Conf-910945--1

PROGRESS IN EVALUATING THE CORROSION OF CANDIDATE HLW CONTAINER METALS IN IRRADIATED AIR-STEAM MIXTURES*

ANL/CP--72982

DE92 000298

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Submitted to
Focus '91, Nuclear Waste Packaging
American Nuclear Society and
American Society of Materials
Las Vegas, Nevada
September 29-October 2, 1991

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*This work was performed under the auspices of the U.S. Department of Energy by Argonne National Laboratory under Contract #W-31-109-ENG-38 and Lawrence Livermore National Laboratory under Contract #W7405-ENG-48. This paper was prepared by Yucca Mountain Site Characterization Project participants as part of the Civilian Radioactive Waste Management Program. The Yucca Mountain Site Characterization Project is managed by the Yucca Mountain Site Characterization Project Office of the U.S. Department of Energy, Las Vegas, Nevada.

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ABSTRACT

Technical issues related to the performance of metals in the air/water vapor environment expected in the potential Yucca Mountain repository are discussed. A status of work to address these issues is given. Preliminary experiments, focused on the atmospheric corrosion of copper-based materials, are summarized. These experiments were performed over a broad range of conditions: temperatures betweens 90 and 150°C; relative humidities of 0, 15, 40, and 100%; and gamma dose rates between 0.01 and 0.3 Mrad/hr. In irradiated moist air, copper-based materials form copper oxides and nitrate phases depending on the dose rate, humidity and temperature. The rates of general corrosion increase with temperature, humidity, and dose rate. Chemical intermediates formed by radiolysis of moist air have been clearly associated with observed corrosion. No significant corrosion was observed for Alloy 825.

INTRODUCTION

The Yucca Mountain Site Characterization Project is evaluating Yucca Mountain in Nye County, Nevada, as a site for a potential high-level nuclear waste repository. Lawrence Livermore National Laboratory is concerned with the development and performance modeling of waste packages for the potential repository. Argonne National Laboratory has performed experimental studies in support of the waste package effort. This effort is currently guided by the Waste Package Plan, which calls for a systems engineering approach to waste package development. Part of this approach involves formulating an approved set of selection criteria to choose the materials to be used in fabricating the waste packages.

The expected environment in a potential repository located in the unsaturated zone of Yucca Mountain is a mixture of air and water vapor. Since the reference design for the waste packages when this work was begun incorporated a relatively thin container wall (about 1 cm thick), it was clear that gamma radiation from the spent fuel or borosilicate glass waste forms would penetrate the wall and irradiate the air-steam mixture in the near-field environment of the waste packages. Preliminary shielding and thermal analyses indicated that the initial gamma dose rates could be as high as 0.1 Mrad/h with temperatures that may exceed 250°C. These depend significantly on waste package design, repository design, fuel burnup, fuel history, and fuel age. Interest in radiolytically-enhanced corrosion is limited to the first 300 y of repository history, since the gamma flux decreases with an effective halflife of 30 y.

Work published in the literature [1] as well as experience [2] in the Climax spent fuel test indicated that the gamma dose rates could influence the corrosion or oxidation of metals in moist air. Since these processes are expected to limit the containment lifetimes of the waste packages, and since a requirement for containment is specified in 10 CFR Part 60, we decided that the area of radiation-influenced corrosion should receive more study. It was also clear, based on the literature, that of the candidate materials then under consideration, the copper-based ones would be most affected by irradiation. Accordingly, we decided to focus the effort primarily on them, although we have included samples of Alloy 825 in our experiments for comparison.

It is also important to note that the work completed to date has focused more on corrosion from the perspective of the gas phase radiation chemistry than the overall and long-term performance of the candidate materials. The literature was reviewed in the areas of the radiation chemistry of air/water vapor systems [3] and oxidation of copper-based materials [4]. We found that most of the experiments in both these areas had been performed at room temperature and at low to moderate humidity. Since the potential repository conditions were expected to include temperatures as high as 250°C, as well as high humidity, we decided to extend the experimental data base through the relevant ranges of conditions. Experimentally, we have also addressed the question of the radiolytic formation of ammonia in oxygen-containing systems [5] and performed NO_x yield studies over a wide range of conditions [6,7].

Most of the work just discussed was performed prior to formulation of the Waste Package Plan, but the data generated have aided in the selection process called for in the Plan. At the time this work was started, six metals were under active consideration for use in the waste packages: Types 304L and 316L stainless steels; high nickel Alloy 825; and three copper-based materials--CDA 102 (oxygen-free copper), CDA 613 (7% aluminum-bronze), and CDA 715 (70-30 cupronickel). To characterize the behavior of the copper-based materials in irradiated moist air systems we performed a series of experiments designed to (1) identify the corrosion products formed as a function of temperature and humidity [4], (2) establish the short-term rates of general corrosion (oxidation) as a function of temperature and humidity [8], and (3) start evaluating the role of radiolytically generated products in the corrosion process. Progress made in these areas is reported herein.

TECHNICAL CONCERNS: RADIOLYTICALLY ENHANCED CORROSION IN AIR-WATER VAPOR MIXTURES

The atmospheric corrosion of copper-based alloys has been relatively well established in nonirradiated systems [9,10]. Exposure to atmospheric conditions results in the following: initially a brown, presumably cuprite

(Cu_2O), film forms, followed by a dull black film, presumably tenorite (CuO), which in time will form a patina that can vary in color depending on the presence of other species in the gas phase. In low relative humidity systems corrosion rates are low, ranging from 0.04 to 0.18 $mg/cm^2/y$. The rates obtained are strongly dependent on the relative humidity and the presence of anionic species such as chloride and sulfide in the gas phase.

In irradiated systems, however, much less is known about the corrosion behavior of copper-based alloys. This point was made previously [8], and existing information about the nature of the phases generated in irradiated moist air systems was summarized. Other work has already been reported in connection with the high-level waste effort [11-13].

The expected environment is an air/water vapor mixture that will likely evolve throughout repository history. A range of compositions from dry air to low humidity air to carbon dioxide-rich air to pure water vapor are considered to be within the realm of anticipated environments. In these environments, the following technical concerns have been identified that relate to the performance of the candidate materials during the initial unsaturated phase of repository history:

1. Radiolytic increase in the uniform corrosion for each anticipated environment. This includes corrosion product identification and species-specific interactions with the candidate metal.

2. Long-term change in uniform corrosion due to radiolytically-induced changes in the protective layer formed.

3. Potential for pitting due to radiolytically generated species in the range of anticipated environments.

4. Radiolytic formation of species that enhance the likelihood of environment assisted cracking (EAC). This is currently known to include ammonia formation (copper-based materials) and nitrite formation.

PROGRESS MADE IN ASSESSING THE EFFECT OF RADIOLYSIS ON METAL CORROSION

Some progress has been made in the first, third, and fourth technical concerns just identified. The identity of the corrosion products observed, initial rates of corrosion and preliminary results related to the relationship between radiolytic products generated and observed corrosion are discussed here.

Identity of Corrosion Products

Our examination of the literature showed that under nonirradiated pure moist air conditions the corrosion or oxidation products found on copper are cuprite (Cu_2O), which is reddish brown in color and tenorite (CuO), which is dull black. Under conditions of atmospheric corrosion where chemical contaminants are present in the air, green or bluegreen corrosion products such as basic chlorides, sulphates, or carbonates are formed. When high radiation fields are present, blue-green dicopper trihydroxy nitrate $Cu_2NO_3(OH)_3$ has been reported.

In experiments performed by Yunker [11], sponsored by the precursor of the Yucca Mountain Site Characterization Project, only cuprite and tenorite were found under irradiated high-humidity air conditions at 95 and 150°C, with gamma dose rates between 1.9×10^3 and 4.9×10^5 rad/h. This was puzzling in view of the literature reports as well as the observation of $Cu_2NO_3(OH)_3$ on copper parts in the LLNL electron linear accelerator target room.

Since a knowledge of the identity of the corrosion products is fundamental to an understanding of the corrosion process we performed experiments to clarify this situation. The general conditions and results obtained are summarized in Table 1. The temperatures used were 90, 120, and 150°C, and relative humidities ranged from zero to 100%. Exposures were a few weeks in duration, at dose rates of 7×10^4 to 2×10^5 rad/hr. The results were reported in 1989 [4]. We found that a key factor was the humidity. Under low to moderate humidity conditions, both the copper oxides and $Cu_2NO_3(OH)_3$ were formed on all three copper-based materials. Under conditions of 100% relative humidity at both 90 and 150°C, only the oxides were found on the oxygen-free copper and the aluminum bronze. The oxides predominated under these conditions on 70/30 copper-nickel, but some of the nitrate phase was found. We were thus able to harmonize the results in the older literature with those of Yunker, and reveal humidity as an important parameter in the irradiation-influenced corrosion of copper-based materials in moist air. It is the buildup of nitric acid concentration on the surface of the metal, which can only occur at intermediate relative humidities, that leads to the formation of nitrate phases.

In later experiments at lower dose rates of 1.1 to 2.1×10^4 rad/hr, we found that the oxides predominated even at moderate humidity. This reflected the competition between the thermal corrosion of the metal, which leads to the formation of oxides, and the oxidation of copper by radiolytically generated NO_x species, which leads to the formation of nitrate corrosion products. Thus, both the humidity and the dose rate are important factors in determining the corrosion products. Alloy 825, the high nickel alloy put in for comparison, showed no observable corrosion. It went in and came out mirror shiny.

General Corrosion Rates as Functions of Temperature and Humidity

The second issue we have explored is the dependence of corrosion rate on temperature and humidity under irradiation [8]. A summary of the corrosion rates we measured are in Table 2. These experiments extended to periods of between 30 and 78 days, and the dose rates were between 1.1×10^4 and 2.1×10^4 rad/hr. Temperatures used were 94 and 152°C. These experiments were performed in the low relative humidity regime (40% RH at 90°C and 0.06% RH at 150°C) although the water vapor content of the gas phase was 30 mole% at both temperatures. The general corrosion behavior we observed for these systems was similar to that observed in nonirradiated systems in that cuprite and tenorite were the predominant corrosion products identified. In all cases cuprite was the initial product observed changing over to tenorite as increased oxidation occurred. This result is significantly different from what we previously observed at gamma dose rates that were effectively ten times higher. In the higher dose rate and total pressure experiments, nitrate phases, in the case of both copper-nickel and aluminum bronze, were the predominant corrosion products and appeared as a minor product on the oxygen-free copper specimens.

The relative rates of corrosion observed decreased in the order of copper $>$ copper-nickel = aluminum bronze in the moist air 90°C experiments. This same order was noted in the long term moist air experiment at 150°C. In the dry air experiments, no consistent trend was observed with all three having the same rate of corrosion at 90°C and copper-nickel undergoing the least corrosion at 150°C. These experiments showed that the corrosion rates of all three materials increased with temperature and were also higher in the humid air as compared to the dry air. The factor of increase between the two temperatures was typically about 4 to 6. The increase due to humidity ranged between 1.35 and 4.0. The general corrosion rates observed ranged between 0.12 and 3.0 mg/cm^2/y (equivalent to between 0.13 and 3.9 microns/y) for the various materials and conditions. Comparison to nonirradiated experiments at similar temperatures and humidities reported in the literature showed that irradiation increased the rates by about 50%.

The vast majority of data for copper-based materials relates to their behavior in outdoor environments. Typical values for the atmospheric corrosion of copper-based materials subjected to long-term exposures of dry air (at ambient temperatures) are in the range of 0.04 to 0.18 mg/cm^2/y. Some data were, however, reported for oxygen-free copper in air-steam conditions in the same humidity regime as in our experiments. At 150°C, corrosion rates of 2.45, 1.39. and 0.88 microns/y were obtained for 1-, 4-, and 10-month exposure times, respectively. These were done at total pressures of 660 ± 10 torr (at the temperature of each experiment). This compares to 3.5 and 2.7 microns/y which we obtained in 1-month and 2.5-month experiments at the same temperature at a gamma dose rate of 0.02 Mrad/h. Although some of the experimental methodology differed, there was a significant enhancement of corrosion, due to the gamma radiation present, by a factor of approximately 1.5.

Role of Radiolytically Generated Intermediates

As discussed in the Introduction, our efforts have encompassed studies of both the radiation chemistry of moist air and the corrosion and oxidation of candidate waste package container materials under relevant temperature, humidity, and dose rate conditions. In order to be able to formulate models that can be used to project long-term behavior, we must be able to couple these two studies together. This involves developing an understanding of the part played by chemical intermediate species generated by the irradiation. Our progress in this area is discussed in this section.

In the experiments we performed, the total pressures of the dry and moist air experiments were approximately the same (660 torr at the temperature of the experiment). The gas phases, however, differed in that, in the moist air experiment, water vapor replaced some of the air (particularly oxygen) as the source of oxidizing species. Radiolytically only ozone, nitrogen fixation products, and associated transients were generated in the dry air system. The presence of water vapor resulted in a partial replacement of these species with the OH and HO_2 free radicals as well as molecular products such as hydrogen peroxide, nitrogen acids and perhaps ammonia.

The effect of replacing air with water vapor can be evaluated, on a preliminary basis, by comparing the corrosion rates observed in the dry and moist air experiments. At 90°C and 150°C, corrosion rates were determined to be significantly lower in the dry air experiments. Although further work is needed, this result is consistent with the more strongly oxidizing environment present when water vapor, rather than oxygen, is subjected to ionizing radiation. This is believed to be a gas phase phenomenon since relative humidities were low.

Some preliminary experiments were also performed to specifically address the question of what radiolytic intermediates led to the corrosion observed. These, along with the irradiation conditions and general corrosion observed are summarized in Table 3. The material in all these experiments was the candidate copper nickel material, with experiments performed at 87°C and dose rates of 1-2 x 10^4 rad/h.

Although these results are very preliminary, some interesting trends were noted. The overall extent of corrosion, in all cases, was relatively small owing to the low temperature and absorbed dose. There were, however, noticeable differences in the general appearance of the coupons in the various atmospheres investigated. No corrosion product analyses were performed because their buildup on the surface of the metal was too small to analyze using X-ray diffraction methods.

In the relative humidity series of experiments (CN-RH-0,40 and CN-PA-100), there was a systematic increase in the extent of bronze coloration associated with cuprite formation as the relative humidity increased. Since the total pressure was fixed at 660 torr at 90°C in these experiments, the NO_x yield decreased with increased relative humidity as the air was replaced by water vapor (NO_x yield is proportional to the partial pressure of N_2). This is consistent with the results from the experiments just summarized.

Two pure gases were investigated. These were pure oxygen and pure water vapor. For pure oxygen, there was little evidence of corrosion even though the oxygen concentration is approximately five times higher than in dry air. This suggests that molecular products of oxygen irradiation, in particular ozone, do not by themselves contribute to the initial corrosion processes observed. Atomic oxygen in this and air-like systems should be present at only small concentrations due to its short lifetime in these systems (reaction with molecular oxygen) and should not contribute to the corrosion observed. The radiolytic products of molecular oxygen, however, may contribute to the overall corrosion of the metal when other gaseous species are present. This can occur because these products will affect the overall radiation chemistry and react with the corrosion products, once they are formed. In dry air, it is NO_x, not ozone or atomic oxygen, that appears to be causing the observed oxidation of the metal.

Significant corrosion was, however, evident in the pure water vapor experiment. The total pressure in this system was set by the water vapor pressure and was 500-525 torr rather than 660 torr. The extent of corrosion was greater in this gas phase and there was clear evidence of tenorite as a corrosion product. It is clear that oxidizing species generated from the radiolytic decomposition of water are contributing to enhanced corrosion rates. Experiments need to be performed at lower relative humidities to distinguish between radiolysis in the gas phase and that occurring in the surface layer of water.

CONCLUSIONS AND RECOMMENDATIONS

Conclusions

We have found that the identity of the corrosion products on copper-based materials in irradiated moist air depends on both humidity and dose rate. The formation of $Cu_2NO_3(OH)_3$ is favored at low to intermediate humidity where the NO_x generated in the gas phase is concentrated in thin layers of water on the surface of the metal. It is also favored by higher gamma dose rate where radiolytic processes compete more effectively with thermal processes. In the absence of water vapor, hence in the absence of nitrogen acid formation, and at high relative humidities, where a thick layer of water is on the metal surface, the formation of Cu_2O and CuO is favored. This is also true as the dose rate is lowered and thermal processes predominate over radiolytically-induced reactions.

Alloy 825, which is the only iron-based alloy we investigated, did not exhibit observable corrosion under the same conditions used to study the corrosion of the copper-based materials. This is consistent with experience, since this material tends to resist attack by NO_x and nitrogen acids. Long-term studies and experiments oriented at EAC in an air-steam environment were not performed, however.

The initial rates of general corrosion on the three copper-based materials (oxygen-free copper, 70/30 copper-nickel, and 7% aluminum-bronze) were found to increase with temperature, humidity, and dose rate in the ranges studied. The radiolytic effect appears to decrease with time. Long-term effects, however, have not been addressed. It is clear that initially rates are significantly affected by radiolytic processes and this effect may alter the nature of uniform corrosion.

We have shown that in low humidity systems, it is primarily NO_x (NO, NO_2) and HNO_3 that contribute to the initial corrosion observed. Replacing air with water vapor enhanced corrosion and led to increased oxidation of the metal (greater relative amount of tenorite vs. cuprite). The effect of relative humidity is more clearly associated with the radiation chemistry in the water layer than that occurring in the gas phase.

Status of Technical Concerns and Recommendations

Although we have performed relatively few experiments, some progress towards addressing the technical concerns identified has been made. With respect to the question of radiolytic enhancement of uniform corrosion, we have observed that (1) initial corrosion rates are significantly enhanced by gamma dose rates in excess of 0.01 Mrad/h, and (2) relative humidity affects the extent of corrosion, the nature of the corrosion products generated and the likelihood of localized corrosion. The long-term implications of these initial trends, which are most important relative to repository licensing concerns, have not been established, however. There is clearly a need for long-term irradiated experiments in an air/water vapor environment and long-term thermal corrosion experiments with coupons that have been pre-exposed to an irradiated air/water vapor environment.

The question of localized corrosion has been addressed only indirectly by observations made in the corrosion experiments performed. We have, on occasion, for the intermediate relative humidities investigated, observed some localized corrosion as the result of localized condensation of NO_x on the surface of the metal. Our results, although few in number, are sufficient to signal to the YMPO that localized corrosion, under some "expected" conditions, may be possible. The overall importance of this concern is therefore closely tied to the expected environment scenario. The question of localized effects clearly warrants further experimental consideration.

No work was performed, in a gas phase environment, that directly addresses the likelihood of environment assisted cracking of the candidate materials. However, in the gas phase radiolysis work, summarized in the companion paper in this symposium, we have discussed the likelihood of ammonia production, nitrite, and organic acids. The implications of the existence of these species on EAC have not been investigated for the expected conditions at the potential Yucca Mountain site. Experiments pertaining to this concern, performed in the expected environments, are recommended.

ACKNOWLEDGMENTS

This work was performed under the auspices of the U.S. Department of Energy by Argonne National Laboratory under Contract #W-31-109-ENG-38 and Lawrence Livermore National Laboratory under Contract #W7405-ENG-48. This paper was prepared by Yucca Mountain Site Characterization Project participants as part of the Civilian Radioactive Waste Management Program. The Yucca Mountain Site Characterization Project is managed by the Yucca Mountain Site Characterization Project Office of the U.S. Department of Energy, Las Vegas, Nevada.

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Table 1. Corrosion Products Identified as a Function of Irradiation Conditions

Metal	Environment	Major Component	Lesser Components
Aluminum Bronze	120°C, 15% RH	$Cu_2(OH)_3NO_3$	Cu (solid solution)
	150°C, dry	Cu_2O	$Cu(OH)_3NO_3$, CuO
	150°C, 15% RH	$Cu_2(OH)_3NO_3$	amorphous $Cu_2(OH)_3NO_3$
	150°C, 100% RH	Cu_2O	CuO
Copper	120°C, 15% RH, yellow	Cu	Cu_2O, $Cu(OH)_3NO_3$
	120°C, 15% RH, red	Cu_2O	$Cu_2(OH)_3NO_3$
	150°C, dry	Cu	Cu_2O, $Cu(OH)_3NO_3$
	150°C, 15% RH, black	Cu_2O	$Cu(OH)_3NO_3$
	150°C, 15% RH, yellow	Cu_2O	$Cu(OH)_3NO_3$
	150°C, 100% RH	Cu_2O	CuO
70/30 Copper Nickel	120°C, 15% RH, green specs	$Cu_2(OH)_3NO_3$	
	120°C, 15% RH, black spots	$Cu_2(OH)_3NO_3$	Cu_2O, Cu solid solution
	150°C, dry air	Cu_2O	CuO, $Cu(OH)_3NO_3$

Table 2. (A) Irradiation Conditions and (B) Corrosion Rates Measured for 70/30 Copper Nickel (CN), 7% Aluminum Bronze (AB) and Oxygen-Free Copper (CU)

A

Experiment Designation (AB-, CN- or CU-)	Environment	Duration (d)	Dose Rate/ Absorbed Dose (Mrad/h)/Mrad
-1R-90M	94°C, 40% RH @ 90°C Air	35	0.021/17.4
-3R-90M	94°C, 40% RH @ 90°C Air	73	0.021/36.6
-3R-90D	94°C, Dry Air	75	0.021/37.6
-1R-150M	152°C, 40% RH @ 90°C Air	30	0.015/10.7
-3R-150M	152°C, 40% RH @ 90°C Air	76	0.011/20.0
-3R-150D	152°C, Dry Air	78	0.015/27.9

B

Experiment Designation	Corrosion Rate $mg/cm^2/y$	Corrosion Products	
		Major	Minor
CU-1R-90M	1.5 ± 0.1	ND	
CU-3R-90M	0.56 ± 0.06	ND	
CU-3R-90D	0.14 ± 0.02	NA	
CU-1R-150M	2.6 ± 0.1	Cu_2O	CuO
CU-3R-150M	2.3 ± 0.1	Cu_2O	CuO
CU-3R-150D	1.6 ± 0.05	Cu_2O	CuO
CN-1R-90M	0.33 ± 0.05	Cu_2O (brown spots)	
CN-3R-90M	0.21 ± 0.03	NA	
CN-3R-90D	0.12 ± 0.04	NA	
CN-1R-150M	1.4 ± 0.1	Cu_2O	CuO
CN-3R-150M	1.1 ± 0.2	CuO	Cu_2O
CN-3R-150D	0.48 ± 0.04	CuO	Cu_2O
AB-1R-90M	0.71 ± 0.13	ND	
AB-3R-90M	0.23 ± 0.05	ND	
AB-3R-90D	0.16 ± 0.01	ND	
AB-1R-150M	3.0 ± 0.4	Cu_2O	CuO
AB-3R-150M	1.2 ± 0.2	CuO	Cu_2O
AB-3R-150D	0.89 ± 0.02	Cu_2O	CuO

Table 3. Irradiation Conditions and Results of Parametric Experiments Performed at P ~ 660 torr (except pure water vapor) and 87 ± 2°C for 70/30 Copper Nickel

Experiment	Environment	Dose Rate (Mrad/h)	Duration (h)	Absorbed Dose (Mrad)	General Appearance of Coupons
CN-RH-0	Dry air	0.021	609.2	13 ± 1	Dull grey, even
CN-RH-40	40% relative humidity air	0.015	469.7	7 ± 0.5	Bronze/grey
CN-PA-100	100% relative humidity air	0.015	609.2	9 ± 0.5	Bronze, very patchy, initial evidence of localized effects
CN-PA-H_2O	Pure water vapor	0.021	609.2	13 ± 1	Blank tarnish, patchy
CN-PA-02	Oxygen	0.021	609.2	13 ± 1	Clear, essentially no corrosion/tarnish

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