MCC

Materials Characterization Center

**State-of-the-Art Report on
Corrosion Data Pertaining to
Metallic Barriers for
Nuclear Waste Repositories**

October 1982

Prepared for the U.S. Department of Energy
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Pacific Northwest Laboratory
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MATERIALS CHARACTERIZATION CENTER

STATE-OF-THE-ART REPORT ON CORROSION DATA
PERTAINING TO METALLIC BARRIERS FOR
NUCLEAR WASTE REPOSITORIES

M. D. Merz

October 1982

Prepared for
the U.S. Department of Energy
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Pacific Northwest Laboratory
Richland, Washington 99352

PREFACE

The Materials Characterization Center (MCC) was established at the Pacific Northwest Laboratory to assemble a standardized nuclear waste materials data base for use in research, systems and facility design, safety analyses, and waste management decisions. This centralized data base will be provided through the means of a Nuclear Waste Materials Handbook. The first issue of the Handbook was published in the fall of 1981 in looseleaf format so that it can be updated as additional information becomes available. To ensure utmost reliability, all materials data appearing in the Handbook will be approved by an independent Materials Review Board (MRB) comprised of materials experts from Department of Energy laboratories and from universities and industry.

In the interim before publication of data in the Handbook there is need for a report summarizing the existing corrosion data on candidate metallic barrier materials. This review summarizes corrosion data for materials that are being considered for canisters and overpacks as part of the waste package. The information is intended to be a good representation (as of September 1982) of the data concerning the corrosion of metals for barriers.

Some general comments about the content of the compilation follow:

Organization. The data in this report are arranged according to testing environment. This organization was chosen primarily because corrosion of metals is very sensitive to the particular environment, and available data already were organized this way. Thus, brine and low-ionic strength waters were convenient classifications and were considered relevant to the various candidate deep geologic sites. An additional section on long-term underground data was included for completeness.

Measurement Methods. Measurement methods used to obtain much of the corrosion data are briefly described in this report. These descriptions are necessary because standard techniques have not yet been adapted for measuring many corrosion properties of barrier materials. The MCC is in the process of codifying such standard measurement techniques.

Relationship of Barrier Corrosion Performance to Criteria. The overall criteria to which barriers must be designed depend upon requirements specified by the Environmental Protection Agency (EPA) and the Nuclear Regulatory Commission (NRC). The requirements, which are in the late draft stages, allow a great deal of flexibility in waste package design since they set specifications only on release to the biosphere (EPA's draft 40 CFR 191) or from the boundaries of a geologic repository (NRC's draft 10 CFR 60). The requirements for the waste package are given in draft 10 CFR 60. These criteria are quite general and specify that the waste package shall provide containment for 1000 yr.

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The author appreciated the comments of several investigators who have studied the corrosion of barrier materials as part of Nuclear Waste Programs. J. E. Mendel and R. L. Dillon contributed significantly to reviewing the document, and the editorial comments of S. R. Gano also were appreciated.

SUMMARY

A compilation of published corrosion data on metals that have been suggested as canisters and overpack materials is presented in this report. The data were categorized according to the solutions used in testing and divided into two parts: high-ionic strength solutions (such as seawater and brine) and low-ionic-strength waters (such as basalt and tuff waters). This distinction was made primarily because of the general difference in aggressiveness of these solutions with respect to general corrosion. Considerable data were reviewed, but limitations of the data were also very evident. For example, in only a few cases were uncertainties in data given, either expressed in definitive statistical terms or presented in a way to permit a meaningful statistical analysis. An exception was the National Bureau of Standards long-term study of underground corrosion of metals. However, the data in that study has very limited applicability to waste disposal in deep geologic sites, mainly because temperature and radiation effects are unknown.

A considerable amount of data indicated that titanium alloys have acceptably low uniform corrosion rates in anticipated repository sites; the other possible corrosion failure modes for titanium alloys, such as stress corrosion cracking and delayed failure due to hydrogen, have not been sufficiently studied to make any similar conclusions about lifetime with respect to these particular degradation processes.

Other data suggested that iron-base alloys are sufficiently resistant to corrosion in basalt and tuff waters, although the effects of radiation and radiation combined with elevated temperature have not been reported in enough detail to conclusively qualify iron-base alloys for any particular barrier thickness in regard to uniform corrosion rate. The effect of overpack size on corrosion rate has been given little attention. However, this effect was beyond the scope of laboratory corrosion studies.

A review of long-term underground data indicated that temperature and accessibility to oxygen were too different for deep geologic repositories to make the underground corrosion data directly applicable. However, the

characteristics of corrosion attack, statistical treatment of data, and kinetics of corrosion showed that corrosion proceeds in a systematic and predictable way. Six-month laboratory data have been correlated with 10-yr underground corrosion, as an example of successful modeling of corrosion and use of short-term data to predict long-term behavior.

Earlier reviews of archaeological data were cited. Although metal objects have survived hundreds of years and even longer than 1000 years, it is very difficult, if not impossible, to relate the environments of archaeological objects to moist or wet conditions that may exist in a geologic repository at elevated temperature and with gamma radiation at the barrier-water interface. Eventually, further testing may make data on archaeological objects relatable to metallic barriers.

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STATE-OF-THE-ART REPORT ON CORROSION DATA
PERTAINING TO METALLIC BARRIERS
FOR NUCLEAR WASTE REPOSITORIES

1.0 INTRODUCTION

The Materials Characterization Center (MCC) had several purposes in preparing this state-of-the-art report on corrosion data. The primary goal was to summarize in one document published data that might predict the long-term survival of metallic structural barriers or aid in identifying deficiencies in such data. The applicability of available data is uncertain primarily because the environment surrounding the metallic barrier has not yet been defined. However, in considering the published data, the MCC has presumed, in line with the Office of Nuclear Waste Isolation and waste package designers, that water might contact the metallic barrier during its designed lifetime. Without water, the general opinion of corrosion experts is that corrosion damage to metallic barriers would be of little consequence. Moreover, it appears unnecessary to use an overpack that need survive only a dry environment, since the waste form itself can provide containment in a dry situation. The metallic barrier is intended to provide containment during the thermal pulse period and to assure that the waste form does not contact repository fluids during the period of relatively high temperatures. The eventual licensing arguments may rest heavily on limited water accessibility as an element of safety. With salt, tuff, and basalt being considered as repositories, the existence of water cannot be discounted. Accordingly, the MCC is working to establish a reference data base for the corrosion of alloys in repository-relevant aqueous environments.

The review of published corrosion data was limited to data applicable to the corrosion of barriers in salt, basalt, and tuff sites. There is, in one sense, a plethora of corrosion data on metallic materials available; however, it is the specificity of corrosion processes, in fact, that has necessitated this "wealth" of data. Corrosion data taken under one set of conditions (e.g., solution chemistry and temperature) are not easily applied to other

conditions, such as might be expected in a repository. Corrosion science has advanced sufficiently to give general appreciation of the effects of test conditions, but quantitative predictions are not possible for parameters such as pH, Eh, and radiation. With serious reservations as to specific applicability of data, the MCC has reviewed approximately 200 corrosion publications identified by a National Bureau of Standards bibliographic search (Sanderson and Kruger 1982) and many other publications identified by an independent MCC review, including corrosion studies completed and published by Nuclear Waste Terminal Storage programs and other Department of Energy (DOE) contractors.

The report is not intended to be an exhaustive presentation of theories of corrosion or a presentation of data on all materials that might be considered. The DOE-funded waste programs have sufficiently narrowed choices of materials to permit focus on the quality of data available as well as the types of data. The selection of references cited in this report was based primarily on whether the reference contained numerical data or not. Hence, theories of corrosion and corrosion mechanisms are not discussed extensively.

Another purpose of this report was to assess the quality of the available published data. The uncertainties associated with published values for corrosion rate were not typically reported. When screening materials is the purpose of testing, accuracy, as defined by ASTM (ASTM-E177 1971), may have marginal utility in light of the cost of testing because good repeatability may be adequate for selecting one alloy over another when one experimenter is testing. But for performance prediction, accuracy of data has direct relevance to licensing criteria, and a measure of the quality of data, as expressed by precision and accuracy statements, is needed. Ultimately, confidence in material performance must be established by quantitative measures of penetration caused possibly by uniform corrosion, pitting, or intergranular corrosion. In addition, metal must be tested in a sufficiently sensitive and repository-relevant way for susceptibility to stress corrosion cracking, crevice corrosion, and hydrogen effects to ensure that these corrosion modes do not cause premature failure.

2.0 CORROSION TESTING

The selection of materials and prediction of metallic barrier integrity over the desired lifetime of the waste package will require data from many kinds of corrosion tests. The identification of corrosion failure modes is a key element in current engineering and scientific application of laboratory data to real service conditions. Laboratory test methods are based on controlling significant test parameters in a way that relates to service and on revealing the extent of degradation due to the various corrosion modes. Screening tests are used to make an initial selection of materials. Data from screening tests are usually only useful for selecting one or two good candidate materials from a list of many. As material selection is narrowed, testing can become more thorough and relevant to qualifying one or two materials.

Corrosion screening tests for alloy development are typically oversevere, e.g., a boiling MgCl_2 stress corrosion test, because such tests reveal in a short time the relative effects of alloy content, processing, and heat treatment. Such severity, unless based on well-known mechanistic effects, cannot be utilized to predict behavior in more benign, real environments. Similarly, use of high radiation fields in oversevere tests may be appropriate for screening. The greatest value of oversevere tests in barrier corrosion testing will be to show absence of any failure or indications of shortened lifetime. Oversevere conditions such as high temperature may be useful in accelerating uniform corrosion to give increased confidence in data at lower temperatures.

2.1 LABORATORY TEST METHODS

The corrosion modes of metal alloys were identified in a MCC workshop (Merz et al. 1981), and the methods for obtaining data were prioritized by the participants with respect to barrier performance. The workshop report did not develop detailed testing methods to the extent that investigators can perform a test and compare data or to the extent that repository-relevant tests can be performed. Only the types of data were identified. Table 2.1 summarizes the data of highest priority for each corrosion mode. The reader is referred to the excellent book by Ailor (1971) for many details of practical problems of corrosion testing and test techniques.

TABLE 2.1. Data of Highest Priority for Important Corrosion Modes

<u>Corrosion Mode</u>	<u>Data</u>
uniform corrosion	<ul style="list-style-type: none"> ● penetration from weight change measurement
pitting	<ul style="list-style-type: none"> ● metallographic examination for pitting ● maximum pit depth, average number of pits/unit area, test on as large a specimen as practicable
crevice corrosion	<ul style="list-style-type: none"> ● metallographic examination for significant penetration or pitting with contrived-crevice specimen
intergranular	<ul style="list-style-type: none"> ● metallographic examination for significant penetration
stress corrosion	<ul style="list-style-type: none"> ● examination for cracking in U-bend specimen ● threshold stress intensity for cracking ● evidence of susceptibility in rising-load or cyclic-precracked test
hydrogen effects	<ul style="list-style-type: none"> ● notched or precracked specimen response to static or slow-rate loading ● crack growth rate under imposed stress intensity factor

2.1.1 Uniform Corrosion

The conversion of metal to an oxide, to dissolved metal ions, or to other corrosion products can result in uniform wastage of the metal barrier. Failure occurs when either the metal is penetrated or the thickness is sufficiently diminished that mechanical strength is compromised. Gravimetric methods are considered most useful to evaluate the mode of attack. The specimen is weighed before and after exposure (with appropriate corrosion-product stripping or accounting for film weight) and the weight change is converted to penetration by a calculation involving metal density, and, if appropriate, stoichiometry of corrosion products that remain attached to the metal, e.g., TiO_2 film on Ti.

Most of the data on uniform corrosion cited herein were obtained by the gravimetric technique. In almost all cases, weight changes were converted to

penetration rate in mm/yr to make comparisons of data easier. Copson (1955), in summarizing a 20-year testing program on atmospheric zinc corrosion, stated that this technique was the best method for evaluating the corrosion. Indeed, the technique persists as the basis for evaluating other techniques and is the most common technique in ASTM test methods. Other methods that can be used are polarization resistance, interference techniques, ellipsometry, metallographic examination, solution analysis, surface analysis, and AC impedance. Metallographic analysis is necessary to qualify the results of gravimetric data, particularly if temperature increases, radiation fields, and pH are used to accelerate corrosion rates.

2.1.2 Localized Corrosion

Various test and material conditions can result in localized attack such as crevice corrosion, intergranular corrosion, and pitting. Crevice corrosion, which is caused by local changes in solution chemistry such as pH decreases or differential oxygen cells, is observed in many metals such as stainless steels, titanium alloys, and iron and steels. Crevice corrosion usually causes pitting and is characterized by attack of small areas which can result in deep penetrations that represent a small areal fraction of the surface. Such pitting is characteristic of cast iron in soils, for example, and some stainless steels in seawater. Intergranular corrosion is characterized by preferential dissolution or penetration at grain boundaries often promoted by a difference in the composition of the boundary region compared with grain interiors.

The assessment of localized corrosion is varied. For pitting, statistical analyses have been proposed for giving probabilities of failure based on measurements of the depth of pits, frequency of pits, and area of specimen exposed (Aziz 1956; Hawn 1977). Intergranular corrosion is a mode that is best avoided since extrapolation of test data to longer times has no good basis. If observed, it may be discounted by measurement of very small penetration or minimized by alloy selection or thermal treatments. Crevice corrosion is a mode that might be extrapolated in time provided the attack produces pitting, e.g., as under rust on iron. Oversevere tests which result in no evidence of crevice corrosion are used to predict satisfactory performance.

Contrived crevices, such as Teflon® pressed against titanium or plates bolted against plates, are examples of specimens to demonstrate absence of susceptibility to crevice corrosion.

2.1.3 Stress Corrosion Cracking

Stress corrosion cracking is failure brought on by the simultaneous action of tensile stress and a specific corrosive environment. Under cyclic loading, cracking is designated as corrosion fatigue. Stresses in barriers such as canisters or overpacks are unavoidable to some extent because of thermal stresses produced when filling canisters (Simonen 1979), inadvertent residual stress due to handling, stresses produced by joining techniques used in closure, and possibly some deformation caused by hydrostatic and lithostatic loading in the repository. The sources of stress can be minimized by design, handling procedures, and process techniques, but there will always need to be some assessment of a barrier material's susceptibility to stress corrosion cracking. An extensive assessment of hydrogen embrittlement in titanium and ferrous alloys for barrier applications was performed by Archbold and Polonos (1981) and Archbold, Bower, and Polonos (1982). Good engineering practice will minimize or eliminate this susceptibility when possible.

Stress corrosion cracking susceptibility is measured by several methods varying from screening tests such as U-bend, C-ring, and CERT (constant extension rate test) or SSRT (slow strain rate technique) tests to crack growth rate tests with fracture-mechanics-type specimens. There is no one accepted test for susceptibility. Specimens for U-bend, C-ring, and CERT tests are smooth initially and cracks must initiate during the test. In the U-bend and C-ring tests the specimen is under a static load. In the CERT or SSRT test, the specimen is slowly strained and changes in elongation or reduction of area are used to indicate susceptibility by comparing the results in and out of the solution of interest. The CERT specimen is forced to fracture by the nature of the test; a decrease in the strain to failure indicates susceptibility.

A more quantitative assessment of crack growth has been attempted by determination of the stress intensity factor K below which cracks appear not to grow. In statically loaded, precracked specimens the threshold K is called

K_{ISCC} . Opinion varies on how K_{ISCC} can best be measured. When a cyclic load is imposed, crack growth is usually dependent on the ΔK imposed during the test, and a threshold ΔK is sought below which cyclic load does not produce crack growth. The value of ΔK_{Th} is always less than K_{ISCC} ; therefore, using a cyclic-load component provides a more conservative threshold than static loads.

2.1.4 Galvanic Corrosion

Contact between unlike metals can increase the corrosion rate of the anodic metal greatly, especially when the ratio of cathodic-metal area to anodic-metal area is large. Galvanic effects often can be avoided by design. The galvanic series available in several references (Uhlig 1971, p. 31; LaQue 1948, p. 416; Sedriks 1979, p. 206) is useful in avoiding galvanic corrosion. Galvanic coupling also can contribute to localized corrosion and stress corrosion cracking. Tylecote (1980) points out that a copper overpack over lead may cause galvanic, preferential, anodic corrosion of the lead once the copper is penetrated. Other data (Hall 1977) indicate that 304 SS corrodes four times faster when coupled to titanium with an area ratio Ti/304 SS = 10/1.

2.2 SIGNIFICANT TEST PARAMETERS

Parameters that are important in barrier corrosion testing include temperature, flow rate, pH, Eh, solution chemistry, and radiation.

2.2.1 Temperature

Because of the expected waste form heat generation, the temperature at the barrier-backfill interface will undoubtedly be above 100°C and possibly as high as 250°C at the boundary between the metal barrier and the backfill interface where corrosion will be occurring. As a general trend, the temperature will decrease with time. Because corrosion rate is very temperature dependent and generally increases with temperature, data at the highest likely temperatures are desirable. Moreover, temperature is the most practical way to accelerate certain corrosion processes to gain confidence for extrapolation of data to times beyond practical test durations. Temperature control to within 10°C of a stated temperature is probably adequate and achievable in most laboratory apparatuses for periods to 1 to 2 years. Power outages and gross

equipment failure are the only circumstances that normally prevent such control. The trend of corrosion rates of titanium and mild steel in Brine A, a synthetic brine, indicate that a variation of 10 to 20°C can cause measurable changes in corrosion rate (Table 2.2).

Estimates could possibly be made for other materials and environments, but the examples in Table 2.2 sufficiently illustrate the need for temperature control in corrosion rate measurements. Such uncertainties in temperature translate directly to uncertainties in corrosion rate. Thus, it can be appreciated that temperature control and data obtained at various relevant temperatures are necessary.

2.2.2 Flow Rate

The sensitivity of corrosion rate to solution flow rate varies from alloy to alloy and arises primarily from the dependence of reactant transport to the metal or corrosion product removal from the metal. There is no evidence that the velocity of solution conceivable in a repository has any significant effect on uniform corrosion aside from transport of species to and from the corroding metal. Maintaining a fairly high flow rate, e.g., one exchange of liquid volume in the vessel per day, has the apparent advantage of environment control (oxygen, pH, and constant solution composition) with attendant understanding of the effect on corrosion rate.

TABLE 2.2. Ratio of Uniform Corrosion Rates for Indicated Temperature Variation

<u>Material</u>	<u>40-60°C</u>	<u>45-55°C</u>	<u>180-200°C</u>	<u>190-200°C</u>
TiCode-12(a, c) in Brine A	2	1.4	1.4	1.2
Mild steel(b) in Brine A	1.3	1.1	1.1	1.06

(a) Based on data with activation energy = 7000 cal/mole.

(b) Based on data with activation energy = 2850 cal/mole.

(c) The use of proprietary, registered trademarks such as "TiCode-12" of TIMET Corp. does not imply endorsement of the company or alloy by Pacific Northwest Laboratory. Other such alloy designators used in this report are given in the Appendix for convenience.

In more complicated tests, where rocks or backfill are introduced to simulate the repository environment, mass transport within the test system may possibly lead to erroneous conclusions unless the mechanisms can be identified. For example, corrosion specimens in a flowing-autoclave test are covered by a protective scale when the water passes over crushed basalt as a "simulation" pretreatment.

The necessity for identifying the origin of the deposited material and the relevance of the mechanism to behavior in more nearly static repository conditions has great bearing on whether such tests give conservative corrosion data. Bounding the repository conditions by performing static immersion corrosion tests seems appropriate. The results from flowing and static tests will be relatable to one another through modeling of transport and corrosion mechanisms. For stress corrosion cracking in steels, there is some evidence that increased flow rates can decrease susceptibility (Choi et al. 1982).

2.2.3 pH and Eh

The pH and Eh of aqueous solutions have decided effects on the stability of metals relative to oxides, hydroxides, and other corrosion products. The effects can be understood in a thermodynamic sense from potential-pH equilibrium diagrams, or Pourbaix diagrams, for unalloyed iron and titanium (Figure 2.1). Although no kinetic information is contained in these diagrams, the appearance of certain phases bears a relation to corrosion and passivity (low corrosion rate) for these two metals. Charlot and Westerman (1981) discuss certain details of iron behavior with respect to oxygen concentration (which is related to Eh). Increased oxygen can decrease the uniform corrosion of carbon steel, for instance, in high purity water but may cause localized corrosion in water with significant impurities. The effect of pH on iron corrosion is well known (Uhlig 1971), and decreasing the pH to less than 4 causes a large increase in corrosion rate. Similarly, low pH, below about pH = 1, causes rapid corrosion of titanium in chloride solutions.

The application of potential-pH equilibrium diagrams to predict corrosion and passivity must be cautiously employed. In general, the corrosion behavior can be mapped on the diagrams (Figures 2.2 and 2.3). As pointed out by

FIGURE 2.1. Potential-pH Diagrams for Iron and Titanium at 25°C
(a) Reduction of water to hydrogen (b) Oxidation of water to oxygen

FIGURE 2.2. Potential-pH Diagram for Titanium at 25°C Showing Regions of Corrosion and Passivation

FIGURE 2.3a-c. Theoretical Conditions of Corrosion, Immunity, and Passivity for Iron with Nonprotective Fe_3O_4 and Protective Ferric Oxides (a) Aged Geothite $\alpha\text{-FeOOH}$ (b) Fresh Geothite $\alpha\text{-FeOOH}$ (c) Lepidocrocite $\gamma\text{-FeOOH}$

Pourbaix (1976) the theoretical boundaries between passivity and corrosion vary with the assumption of which oxides and oxyhydroxides of iron are protective (Figure 2.3). Pourbaix suggests that corrosion of iron may proceed from behavior initially governed by Figure 2.3c to behavior shown in Figure 2.3a. An indication of actual corrosion rates for iron is given in Figure 2.4 (Pourbaix 1966).

2.2.4 Solution Chemistry

The composition of test solutions is an important parameter in corrosion testing because the presence of minor species in low-ionic-strength water can affect passive film formation and the tendency to pitting and stress corrosion cracking. The high chloride content of seawater and brines can prevent passive behavior.

Solutions appropriate for corrosion testing for various sites will probably be based on groundwaters sampled at the site and/or similar waters that have been modified to simulate interactions that can occur near the waste packages. A number of solutions typical of the sites are given in Table 2.3.

FIGURE 2.4. Actual Corrosion Behavior of Iron Shown on Potential-pH Diagram (Pourbaix 1966)

The composition, E_h , and pH of groundwaters and possible intrusion solutions in the repository sites are of great interest and have been suggested by investigators studying potential sites. There has been no acceptance of a particular composition as representative of a site, although certain compositions of waters are being used in testing. The Waste Isolation Pilot Plant (WIPP) Brines A and B were adopted by the Sandia workers for corrosion testing for bedded salt (Braithwaite and Molecke 1978). The composition given here for tuff water is based on J-13 well water analyses and is a reference composition adopted by Los Alamos investigators for sorption experiments for the Nevada Test Site (Wolfsberg et al. 1979). This composition is not based on a selected repository horizon. The composition of Grande Ronde Basalt water was suggested as a reference water for the Basalt Waste Isolation Project (Wood 1980).

Oxygen concentration can markedly affect the various corrosion modes in most metals, especially iron and nickel-base alloys in aggressive environments such as brine and seawater, where corrosion rates increase with oxygen level.

TABLE 2.3. Composition of Groundwaters for Various Geologic Media

Component	Concentration (ppm)						
	Seawater (Braithwaite and Molecke 1978)	Tuff, Nevada (Wolfsberg et al. 1979)	GR Basalt, Hanford (Wood 1980)	Shale, Nevada (Doesch and Lynch 1979)	Granite, Sierra (Erdal et al. 1979)	Brine A, WIPP (Braithwaite and Molecke 1978)	Brine B, WIPP (Braithwaite and Molecke 1978)
Na ⁺	10,651	50.2	358	30	7	42,000	115,000
K ⁺	380	4.9	3.4	7	2	30,000	15
Mg ⁺²	1,272	2	0.032	25	2	35,000	10
Ca ⁺²	400	14.2	2.8	79	8	600	900
Sr ⁺²	13	0.045		0.4		5	15
Cl ⁻	18,980	6.4	312	13	2	190,000	175,000
SO ₄ ⁻²	884	18	172	62	3	3,500	3,500
I ⁻	0.05					10	10
HCO ₃ ⁻	146	130	55	280		700	10
Br ⁻	65					400	400
BO ₃ ⁻³						1,200	10
Ba		0.004					
Li		0.06					
H ₃ SiO ₄ ⁻			120				
F ⁻		2	33	0.6			
CO ₃ ⁻²			27				
Fe ⁺³		0.039		0.03			
Si		29-37		31	13		
NO ₃ ⁻		9					
pH (RT)	8.1	7.3-8.4	9.9	7.2	8	6.5	6.5

In high purity water, corrosion rate can increase with decreasing oxygen level at low oxygen levels and may exhibit the smallest corrosion rates at intermediate oxygen levels between 0.002 and 1 ppm (Weinstein 1982). Differential aeration can cause increased corrosion in metals that form passive films, such as iron and aluminum, but has little effect on other metals such as copper (Haynes and Baboian 1979). This effect may be aggravated by uneven contact of the barrier with the backfill. In a sealed repository, limited oxygen may limit corrosion for some metals. Data on oxygen effects, especially in the presence of radiation, must be developed.

2.2.5 Radiation

Radiation from the waste form will reach the region near the barrier-backfill interface and may affect corrosion processes by radiolysis of the liquid present. The radiation reaching this region will be primarily gamma radiation and the level may be as high as 10^5 to 10^6 R/h for commercial high level waste and decrease to less than 10^4 R/h by 100 years (Mendel 1977). Other wastes, i.e., defense high level waste, will probably have a significantly lower radiation level. The overpack (metal barrier) may be thick enough to attenuate the radiation significantly.

The effects of radiation on corrosion of the metal barrier are largely unknown and appear to differ considerably with metal and solution. The effect appears large for iron in brine but small for titanium in brine and small for iron in basalt water.

Radiolysis may have an effect on stress corrosion cracking; a study by Fujita, Akiyama, and Tamura (1981) has shown an increase in cracking of 304 SS when oxygen is present. Radiolysis can produce hydrogen which may react with ferritic steels and hydride-forming metals such as zirconium and titanium and cause delayed failure, a failure mode due to hydrogen intrusion into the metal.

3.0 CORROSION DATA ON METALS IN SALT, BRINE, AND SALINE WATER

The data presented in this section were selected to provide a review of corrosion rates and effects of test parameters on corrosion rates that have some relevance to salt and bedded salt deep geologic repositories. Data from tests in seawater and from shallow underground tests in salt water are given for completeness. These data indicate the extent of the understanding of the corrosion modes in Table 2.1. Much can be inferred from this variety of corrosion data to select additional tests to qualify metallic barrier materials.

The data were drawn from three broad categories:

- screening studies for metallic barriers for service in repositories
- screening studies for service in geothermal environments related to programs to extract heat from natural geothermal sources
- corrosion studies in salt water and concentrated chloride solutions related to near-surface service of metals

3.1 SCREENING STUDIES IN SALT, BRINE, AND SALINE WATER

Early studies by Kegley and Empson (1969) on 304L stainless steel, as part of Project Salt Vault, revealed stress corrosion cracking as an important failure mode for waste containers after emplacement. For stress corrosion cracking to occur in austenitic stainless steel, the earlier criteria of Marshall (1958) were cited:

- presence of tensile stresses
- aqueous liquid phase
- temperature greater than 50°C
- presence of Cl^- ions
- presence of oxygen.

Not surprisingly, a carbon steel heater suffered little loss of thickness under the relatively dry, limited water conditions of the testing. These early studies are difficult to interpret because of very high temperature excursions (~690°C) and other fluctuations such as heating cycle, oxygen, and moisture.

Later, more extensive screening studies by Clark et al. (1967) with a variety of environments in limited amounts of liquid gave the relatively low corrosion rates shown in Table 3.1. Tests of welded 304L in dry salt, 98% NaCl-2% H₂O, and 95% NaCl-2% H₂O showed corrosion rates less than 0.003 mm/yr for tests of 30, 60, 90, and 168 d at 300°C. Small additions of MgCl₂ raised the corrosion rate to 0.01 mm/yr under the same conditions. Other tests showed no reaction of 304L with Carlsbad Salt at 80°C, 225°C, and 600°C for times as long as 1000 h (Rankin 1980). Carbon steel was severely attacked.

Investigators at Sandia National Laboratories (Braithwaite 1978; Braithwaite and Magnani 1979, 1980; Braithwaite and Molecke 1980) have performed extensive screening tests on materials to identify candidate barrier materials for bedded salt, seawater, and dome salt. The corrosion studies were directed toward wet salt because it has been generally acknowledged that corrosion in dry salt and salt with small amounts of moisture does not

TABLE 3.1. Corrosion Rates, mm/yr, of Various Metals Under Conditions Simulating Salt Mine Storage, 30-d Duration (Clark et al. 1967)

Alloy	99.5% NaCl + 0.5% H ₂ O		99.75% NaCl + 0.25% MgCl ₂	
	200°C	300°C	200°C	300°C
304L SS	0.005 (.010)	0.013 (0.056)	0.018 (0.005)	0.02
Hastelloy C	0.0025	0.0025	0.0025	0.0025
Carbon Steel ASTM A108	0.033 (0.046)	0.038 (0.066)	0.069	0.089
Nickel	0.0025	0.0025	0.0076	0.015
1100 Aluminum	0.0051 (0.053)	wt gain (0.084)	wt gain	wt gain (0.091)
Ti 45 A	0.0025	0.0025	0.0051	0.0025

NOTE: Numbers in parentheses are maximum pitting penetration, mm. Tests were performed in autoclaves with specimens packed in various salts. Other materials reported were 310 SS, Haynes 25, Inconel, Monel, and 347 SS.

constitute a corrosion threat to most metals. One exception was 90 Cu-10 Ni alloy which was reported to corrode significantly, even in dry salt (Molecke et al. 1981). For the most part the laboratory corrosion testing for salt repositories has not been performed under "expected conditions" and has emphasized what is purported to be "overtesting," e.g., testing in saturated conditions and low oxygen for titanium alloys.

An extensive screening study was reported by Braithwaite and Molecke (1980) on corrosion in two synthetic brines and seawater (Table 3.2). The two brines, Brine A and Brine B (Table 3.3), were designed to simulate possible, though very improbable, intrusion liquids for bedded salt at the Waste Isolation Pilot Plant Site in New Mexico. Oxygenated tests were also conducted (Table 3.4). These early screening tests led to a more thorough testing of titanium alloys since this material was preferred over others (Abrego and Braithwaite 1980; Abrego and Rack 1981; Farrow et al. 1979; Braithwaite et al. 1980; Molecke et al. 1981; Molecke and Abrego 1980). For example, increased oxygen decreased the corrosion rate of titanium (Table 3.5), and the temperature dependence of corrosion rate was found to obey an Arrhenius equation (Table 3.6 and Figure 3.1). The corrosion rate of Ti-50A decreased with time dramatically, at least for the limited result of 30-d tests (Figure 3.2). Generally, it appears that TiCode-12 in Brine A, Brine B, and seawater will have a low uniform corrosion rate over long periods even if the total metal penetration, as derived from Figure 3.2, is repeated for each similar multiple of time if for some reason the TiO_2 film were disrupted. Molecke et al. (1981) reported results showing the sensitivity of corrosion rate to processing conditions (Figure 3.3).

Titanium readily forms a TiO_2 film under a wide range of Eh and pH conditions (Figure 2.1b in water). The details of such diagrams for high-ionic-strength solutions such as Brine A, Brine B, and seawater are unknown, but general characteristics can be inferred. Farrow et al. (1979) confirmed TiO_2 (rutile) formation by use of Raman spectroscopy and thus documented the formation of a stable oxide film in brine. Little data were reported under expected near-dry and oxidizing conditions or with backfill or materials expected to contact the waste canister or overpack. Such tests will be required on the selected alloy.

TABLE 3.2. Corrosion Rates of Candidate Alloys in Deoxygenated Solutions (250°C, P = 5 MPa, t = 28 d)
(Braithwaite and Molecke 1980)

<u>Alloy</u>	<u>Brine A (mm/yr)</u>	<u>Brine B (mm/yr)</u>	<u>Seawater (mm/yr)</u>
1018 Steel, 25°C	0.03	0.03	--
1018 Steel, 70°C	0.07	0.036	--
1018 Steel	1.7	0.07	0.4
Corten A Steel	0.9	0.05	0.2
2 1/4 Cr-1 Mo Steel	1.0 ^(a)	0.1 ^(a)	0.2
Lead	0.5	0.3	0.3
Copper	0.07	0.05	0.05
Naval Brass	1.0	--	1.0
90 Cu-10 Ni	0.14	--	0.07
304L SS	0.018	0.01	0.006
316L SS	0.015	--	0.005
Nitronic 50	0.018	--	0.003
20Cb3	0.007	--	0.005
Ebrite 26-1	0.016	--	0.005
Monel 400	0.03	--	0.1
Incoloy 825	0.006	--	0.004
Inconel 600	0.009	0.007	0.005
Inconel 625	0.005	0.001	0.012 ^(b)
Hastelloy C-276	0.007	--	0.0015
Zircaloy-2	0.001	--	--
Titanium C.P.	0.014	--	0.012
TiCode-12	0.003	--	0.001

(a) Crevice corrosion.

(b) Pitting corrosion.

The corrosion behavior of titanium alloys as inferred from electrochemical polarization curves was also reported, but no corrosion rates were derived from the curves (Braithwaite et al. 1980). Passive film formation and low corrosion currents at relatively high anodic potentials indicated the possibility of excellent corrosion resistance.

TABLE 3.3. Solution Compositions of Seawater, Brine A and Brine B

<u>Ion</u>	<u>Seawater, ppm</u>	<u>Brine A, ppm</u>	<u>Brine B, ppm</u>
Na ⁺	10,651	42,000	115,000
K ⁺	380	30,000	15
Mg ⁺²	2,272	35,000	10
Ca ⁺²	400	600	900
Sr ⁺²	13	5	15
Cl ⁻	18,980	190,000	175,000
SO ⁻²	884	3,500	3,500
I ⁻	- 05	10	10
HCO ₃ ⁻	146	700	10
Br ⁻	65	400	400
BO ₃ ⁻³	--	1,200	10
pH	8.1	6.5	6.5
<hr/>			
Total Dissolved Solids	35 g/l	306 g/l	297 g/l

TABLE 3.4. Corrosion Rates of Candidate Alloys in Oxygenated Solutions^(a)
(250°C, P = 7 MPa, t = 14 d) (Braithwaite and Molecke 1980)

<u>Alloy</u>	<u>Brine A, mm/yr</u>	<u>Seawater mm/yr</u>
1018 Steel	7.0	11.0
Copper	1.2	5.0
Lead	1.2	1.0
90 Cu-10 Ni	0.4	0.7
Ebrite 26-1	0.24	--
20Cb3	0.1 ^(b)	--
Inconel 600	--	0.1
Hastelloy C-276	0.06 ^(c)	0.2 ^(c)
TiCode-12	0.0004	0.0006

(a) (O₂) = 600 ppm in Brine A and 1750 ppm in seawater.

(b) Crevice corrosion.

(c) Pitting and crevice corrosion.

TABLE 3.5. Effect of Dissolved Oxygen on the Uniform Corrosion Rate of Titanium Alloys at 250°C in Brine and Seawater (30-d tests) (Molecke et al. 1981; Braithwaite and Molecke 1980; Braithwaite, Magnani and Munford 1980)

	Corrosion Rate, mm/yr				
	Brine A		Brine A	Seawater	
Oxygen Content:	30 ppm	450 ppm	600 ppm	30 ppb	500 ppm
<u>Alloy</u>					
Ti-50A	0.014	3.2	--	0.0117	0.0162
TiCode-12	0.0032	0.0018	0.0004	0.00110	0.00060
Ti-0.2 Pd	0.0024	0.0004	--	0.00114	0.000062

TABLE 3.6. Effect of Temperature on the Uniform Corrosion Rate of Titanium Alloys in Bedded Salt Brine A (O₂ ~30 ppb) (30-d tests) (same ref. as Table 3.5)

<u>Alloy</u>	<u>70°C</u>	<u>150°C</u>	<u>250°C</u>
Ti-50A	<0.00006	0.0026	0.014
TiCode-12	<0.00007	0.0009	0.0032
Ti-0.2 Pd	<0.00009	0.0003	0.0024

The corrosion evaluation of the superior titanium alloys such as TiCode-12 and Ti-0.2% Pd has included an evaluation of whether the materials are sensitive to crevice corrosion (Abrego and Braithwaite 1980) and stress corrosion (Abrego and Rack 1981). No evidence of crevice corrosion was found in TiCode-12 and Ti0.2% Pd up to 300°C in seawater, with and without oxygen. Stress corrosion testing (CERT tests) showed no indication of stress corrosion cracking for TiCode-12 in synthetic seawater, but at very low strain rates there was some indication of susceptibility in Brine A.

Data from a screening study in Brine A reported by Westerman 1980 are shown in Table 3.7. The corrosion data were obtained on U-bend specimens that were weighed before and after testing. In these tests, the solution was nearly static and oxidizing, although no replenishment of oxygen was deliberately provided.

FIGURE 3.1. Corrosion Rate of Ti-50A in Deoxygenated Seawater Versus Test Temperature, 14-d Test (after Molecke et al. 1981)

FIGURE 3.2. Corrosion Rate of Ti-50A in Deoxygenated Seawater Versus Time at 250°C (Corrosion rate is average rate over each time period) (after Molecke et al. 1981)

FIGURE 3.3. Corrosion Rate of TiCode-12 Versus HCl Molarity Before and After Sensitization Treatment for 30 min at 680°C (Molecke et al. 1981)

TABLE 3.7. Corrosion Rate of Samples in MgCl₂-NaCl Brine at 250°C, Brine A (after Westerman 1980)

Material	Rate, mm/yr	Duration, d
304L SS	0.0003	72
410 SS	0.023	72
Nickel 200	0.034	58
Inconel 600	0.0009	58
70 Cu-30 Ni	0.203	20
Copper	0.414	38
Hastelloy C-276	0.00042	58
TiCode-12	0.00019 ^(a)	58
Zircaloy-2	0.0022 ^(a)	58
Ductile cast iron	1.39 to 1.95	58

(a) Based on weight gain and assumption of TiO₂ or ZrO₂ formation

The results on some materials can be compared with the results of Braithwaite and Molecke in Table 3.8, even though in most cases Westerman tested for longer times, ~60 d versus 28 d. The specimens were placed together in one 6-liter autoclave, and undoubtedly the rapid iron and copper corrosion rates had a suppressing effect on other rates, e.g., compare Hastelloy C-276 rates. In once-through-flowing autoclave tests, such effects would not be as pronounced.

Only 400 series stainless steel specimens showed any stress corrosion cracking in these screening tests. An interesting observation on the two sets of data is that even though the test conditions were different, the materials were ranked the same except that 304L, Inconel 600, and Hastelloy C-276 were interchanged in their intermediate positions.

The temperature dependence of cast iron and cast steel can be estimated by plotting selected data and comparing them with data on 1020 steel (Braithwaite and Molecke 1980). In this case, the 16-year corrosion data of Southwell (1969) in seawater appears to match with the other oxic (high oxygen level) data (Figure 3.4) (Davis and Munir 1977), and the anoxic (low oxygen level) data conform to parallel lines. The composite graph can be used to estimate that corrosion rate is given by:

$$R = 2500 \exp [-2800/T] \text{ (oxic)}$$

$$R = 370 \exp [-2800/T] \text{ (anoxic)}$$

TABLE 3.8. Comparison of Corrosion Rates in Screening Tests in Brine A for Two Investigations at 250°C

<u>Material</u>	<u>Corrosion Rate, mm/yr</u>	
	<u>Westerman (1980)</u>	<u>Braithwaite and Molecke (1980)</u>
304L SS	0.0003	0.018
Inconel 600	0.0009	0.009
Copper	0.415	0.07
Hastelloy C-276	0.00042	0.007
TiCode-12	0.00019	0.003
Ductile cast iron	1.39 to 1.95	1.7(a)
<u>(a) 1018 steel.</u>

FIGURE 3.4. Corrosion Rates of Various Cast Irons and Steels in Brine and Seawater Versus Reciprocal Temperature (data from Braithwaite and Molecke 1980, Westerman 1980, Southwell 1969)

These equations and Figure 3.4 illustrate the consistency of various data on iron and iron-based materials.

The data in Table 3.9 illustrate the degree of agreement in corrosion rates reported by five investigators for seawater at various locales and for a range of cast irons of considerably different compositions and thermal histories. These measurements indicate that corrosion rates, as measured by weight loss, for tests in seawater are probably very reproducible.

TABLE 3.9. Corrosion Rates, mm/yr, for Cast Irons in Seawater
(after Angus 1978)

Reference:	Hudson (1950)	Kress (1953)	Paris and de la Bruniere (1957) Laboratory ^(a)	Speidel and Wittmoser (1960) Laboratory	LaQue (1959) Harbor Island USA
Location:	Emsworth UK	Cuxhaven Germany			
<u>Duration of Test:</u>	<u>2 yr</u>	<u>0.5 yr</u>	<u>1 yr</u>	<u>0.6 yr</u>	<u>3 yr</u>
Steel	--	0.10	0.11	0.084	--
Gray Iron	0.061	0.086	0.086	0.030	0.81 ^(b)
White Iron	--	--	--	0.043	--
Malleable Iron	0.079	--	--	0.043	--
Pearlitic Nodular Cast Iron	--	0.051	--	0.071	0.28 ^(b)
Ferritic Nodular Cast Iron	--	0.11	0.082	0.026	--

(a) Artificial Seawater

(b) Rate of pitting

The corrosion of copper in saline groundwater with 6500 ppm Cl was measured by King et al. 1981. Results were reported for temperatures of 75°C and 150°C (Tables 3.10 and 3.11).

3.2 GEOHERMAL BRINE STUDIES

Considerable corrosion data have been produced by corrosion engineers and scientists in support of geothermal energy development. Because of the high temperatures and ionic strength of the solutions, these data have relevance to barrier service. In particular, the results can be used to screen materials, to indicate corrosion modes, and to increase confidence in the resistance of metallic barriers to corrosion. Moreover, the data indicate major effects of

TABLE 3.10. Corrosion Rate of Copper in Saline Groundwater at 75°C (1/20 bentonite/water ratio; electrochemical measurement) (King et al. 1981)

<u>Time of Immersion, d</u>	<u>Corrosion Rate, mm/yr</u>	
	<u>Air-Saturated</u>	<u>Air-Free</u>
5	0.040	0.00095
8	0.075	0.00063
9	0.105	0.00050
10	0.100	0.00050
11	0.165	0.00055
12.5	0.165	--
15	0.200	0.00055

TABLE 3.11. Corrosion Rate of Copper in Saline Groundwater at 150°C (air-free) (King et al. 1981)

<u>Time of Immersion, d</u>	<u>Average Penetration, mm^(a)</u>	<u>Corrosion Rate, mm/yr</u>
<u>No bentonite</u>		
7	0.00010	0.0052
10	0.00026	0.0095
40	0.00035	0.0032
50	0.00036	0.0026
80	0.00046	0.0021
90	0.00060	0.0024
<u>With bentonite/water 1/20</u>		
20	0.00020	0.0036
50	0.00043	0.0031
100	0.00038	0.0014

(a) Pitting occurred and was as deep as 0.036 mm in some cases. The cause was unresolved, but was suspected to be due to crevice corrosion between touching specimens.

test parameters that were varied by the investigator. The results also increase confidence through agreement between field test and laboratory test data. As far as representing expected conditions in repositories, these tests must be considered as severe overtests in most cases, especially for iron-base material that corrode much faster as flow rate is increased. The flow rates in these tests are much higher than any anticipated condition in a geologic repository.

Data obtained by Davis and Munir (1977) were reported in terms of maximum corrosion rate at 95% confidence (Table 3.12). This confidence was based strictly on repeatability, but a similar approach in reporting barrier corrosion data would permit confidence statements to be given for barrier corrosion performance, provided enough replicate tests were conducted. Currently, there are no available data to warrant such statements for materials tested in simulated-repository environments, even for screening laboratory tests of short test durations. One feature of 304 stainless steel noted by Davis and Munir was the decreased corrosion rate at 95°C compared with 60 and 23°C, which they attributed to an active-passive transition. Scale formed at 95°C but not at 60 and 23°C. Similar, previously reported behavior was referenced by the authors. These tests were too short for any conclusion on pitting corrosion. The corrosion rate of AISI 1010 increased with time but the 0.75-d test duration was much too short to attach any significance to this observation.

A very extensive study of the corrosion of metals in geothermal brines was conducted by the Bureau of Mines (Needham et al. 1979; Cramer and Needham 1978; Banning and Oden 1973; Carter et al. 1979; Carter, Cramer, and Conrad 1980;

TABLE 3.12. Maximum Corrosion Rate, mm/yr, in Synthetic Salton Sea Brine, pH = 3, 8 to 10 ppm oxygen, 95% Confidence Level (Davis and Munir 1977)

<u>Material</u>	<u>95°C</u>	<u>60°C</u>	<u>23°C</u>	<u>Test Duration, d</u>
AISI 1010 Steel	1.1	0.69	0.32	0.75
304 SS	0.0507	0.2446	0.0842	2.5
316 SS	0.0449	0.0421	0.0393	2.0
Monel 400	0.0897	0.0481	0.0228	--
Titanium	0.00129	--	--	7.0

Carter and Cramer 1980; Cramer and Carter 1981; Cramer and Carter 1980; McCawley et al. 1981; Carter, Cramer, and Conrad 1981) and others (Shannon 1977; Shannon 1978; Shannon, Morrey, and Smith 1977). Extensive field and laboratory testing was conducted. These tests were very severe compared with any expected conditions in repositories and thus the data cannot easily be related directly to barrier performance, although in some cases such as titanium the corrosion rates were very low.

In the laboratory tests, an extensive screening study for uniform corrosion, crevice corrosion, pitting, weld corrosion, and stress corrosion was reported (Cramer and Carter 1980). The data are given in Tables 3.13 through 3.16. In Table 3.14, stress corrosion cracking is shown to occur in many of the alloys, especially with 100 ppm O_2 . Ti-0.2Pd, Ti-2Ni, 1020 steel, and E-Brite 26-1 resisted cracking in these U-bend tests. The 316 SS autoclave failed due to stress corrosion cracking in the 100 ppm O_2 brine solution after 7 d. For low oxygen, a number of materials have low uniform corrosion rates (Table 3.13), although carbon steel corroded rapidly in deaerated brine at 232°C; again it must be pointed out that the flow rate is rather severe in these tests. Titanium alloys generally corroded very little.

Results on weld corrosion (Table 3.17) indicated almost no effect for the deaerated brines, but corrosion rates increased by a factor of about 4 for the oxygenated brine as a result of localized corrosion near the weld. Stress corrosion cracks appeared across the welded 316L stainless steel in the oxygenated brine. The effect of pH on the corrosion rate of 1020 steel was also studied; the corrosion rate increased sharply for pH less than 3.0 (Figure 3.5).

In field studies, the effect of a steam environment as opposed to a liquid environment was indicated to be significant for iron and stainless steel for field corrosion tests in geothermal brines (Tables 3.17 and 3.18). Corrosion rates were approximately a factor of 2 greater for steam in some cases but relatively unaffected or smaller in others. No differences were observed in U-bend stress corrosion cracking tests (Table 3.19). The 1020 steel and the steel and the nickel-base alloys were resistant to U-bend stress corrosion cracking in these field tests, whereas some of the nickel-base alloys cracked in laboratory tests (Table 3.14).

TABLE 3.13. Corrosion Rate, mm/yr,^(a) in Salton Sea KGRA-Type Brine
(Cramer and Carter 1980)

	105°C - 15 d pH = 6.1 Cl = 140,000 ppm		232°C - 15 d pH = 5 Cl = 148,000 ppm	
	<u>Deaerated</u>	<u>Aerated</u>	<u>Deaerated</u>	<u>Aerated (100 ppm)</u>
<u>Iron Base</u>				
1020 carbon steel	0.106	0.147	0.419	26.92
E-Brite 26-1	0.000	0.0914	0.0025	0.508
316 SS	0.000	0.102	0.000	4.06
<u>Nickel Base</u>				
Monel 400	0.071	0.094	0.500	7.37
Inconel 625	0.000	0.000	0.000	0.488
Hastelloy C-276	0.000	0.000	0.000	0.150
<u>Copper Base</u>				
Copper	0.079	0.318	--	--
70 Cu-30 Ni	0.015	0.147	0.239	5.72
<u>Titanium Base</u>				
Ti-50A	0.000	0.000	0.005	0.025
Ti-2Ni	0.000	0.000	0.003	0.000
TiCode-12	--	--	0.0	0.0
<u>Aluminum Base</u>				
2024-T3	0.033	0.886(b)	--	--
6061-T6	0.772(b)	1.438(b)	--	--

(a) Reported to nearest 0.1 mil/yr, converted to mm/yr for this report.

(b) Also 1.5 mm pits.

TABLE 3.14. Crevice Corrosion and Stress Corrosion in Salton Sea KGRA-Type Brine at 232°C (Cramer and Carter 1980)

Crevice Susceptibility

0 = None

1 = slight (<0.003 mm/yr)

2 = moderate (0.003 to 0.015 mm/yr)

3 = Severe (>0.015 mm/yr)

Material	Deaerated 15 d	100 ppm O ₂ 7 d	Deaerated 15 d	100 ppm O ₂ 7 d
1020 Steel	0	0	none	--
E-Brite 26-1	0	3	no cracking	no cracking
316L SS	1	3	cracking	cracking
Monel 400	0	0	no cracking	--
Inconel 625	2	3	cracking	cracking
Hastelloy C-276	0	3	no cracking	cracking
70 Cu-30 Ni	0	3	no cracking	--
Ti-50A	3	0	no cracking	cracking
Ti-2Ni	2	0	no cracking	--
Ti-0.2Pd	0	0	no cracking	no cracking
TiCode-12	-	0	--	--

TABLE 3.15. Pitting Corrosion, mm/yr, in Salton Sea KGRA-Type Brine at 232°C (Cramer and Carter 1980)

Material	Deaerated 15 d	100 ppm O ₂ 7 d
1020 Steel	1.7	detected
E-Brite 26-1	not detected	243
316L	not detected	detected
Monel 400	detected	detected
Inconel 625	not detected	13.5
Hastelloy C-276	not detected	16.3
70 Cu-30 Ni	not detected	detected
Ti-50A	5.46	detected
Ti-2Ni	detected	not detected
Ti-0.2Pd	detected	not detected
TiCode-12	--	not detected

TABLE 3.16. Weld Corrosion in Salton Sea KGRA-Type Brine at 232°C - 15-d Exposure (Cramer and Carter 1980)

Alloy	Corrosion Rate, mm/yr			
	Deaerated		100 ppm Oxygen	
	Weld	Nonweld	Weld	Nonweld
1020 Steel	0.391	0.491	29.97	26.92
316L SS	0.005	0.000	3.683	4.064
E-Brite 26-1	0.000	0.003	3.683	0.531
Inconel 625	0.003	0.000	2.209	0.488
Ti-50A	0.071	0.005	0.000	0.000
Ti-0.2Pd	0.010	0.000	0.000	0.000

NOTE: Most values reported to nearest 0.1 mil/yr.

TABLE 3.17. General Corrosion Rates, mm/yr, in MESA 6-1 Steam and Liquid Brine for 22-d Exposures (McCawley et al. 1981)

Material	Liquid 152°C(a) Brine pH = 5.2	Steam(b) 140°C pH = 5.2
1020 Steel	0.29	0.422
E-Brite 26-1	0.249	0.041
Ti-2 Ni	<0.003	<0.003
Hastelloy C-276	<0.003	<0.003
Hastelloy S	<0.003	<0.003
Inconel 625	<0.003	<0.003

NOTE: None of U-bends (all materials) exposed for 22 d in steam had cracks. 316L and Inconel 625 had pits, but the depths were too shallow to measure by optical microscopy. The brine was presumably a low-oxygen solution, though oxygen level was not given.

(a) 11000 ppm Cl⁻.

(b) 3000-5000 ppm Cl⁻.

The results in Table 3.20 were cited in this report to give an appreciation for the small variation in corrosion rate for various carbon steels. Even though carbon varied from 0.10 to 0.44 wt%, and other minor compositional differences existed, the corrosion rates varied little for a given solution.

FIGURE 3.5. Corrosion Rate Versus pH for 1020 Carbon Steel (Cramer and Carter 1980)

TABLE 3.18 . Maximum and Average Pit Penetration in Salton Sea KGRA Brine and Brine-Derived Steam at 207°C. Flow rate, 170 l/min. (Brine, 123,000 ppm Cl⁻; pH = 5.7; steam, 8500 ppm Cl⁻, pH = 5.9) (Carter, Cramer, and Conrad 1981)

Material	15-d Test		30-d Test		45-d Test	
	Rate (a)	Factor (b)	Rate (a)	Factor (b)	Rate (a)	Factor (b)
E-Brite 26-1:						
Brine	7.7/2.7/0.018	430	3.8/1.5/0.031	120	1.1/0.61/0.010	110
Steam	7.4/2.7/0.023	320	6.7/1.2/0.033	200	2.6/0.87/0.020	130
316L SS:						
Brine	8.2/3.1/0.084	98	3.0/1.1/0.064	47	1.7/0.62/0.020	85
Steam	5.6/2.6/0.053	110	3.7/1.9/0.048	77	1.8/1.3/0.048	38

(a) Rates are maximum pitting rate/average pitting rate/uniform corrosion rate.

(b) Factor is ratio of maximum pitting rate to uniform corrosion rate.

TABLE 3.19. Stress Corrosion Test Results, 30-d U-Bend Field Tests in Salton Sea KGRA Environments, 207°C (see Table 3.19 for conditions) (Carter, Cramer, and Conrad 1981)

<u>Alloy</u>	<u>Liquid Brine</u>	<u>Steam</u>
AISI 1020 Steel	no cracking	no cracking
AISI 430 SS	cracking	cracking
E-Brite 26-1	cracking	cracking
AISI 316L SS	cracking	cracking
Hastelloy G, C-276 and Inconel 625	no cracking	no cracking

NOTE: Results on nickel-base alloys should not be taken as conclusive evidence since laboratory tests, Table 3.12, showed cracking in some cases. (Cramer and Carter 1980)

TABLE 3.20. Corrosion Rate, mm/yr, of Various Steels in Brines Showing Effect of H₂S and Dissolved Silica (Shannon 1977)

Temperature: 150°C Pressure: 68.9 Bar
 25°C pH: 4.6 Oxygen: <0.01 ppm
 NaCl: 1%
 Flow Rate: 1 exchange/3.3 h

<u>Steel</u>	<u>Average Corrosion Rate, mm/yr (2-week exposure)</u>		
	<u>1% NaCl-CO₂</u>	<u>1% NaCl-CO₂ + H₂S 10 mg/l</u>	<u>1% NaCl-CO₂ + SiO₂ 400 mg/l</u>
A570	0.53	0.53	0.41
A53B	0.53	0.41	0.36
1010	0.38	0.28	0.36
A106	0.41	0.43	0.36
C75	0.51	0.28	0.38
C95	0.58	0.33	0.38
K55	0.58	0.28	0.41
L80	0.58	0.33	0.38
P110	0.58	0.30	0.41

For example, seven of the nine steels were within the range of 0.51 to 0.58 mm/yr for 1% NaCl, and within 0.28 to 0.41 for 1% NaCl + H₂S. All were within 0.36 to 0.41 mm/yr for 1% NaCl + 400 ppm SiO₂. Silica dissolved in the water apparently decreases the corrosion rate of iron. In these tests, where the corrosion film controlled metal wastage, the corrosion rates were apparently quite reproducible and controlled by transport through films rather than by the metal. These results confirm the assertion by Uhlig (1971) that "so long as oxygen diffusion is controlling, which is the case within pH 4 to 10, a small variation in composition of a steel and its heat treatment, or whether it is cold worked or annealed, has no bearing on corrosion properties. Oxygen concentration, temperature and velocity of the water alone determine the reaction rate."

Other data (Shannon 1977) indicated an increase in corrosion rate of carbon steels with Cl concentration (Table 3.21 and Figure 3.6) and the smaller increase for high chromium steels and nickel-base alloys. The corrosion rates of titanium alloys and zirconium were also low in related environments, as shown in Table 3.22. Figure 3.7 illustrates that the corrosion rate of carbon steels increased with decreasing pH. The corrosion rate of steel actually decreased with increasing temperature (Figure 3.8) as more-protective Fe₃O₄ films were formed at higher temperature. This trend was reversed, however, at very high salinity and 250°C. Tables 3.23 and 3.24 give a comparison between laboratory tests and field tests. Agreement of data was good except at 50°C where a different kind of silica film apparently formed in the field tests and caused lower corrosion rates (Table 3.23).

A study of the corrosion of A212B 0.35% carbon steel in 4M NaCl with pH 2 to 7 in a refreshed autoclave was reported by Posey and Palko (1979) and Posey, Palko, and Bacarella (1978) (Figures 3.9 and 3.10). These electrochemical measurements yielded corrosion rates that were generally consistent with rates reported elsewhere, e.g., compare with Figure 3.4 anoxic data. A thorough literature review of electrochemical measurements of iron corrosion was also provided (Posey et al. 1978).

TABLE 3.21. Uniform Corrosion Rate of Various Alloys in Geothermal Brine (Shannon 1977)

Temperature: 250°C
 Pressure: 68.9 Bar (1000 psi)
 Oxygen: <0.01 ppm

pH: 4.6 to 4.8
 Flow Rate: 1 exchange/3.3 h

Alloy	Corrosion Rate, mm/yr			
	1% NaCl	5% NaCl	10% NaCl	20% NaCl
A570 Steel	0.3	0.9	1.1	2.8
A53B Steel	0.3	0.6	0.9	2.8
C75 Steel	0.2	0.4	0.2	2.0
1010 Steel	0.4	1.0	1.0	3.5
2-1/4 Cr-1 Mo Steel	0.08	0.6	0.3	1.8
410 SS	0.08	0.01	0.08	3.5
E-Brite 26-1 Heat 1	0.05	0.01	0.02	0.08
E-Brite 26-1 Heat 2	--	0.02	0.02	0.02
Hastelloy C-276	0.02	0.03	0.04	0.04
Inconel 625	0.01	0.01	0.01	0.02
Inconel 600	0.02	0.02	0.05	0.04
Incoloy 825	0.01	0.01	0.02	0.05
6X	0.01	0.01	0.01	0.03

In an electrochemical study of pitting in low-salinity geothermal brine with ~9700 ppm [Na, Ca, K] Cl, Syrett, Macdonald, and Shih (1980) reported no pitting of TiCode-12 and Titanium 50A for tests as long as 42 days at 200°C. Pitting could not be produced even by electrochemical methods for these two alloys.

3.3 RADIATION CORROSION STUDIES IN BRINE AND SEAWATER

Only a small number of investigators have measured corrosion in a radiation field and results have indicated that radiation can accelerate attack in some cases. An early theoretical consideration of radiation effects in salt brines by Jenks (1972) concluded that the important radiolysis products were H₂, O₂, and possibly ClO₃⁻ and BrO₃⁻. Hydrolysis of MgCl₂ in brine was

FIGURE 3.6. Average Corrosion Rate of Carbon Steels Versus Salinity (Shannon 1977)

postulated to produce HCl. The analyses were very detailed, and the conclusions were tied to estimates of amounts of brine, temperatures, and power (heating rate) of the canister. The treatment did not attempt any analysis of transient species on corrosion rate per se and dealt with "stable" species. A revealing comparison was made between the amounts of H_2 produced by radiolysis of brine around the canister (~ 0.8 mole/yr) and by an assumed corrosion rate of 1.3 mm/yr of the canister (400 mole/yr). Thus, the amount of radiolytically produced hydrogen was predicted to be small compared with corrosion-produced hydrogen. However, if corrosion rates are much less than 1.3 mm/yr, the amounts of hydrogen produced by radiolysis become relatively more significant.

Molecke et al. (1981) reported increases in corrosion rate by a factor of 2000 for 304L SS in Brine A in going from 10^5 to 10^7 R/h ^{60}Co radiation at 90°C (Table 3.25). A factor of 10 increase was found for 1018 steel. An estimate of the effect of radiation for TiCode-12 from Table 3.6 and

TABLE 3.22. Corrosion Rate, mm/yr, of Alternate Alloys and Carbon Steels in Oxygen-Free Brines - 68.9 Bar (Shannon 1977)

	250°C, 1% NaCl pH 4.8, H ₂ S 10 mg/l	250°C, 20% NaCl pH 4.6
Average of ten carbon steels	0.18	2.2
<u>Chromium Steels</u>		
2-1/4 Cr-1 Mo Steel	0.08	0.48
E-Brite 26-1	0.02	0.02
410 SS	0.04	3.0
430 SS	0.04	1.6
446 SS	0.02	0.02
29 Cr-4 Mo Steel	0.02	0.02
6X	0.003	0.01
<u>Nickel Alloys</u>		
Hastelloy C-276	0.04	0.01
Inconel 625	0.005	0.003
Inconel 600	0.23	0.01
Inconel 825	0.005	0.008
<u>Titanium Alloys</u>		
Ti35A	<0.005	<0.005
Ti1.5 Ni	<0.005	<0.005
Ti0.2 Pd	<0.005	<0.005
Ti6Al 4V	<0.005	<0.005
<u>Zirconium</u>	<0.005	<0.005

Table 3.25 gives an increase by a factor of ~14 in going from no radiation to 10^7 R/h ^{60}Co . An unusual observation of an increase in corrosion rate with test duration was also reported for TiCode-12 in Brine A and Inconel 600 in seawater (Table 3.25). This result needs to be confirmed by additional testing because of the significance of increasing corrosion rate with time. A field of 10^7 R/h is at least one order of magnitude higher than expected in commercial waste levels and probably three orders of magnitude greater than the maximum level expected for defense waste. Thus, these tests must be considered as very severe with respect to radiation.

FIGURE 3.7. Average Corrosion of Ten Carbon Steels in NaCl-CO₂ Brines Versus pH (Shannon 1977)

FIGURE 3.8. Average Corrosion of Ten Carbon Steels in CO₂ Bicarborate Brines (Shannon 1977)

TABLE 3.23. Comparison of Corrosion in Refreshed Autoclave Tests with Corrosion in Actual Geothermal Brines (Shannon 1977)

Corrosion Rate in mm/yr (2-week exposure)				
Alloy	150°C		50°C	
	Autoclave Test (a)	Geothermal Brine (b)	Autoclave Test (a)	Geothermal Brine (b)
1010 Steel	0.11	0.15	0.36	0.042
A53B Steel	0.12	0.14	0.46	0.045
4130 Steel	0.17	0.17	0.32	0.088
410 SS	0.04	0.03	0.015	0.03
E-Brite 26-1	0.003	0.003	0.0067	0.0076

- (a) Synthetic Brine: pH 5.6-5.8; NaCl-24,000 mg/liter; SiO₂-320 mg/liter; H₂S-1 mg/liter; O₂ <0.01 ppm.
 (b) East Mesa Well 6-1: pH 5.6-5.8; Salinity-22,000 mg/liter; SiO₂-300 mg/liter; H₂S-1.3 mg/liter; O₂ <0.01 ppm.

TABLE 3.24. Effect of Acidification on Corrosion in Refreshed Autoclave Tests and Actual Geothermal Brine (Shannon 1977)

Alloy	Corrosion Rate, mm/yr, in East Mesa 6-1 Geothermal Brine 150°C-1 ft/s		Corrosion Rate, mm/yr, in Refreshed Autoclave Tests 150°C - 1 exchange/3.3 h	
	Wellhead pH 5.6-5.8	Acidification pH 4.6-4.8	pH 5.6-5.8	pH 4.6-4.8
1010 Steel	0.15	0.64	0.11	0.41
A53B Steel	0.14	0.39	0.12	0.53
4130 Steel	0.17	0.56	0.47	--
410 SS	0.03	0.04	0.04	--
E-Brite 26-1	0.003	0.003	0.003	--

A review by Glass (1981) presented a perspective on the effect of radiation on corrosion of waste canisters for the Nevada Test Site, bedded salt, and subseabed disposal. No new data were presented, and the only corrosion data referenced were the earlier results reported by Braithwaite and Molecke. Glass cited a factor of 2 increase in corrosion rate of TiCode-12, which is

FIGURE 3.9. Corrosion Rate, mm/yr, Versus Reciprocal Temperature for 0.35 wt% Carbon Steel in 4M NaCl Brine for Various pH values

lower than the factor of approximately 14 from interpolation of the data in Table 3.6 and the value of 2×10^{-3} mm/yr at 90°C in Table 3.25. There appears to be insufficient data to quantify the radiation effect on TiCode-12 corrosion in brine, although the increase does not appear to be large for anticipated radiation levels in defense waste.

FIGURE 3.10. Corrosion Rate, mm/yr, Versus pH for Various Temperatures for 0.35 wt% Carbon Steel in 4M NaCl Brine

TABLE 3.25. Corrosion Rate of Various Alloys in ^{60}Co Radiation in Brine A, Brine B, and Seawater at 90°C (Molecke et al. 1981)

<u>Alloy</u>	<u>Dose Rate, R/h</u>	<u>Solution</u>	<u>Test Duration, d</u>	<u>Corrosion Rate, mm/yr</u>
TiCode-12	10^7	Brine A	49	0.002
	10^7	Brine A	87	0.02
	10^7	Seawater	49	0.002
	10^7	Seawater	87	0.001
304L SS	10^7	Brine A	79	0.2
	10^5	Brine A	49	0.0009
	10^5	Seawater	49	0.0007
1018 steel	10^7	Brine B	79	1
	10^7	Brine A	79	1
	10^5	Brine A	49	0.1
	10^5	Seawater	49	0.03
Inconel 625	10^7	Brine A	87	0.002
	10^7	Seawater	87	0.009
	10^7	Seawater	49	0.002

4.0 CORROSION DATA ON METALS IN LOW-IONIC-STRENGTH WATERS

The corrosion of metals in solutions such as Hanford groundwater, Grande Ronde groundwater, J-13 well water, and granitic waters, which are characteristic of potential repository sites, might be expected to be similar upon cursory examination. However, two important considerations prevent a completely common treatment of corrosion. First, corrosion rate and especially stress corrosion can be very sensitive to minor ionic species such as chloride, fluoride, dissolved oxygen, and sulfate. Amounts of dissolved oxygen may depend on the sealing characteristics of the repository. Secondly, the effects of radiation and temperature on these waters are unknown and must be assessed by appropriate corrosion testing. However, these kinds of effects have not been investigated to any large degree, and the review of data in this report is primarily of data that appear to relate to all of the low-ionic-strength waters mentioned above. The reader is referred to a recent review of the corrosion resistance of cast irons and titanium by Charlot and Westerman (1981) for a more detailed discussion relative to the basalt geologic environment. Much of the data cited are also summarized herein.

4.1 SCREENING STUDIES IN REPOSITORY WATERS

Two investigations to rank materials and select a more limited number of candidate materials were performed for Hanford groundwater (Westerman 1980) and Grande Ronde water (Anderson 1981). Tables 4.1 and 4.2 give the results of these tests.

Because the weight changes were obtained on U-bend specimens with welds, the reported values in Westerman (1980) are probably not very accurate, especially for rates less than about 0.0001 mm/yr; no claims otherwise are made for these screening studies. The corrosion rates for the cast irons were probably more representative of values on rectangular specimens of well-defined surface area since the corrosion rates were relatively high. No cracking was reported for any of the materials in Table 4.1. Tests with cyclic-loaded, precracked specimens and residual stress specimens in a 44 ppm Cl simulated repository water indicated no susceptibility to stress corrosion cracking for Grade 2 and Grade 12 titanium (Pitman, Griggs, and Elmore 1980; Westerman et al. 1981).

TABLE 4.1. Corrosion Rate of Metal Alloys in Simulated Hanford Groundwater at 250°C (oxygen <1 ppm first 90 d, <50 ppb second 90 d) (adapted from Westerman 1980; Pitman et al. 1980)

<u>Material</u>	<u>Corrosion Rate, mm/yr 1st 90 d</u>	<u>Corrosion Rate, mm/yr 2nd 90 d</u>
304L SS	0.000046	0.000077
410 SS	0.00027	0.00024
Nickel 200	--	0.0157
Inconel 600	0.00016	0.00020
70 Cu-30 Ni	0.0040	0.0263
Copper	--	0.00776
Hastelloy C-276	0.00008	0.000160
TiCode-12	nil	nil
Ductile Cast Iron	0.02	0.02 to 0.03
Gray Iron	0.01 to 0.02	0.03

The results reported by Anderson (1981) for simulated Grande Ronde basalt groundwater (Table 4.2) were also obtained on U-bend specimens. These results were intended for screening and cannot be used to predict corrosion rates for extended periods. The calculated corrosion rates also were in error because corrosion films were not completely removed. The relevance of the experiments to very low oxygen environments is uncertain because the attempt to achieve extremely low oxygen fugacity was apparently unsuccessful based on the reported significant oxygen level in the cover gas, which did not equilibrate to the desired low-oxygen fugacity.

Westerman, Pitman, and Nelson (1982) have recently reported very low corrosion rates for various cast irons and steels in basalt groundwater (Table 4.3). In basalt groundwater, cast iron corroded at a maximum of 0.0076 mm/yr in 6 months at 250°C, and no gross pitting or other significant nonuniform corrosion was reported. The two cast steels corroded at somewhat lower rates. As

TABLE 4.2. Corrosion Rates of Metal Alloys in Simulated Grande Ronde Groundwater at 250°C (adapted from Anderson 1981)

<u>Alloy</u>	<u>Corrosion Rate, mm/yr</u>
Inconel 600	0.00015
Hastelloy G	0.00028, 0.00029
Inconel 625	0.00031
Titanium Grade 2	0.00037, 0
Titanium Grade 12	0.000214, (a) 0.00043(a)
Ferallium 25-5	0.0035, 0.0036
Hastelloy C-276	0.000089
90 Cu-10 Ni	0.00095
Inconel 825	0.00034

(a) Based on weight loss.

expected the corrosion rates were lower at 150°C. The specimens were coated (probably protectively) with $\text{NaAlSi}_2\text{O}_6 \cdot \text{H}_2\text{O}$ and $(\text{Fe}, \text{Al})\text{Si}_2\text{O}_5(\text{OH}) \cdot n(\text{H}_2\text{O})$; apparently material transferred from the crushed basalt in the bottom of the autoclave. No Fe_2O_3 or Fe_3O_4 corrosion products were found. Such transfer, as well as oxygen depletion by the basalt, are key factors that must be assessed in order to apply the corrosion rates measured.

In the tuff groundwater, the rates were even lower than in the basalt groundwater, although the amount of data was more limited (Table 4.3). The type of coating that formed on the specimens was not determined.

The corrosion rates in deionized water at 250°C were within the ranges observed for basalt water. This similarity in rates indicates that basalt water and deionized water are about equal in corrosiveness even though a different film, Fe_3O_4 , formed in deionized water. It remains to be shown whether this conclusion applies to corrosion at lower temperatures.

TABLE 4.3. Corrosion Rates of Cast Iron and Cast Steels in Groundwaters
in Flowing Autoclave Tests (Westerman, Pitman, and Nelson 1982)

Material	Solution	Temperature, °C	Test Duration, mo	Rate, mm/yr
Ductile Cast Iron	Basalt(a) Groundwater	150	1	0.0089, 0.0071
		150	3	0.0038
		150	6	0.0036
		250	1	0.026, 0.023
		250	3	0.0079
		250	6	0.0069, 0.0048
Cast Steel 2-1/2 Cr 1 Mo	Basalt(a) Groundwater	150	1	0.0033
		150	3	0.0020
		150	6	0.0025, 0.0012
		250	1	0.014, 0.015
		250	3	0.0041
		250	6	0.0028, 0.0015
Cast Steel 1-1/4 Cr 1/2 Mo	Basalt(a) Groundwater	150	1	0.00203, 0.0198
		150	3	0.0025
		150	6	0.0024, 0.0030
		250	1	0.014, 0.025
		250	3	0.0043
		250	6	0.0058, 0.0018
Ductile Cast Iron	Tuff Groundwater(b)	250	1	0.0046, 0.0069
Cast Steel 2-1/2 Cr 1 Mo	Tuff Groundwater	250	1	0.0053, 0.0064
Cast Steel 1-1/4 Cr 1/2 Mo	Tuff Groundwater	250	1	0.0147, 0.0046
Ductile Cast Iron	Deionized Water	250	1	0.0097, 0.0104
		250	3	0.0076, 0.0078
Cast Steel 2-1/2 Cr 1 Mo	Deionized Water	250	1	0.0135, 0.0112
		250	3	0.0102
Cast Steel 1-1/4 Cr 1/2 Mo	Deionized Water	250	1	0.0112, 0.0112
		250	3	0.0079, 0.0074

(a) Synthetic Grande Ronde groundwater (Wood 1980) passed through crushed basalt; pH 9.8 to 10, inlet oxygen 6-8 ppm, outlet ~0.3 ppm

(b) J-13 Well water from Nevada Test Site flowed through Bullfrog Horizon tuff rock; inlet oxygen 6-8 ppm

Henrikson and Pourbaix (1979) reported corrosion tests on a titanium alloy (ATi-24) similar to titanium Grade 1 in "severe" solutions (4000 ppm Cl^- and $\text{pH} = 4.5$) and found very low corrosion rates, less than 0.00001 mm/yr, for times as long as 600 days (Table 4.4). There was no indication that corrosion rates increased with time. No pickup of hydrogen was found; in these tests higher oxygen caused a slightly higher corrosion rate. Some specimens placed in bentonite exhibited about the same corrosion rates. These tests are very convincing that titanium corrodes slowly in the test environment and that corrosion measurements have good repeatability.

Titanium and zirconium corrosion rates were measured in air-saturated artificial groundwater (Videm 1981) by an electrochemical method to give corrosion rates for times to 434 days (Figure 4.1). The corrosion rates in Figure 4.1 are very low, e.g., for times greater than a year, $<10^{-6}$ mm/yr, but rates were measured only at 25°C.

TABLE 4.4. Corrosion Rates of Titanium in Synthetic Water, 4000 ppm Cl^- ; $\text{pH} = 4.5$ (Henrikson and Pourbaix 1979)

Temperature	Time, d					
	30	100	200	270	300	600
<0.01 ppm O_2						
100°C	0.00008	0.00006	0.00002	≤ 0.00001 (a)	≤ 0.00001 (a)	≤ 0.00001 (a)
130°C	0.00005	0.00004	0.00002	0.00002	≤ 0.00001	--
8 ppm O_2						
100°C	0.00005	0.00004	0.00004	0.00002	0.00004	--
130°C	0.00003	0.00003	≤ 0.00001 (a)	< 0.00001 (a)	0.00002	--

(a) The sensitivity of the method was approximately 0.00001 mm/yr

FIGURE 4.1. Corrosion Rates of Titanium (commercially pure) and Zirconium Versus Time of Exposure. (Solution in ppm: 120 Na⁺, 1.5 F⁻, 6 Ca⁺⁺, 3 Mg⁺⁺, 2 K, 300 HCO₃, 12 Cl⁻, 12 SO₄, 10 SiO₂, 8 O₂) (Videm 1981)

Corrosion rates of 23 alloys in a clay environment to temperatures as high as 300°C for periods as long as 16 months were reported by Casteels et al. (1980). The experiments involved loading air with the corrosive agents expected from pyrolysis of specific clays. The sulfur and chloride in the gas were particularly aggressive and resulted in localized attack in some cases (Table 4.5). Carbon steel specimens were destroyed in 9 months at 50°C. Titanium, Hastelloy B, and Hastelloy C had excellent corrosion resistance in these tests.

4.2 RADIATION CORROSION STUDIES IN WATER

A computer calculation of radiolysis of groundwater was reported by Christensen 1978, which included the amounts of oxygen, hydrogen, and hydrogen peroxide expected from an 18 to 1.8 × 10⁶ R/h dose rate. The levels were in the range of 10 to 30 ppb and increased only moderately with dose rate, even though hydrogen peroxide was significantly lower at low dose rates.

Hendrickson, et al. (1978) reported that titanium remained passive in a simulated gamma-radiation environment for aqueous solutions characteristic of bentonite, quartz, and groundwater.

TABLE 4.5. Depth of Localized Penetration on Alloys in a Wet, Simulated Clay-Gas Environment at 49°C (Casteels et al. 1980)

Alloy	9 Month		16 Month	
	Depth, mm	mm/yr	Depth, mm	mm/yr
304 SS	0.078	0.104	0.060	0.045
316 SS	0.020	0.027	0.010	0.008
UHB 904L	No localized attack	--	--	--
Ni-200	0.026	0.035	0.020	0.015
Inconel 625	0.036	0.048	--	--
Hastelloy C	No localized attack		--	--
Al	0.076	0.101	--	--
Ti, Ti6Al-4V and Ti-Pd	No localized attack		--	--

Westerman, Pitman, and Nelson (1982) have reported radiation-corrosion results in basalt groundwater (Table 4.6) and have observed significant differences in both rate and film formed on the specimen compared with tests without radiation. For example, the corrosion rate in the radiation field was as much as four times greater after 3 months for ductile cast iron and a cast steel. Moreover, in addition to considerable $\text{Na AlSi}_3\text{O}_6 \cdot \text{H}_2\text{O}$ (which occurred in only small quantities on the unirradiated specimens) Fe_2O_3 formed but not $(\text{Fe, Al}) \text{Si}_2\text{O}_5(\text{OH}) \cdot n(\text{H}_2\text{O})$. Moreover, Fe_3O_4 also formed on the cast steel. Thus, corrosion rates apparently were increased by radiation, and the corrosion mechanisms also were changed.

Westerman et al. (1982) observed no formation of TiO_2 on TiCode-12 specimens in irradiation-corrosion tests in basalt water but CaTiSiO_5 formed instead. The corrosion rate increased compared to tests with no radiation, although the corrosion rates were still very low as shown in Table 4.6.

Studies by Fujita, Akiyama, and Tamura (1981) on the stress corrosion of austenitic stainless at 250°C in oxygenated (8 ppm) and deoxygenated (≤ 20 ppb) water indicated a large effect of radiation in the presence of oxygen at 4.5×10^4 R/h. They showed that in the presence of 8 ppm oxygen 304 stainless

TABLE 4.6. Corrosion Rates of Cast Iron, Cast Steel, and Titanium in ^{60}Co Radiation Field in Basalt Groundwater (Dose Rate, 2×10^5 R/h, 8 ppm inlet oxygen) (Westerman, Pitman, and Nelson 1982)

Material	Temperature, °C	Test Duration, Mo	Corrosion Rate, mm/yr
Ductile Cast Iron	250	1	0.012, 0.014
Ductile Cast Iron	250	3	0.0097, 0.031
Cast Steel 2-1/2 Cr-1Mo	250	1	0.0089, 0.0076
Cast Steel 2-1/2 Cr-1Mo	250	3	0.014, 0.022
Ti Grade 2	250	3	~0.001(a)
Ti Grade 12	250	3	<0.001

(a) Assuming corrosion film is CaTiSiO_5

steel became more susceptible to stress corrosion cracking under 4.5×10^4 R/h ^{60}Co radiation than without radiation (Table 4.7). No other materials were examined. No effect of radiation occurred for tests in water with ≤ 20 ppb oxygen. While the studies produced no data on corrosion rate, observations by Fujita et al. indicated that for oxygen ≤ 20 ppb protective oxides formed more readily under radiation than in the same solution without radiation. They concluded that radiolysis has more effect in the presence of oxygen because of the secondary radiolysis products O_2^- , HO_2^- , and H_2O_2 .

TABLE 4.7. Constant Extension Rate Tests on Type 304 Stainless Steel With ^{60}Co Radiation in Water at $T = 250^\circ\text{C}$, $P = 5 \times 10^7$ Pa (Fujita et al. 1981)

	Extention Rate, mm/min	^{60}Co Radiation Dose Rate, R/h	Time to Fracture, h	Elongation, %	Maximum Stress, kg/mm ²
Dissolved oxygen ≤ 20 ppb	5×10^{-4}	no exposure	203	30.4	38.5
		4.5×10^5	221	33.2	39.0
	1×10^{-3}	no exposure	109	32.7	39.9
		4.5×10^5	110	33.0	39.2
Dissolved oxygen = 8 ppm	5×10^{-4}	no exposure	56	8.4	22.5
		4.5×10^5	42	6.3	17.1
	1×10^{-3}	no exposure	55	16.5	30.6
		4.5×10^5	36	10.8	22.3

5.0 LONG-TERM CORROSION DATA

Certain long-term data and observations on archaeological objects are of particular interest because of the very long containment times expected of metallic barriers. The archaeological data, in particular, are intriguing because metal objects are known to have resisted corrosion for 1000 yr and longer.

5.1 UNDERGROUND CORROSION DATA

Several aspects of underground corrosion studies warrant inclusion of the data in this compilation. The significant attributes were:

- Very long term studies were systematically made by the NBS for periods as long as 31 yr on cast-irons, steels, copper, zinc, and lead (Romanoff 1957; Gerhold, Escalante, and Sanderson 1981).
- Large numbers of specimens (>30,000) were studied which facilitated statistical analyses.
- 10-yr data and 6-month data were correlated, and both general corrosion and pitting penetration were predicted from short-term data.

However, the underground data are not directly applicable to deep geologic burial of nuclear waste because:

- The studies were made in varied soils but none were representative of proposed deep geologic sites.
- The moisture content and oxygen content were not controlled or known and varied seasonally. Thus, how the data relate to other laboratory tests is difficult to assess. The oxygen concentration in the 1 1/2- to 6-ft-deep burial horizons was probably higher than in a sealed repository.
- The studies contain no data on the effects of temperature or radiation.

In spite of these limitations, it was considered appropriate to include long-term underground data because of the statistical treatment given the data in the study.

Exceptional corrosion resistance of titanium was observed in NBS soil tests in a variety of soils of varying pH (4.0 to 8.8), varying water content, soil electrical resistivities from 55 to 30,000 Ωcm , and chemistries (Sanderson and Romanoff 1969). No corrosion occurred for tests as long as 8 yr with no indication of pitting or stress corrosion cracking associated with welding. These results were reported on 88 specimens and are noteworthy. Carbon steel had pit depths from 1.5 to 3.2 mm in the same 8-yr test with weight losses corresponding to 0.017 to 0.21 mm general penetration (Figures 5.1 and 5.2).

In clay and marsh soils, pitting appeared to proceed at a high linear rate only after 2 yr. Two-year tests could be misleading in these poorly drained, low resistivity soils.

In other presentations of the NBS data, the long-term data were summarized in comparisons of steel, lead, copper, and zinc in soils with various degrees of aeration, soluble salt content, sulfides, and pH (Figure 5.3). Steel

FIGURE 5.1. General Penetration Derived from Weight Losses Reported for Carbon Steel Pipe in a Variety of Soils (after Sanderson and Romanoff 1969) A. Sandy Loam; B. Loam; C. Clay; D. Sand; G. Tidal Marsh

FIGURE 5.2. Pit-Depth Versus Exposure Time for Carbon Steel in Various Soils (after Sanderson and Romanoff 1969) A. Sandy Loam; B. Loam; C. Clay; D. Sand; G. Tidal Marsh

corroded slowest in well aerated, acid, low-salt soils and more rapidly in alkaline soil. Chloride passivated lead (Figure 5.3), and copper exhibited a low corrosion rate in every case, except in the presence of sulfides. Later, 3-yr studies by Haynes and Baboian (1979) gave corrosion rates for copper that were in the range of 0.0013 to 0.0088 mm/yr in similar soils.

A methodology for analyzing pitting penetration in the NBS study should be mentioned (Romanoff 1957, p. 73). For example, using equations derived from the data on iron and steel, many soils were predicted to cause pitting penetration in a 1000 ft of 215-mm-diameter pipe of 8.5-mm wall thickness only after times far exceeding 1000 yr. However, many soils were expected to produce penetration in less than 100 yr.

The correlation of 6-month laboratory weight-loss data with 10-yr field data was also reported. The correlation was empirical but gave good results across many soils using the same constants in a linear equation that related laboratory data to field test data. The 10-yr general penetration, based on

FIGURE 5.3. Average and Maximum Penetration Time Curves of Steel, Copper, Lead, and Zinc in Soils of Indicated Characteristics (Romanoff 1957)

weight loss, was much less than 20 times the 6-month penetration. The depth of pitting after 10 years also correlated linearly with the short-term weight loss.

The uncertainties of corrosion rates were reported in terms of standard deviations for some of the NBS data. It was not readily apparent how many specimens were tested for each case. Generally two to four replicate specimens were measured for each soil and exposure time. Relative standard errors were in the range 0.1 to 30% for wrought steels and 6 to 50% for cast irons. Certain soils gave much larger relative standard errors than others, but no reasons were given for the differences.

5.2 ARCHAEOLOGICAL DATA

Several reviews of archaeological objects have been directed toward the corrosion of metals with respect to barriers for nuclear waste repositories (Tylecote 1977, 1979, 1980; Johnson and Francis 1980). The derived corrosion rates were varied; e.g., estimated corrosion rates of iron varied from less than 0.00001 to 1 mm/yr and copper from 0.00001 to 0.005 mm/yr (Johnson and Francis 1980). The variations can be ascribed to the different environments where the objects were found. Thus, in ascribing any significance to the data, the environment must somehow be matched or related to the repository environment since there was such a large variation in corrosion rates (see Table 5.1). The data in Table 5.1 illustrate the sensitivities to environment. The table also shows that the corrosion rate of iron can be quite low in seawater, mud, and dry conditions.

Tylecote (1979) indicated that tin bronzes have lasted over 1000 yr in 8 to 8.5 pH soil and estimated a corrosion rate of 0.45 mm/2000 yr. Other reports (Tylecote 1980 and Krysko 1980) suggested that lead corrodes as slowly as 0.0085 mm/yr based on 333 yr corrosion of a lead ball in polluted water. Small grain size in lead is a requisite for such corrosion resistance. Another observation (Tylecote 1977) was that copper sheet lasted for 300 yr and 2000 yr after shipwrecks in seawater, and no evidence was found of intergranular attack. The copper sheet thinned uniformly during the long times of exposure.

TABLE 5.1. Corrosion Rates Estimated from Various Archaeological Objects (after Johnson and Francis 1980)

<u>Metal</u>	<u>Environment</u>	<u>Corrosion rate, mm/yr</u>
Iron	Arid climate, tomb	0.001 to 0.005
Iron	Seawater	0.4
Iron	Seawater Mud	0.002

The archaeological data leave many questions unanswered especially with regard to the relation of the observations to possible wet conditions in deep geologic repositories. There is no doubt that metals can survive long periods of time. In particular, dry or mildly moist conditions appear to be optimum for metal preservation. Again, elevated temperature and radiation in a nuclear waste repository make application of the data difficult without relating the data in some way to a broader data base that includes temperature and radiation as test parameters.

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APPENDIX

ALLOY DESIGNATION AND NOMINAL COMPOSITIONS

APPENDIX

ALLOY DESIGNATION AND NOMINAL COMPOSITIONS

For the reader's convenience the nominal compositions of various alloys included in this report are given in Table A.1. Many designations are registered trademarks of metal alloy producers and these are noted where appropriate. Use of manufacturers' names or trademarks does not imply endorsement by the Materials Characterization Center or Pacific Northwest Laboratory.

Specifications on other minor impurities are required to completely describe many of these materials. In many cases, these were omitted for the purpose of this report. The reader should check the original reference for more details since minor constituents may have strong effects on stress corrosion cracking in some cases. Compositions given are not necessarily based on analyses of the materials used in studies cited in this report.

TABLE A.1. Nominal Compositions and Trademarks

<u>Alloy</u>	<u>Nominal Composition, wt%</u>	<u>Origin of Trademark</u>
<u>Steels</u>		
Carbon Steel ASTM A108	0.13–0.20 C, 0.3–0.9 Mn, bal Fe	
Corten A Steel	0.1 C, 0.4 Mn, 0.5 Si, 1 Cr, 0.02 Ti, bal Fe	U.S. Steel
2 1/4 Cr–1 Mo Steel	0.2 C, 0.8 Mn, 0.3 Si, 2.2 Cr, 1 Mo, bal Fe	
Cast Steel 2 1/2 Cr 1 Mo	0.12 C, 0.6 Mn, 0.6 Si, 1 Mo, 2.5 Cr, bal Fe	
Cast Steel 1 1/4 Cr 1/2 Mo	0.12 C, 0.8 Mn, 0.6 Si, 0.5 Mo, 1.3 Cr bal Fe	
A570	0.13 C, 0.42 Mn, bal Fe	
A53B	0.20 C, 0.55 Mn, bal Fe	
1010	0.10 C, 0.3 Mn, bal Fe	
1020	0.18–0.23 C, 0.3–0.6 Mn, bal Fe	
4130	0.3 C, 0.5 Mn, 0.3 Si, 0.8 Cr, bal Fe	
A106	0.20 C, 0.55 Mn, 0.15 Si, 0.1 P, bal Fe	
C75	0.44 C, 1.6 Mn, 0.17 S, 0.2 Si, 0.2 Mo, bal Fe	
C95	0.29 C, 1.4 Mn, 0.013 P, 0.025 S, 0.21 Si, 0.14 Cu, 0.13 Cr, 0.15 Mo, bal Fe	
K55	0.32 C, 1 Mn, bal Fe	
L80	0.29 C, 1.2 Mn, bal Fe	
P110	0.28 C, 1.4 Mn, 0.017 P, 0.016 S, 0.12 Si, bal Fe	
A212B	0.35 C, 0.62 Mn, 0.18 Si, bal Fe	

<u>Alloy</u>	<u>Nominal Composition, wt%</u>	<u>Origin of Trademark</u>
<u>Cast Irons</u>		
Ductile Cast Iron	2.8–3.8 C, 2–3 Si, 0.2–0.6 Mn, 0–2 Ni, bal Fe	
Gray Cast Iron	3.0–3.75 C, 1.8–2.8 Si, 0.5–1.0 Mn, bal Fe	
White Iron	3.4 C, 0.7 Si, 0.6 Mn, bal Fe	
Malleable Iron	2.5 C, 1 Si, 0.5 Mn, bal Fe	
Pearlite Nodular Cast Iron	2.4–2.5 C, 1–1.5 Si, 0.28–0.48 Mn, bal Fe	
Ferritic Nodular Cast Iron	2–2.45 C, 0.8–1.4 Si, 0.55 Mn max, bal Fe	
<u>Stainless Steel</u>		
304L SS	0.030 C max, 18–20 Cr, 8–12 Ni, bal Fe	
316L SS	0.030 C max, 16–18 Cr, 10–14 Ni, 2–3 Mo, bal Fe	
Nitronic 50	0.06 C max, 22 Cr, 13 Ni, 2.25 Mo, 0.2 Nb, 0.2 V, bal Fe	Armco Steel Corp
20Cb3	0.07 C max, 20 Cr, 13 Ni, 2.25 Mo, bal Fe	Carpenter Technology
Ebrite 26–1	0.01 C max, 26 Cr, 1 Mo, bal Fe	Allegheny Ludlum Corp
310 SS	0.25 C max, 1.5 Si max, 24–26 Cr, 19–22 Ni, bal Fe	
347 SS	0.08 C max, 2 Mn, 17–19 Cr, 9–13 Ni, bal Fe	
430 SS	0.12 C max, 16–18 Cr, bal Fe	
316 SS	0.08 C, 2 Mn, 1 Si, 16–18 Cr, 10–14 Ni, 2–3 Mo, bal Fe	
6X	0.03 C, 1.5 Mn, 24 Ni, 29 Cr, 6.5 Mo, bal Fe	Allegheny Ludlum
446 SS	0.20 C max, 18–23 Cr, bal Fe	

<u>Alloy</u>	<u>Nominal Composition, wt%</u>	<u>Origin of Trademark</u>
<u>Stainless Steel (contd)</u>		
29 Cr-4 Mo	0.01 C max, 24 Cr, 4 Mo, bal Fe	
410 SS	0.15 C max, 11.5-13.5 Cr, bal Fe	
Ferrallium 25-5	5 Ni, 25.5 Cr, 3.5 Mo, 1.7 Cu, 0.17 N, bal Fe	Langley Alloys Ltd
UHB 904L	25 Ni, 20 Cr, 4.5 Mo, 1.5 Cu, 0.03 C, bal Fe	
<u>Copper Alloys</u>		
Naval Brass	60 Cu, 39 Zn, 1 Sn	
<u>Aluminum Alloys</u>		
1100 Al	0.12 Cu, 99.00 Al min	
6061-T6	0.6 Si, 0.28 Cu, 1 Mg, 0.2 Cr, 97.9 Al min	
2024-T3	4.4 Cu, 0.6 Mn, 1.5 Mg, 93.5 Al min	
<u>Titanium Alloys</u>		
Ti 45A	0.25 Fe, 0.08 C max, 0.05 N max, 0.015 H max, bal Ti	
Ti 35A	0.18 C, 0.01 N, 0.04 Fe, 0.09 O	
Ti 1.5 Ni	0.01 C, 1.5 Ni, 0.04 Fe	
Ti 6Al4V	6 Al, 4 V, bal Ti	
Ti C.P.	99.5 Ti	
TiCode-12	0.03 N max, 0.10 C max, 0.015 H max, 0.30 Fe max, 0.25 O max, 0.8 Ni, 0.3 Mo, bal Ti	TIMET
Ti-50A	Commercially pure Ti, ASTM Grade 2, 0.03 N max, 0.10 C max, 0.015 H max, 0.3 Fe max, 0.25 O max	

<u>Alloy</u>	<u>Nominal Composition, wt%</u>	<u>Origin of Trademark</u>
<u>Titanium Alloys (contd)</u>		
Ti-0.2% Pd	Same as ASTM Grade 7, 0.03 N max, 0.10 C max, 0.015 H max, 0.3 Fe max, 0.25 O max, 0.2 Pd	
Ti-2 Ni	2 Ni, bal Ti	
Ti Grade 2	0.03 N, 0.10 C, 0.015 H, 0.30 Fe, 0.25 O	
Ti Grade 12	Nominally same as TiCode-12	
Ti Grade 1	0.003 C, 0.006 N, 0.014 H, 0.04 Fe, 0.07 O	
<u>Nickel Alloys</u>		
Hastelloy C	0.08-0.12 C, 4-7 Fe, 3-5.25 W, 15-18 Mo, 14.5-17.5 Cr, 0.2-0.4 V, bal Ni	Cabot Corp
Monel 400	66.5 Ni, 31.5 Cu, 1.25 Fe, 1 Mn	Huntington Alloys
Incoloy 825	0.03 C, 42 Ni, 30 Fe, 21.5 Cr, 3 Mo, 0.9 Ti	Huntington Alloys
Hastelloy S	0.02 C max, 15.5 Cr, 15.5 Mo, bal Ni	Cabot Corp
Hastelloy G	0.05-0.12 C, 1-2 Mn, 18-21 Fe, 5.5-7.5 Mo, 21-23 Cr, 1.5-2.5 Cu, 1.75-2.5 Nb + Ta, bal Ni	Cabot Corp
Inconel 600	0.08 C, 76 Ni, 15.5 Cr, 8 Fe	International Nickel Corp
Inconel 625	0.05 C, 61 Ni, 21.5 Cr, 9 Mo, 2.5 Fe	International Nickel Corp
Hastelloy C-276	0.02 C, 4-7 Fe, 3-4.5 W, 15-17 Mo, 14.5-16.5 Cr, bal Ni	Cabot Corp
Nickel 200	99.0 Ni	
Hastelloy B	0.05-0.12 C, 4-6 Fe, 26-30 Mo, 0.2-0.4 V, bal Ni	Cabot Corp

<u>Alloy</u>	<u>Nominal Composition, wt%</u>	<u>Origin of Trademark</u>
<u>Zirconium Alloys</u>		
Zircaloy-2	0.12 C, 0.1 Cr, 0.05 Ni, 1.5 Sn, 98.2 Zr	
<u>Cobalt Alloys</u>		
Haynes 25	0.1 C, 54 Co, 20 Cr, 10 Ni, 1 Fe, 15 W	Cabot Corp

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