCC) are forms of corrosion that can develop on 304L in concentrated electrolytes -- particularly in high chloride solutions.

While the corrosion literature abounds in information on passive film breakdown and localized and stress corrosion initiation, it is difficult to apply this information without qualification to new situations in an attempt to establish critical levels of ionic concentrations, temperature, and stress below which the stainless steel is "safe" from these kinds of attack. The review by Nuttall and Urbanic (8) on materials for nuclear waste containment summarized available data from many sources. Their review indicates that concentrations of chloride in excess of several hundred ppm are needed to initiate localized and transgranular stress corrosion cracking. Other chemical species in the water may accelerate or inhibit the initiation process. Experimental work in which the chlorides are allowed to concentrate (e.g., wetting and drying situations) often indicate stress corrosion initiation at lower initial levels of chloride in the water (9).

Alternative materials were thus chosen for experimental testing should 304L stainless steel be subject to the kinds of corrosion discussed above. The alternative materials were chosen on the basis of their known improvements in resistance to specific forms of corrosion. If sensitization effects and resulting IG/IGSCC develop, a stabilized grade of stainless steel such as Type 321 or Type 347 may show improved resistance. Premium grades of 304 with even lower C contents than 304L (0.03% C maximum) would also be expected to have greater resistance. If concentrated electrolyte effects and resulting pitting/crevice corrosion develops, 316L stainless steel may be the choice, as Mo additions are known to combat these forms of corrosion. If the concentration effects are severe, a higher molybdenum content alloy, such as 317L, may be required. The low carbon content of these alloys should provide resistance to sensitization-induced corrosion effects comparable to that of 304L. If the 304L is subject to TGSCC because of high residual stress in the canister plus high chloride levels in the environment, then a high-nickel alloy, such as Alloy 825, may fill the need. As this alloy is Ti-stabilized, it should have resistance to sensitization-induced corrosion effects comparable to that of 321. (The high Ni content of 825 makes it more susceptible to sensitization, so a higher level of Ti than in 321 is needed to offset this effect). As Alloy 825 contains 2.5 to 3.5% Mo, it is expected to show resistance to pitting and crevice attack comparable to that of 316L. The logic in this choice of candidate materials is to proceed in a forward direction in overcoming a particular corrosion problem while not losing ground in resistance to another form of corrosion. The text by Sedricks (10) gives an expanded discussion of

corrosion problems and material selection approaches to overcome these problems.

In order to reduce the test matrix to manageable dimensions, 304L, 321, 316L, and 825 are the alloys of focused interest. Premium grades of 304L (with careful control of carbon and possibly other interstitial elements: N, P, and S) will be pursued if 304L generally performs well, but test results indicate that tighter controls on the chemical composition could substantially improve the corrosion performance. In addition to the generally good corrosion resistance, the reference and alternative canister materials possess good fracture toughness properties. While the canister need not be a high strength material, fracture toughness is important in the handling and emplacement operations of waste packages. A discussion of the mechanical properties of these materials relative to the requirements in a tuff repository is available: (11)

TEST RESULTS

A test program was begun in October 1982 to resolve some of the corrosion-related concerns discussed above. The test program focuses on 304L stainless steel as the reference material and other austenitic alloys as alternative materials. The test environments fall into three general categories: (1) hot, "dry" air with controlled humidity levels, but well below saturation, at temperatures 95-300°C. This environment corresponds to the initial conditions where the thermally hot waste package drives moisture away from its vicinity. (2) "Moist" air with humidity levels at or near saturation. This environment corresponds to the period when the rock has cooled to about 95°C and water can re-enter the vicinity of the waste package. (3) Water immersion (J-13 well water). This environment corresponds to possible episodic intrusions of water into the repository environment.

Emphasis was placed on environmental conditions (2) and (3), because these are believed to be more instructive in guiding the material selection process. Survey experiments were begun with J-13 water in contact with crushed Topopah Spring tuff and in the vapor phase over this solution. Some tests were conducted in the presence of a Co-60 source to produce gamma radiation. Test results available as of this writing (November 1983) are now discussed. Because of space limitations imposed by this symposium, not all test results and experimental details can be presented here. They are available elsewhere (12).

A coupon immersion test without irradiation is in operation. After slightly more than 1000 hours of exposure time, the first group of specimens was removed from the 100°C test in tuff-conditioned water and saturated steam. Water from Well J-13 was conditioned by contact with crushed Topopah Spring tuff (2 mm average particle size). Triplicate specimens were used for each alloy tested. Rectangular coupons were nominally 2-inch by 1-inch and were fitted with specially made creviced teflon washers so that incipient crevice attack could be observed. The coupons were suspended so that some were immersed in the water while others were exposed to the steam phase at the top of the enclosed cell. The weight loss of each exposed coupon was determined (ASTM G-1 Test Procedure), and the coupon surfaces were examined for evidence of localized corrosion both on the creviced surface and on the freely exposed surface. The corrosion rates for the austenitic stainless steels are presented in Table 2. As expected, the results show a very low general corrosion rate. No evidence of pitting or crevice corrosion

attack was observed. This test is continuing for longer exposure periods (several months to a year or more).

TABLE 2 Corrosion Rates ($\mu\text{m}/\text{yr}$) for Stainless Steel Coupons.
(1000-hour Exposure of Triplicate Coupons)

Material	100°C Saturated Steam at Atmospheric Pressure	100°C J-13 Water Conditioned with Crushed Tuff Rock
304L stainless steel	nil*; 0.25; nil	nil; 0.25; nil
316L stainless steel	nil; nil; nil	0.51; 0.51; 0.51
317L stainless steel	0.25; nil; 0.13	0.25; 0.51; 0.76

Surface Condition After Exposure: no evidence of crevice or other localized attack.

* nil is less than $0.13 \mu\text{m}/\text{year}$, which is based on our weight loss detection limit.

Corrosion Tests in Irradiated Environments

Tests were conducted at the Pacific Northwest Laboratory in J-13 water irradiated with a Co-60 source. Test vessels are placed at the bottom of the "gamma pit". Air-sparged J-13 water was flowed at a rate of 35 ml/hr through the test vessels. Crushed tuff was placed in the bottom of each vessel so that some test specimens could be embedded in this rock + water environment in an effort to accentuate "crevice effects" around the coupon. One test vessel was maintained at 105°C (under pressure) and the calculated radiation dose rate in the center of this vessel was 3×10^5 rads/hr; the other test vessel was maintained at 150°C with a radiation rate dose of 6×10^5 rads/hr. (For comparison, the estimated maximum dose rate at the canister surface for a CHLW waste package is 1×10^5 rads/hr.) The 304L coupons were solution-annealed, and some coupons were subsequently given a treatment in the usual sensitization range. The test results for the individual coupons are presented in Table 3. Comparison of the results in Tables 2 and 3 indicates some mild enhancement of the general corrosion rate of 304L stainless with irradiation. However, none of the irradiated coupons exhibited localized attack (especially intergranular), even when the alloy was given the sensitizing treatment. These irradiation tests are continuing.

Stress Corrosion Test Results

As survey tests to indicate which combinations of environments and alloy compositions/metallurgical conditions lead to stress corrosion susceptibility, both the "slow strain rate" test and four-point loaded bent beam configuration are used. Some results from the slow strain rate tests are available and are summarized in Table 4. Type 304L specimens are tested in both the solution-annealed and in the "sensitized

Table 3 Corrosion Test Results for Stainless Steel Coupons
in Irradiated Environments (2-month Exposure Data)

Material	Environment	Corrosion Penetration Rate ($\mu\text{m/yr}$)	
		1050°C $3 \times 10^5 \text{ rads/hr}$	1500°C $6 \times 10^5 \text{ rads/hr}$
304L (Solution-annealed)	J-13 water	0.31; 0.31	0.36
	J-13 + tuff	0.29; 0.32	0.31
304L (solution annealed + "sensi- tizing" condition)	J-13 water	0.23; 0.37	0.51
	J-13 + tuff	0.25; 0.30	0.55

conditions. The heat treatment conditions are given in Table 4. Note that these times and temperatures are representative of the reprocessed glass waste pouring operation into a stainless steel canister; they would readily sensitize the higher-carbon 304 stainless steel. The actual carbon level in the tested steel was 0.024%. The slow strain rate tests were conducted at 10^{-4} and $2 \times 10^{-7} \text{ sec}^{-1}$ and at 1500°C in air-sparged tuff-conditioned J-13 water. The initial tests were conducted at 1500°C to accelerate any stress corrosion susceptibilities. The limited number of results in Table 4 indicate that neither metallurgical condition and neither strain rate provoked stress corrosion effects. All fractures were ductile. Tests are continuing at other temperatures and strain rates.

Table 4 Slow Strain Rate Test Results for 304L Stainless Steel in
 1500°C Tuff-Conditioned J-13 Water

Metallurgical Condition	Environment	Strain Rate (sec^{-1})	Elongation (%)	Yield Stress (ksi)	Comments
SA(a)	J-13	10^{-4}	54.0	25.8	replicate tests ductile fractures
SA	J-13	10^{-4}	52.0	27.1	
SA + S(b)	J-13	10^{-4}	49.6	--	replicate tests ductile fractures
SA + S	J-13	10^{-4}	51.9	29.6	
SA + S	air	10^{-4}	49.0	29.4	ductile fracture
SA + S	J-13	2×10^{-7}	49.0	26.6	replicate tests ductile fractures
SA + S	J-13	2×10^{-7}	48.0	27.2	

(a) SA = Solution-annealed (1050°C , 15 minutes; water quench)

(b) SA + S = Solution-annealed + "sensitizing" treatment (as above, then 600°C , 10 hours, air cool)

Electrochemical Polarization Test Results

Electrochemical polarization testing of the reference and alternative canister materials complements the corrosion coupon testing. We are performing a number of electrochemical tests. Here we show an example of the test results obtained from an anodic potentiodynamic scan in the

forward and reverse directions. From such a scan, the so-called "breakdown potentials" and "protection potentials" can be obtained and compared to the "corrosion potential". Useful predictions of the tendency of alloy/environment combinations toward pitting and crevice corrosion are obtained from the comparison. In Figure 2, we show the protection and corrosion potentials obtained for 304L in J-13 water conditioned with crushed tuff at different temperatures. As the protection potential is considerably more noble (more anodic) than the corrosion potential, the alloy should not be susceptible to pitting or crevice corrosion in this environment. Alternative alloys (316L, 321, 825) likewise show E_{prot} more positive than E_{corr} in J-13 water in the 50-100°C temperature range. However, concentration of the J-13 water (as in a wetting and drying cycle) could lower the value of the protection potential. Radiolysis of the water and dissolved species in it could raise the value of the corrosion potential by making the environment more oxidizing. Under these circumstances the stainless steel could undergo localized attack. We are, therefore, conducting electrochemical tests where we concentrate electrolytic species in the J-13 water and where we make deliberate additions of the more stable radiolysis products (nitrate ion, hydrogen peroxide).

Figure 2. Corrosion Potentials and Protection Potentials for 304L Stainless Steel in Tuff-Conditioned J-13 Water at Different Temperatures

Conclusions and Future Work

All of the results to date indicate that 304L stainless steel performs suitably as a containment material under the environmental conditions tested, but much more testing is required before definitive conclusions can be drawn. The very limited number of corrosion test results confirms earlier predictions of generalized corrosion behavior. As localized corrosion (intergranular, pitting, crevice) and stress corrosion cracking concerns are more restrictive in the use of 304L (and other austenitic alloys), these forms of corrosion attack will be addressed more thoroughly in future work. Future work will bracket the

response of the candidate alloys to the expected environmental conditions at Yucca Mountain, as well as to possible episodic environmental conditions. These tests will provide an important part of the data base from which the final selection of materials and fabrication processes will be specified and on which the models for extrapolation to long term corrosion behavior will be based.

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