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RADIATION INDUCED CORROSION OF METALS

Armen R. Kazanjian

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RADIATION INDUCED CORROSION OF METALS

Armen R. Kazanjian

David R. Horrell

Abstract. A general survey has been made of a number of metals and organic liquids to determine their susceptibility to radiation induced corrosion. The metals were tantalum, tungsten, stainless steel 304, platinum, copper, zirconium, niobium, titanium, uranium, thorium, aluminum, silver and molybdenum. The liquids were carbon tetrachloride, trichloroethylene, chloroform, perchloroethylene, trichlorotrifluoroethane, hexane, and Texaco Regal A oil. These metal-liquid combinations were gamma irradiated with doses on the order of 2×10^{21} eV/ml and then examined for corrosion. The metals that showed appreciable corrosion were copper, thorium, and stainless steel in most of the liquids, uranium in chloroform, and aluminum in carbon tetrachloride. The other systems were essentially unaffected by radiation.

INTRODUCTION

Since radiation changes the composition of liquids and produces free radicals and ions in the process, it is very likely that radiation will change the corrosion behavior of some metals. This has been found to be true in many instances, especially for aqueous solutions in which most of the studies have been made. Although a few broad studies have been made,^{1,2,3} there are very few generalizations because most of the research has been very specific.

Because of the scarcity of information on radiation induced corrosion in nonaqueous media, we have started an investigation in this area. The first part (this report) has been a general survey of a number of metals and organic liquids to determine their susceptibility to radiation induced corrosion. The purpose was to screen a large number of systems rather than obtain accurate corrosion rates. Those metal-liquid combinations that experienced corrosion will be investigated further. Although quantitative values are presented, they can be considered estimates because the tests were not made in replicate and the experimental conditions were somewhat

varied. Nevertheless, the screening operation was successfully accomplished.

The metals were selected on the basis of their availability and their representation of the periodic table. The liquids fall into two categories — lubricants and halogenated solvents. These are used for machining and cleaning at Rocky Flats.

EXPERIMENTAL

Liquids:

Carbon tetrachloride, CCl_4 — Baker Analyzed Reagent
 Trichloroethylene, C_2HCl_3 — Baker Analyzed Reagent
 Chloroform, CHCl_3 — B & A Analyzed Reagent
 Perchloroethylene, C_2Cl_4 — Dow unstabilized solvent
 1,1,2-Trichlorotrifluoroethane, $\text{C}_2\text{Cl}_3\text{F}_3$ — Genesolv-D (99.999%, Allied Chemical Corp.)
 Hexane, C_6H_{14} — Baker Analyzed Reagent
 Texaco Regal A Oil — light hydraulic oil

Metals:

Tantalum, Tungsten, Stainless Steel 304, Platinum, Copper, Zirconium, Niobium, Titanium, Uranium, Aluminum, Molybdenum, Silver, Thorium

The metal purities are given in Table 1.

Cleaning Procedure:

A six-step cleaning procedure was used before the initial weighing. The first step was omitted in the procedure before the final weighing at the end of the experiment.

1. Coupons were sanded with 280-grit silicon carbide paper.

2. Coupons were immersed in trichloroethylene and swabbed with cotton.
3. Coupons were immersed in methyl alcohol and swabbed with cotton.
4. Coupons were immersed in water and swabbed with cotton.
5. Coupons were immersed in acetone and swabbed with cotton.
6. Leave in desiccator, weigh.

Blanks were run along with the corrosion tests. Coupons were cleaned, weighed, and stored in air for the same period as the corrosion tests. They were then cleaned and weighed again. The change in weight averaged 0.1 mg, which was the sensitivity of the balance used. This showed that the cleaning procedure was not responsible for any weight change that occurred in the corrosion tests.

Irradiation:

A commercial Cobalt-60 gamma source (Gammacell-220) was used for all the irradiations. The source strength was increased almost 10-fold at the midpoint of the investigation, and the exposure conditions were changed to minimize this difference. The dose rates of the two sources were approximately 2×10^{17} and 1.5×10^{18} eV/ml minute. The exact values depend on the position of the glass containers in the irradiation chamber and on the electron density of the particular liquid and metal being irradiated. The exact doses are presented in Table 1. There are differences in the doses absorbed by a given liquid in contact with the various metals because of slightly varying irradiation times and the two source strengths. The difference in doses are considered to be negligible for the purpose of the investigation.

Test Procedure:

Metal coupons, formed by machining oversized disks down to a one-inch diameter, were cleaned then immersed in 100 ml of liquid contained in narrow glass beakers. The coupons rested on holders made from glass rod. In experiments without irradiation, the beakers were held in a constant temperature water bath of 30°C for a period of 7 days. The beakers were covered and taped to exclude water vapor from the contents. The only liquid to acquire an appreciable amount

of water was chloroform; 300 to 400 ppm at the end of the test. It was not determined whether the water absorption took place during the immersion or during the handling period before and after the immersion. The liquids were not stirred in any of the experiments.

In those experiments (stainless steel, Ta, W, Cu, Pt) using the weaker gamma source, the irradiations were from 65- to 73-hours duration. The coupons were examined for corrosion after this period. The temperature in the irradiation chamber was 29°C. In those experiments (Ag, Zr, Nb, Ti, U, Al, Mo, Th) using the stronger gamma source, the irradiation periods were 24 hours at 48°C. The coupons were then left immersed (nonirradiated) in the liquids for 4 or 5 days at 30°C. The beakers, holders, etc. were the same as those used in the nonirradiation tests.

The corrosion rates are reported as milligrams per square decimeter per day (mdd). The variation in the minimum corrosion rates that could be measured is due mainly to the differences in total immersion time.

RESULTS

The corrosion rates determined in these tests are shown in Table 1. The visual changes associated with minor corrosion have not been reported here as only the gross effects were of interest. The liquids chosen for study are relatively inert and it is not surprising that, with the exception of uranium, the metals were not appreciably corroded in the absence of radiation. Uranium is very reactive and is known to be slightly soluble in a variety of organic liquids.

With radiation, only Cu, Th, and stainless steel experienced increased corrosion rates in most liquids. It can be seen, with two exceptions, that those metals that experienced corrosion did so in most liquids. This implies that although the tests were run singly, they can be considered to have been run in replicate. The five halogenated solvents can be expected to behave similarly; and the hexane and Texaco oil, which is a mixture of hydrocarbons, should also behave similarly.

One of the exceptions was aluminum in CCl_4 where the coupon was completely dissolved. This phenomenon is closely related to the high reactivity in CCl_4 at elevated temperatures. The

mechanism for this reaction is postulated⁴ to originate with the breaking of a carbon chlorine bond to form two free radicals ($\text{Cl}\cdot + \text{CCl}_3\cdot$). This is also the primary step in the radiolytic decomposition of CCl_4 so that the effects of radiation and high temperature are probably identical.

The other exception was uranium in CHCl_3 where the radiation significantly enhanced the corrosion. The increased rate is probably the result of dissolution by HCl , which radiation produces in CHCl_3 in a very high yield. The dissolution is probably greatly aided by the moisture in CHCl_3 . This is an important variable that will be investigated further.

The relatively high temperature (48°C) in the source chamber resulted in an excessive evaporation rate for the Genesolv-D ($\text{C}_2\text{Cl}_3\text{F}_3$) with its low boiling point of 48° . The liquid had to be replenished during the 24-hour irradiation. The experiments with Al, Ag and U were repeated while cooling the Genesolv-D to a constant temperature of 23°C . The corrosion rates for Ag and Al were unchanged, whereas the corrosion rate of U increased from 2.9 to 36.5 mdd. This increase was due to the longer contact time between U and Genesolv-D and its radiation products.

It should be noted that the radiation dose administered in these tests would convert about 1% of the liquid to products. Further irradiation would increase this fraction and thereby increase the corrosion rate if the metal were susceptible to corrosion in that medium. This means that corrosion rates obtained with irradiation are dependent on the total dose, dose rate, and the relationship between irradiation periods and immersion periods if they are not completely overlapping, as well as the commonly encountered variables in corrosion testing.

DISCUSSION

One of the primary goals for our research is to determine exactly how radiation can influence corrosion. A discussion of these considerations follows.

It is possible for radiation to enhance the corrosion by affecting three different areas in the system; the metal, the metal surface, and the

liquid. The important radiation effect in metals is the displacement of atoms to cause defects. Since these displacements occur mainly with neutron and heavy particle bombardment and very rarely with gamma radiation, the effects of metal irradiation in these experiments are considered to be negligible. The metal surface or solid interface is obviously an important area for reactivity. The interface could be either a metal oxide that was previously formed or less likely a compound (for example, the chloride) that is formed after immersion in the liquid. As in the case of metal, there would be very few displacements. However, changes in the electronic properties, such as ionic conductivity, could also take place because the solid would be an ionic compound. Unfortunately very little is known about this subject in relation to corrosion, so its contribution can not be assessed.

The third area, the liquid, can be considered to have the greatest influence on the corrosion. The radiation chemistry of the liquids used in this study is fairly well known and will not be discussed here except for a few general comments. The gamma radiation generates stable products as well as transient ions and free radicals which can react with the metal. The products from these liquids include halogens, halogen acids, and hydrogen which are very reactive with metals. At a production rate of only 0.2 molecule per 100 eV of absorbed energy the amount of product generated in 100 ml liquid in the 24 hour irradiations would be 0.001 mole; much more than sufficient to cause the corrosion observed in these experiments.

The effect of organic ions and free radicals can be considered negligible. Although these species are very reactive, they will only react with those metals that are able to form organometallic compounds. The metals that corroded the most were not those that have the highest tendency to form organometallics. A more important restriction is the limited concentration of ions and free radicals at the metal surface. Although the production rate may be as large as that for the stable products, the transient species are too reactive to be able to diffuse very far from their point of origin. Therefore, the concentration at the metal surface is very small and insufficient to cause the corrosion observed in our experiments. It is also possible for these reactive species to cause corrosion by serving as catalysts; however, not enough is known about this for comment.

High Temperature Corrosion

The metals were also immersed for 4 hours in CCl_4 at 75°C and Texaco oil at 90°C to briefly examine the effect of temperature because some of the irradiations had been carried out at 48°C . The CCl_4 was selected to be representative of the halogenated solvents and the Texaco oil would behave similarly to the hexane. Tests of such short duration would only indicate a very rapid attack. As Table 2 shows, there were only two cases of excessive corrosion; uranium in Texaco oil and aluminum in CCl_4 . Uranium did corrode a little at 30° (12.0 mdd) in oil and the temperature coefficient is obviously very large (large activation energy) to effect such a large increase in corrosion rate (2300 mdd). The very high reaction rate of Al with CCl_4 at high temperatures has been previously investigated.^{4,5}

SUMMARY

These experiments show, that of the combinations tested, the only examples of radiation enhanced corrosion were copper, thorium, and stainless steel in most of the liquids, uranium in chloroform, and aluminum in carbon tetrachloride. These metal-liquid systems will be subjects of further investigation.

TABLE 1. Results of Corrosion Tests.
(Corrosion rates in milligrams
per square decimeter per day.)

Tantalum

Purity 99.99%, thickness 0.0055 in.

	No Irradiation	With Irradiation	Absorbed Dose, eV/ml
CCl_4	<0.3	<0.7	1.2×10^{21}
C_2HCl_3	<0.3	<0.7	1.1×10^{21}
CHCl_3	<0.3	<0.7	1.2×10^{21}
C_2Cl_4	<0.3	<0.7	1.2×10^{21}
$\text{C}_2\text{Cl}_3\text{F}_3$	<0.3	<0.7	1.3×10^{21}
C_6H_{14}	—	<0.7	0.66×10^{21}
Texaco oil	<0.3	<0.7	0.87×10^{21}

Temperature and Contact Time— 30°C , 168 hr; 29°C , 73 hr.

Comments — No corrosion observed.

Tungsten

Purity >99.9%, thickness 0.0107 in.

	No Irradiation	With Irradiation	Absorbed Dose, eV/ml
CCl_4	<0.3	<0.7	1.1×10^{21}
C_2HCl_3	<0.3	<0.7	1.0×10^{21}
CHCl_3	<0.3	2.2	1.0×10^{21}
C_2Cl_4	<0.3	<0.7	1.1×10^{21}
$\text{C}_2\text{Cl}_3\text{F}_3$	<0.3	<0.7	1.2×10^{21}
C_6H_{14}	—	—	—
Texaco oil	<0.3	<0.7	0.77×10^{21}

Temperature and Contact Time— 30°C , 168 hr; 29°C , 65 hr.

Comments — No corrosion observed except in CHCl_3 and this low rate is questionable.

Stainless Steel 304

Purity 19% Cr, 10% Ni, thickness 0.0050 in.

	No Irradiation	With Irradiation	Absorbed Dose, eV/ml
CCl_4	<0.3	34	0.80×10^{21}
C_2HCl_3	<0.3	2	0.74×10^{21}
CHCl_3	<0.3	58	0.76×10^{21}
C_2Cl_4	<0.3	<1	0.81×10^{21}
$\text{C}_2\text{Cl}_3\text{F}_3$	<0.3	1.5	0.86×10^{21}
C_6H_{14}	—	25	0.44×10^{21}
Texaco oil	<0.3	2	0.57×10^{21}

Temperature and Contact Time— 30°C , 168 hr; 29°C , 48 hr.

Comments — Appreciable attack in CCl_4 , CHCl_3 , and C_6H_{14} when irradiated.

Platinum

Purity >99.9%, thickness 0.0024 in.

	No Irradiation	With Irradiation	Absorbed Dose, eV/ml
CCl_4	<0.3	4.5	1.2×10^{21}
C_2HCl_3	<0.3	<0.7	1.1×10^{21}
CHCl_3	<0.3	<0.7	1.1×10^{21}

Uranium (continued)

C ₂ Cl ₄	<0.3	<0.7	1.2 x 10 ²¹
C ₂ Cl ₃ F ₃	<0.3	<0.7	1.2 x 10 ²¹
C ₆ H ₁₄	<0.3	<0.7	0.64 x 10 ²¹
Texaco oil	<0.3	<0.7	0.83 x 10 ²¹

Temperature and Contact Time—30°C, 168 hr; 29°C, 70 hr.

Comments — Slight corrosion observed in irradiated CCl₄.

Copper

Purity 99.99%, about 0.01% Pb, thickness 0.0013 in.

	No Irradiation	With Irradiation	Absorbed Dose, eV/ml
CCl ₄	<0.3	35	1.2 x 10 ²¹
C ₂ HCl ₃	<0.3	10	1.1 x 10 ²¹
CHCl ₃	<0.3	130	1.1 x 10 ²¹
C ₂ Cl ₄	<0.3	13	1.2 x 10 ²¹
C ₂ Cl ₃ F ₃	<0.3	28	1.3 x 10 ²¹
C ₆ H ₁₄	<0.3	7	0.66 x 10 ²¹
Texaco oil	<0.3	7	0.85 x 10 ²¹

Temperature and Contact Time—30°C, 168 hr; 29°C, 72 hr.

Comments — Appreciable corrosion observed when irradiated.

Zirconium

Purity 99.9%, 800 ppm Fe, 300 ppm Cr, 200 ppm Si,
thickness 0.0205 in.

	No Irradiation	With Irradiation	Absorbed Dose, eV/ml
CCl ₄	<0.3	<2	2.8 x 10 ²¹
C ₂ HCl ₃	<0.3	<2	2.6 x 10 ²¹
CHCl ₃	<0.3	<2	2.7 x 10 ²¹
C ₂ Cl ₄	<0.3	<2	2.9 x 10 ²¹
C ₂ Cl ₃ F ₃	<0.3	<2	4.0 x 10 ²¹
C ₆ H ₁₄	<0.3	<2	2.0 x 10 ²¹
Texaco oil	<0.3	<2	2.7 x 10 ²¹

Temperature and Contact Time—30°C, 168 hr; 48°C, 24 hr.

Comments — No corrosion observed.

Niobium

Purity 99.9%, 600 ppm Fe, 300 ppm Si,
<50 ppm other metals, thickness 0.0360 in.

	No Irradiation	With Irradiation	Absorbed Dose, eV/ml
CCl ₄	<0.3	<2	2.8 x 10 ²¹
C ₂ HCl ₃	<0.3	<2	2.6 x 10 ²¹
CHCl ₃	<0.3	<2	2.7 x 10 ²¹
C ₂ Cl ₄	<0.3	<2	2.9 x 10 ²¹
C ₂ Cl ₃ F ₃	<0.3	<2	4.0 x 10 ²¹
C ₆ H ₁₄	<0.3	<2	2.0 x 10 ²¹
Texaco oil	<0.3	<2	2.7 x 10 ²¹

Temperature and Contact Time—30°C, 168 hr; 48°C, 24 hr. irradiated.

Comments — No corrosion observed.

Titanium

Purity 99.5%, 0.4% Fe, 0.1% Si, trace other metals,
thickness 0.0020 in.

	No Irradiation	With Irradiation	Absorbed Dose, eV/ml
CCl ₄	<0.3	<0.4	2.8 x 10 ²¹
C ₂ HCl ₃	<0.3	<0.4	2.6 x 10 ²¹
CHCl ₃	<0.3	<0.4	2.7 x 10 ²¹
C ₂ Cl ₄	<0.3	<0.4	2.9 x 10 ²¹
C ₂ Cl ₃ F ₃	<0.3	<0.4	4.0 x 10 ²¹
C ₆ H ₁₄	<0.3	<0.4	2.0 x 10 ²¹
Texaco oil	<0.3	<0.4	2.7 x 10 ²¹

Temperature and Contact Time—30°C, 168 hr; 48°C, 24 hr. irradiated,
then 30°C, 120 hr.

Comments — No corrosion observed.

Uranium

Purity >99.99%, thickness 0.0090 in.

	No Irradiation	With Irradiation	Absorbed Dose, eV/ml
CCl ₄	40	15	2.8 x 10 ²¹
C ₂ HCl ₃	8	15	2.6 x 10 ²¹
CHCl ₃	5	210	2.7 x 10 ²¹
C ₂ Cl ₄	80	17	2.9 x 10 ²¹

C ₂ Cl ₃ F ₃	5	36	4.0 x 10 ²¹
C ₆ H ₁₄	50	2	2.0 x 10 ²¹
Texaco oil	12	13	2.7 x 10 ²¹

Temperature and Contact Time—30°C, 168 hr; 48°C, 24 hr. irradiated then 30°C, 120 hr.

Comments — Corrosion was observed in all specimens. In all cases, except CHCl₃, no significance can be attached to the differences in rates between the nonirradiated and irradiated systems.

Aluminum—1100

Purity ~1%Fe; 0.1–0.5% Si, Cu, Ni, 0.01–0.1% Cr, Mn, V, Zn; thickness 0.0030 in.

	No Irradiation	With Irradiation	Absorbed Dose, eV/ml
CCl ₄	<0.3	dissolved	2.8 x 10 ²¹
C ₂ HCl ₃	<0.3	<0.4	2.6 x 10 ²¹
CHCl ₃	<0.3	<0.4	2.7 x 10 ²¹
C ₂ Cl ₄	<0.3	<0.4	2.9 x 10 ²¹
C ₂ Cl ₃ F ₃	<0.3	<0.4	4.0 x 10 ²¹
C ₆ H ₁₄	<0.3	<0.4	2.0 x 10 ²¹
Texaco oil	<0.3	<0.4	2.7 x 10 ²¹

Temperature and Contact Time—30°C, 168 hr; 48°C, 24 hr. irradiated then 30°C, 96 hr.

Comments — No corrosion observed except for irradiated CCl₄ in which the coupon was completely dissolved.

Molybdenum

Purity 98%, 1% Fe, 0.4% Pb, 0.3% Cu, 0.3% Ni, thickness 0.0250 in.

	No Irradiation	With Irradiation	Absorbed Dose eV/ml
CCl ₄	<0.3	6	2.8 x 10 ²¹
C ₂ HCl ₃	<0.3	<0.4	2.6 x 10 ²¹
CHCl ₃	<0.3	2	2.7 x 10 ²¹
C ₂ Cl ₄	<0.3	<0.4	2.9 x 10 ²¹
C ₂ Cl ₃ F ₃	<0.3	<0.4	4.0 x 10 ²¹
C ₆ H ₁₄	<0.3	<0.4	2.0 x 10 ²¹
Texaco oil	<0.3	<0.4	2.7 x 10 ²¹

Temperature and Contact Time—30°C, 168 hr; 48°C, 24 hr. irradiated then 30°C, 96 hr.

Comments — Slight corrosion observed in irradiated CCl₄ and CHCl₃. Low rate in CHCl₃ is questionable.

Silver

Purity 99.8%, 0.2% Cu, <0.02% other metals, thickness 0.0305 in.

	No Irradiation	With Irradiation	Absorbed Dose, eV/ml
CCl ₄	approx. 1	approx. 1	2.8 x 10 ²¹
C ₂ HCl ₃	approx. 1	approx. 1	2.6 x 10 ²¹
CHCl ₃	approx. 1	approx. 1	2.7 x 10 ²¹
C ₂ Cl ₄	approx. 1	approx. 1	2.9 x 10 ²¹
C ₂ Cl ₃ F ₃	approx. 1	approx. 1	4.0 x 10 ²¹
C ₆ H ₁₄	approx. 1	approx. 1	2.0 x 10 ²¹
Texaco oil	approx. 1	approx. 1	2.7 x 10 ²¹

Temperature and Contact Time—30°C, 168 hr; 48°C, 24 hr. irradiated then 30°C, 96 hr.

Comments — The silver was tarnished in all cases. The 1 mdd is a weight gain rather than the usual weight loss.

Thorium

Purity >99.9%. 150 ppm Ni, 100 ppm Fe, thickness 0.0158 in.

	No Irradiation	With Irradiation	Absorbed Dose, eV/ml
CCl ₄	<1	3	2.8 x 10 ²¹
C ₂ HCl ₃	2	22	2.6 x 10 ²¹
CHCl ₃	<1	940	2.7 x 10 ²¹
C ₂ Cl ₄	<1	20	2.9 x 10 ²¹
C ₂ Cl ₃ F ₃	<1	<1	4.0 x 10 ²¹
C ₆ H ₁₄	<1	<1	2.0 x 10 ²¹
Texaco oil	<1	20	2.7 x 10 ²¹

Temperature and Contact Time—30°C, 168 hr; 48°C, 24 hr. irradiated then 30°C, 96 hr.

Comments — Irradiation appreciably enhanced the corrosion in most of the liquids. The very high rate in CHCl₃ (as for Cu and U) may be partly caused by the water (about 300 ppm) which was absorbed by the CHCl₃.

TABLE 2. High Temperature Corrosion.

<u>Metal</u>	<u>CCl₄</u>	<u>Texaco oil</u>	<u>Remarks</u>
U	nil	2300 mdd	surface darkened
Th	nil	nil	surface slightly discolored
Al	dissolved	nil	
Ti	nil	nil	
Cu	nil	nil	surface darkened
Ag	nil	nil	tan residue on surface
S.S.	nil	nil	light yellow coloration
Ta	nil	nil	
W	nil	nil	
Pt	nil	nil	
Mo	nil	nil	
Zr	nil	nil	yellow coloration
Nb	nil	nil	

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