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Potential Mechanisms of Corrosion and Stress Corrosion Cracking Failure
of 3013 Storage Containers Composed of 316 Stainless Steel

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The degradation of 316 stainless steel (SS) storage container materials is a potential problem for radioactive waste disposition. Container materials will be exposed to significant ionizing radiation, elevated temperatures, embrittling and/or alloying agents (e.g., gallium), chloride-containing compounds (as much as 20 wt% Cl or Cl⁻), oxidizing compounds, and a limited quantity of moisture. Additionally, containers will contain welds that have heterogeneous composition due to solute segregation and that may retain significant residual stress. All of the above-listed environmental and material conditions have been shown to be deleterious to material integrity under certain conditions. Unfortunately, the precise conditions within each container and environment is unknown and may vary widely from container to container. Thus, no single test or set of tests will be able to mimic the broad range of storage container conditions. Additionally, material behavior cannot be predicted at this time because the synergistic effects of temperature, time, chloride, moisture, sensitization, weldments, salt formation, etc., have not been fully studied. The complexity and uncertainty of storage conditions currently precludes any detailed recommendations. The objective of this document is to detail selected previous studies and to suggest some general guidelines for storage of radioactive waste. Because of the voluminous research in this area, this review cannot be considered to be comprehensive. Readers are directed to references that contain detailed reviews of particular processes for more information. Note that the effect of gallium on the degradation of SS storage containers has been discussed elsewhere by the authors and will not be discussed here.¹

Atmospheric or Gaseous Corrosion

The corrosion of 3013 containers by contained gas may be considered a rather special case of atmospheric corrosion. Although there are no specific fundamental studies relevant to the 3013 environment, there have been many studies of atmospheric corrosion of materials from which we can gain insight (see for example references in Leygraf²). The majority of the research that has been

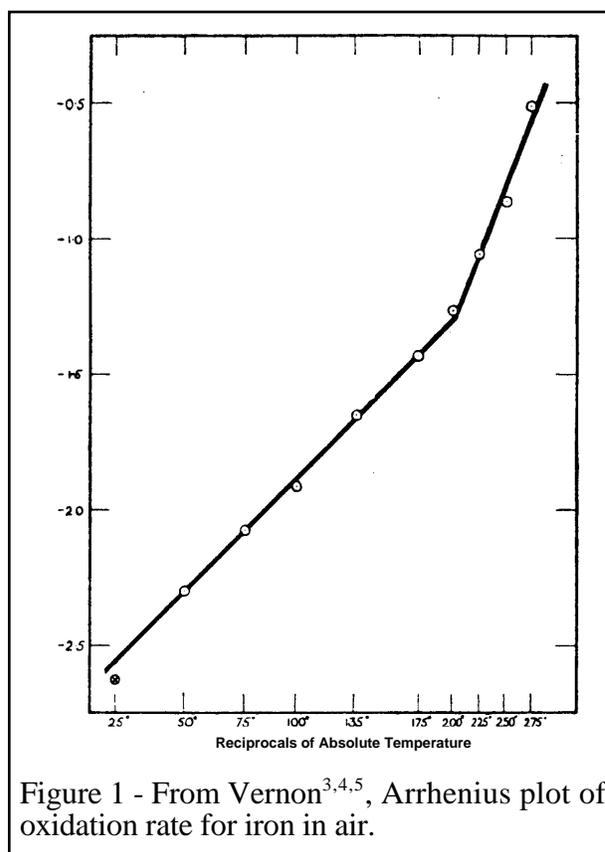
done has been concerned with outdoor atmospheric corrosion where humidity levels can be high and variable, and where there are a wide variety of corrosive pollutants such as SO₂ and salts, and various particulate. It has only been relatively recently that people have become concerned with indoor atmospheric corrosion, due largely to increased concern for the life of electronic components. The 3013 storage container condition is a rather unique hybrid having certain corrosive elements similar to both indoor and outdoor environments.

Corrosion by the earth atmosphere is defined by the presence of a very thin, sometimes discontinuous, aqueous electrolyte that forms on the surface of a material. This electrolyte often contains impurities from the environment in a concentrated form. Atmospheric corrosion differs from aqueous corrosion in that the thin layer of electrolyte readily allows oxygen (the cathodic reaction species) from the atmosphere to diffuse to the surface of the material. During atmospheric corrosion, higher oxygen concentrations at the surface cause the corrosion processes to occur at higher rates than would ordinarily occur in an otherwise similar aqueous solution, where rates are often cathodically limited. For example, corrosion at the bottom of the sea occurs much more slowly than at the surface. Therefore, atmospheric corrosion can be significantly more severe than aqueous corrosion if there is a sufficient quantity of electrolyte present.

There are many variables that affect the rate of atmospheric corrosion. The most important variables are material, water vapor partial pressure (p_{H_2O}), temperature (T), time (t), and impurities. Relationships have been developed that describe the corrosion rate of materials in water vapor environments, where impurities are absent or controlled, that essentially have the form:

$$\text{corrosion rate} = \text{constant} + f(p_{H_2O}) \cdot f(T) \cdot f(t)$$

Thorough studies of atmospheric corrosion were done by W.H.J. Vernon more than 60 years ago.^{3,4,5} He found that there was a critical water vapor partial pressure, $p_{H_2O}^*$, above which the rate of atmospheric corrosion became appreciable (on the time scale of less than one year). It has



been stated that, as a crude rule of thumb, if the relative humidity is 20%, there will be about 1 monolayer of water on the surface of a metal, at 60% there will be 2-5 layers, and at 80% there will be 5-10 layers.^{6,7} However, this can be substantially different for different metals. For example, Dante and Kelly observed approximately 5 monolayers of water on the surface of gold at only 15% relative humidity and more than 40 monolayers at 95% humidity.⁸ Experimental measurements of the properties of thin adsorbed layers of water indicate that when approximately 3 monolayers (or more) of water are present the layer is an effective electrolyte.⁹ These results are in relatively good agreement with the studies of Vernon.^{3,4,5} Above $p_{\text{H}_2\text{O}}^*$, the rate of corrosion tends to increase as some function of $p_{\text{H}_2\text{O}}$. Further, Vernon found (as have many others) that above $p_{\text{H}_2\text{O}}^*$, the atmospheric corrosion typically exhibits Arrhenius behavior. As shown in Figure 1, in the case of iron exposed to moist air, Vernon found that an Arrhenius relationship was obeyed from room temperature to 200°C. Therefore, the rate of atmospheric corrosion increases exponentially with temperature.

In general, based on a variety of reported studies, increasing the temperature of a metal by 50°C will increase the atmospheric oxidation rate by a factor of 2-20. The effect of relative humidity on oxidation can not be predicted unambiguously. However, above $p_{\text{H}_2\text{O}}^*$, a doubling of the relative humidity level, for example from 35 to 70%, can roughly double the rate of oxidation. Thus, the key to preventing oxidation or corrosion is to keep the relative humidity and temperature as low as possible.

In addition to reducing $p_{\text{H}_2\text{O}}$ and T, it is very important to keep the environment as free of impurities as possible. If corrosive impurities are present, the effects of T and $p_{\text{H}_2\text{O}}$ may be comparatively negligible. For example, the presence of halogens such as chloride can lead to breakdown of any otherwise protective oxide film. This breakdown of the oxide, can be autocatalytic and can lead to the evolution of relatively large corrosion features such as pits. Another example is the corrosion of metals exposed to NO_2 vapor. NO_2 vapor has been shown to react with water to form a thin nitric acid solution on a surface that may be as concentrated as 1 M.⁸

Vapor corrosion rates, the propensity for pitting, stress corrosion cracking susceptibility, etc. cannot be predicted based on existing data but must be evaluated case-by-case due to the complex synergy of material, environment, dew point effects, etc. However, it is clear from previous studies that the material in contact with chloride based high level waste will react. It can be stated almost without exception that reducing the water vapor partial pressure, temperature and chloride concentration will have a positive effect.

It is beneficial that the 3013 environment is a closed system because available reductants (e.g., water) will tend to be consumed in the metal oxidation reaction. However, it is unclear whether the water will remain fixed (for example in a stable hydrated oxide) or if it will be recycled and available for further reactions. The sections below attempt to address some of the potential specific effects of variables such as radiation, welding, and chloride concentration on atmospheric corrosion.

Radiation effects

The effects of ionizing radiation on the corrosion and stress corrosion cracking (SCC) of SS can be divided into three primary components - modifications of the structure of the underlying substrate (including segregation and the introduction of defects), alteration of the diffusive nature of the passive film, and changes in the environment. Considering SCC, it has been stated that "The possible interactions between radiation and environmental cracking are numerous, and many are poorly quantified and/or completely unknown. In addition to changes in mechanical properties, microstructural evolution, creep, and radiation-induced segregation, many associated phenomena may be influential in irradiation-assisted SCC (IASCC), including hydrogen trapping, localized shear, inhomogeneous deformation near interfaces due to hardening and radiation induced segregation, phase transformations, transmutations (to form hydrogen, helium, etc.) and damage to the protective oxide. While many of these radiation phenomena are hypothetically important in IASCC, the paucity of and difficulty in obtaining data dictate that key radiation phenomena be identified and studied; to date, the international consensus is that key elements include radiation-induced segregation (RIS) and radiation water chemistry."¹⁰ It should be noted that alteration of the diffusive nature of the passive film may also be important, in light of research which showed that pre-oxidation of 304 SS can reduce IASCC susceptibility. Regardless, a huge volume of work exists on radiation effects on materials and environments. For more information, readers are directed to annual series and journals that contain extensive examinations of radiation effects on 304 and 316 SS.^{11,12,13} For reference, the compositions of 304 and 316 SS are shown in Table 1.

	Fe	Cr	Ni	Mo	Mn	Si	N	C	P	S
304 SS	bal.	18.0-20.0	8.0-10.5	-	2.00	1.00	0.10	0.08	.045	.03
316 SS	bal.	16.0-18.0	10.0-14.0	2.0 - 3.0	2.00	1.00	0.10	0.08	.045	.03

Table 1 - SS compositions in wt%. All single values are maximum values.

Structure / composition alterations

Radiation affects mechanics, microstructure, and environment by causing relaxation and hardening, and depletion and enrichment at grain boundaries.^{14,15,16,17,18,19,20} Radiation affects mechanics and microstructure, in part, by producing vacancy clusters. Point and clustered defects cause an increase in hardness, yield strength, and UTS as well as a loss of ductility and fracture toughness. Defects may also result in heterogeneous deformation realized in the form of regions of intense shear. These regions of intense shear increase the susceptibility to SCC by the mechanism of slip - film rupture - dissolution. Moreover, IASCC can be severe enough to result in grain dropout.²¹ Structure / composition alterations of the 304 SS substrate are more likely to affect SCC properties than corrosion properties, although segregation effects can be significant if they result in sensitization of the SS due to Cr depletion at the grain boundaries.^{15,16,17,18} The difference between classical thermal sensitization and sensitization by RIS is that the RIS sensitization does not result from carbide formation and has a narrower composition profile due to the lower temperatures involved¹⁰. It has been stated that "The absence of a clear correlation [between impurity (e.g., Si, N, H, S, P, C, B) and alloy element segregation and IASCC, with the exception of sensitization] may suggest that other factors dominate, such as complex interactions among alloying elements and impurities, or among RIS, radiation hardening, and creep."¹⁰ Thus, with the exception of sensitization by RIS, the effects of radiation on the structure / composition relationship with IASCC are not fully understood.^{15,22}

Radiation effects on the storage environment

The vast majority of studies examining the effects of radiation on corrosion incorporate some form of water, whether in the liquid or vapor state. There are a variety of review articles which extensively cover radiolysis effects.^{10,23,24,25,26} In short, radiolysis produces a variety of reactive unstable and metastable compounds, which may be oxidizing or reducing. The most significant product of radiolysis is hydrogen peroxide (H_2O_2) which is a strong oxidizer. Therefore, radiation typically has the effect of increasing the corrosion rate of SS although exceptions (e.g., already strongly oxidizing environments) may exist.²³ One potential benefit of H_2O_2 production is the prevention of localized corrosion (crevice corrosion, pitting corrosion) in the case where significant radiolysis occurs in the localized environment.^{23,27} However, particular conditions are required to obtain this benefit.

In general, the atmospheric corrosion rate of 304 SS increases with increasing radiation dose rate, but no increase in the corrosion rate is typically observed in cases in which the radiation

dose absorbed in the metal causes heating of the metal, thereby preventing the formation of a condensed moisture film on the metallic surface.

The review of earth atmospheric corrosion in radiation environments by Byalobzheskii is helpful in understanding potential degradation mechanisms of SS containers.²³ It has been found that no corrosion of 18 Cr - 8 Ni SS (analogous to 304 SS) was observed during exposure to an electron accelerator (98% humid air, 20°C, 16 hrs.). In contrast Cu, Al, Zn, and Fe corroded under these conditions. The SS results were ambiguous, however, because samples may have been free of condensate due to heating by the electron beam. A similar result was observed by Shatalov using a Po²¹⁰ source (α radiation).²³ Primak and Fuchs also noted a lack of corrosion of Au and SS where Al, Co, Cu, and Ni corroded.²⁸ The apparent corrosion resistance of Au and SS is likely due to HNO₃ production from atmospheric nitrogen which attacks Al, Co, Cu, and Ni but not Au and SS. Given that the atmosphere inside of storage containers is quite different from that of earth atmosphere, SS would not be expected to be immune to corrosion. It has been stated that "the corrosive effect of radiation is much greater than the effect of small amounts of contaminants (CO₂, SO₂, Cl₂, dust, organic substances) in the air, and that the effect of radiation on the atmospheric corrosion of metals is not associated with the effect of radiation on the above-mentioned contaminants."²³ Again, this is for the earth atmosphere and not the atmosphere inside of a storage container.

Earth atmospheric corrosion under irradiation occurs only in the presence of moisture. In a dry atmosphere (at room temperature), SS samples have shown no signs of corrosion after irradiation under any conditions.²³ However, given the presence of some moisture, irradiation has been shown to shift the critical humidity for atmospheric corrosion of iron to 15-20% as compared w/ 60-70% in the absence of radiation.^{29,30,31} Increasing humidity was shown to increase the corrosion rate for long-time tests. Observed increases in corrosion rate in moist atmospheres are attributable to the production of short-lived radiolysis products and to damage to the metal, with short-lived species having the predominant effect.²³

The composition of the storage container atmosphere exclusive of water content is also important during irradiation. N₂ and O₂ have the potential to increase the corrosion rate due to production of nitrogen-based oxidizers and ozone, respectively. As stated above, a significant quantity of nitrogen and nitrogen compounds has the potential to be beneficial if significant quantities of HNO₃ are produced, but conditions must be tightly controlled.

Byalobzheskii summarizes atmospheric corrosion of irradiated metals as follows: "The following mechanism of the effect of radiation on the atmospheric corrosion of metals may be proposed on the basis of the experimental data: in principle, irradiation causes no change in the mechanism of atmospheric corrosion of metals; the corrosion is electrochemical and takes place within a thin (condensed or adsorbed) layer of moisture on the metallic surface. The radiation-induced acceleration of the corrosion processes is caused by the formation (in the atmosphere) of various products as a result of radiation-chemical transformations of the oxygen, water, and nitrogen. These products consist mainly of strong oxidizing agents which serve as cathodic depolarizers. Short-lived radiolysis products have a particularly strong effect on the above process."²³

Influence of chloride on container corrosion

It is well known that the presence of chlorides in aqueous solution promotes the general and localized corrosion of materials, including 316 SS. The literature is replete with studies of radiation-enhanced aqueous corrosion of 316 SS exposed to chloride solutions.^{11,12} These studies find that increasing chloride and increasing radiation are detrimental to the corrosion resistance of SS. High temperature oxidation studies of SS exposed to Cl_2 and chlorine-containing gases have also been studied.^{32,33,34,35,36,37} At 340°C, a corrosion rate of 30 mils per year (mpy) and 120 mpy were noted for 304 SS exposed to dry Cl_2 and HCl, respectively.³⁴ Small additions (0.4%) of water may increase the corrosion rate orders of magnitude (Figure 2). The effect is largest at low temperatures but effectively disappears above 370°C. Studies on 310 SS, an austenitic SS containing more Cr and Ni than 316 SS, also indicate that increasing temperature and Cl_2 increase the corrosion rate.³⁸ Given the excellent atmospheric corrosion and SCC resistance of 316 SS in the absence of radiation and weldments, the presence of an aggressive element or compound such as chloride is required for container failure by corrosion or SCC. In the case of dry chlorine gas corrosion, scaling rates are probably determined by the diffusion through the chloride scale and the protectiveness of this scale.³³ In his review,

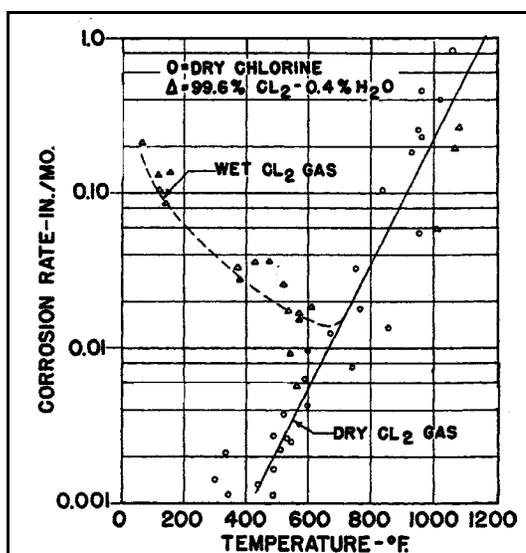


Figure 2 - Corrosion rate (inch/month) in wet and dry Cl_2 gas as a function of temperature ($^{\circ}\text{F}$).³⁴

Dillon notes that temperature cycling promotes spallation of these films and thus promotes increased corrosion.³⁹ Further, in the presence of moisture, radiolysis products can combine with halogenated organics to produce HCl²³, which is extremely aggressive in thin aqueous surface films.

Low melting temperature salt formation

The formation of molten salts is conceivable, given that large amounts of chloride (as much as 20 wt%) may be present. Molten salts are known to be extremely aggressive with respect to SS, as indicated in a review article.⁴⁰ Indeed, a standard accelerated test to examine stress corrosion cracking susceptibility of SS materials involves immersion in boiling MgCl₂ (155°C).⁴¹ Corrosion rates in these environments can be unacceptably large. For instance, Karlsson indicated that a particular KCl / NaCl mixture at 310°C yielded corrosion rates of 5 - 10 mm/month.⁴² Thus, the formation of low temperature molten salts can result in extremely rapid material degradation and should be avoided.

Weld effects

The effect of welding on materials has been well studied and is the topic of many journals.^{43,44} In short, weldments are commonly failure sites for SS materials.^{45,46} A review of SCC of weldments in boiling water reactor (BWR) service has stated that "The basis (for SCC) is the coexistence of three major contributors in the weldment: (1) tensile stresses above the yield stress of the base material, (2) a sensitized microstructure, (3) the BWR water coolant."⁴⁷ With the exception of (1), the same may be said for corrosion.⁴⁵ It should be noted that sensitization is not required for SCC or enhanced corrosion of weldments because brittle phase formation in the heat-affected zone can promote SCC and general and localized corrosion.⁴⁸ Regardless, it is clear that the synergy between radiation and weldments can promote material degradation and failure. It should be noted that some weldments can be more resistant to SCC and corrosion than the base material when high alloy filler materials are used in the weld.

Recommendations for storage conditions

Given that the contents of storage containers are not precisely known and that the contents will vary between containers, specific recommendations cannot be offered. Although the synergistic effects of humidity, temperature, radiation, salt formation, weldments, etc., have not been fully explored, it is probable that the following recommendations can be used as general guidelines for storage:

- 1) The water content within the container should be kept to a minimum. It is clear that the presence of water is detrimental to the corrosion and SCC resistance of 316 SS, especially within the likely wall temperature range of storage containers (150°F - 200°F)⁴⁹. The effect of water and some of its radiolysis products (most importantly H₂O₂) is to oxidize the SS container, enhancing SS metal loss and promoting SCC. One benefit of a closed container system is that a limited supply of reactant (water) is present, in the absence of compounds which may oxidize the water reduction products back to water. Backfilling containers with dry gas or adding desiccants may help to reduce corrosion and SCC. In the absence of humidity or a low melting temperature salt, corrosion is likely a smaller concern than radiation embrittlement, weld embrittlement, etc.
- 2) Container walls should be maintained at the lowest temperature possible, assuming that no significant humidity is present within the container. Because corrosion is an electrochemical reaction, a reduction in temperature will reduce the reaction rate, thereby reducing the rate of metal loss. There is a notable exception to this recommendation: If there is appreciable water within the container, the possibility of dew point corrosion exists. That is, water may vaporize within the container and then condense on a low temperature surface, i.e., the container walls. This is typically prevented in industry by keeping the container surfaces at a temperature equal to the environment interior. However, if a lack of condensation cannot be assured, it is best to maintain the lowest temperature possible to reduce the corrosion reaction rate. Additionally, temperature cycling may deleterious in the presence of a chloride scale and thus should be avoided.
- 3) Reductions in radiation flux and energy reduce corrosion by reducing radiolysis, material damage, and material temperature. For a given temperature, humidity, and atmosphere, the radiation threshold for safe storage can be determined via experimentation. In the absence of this information, determination of a radiation limit is impractical.

- 4) The thermodynamics of the waste and container should be well understood. For example, there exists the possibility that, combined with elements of the metal such as Fe, molten salts could form, leading to more rapid corrosion.
- 5) While the effect of chlorine-containing additions to dry atmospheres is not as dramatic as additions to humid atmospheres, the effect can be significant at elevated temperatures. There are almost no circumstances in which the additions of halide-containing compounds are beneficial to the corrosion or SCC resistance of 316 SS. Thus, halide-containing compound concentrations should be minimized wherever possible.
- 6) Avoiding weldments may significantly reduce the failure probability of storage containers. Where welding is required, the use of high alloy filler material along with careful control of heat-affected zone temperature profile can eliminate corrosion and SCC failures at the weld.
- 7) The use of different container materials may reduce or eliminate corrosion and SCC failure concerns. Ali and Moccari ranked the corrosion resistance of different austenitic SS exposed to 95% O₂ / 5% wet Cl₂ at 650°C. They found that the corrosion resistance of 310 SS > 316 SS > 304 SS > 321 SS.⁵⁰ Thus other stainless steels may afford greater corrosion protection. Other noniron-based materials may yield large improvements in corrosion and SCC resistance.

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