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CORROSION TESTS ON TANTALUM, HASTELLOY C AND DURIRON IN 234-5 PROJECT SOLUTIONS

INTRODUCTION

Room temperature and elevated temperature, static immersion and vapor suspension, corrosion tests were conducted with Duriron, Hastelloy C, and tantalum in hydriodic acid and 234-5 project process supernatant solution (synthetic environments. The data relevant to these tests are contained herein.

SUMMARY

Corrosion tests indicate that tantalum and Duriron can be employed in 234-5 project process streams. Of the two, tantalum is recommended. Duriron has limiting fabrication disadvantages. Hastelloy C is not recommended for this use.

DETAILS

The corrosion tests discussed in this report were of two types: (a) complete immersion tests - in which the test specimen was suspended by means of a glass holder in the corrosive, and (b) vapor suspension tests - in which the test specimen was suspended by means of a glass holder in the vapor above the corrosive. Both types were conducted at room temperatures and/or at boiling temperatures depending upon the test requirements for any one specimen.

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Apparatus for the room temperature tests consisted of quart Mason jars, the lids of which were fitted with polythene[1] gaskets to avoid contact of the corrosive vapors with foreign metallic ions from the metal lids and consequent contamination of the test corrosives. One complete immersion and one vapor suspension specimen was contained per Mason jar. Tests at boiling temperatures were carried out in 500 ml Erlenmeyer flasks to which 300 ml Allihn condensers were fitted with 45/50 T ground glass joints. As in the room temperature tests each unit contained two test specimens, one in the corrosive and one in the vapors.

The volume of the test corrosive was in excess of 250 ml per square inch test specimen. No attempt was made to aerate the corrosives since no external aeration is anticipated in process. All room temperature tests were static. The elevated temperature (boiling) tests were agitated in so far as convection currents and solution "bumping" agitate a solution at its boiling point.

Test samples were degreased, alcohol rinsed and air dried prior to weighing and exposure. Post-exposure cleaning was with water and bristle brush for the periodic inspections. In such instances where adherent films were encountered, additional cleaning was accomplished after the final exposure period by pickling in a 33% HNO_3[2] solution for 2-3 minutes at room temperature.

Corrosives were reagent grade hydriodic acid and 234-5 project supernatant solution (synthetic) prepared from reagent grade chemicals. See Table II, "Composition of Corrosives," Appendix, p. 7. In those tests conducted at elevated temperatures (boiling) the solutions were renewed at 24 hour intervals to compensate for iodine losses.

The rate of corrosion was calculated in terms of mils penetration per year[3] and recorded to the nearest mil. Any corrosion rate found to be less than one mil was recorded as "Nil" since corrosion rates of less than one mil fall beyond the limits of experimental error. Calculations:

$$C_m/yr. = \frac{527,000 \times W}{DAT}$$

where W = wt. loss in grams
D = density of material
A = area in sq. in.
T = exposure time in hrs.

(1) The polythene plastic, normally milky-white, acquired a violet hue, indicating iodine absorption. See Koenig, W.W., "234-5 Project Static Corrosion Tests - Plastics and Synthetic Rubber in HI and/or Process Supernatant Solution," Doc. No. HW-12172, January 13, 1949.

(2) By volume.

(3) Assumes uniform corrosion.

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Evaluation of Results

See Table II, "Specimen Exposure and Corrosion Rate Data," Appendix, pp. 8-9.

Duriron

Data from these tests (complete immersion and vapor suspension) revealed very little change in the physical characteristics of this material following exposure to 47% HI (1). Both the complete immersion and vapor suspension tests were characterized by a measurable initial (first 48-hr.) corrosion rate accompanied by the formation of an adherent dull grey film. Subsequent exposure periods failed to produce additional corrosion which could be measured by the methods employed. In addition to the grey film the vapor suspension test specimen showed slight evidence of rusting in the form of scattered rust spots, varying in size from pinpoint to pinhead.

This material (Duriron) is acceptable from the corrosion standpoint for use where process conditions similar to these test conditions may be encountered.

Hastelloy C

All samples of this material showed evidence of corrosion although not all to an undesirable degree. However, any use of this material would be closely limited by conditions which would make its use impractical.

The corrosion products encountered were of two types: (a) a loose, finely granular black deposit easily removed with water and bristle brush, and (b) an adherent, smooth, greenish deposit which could not be removed with water and bristle brushing. This was apparently denickelification type of corrosion.

The specimens exposed to process supernatant solution at elevated temperatures (boiling) had a faster corrosion rate than those exposed at room temperature. Four specimens (HC-10, HC-16-18) exposed to the S.N. solution showed evidence of limited pitting while only one specimen (HC-14) exposed to HI pitted. Of these all but HC-10 (rolled-welded-sandblasted) were cast-ground specimens; HC-10 and 14 were room temperature tests while the others were run at elevated (boiling) temperatures.

On the basis of the results from these tests, i.e., erratic pitting of specimens and the formation of corrosion products which would likely contaminate the process, the use of Hastelloy C as a material of construction for the 234-5 project is not recommended.

Tantalum

This metal proved to be most resistant to these test corrosives and conditions and can be recommended for use with the 234-5 project process streams.

WWK/so

(1) By weight.

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BIBLIOGRAPHY

Uhlig, H. H., Corrosion Handbook, John Wiley & Sons, Inc., New York, N.Y.

Koenig, W. W., HW-2351-T - Metallurgy Laboratory 234-5 Project Corrosion Data, (Notebook).

Durco Alloys and Equipment, The Duriron Company, Dayton, Ohio

Hastelloy, The Haynes Stellite Company, Kokomo, Ind.

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TABLE I

METAL AND ALLOY DATA

Metal or Alloy	Nominal Composition	Physical and Mechanical Characteristics	Special Characteristics
DURIRON (High silicon iron)	Silicon 14.50% Carbon 0.85% Manganese 0.50% Sulfur 0.08% Phosphorus 0.20%	Density 7.0 Rockwell C 52 Tensile strength psi ($\frac{1}{2}$" dia. bar): 16,000 Casting shrinkage/ft.: 3/16" Coefficient of expansion: 12.2×10^{-6} per deg. C. 20° to 100°C. Nat. Bur. Stds.	Very hard. Machined by grinding. Available in cast form. Relatively inexpensive.
HASTELLOY C (Nickel-molybdenum-chromium-iron alloy)	Nickel 54.5-59.5% Molybdenum 15-19% Carbon 0.04-0.15% Iron 4-7% Chromium 13-16% Tungsten 3.5-5.5%	Density 8.94 Brinell: Cast: 175-215 Rolled annealed: 160-210 Ultimate tensile strength, lb. per sq. in.: Cast: 72,000-80,000 Rolled-annealed: 115,000-128,000 Yield point, lb. per sq. in.: Cast: 45,000-48,000 Rolled-annealed: 55,000-65,000 Elongation, % in 2": Cast: 10-15 Rolled-annealed: 25-50 Melting range, °C: 1,270-1,305 Thermal conductivity, cgs. 0.03 Specific heat: 0.092 Mean Coeff. of thermal expansion, per °C.: 0-100°C. 0.0000113 0-1000°C. 0.0000153 Casting shrinkage, in. per ft. $\frac{1}{4}$"	Machinable at moderate speeds and can be welded by the oxy-acetylene, atomic hydrogen, or metallic arc method. Corrosion resistant to strong oxidizing agents such as nitric acid (conditions important), free chlorine, aqueous solutions containing chlorine or hypochlorites, and acid solutions of ferric or cupric salts. Resists hydrochloric and phosphoric acid; is highly resistant to acetic, formic, and sulphurous acids; and has excellent resistance to dry battery mix. Available as castings and hot-rolled sheet or plate.

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TABLE I (continued)

METAL AND ALLOY DATA

Metal or Alloy	Nominal Composition		Physical and Mechanical Characteristics		Special Characteristics
TANTALUM	Tantalum	99.96%	Density 16.6		Corrosion resistant metal used in heat-transfer area in acid-proof chemical equipment. It oxidizes in air above 300°C (570°F), is soluble in hydrofluoric acid, in strong alkalies and in solutions that contain free sulfur trioxide. In general, it is corrosion resistant except when subject to galvanic couple action. Tantalum can be spot and seam resistance welded.
	Chromium	0.03%	Melting point	2996°C	
	Iron	0.01%	Specific heat	0.036	
			Thermal conductivity, cgs.	0.130	
			Thermal expansion, per °C	65 x 10^{-7}	
			Machinability - like cold rolled steel.		
			Rockwell E:		
			Sheet, annealed	60	
			Sheet, worked	95	
			Elongation, % in 2":		
			Sheet, annealed	40	
			Sheet, worked	1	
			Tensile strength, psi:		
			Sheet, annealed	50,000	
			Sheet, worked	110,000	

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TABLE II

COMPOSITION OF CORROSIVES

1. 234-5 Project Process Supernatant Solution (Synthetic):

$Fe(NO_3)_3 \cdot 9H_2O$	1.616 gm/liter
$Ce(NO_3)_3 \cdot xH_2O$	0.362 "
$NiSO_4 \cdot 6\ H_2O$	0.184 "
$KHSO_4$	8.17 "
KH_2PO_4	0.272
KNO_3	36.4 "
HI (Anhydrous basis)	103.7 "
I_2	33.0 "
$HO_2C \cdot CO_2H$	27.7 "

2. Hydriodic acid: Merck reagent, Sp. Gr. 1.7:

Assity, HI	55-58%
Maximum impurities:	
Non-volatile	0.010%
Chlorine, bromine (as Cl)	0.050%
Phosphorus	0.003%
Sulfate (SO_4)	0.005%
Heavy metals (as Pb)	0.001%
Iron (Fe)	0.001%
Mol. Wt.	127.93

3. Hydriodic Acid (Stabilized): Baker's Reagent, Sp. Gr. 1.7:

Assay, HI (min.)	56%
Maximum impurities:	
Chlorine and bromine (as Cl)	0.05%
Sulfate (SO_4)	0.005%
Iron (Fe)	0.001%
Heavy metals (as Pb)	0.001%
H_3PO_2 (Preservative) approx.	3.0%

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TABLE III

SPECIMEN, EXPOSURE AND CORROSION RATE DATA FOR DURIRON, HASTELLOY C AND TANTALUM IN HYDRIODIC ACID AND 234-5 PROJECT SUPERNATANT SOLUTION (SNYTHETIC)

Specimen Data		Exposure Data				Corrosion Data (Rate Expressed in MPY-Mils Penetration per Year to Nearest Mil)						
Material(1)	Preparation(2)	Test	No.	Temp.	Corrosive	48 Hrs.	48 Hrs.	72 Hrs.	217 Hrs.	744 Hrs.	Cumulative	Remarks(3)
Duriron	C.S.	ST.,C-I.	HC-26	Rm.	47% HI(4)	10	X	+ 1	Nil(<1)	Nil	Nil(1079 hrs.)	Dull grey film
	C.S.	V.	HC-27	Rm.	"	53	X	+ 1	Nil	Nil	Nil(")	Dull grey film > HC-26, slight rust formation.
Hastel-	R.S.	ST.,C-I.	HC-1	Rm.	"	19	X	Nil	Nil	3	Nil(")	Grayish-green corrosion products (adherent)
loy C	R.S.	V.	HC-2	Rm.	"	7	X	2	5	5	2 (")	" " " , gradual etching with disappearance of deposits.
	R.S.	ST.,C-I.	HC-3	Rm.	S-N.	26	X	3	1	3	2 (")	Black and light-green deposits (adherent).
	R.S.	V.	HC-4	Rm.	"	33	X	10	Nil	7	1 (")	" " " " " , gray oxide spots.
	R.S.	C-I.	HC-5	Boiling(5)	"	77	35	X	X	X	56 (96 hrs.)	Green deposits, etching and partial deposit removal.
	R.S.	V.	HC-6	"	"	29	4	X	X	X	17 (96 hrs.)	Light green deposits (adherent).
	R.W.S.	ST. C-I.	HC-8	Rm.	47% HI	89	X	110	88	53	65 (1079 hrs.)	Black deposit.
	"	V.	HC-9	"	"	5	X	4	5	5	4 (")	Yellow-gray and green deposits, some rust.
	"	ST.,C-I.	EC-10	"	S-N.	46	X	22	3	13	4 (")	Black and green deposits (adherent), etch, weld pit.
	"	V.	HC-11	"	"	29	X	18	12	Nil	5 (")	Black and greenish deposits (adherent).
	"	C-I.	HC-12	Boiling	"	139	88	X	X	X	114(96 hrs.)	Light green deposits (adherent).
R.WX.S.	V.	HC-13	"	"	21	Nil	X	X	X	11 (")	Green deposits (adherent), weld macro-etched.	
C.G.	ST.,C-I.	HC-14	Rm.	47% HI	6	X	1	1	1	Nil(1079 hrs.)	Pc. slightly dulled, limited pitting, as cast hole attacked	
"	V.	HC-15	"	"	3	X	2	2	2	Nil(")	" " " , blackish deposits, " " "	
"	ST.,C-I.	HC-16	"	S-N.	4	X	2	2	2	1 (")	" " " , limited pitting, " " "	
"	V.	HC-17	"	"	Nil	X	Nil	Nil	Nil	Nil(")	" " " " " " " "	
"	C-I.	HC-18	Boiling	"	70	73	X	X	X	72 (96 hrs.)	" " " " " " " "	
"	V	HC-19	"	"	21	3	X	X	X	12 (")	Pc. dulled, greenish deposits (adherent) " " "	
C.W.S.	ST.,C-I.	HC-20	Rm.	47% HI	4	X	2	1	2	Nil(1079 hrs.)	Blackish deposits, brightening.	
"	V.	HC-21	"	"	6	X	7	2	3	Nil(")	" " , some etching.	
C.WX.S.	ST.,C-I.	HC-22	"	S-N.	4	X	1	Nil	1	Nil(")	Etching.	
"	V.	HC-23	"	"	Nil	X	Nil	+ 1	+ 1	Nil(")	Grayish and greenish deposits.	
"	C-I.	HC-24	Boiling	"	59	63	X	X	X	61 (96 hrs.)	Green deposits, macro-etch.	
"	V.	No sample available			-	-	-	-	-	-	-	

TABLE III (continued)

SPECIMEN, EXPOSURE AND CORROSION RATE DATA FOR DURIRON, HASTELLOY C AND TANTALUM IN HYDRIODIC ACID AND 234-5 PROJECT SUPERNATANT SOLUTION (SYNTHETIC)

Specimen Data		Exposure Data				Corrosion Data (Rate Expressed in MPY-Mils Penetration Per Year to Nearest Mil)						
Material(1)	Preparation(2)	Test	No.	Temp.	Corrosive	48 Hrs.	48 Hrs.	48 Hrs.	120 Hrs.	384 Hrs.	Cumulative	Remarks(3)
Tantalum	Sheet, as rec'd.	ST.,C-I.	T-1A	Rm.	HI (1.5% H_3PO_2)	Nil(<1)	Nil	X	X	Nil	Nil (480 Hrs.)	
"	"	ST.,C-I.	T-1B	Rm.	47% HI	"	"	X	Nil	X	Nil (216 Hrs.)	
"	"	V.	T-2A	Rm.	HI (1.5% H_3PO_2)	"	"	X	X	Nil	Nil (480 Hrs.)	
"	"	V.	T-2B	Rm.	47% HI	"	"	X	Nil	X	Nil (216 Hrs.)	
"	"	ST., C-I.	T-3A	Rm.	S-N(1.5%H_3PO_2-HI)	"	"	X	X	Nil	Nil (480 Hrs.)	
"	"	ST.,C-I.	T-3B	Rm.	S-N	"	"	X	Nil	X	Nil (216 Hrs.)	
"	"	V.	T-4A	Rm.	S-N(1.5%H_3PO_2-HI)	"	"	X	X	Nil	Nil (480 Hrs.)	
"	"	V.	T-4B	Rm.	S-N	"	"	X	Nil	X	Nil (216 Hrs.)	
"	"	C-I.	T-5A	Boiling	HI (1.5% H_3PO_2)	"	"	Nil	X	X	Nil (144 Hrs.)	
"	"	C-I.	T-5B	Boiling	47% HI	"	"	"	X	X	"	
"	"	V	T-6A	Boiling	HI (1.5% H_3PO_2)	"	"	"	X	X	"	
"	"	V.	T-6B	Boiling	47% HI	"	"	"	X	X	"	
"	"	C-I.	T-7A	Boiling	S-N(1.5%H_3PO_2-HI)	"	"	"	X	X	"	
"	"	C-I.	T-7B	Boiling	S-N	"	"	"	X	X	"	
"	"	V.	T-8A	Boiling	S-N(1.5%H_3PO_2-HI)	"	"	"	X	X	"	
"	"	V.	T-8B	Boiling	S-N	"	"	"	X	X	"	

(1) See Table I, Appendix, pp. 5-6, "Metal and Alloy Data."

(2) Legend: C. = cast; C-I. = complete immersion; G. = ground finish; HI. = Hydriodic acid, see Table II, Appendix, p.; R. = Rolled; S. = Sandblasted; S-N. = Process supernatant solution (synthetic). See Table II, Appendix, p. 7.; ST. = static test; V. = vapor; W. - welded; WX. - weld ground flush.

(3) Adherent as used in remarks signifies that the corrosion products could not be removed by water and bristle brush. In general, the black corrosion products could be washed off, the greenish corrosion products were adherent (apparently denickelification).

(4) All percentages are calculated on a by weight basis. See Table II, Appendix, p. 7, "Composition of Corrosives."

(5) Boiling tests are naturally agitated by convection currents and solution bumping. All boiling solutions were changed every 24 hours.

END