CORROSION STUDIES ON TITANIUM AND ZIRCONIUM METALS

L. B. Golden, I. R. Lane, Jr., E. V. Rhoderick, J. T. Pons, W. R. Acherman and W. Mace

Semiannual Report for December, 1950:

This investigation was initiated in June, 1947 to obtain quantitative data on the corrosion resistance of titanium and zirconium metals and their alloys to acids, bases, salts, and various organic compounds. Since, from the utilitarian viewpoint, titanium may become a successful replacement for stainless steel, one of the most corrosion-resistant varieties, Carpenter No. 20, has been used in parallel tests to obtain a basis for comparison. Also, since for certain purposes, zirconium may become a replacement for the much less abundant tantalum metal and in addition possesses unique properties of its own, extensive tests have been made on the corrosion resistance of this metal.

During the past six months tests have been made on the corrosion resistance of zirconium and zirconium alloys in solutions of hydrochloric acid (embrittlement tests), sulfuric acid, phosphoric acid, and organic acids. A comparison was made of the relative corrosion resistance of arc-melted zirconium and zirconium induction-melted in graphite. Titanium was tested in sulfuric and nitric acids and in trisodium phosphate and stannic chloride solutions. Stainless steel was tested in sulfuric, nitric, hydrochloric, and phosphoric acids, in sulfuric-nitric acid mixtures and in aqua regia. The following compounds were used in carrying out tests on titanium, zirconium, and stainless steel: cupric, mercuric, nickel, manganese, stannic, aluminum, calcium, sodium, and ammonium chlorides. These three metals were also tested in various chlorinated hydrocarbon-water mixtures. A series of zirconium alloys was tested in various rocket fuels and also submitted to thirty day salt spray tests in 3 percent sodium chloride solution and in synthetic ocean

water. A discussion of the various tests follows.

Zirconium and zirconium alloys - hydrochloric acid (embrittlement tests):

Previous tests at 35°C, 60°C, and 100°C in aerated hydrochloric acid solutions from 1 to 20 percent concentration gave negligible corrosion rates and no signs of embrittlement for ordinary purity zirconium induction melted in graphite. However, when tested in non-aerated and static concentrated (37 percent) acid at 35°C for six days the metal was embrittled although the corrosion rate was low (3.02 m.p.y.). In order to determine whether this embrittlement was typical of zirconium in all non-aerated hydrochloric acid solutions or whether the susceptibility to this type of corrosive attack was confined to the particular lot of metal tested, a series of tests was run in non-aerated and static 1, 5, 10, 15, and 20 percent hydrochloric acid solutions at 35°C, 60°C, and 100°C for thirty days. Corrosion rates for these tests were all less than 0.11 m.p.y. and there were no signs of embrittlement (Table I). The test in non-aerated and static concentrated (37 percent) acid at 35°C was repeated and ran for thirty days instead of six days using a more recent lot of zirconium metal. The rate for this thirty day test was only 0.32 m.p.y. as compared to 3.02 m.p.y. for the earlier six day test, and there was no evidence of embrittlement (using a manual bending test).

The difference in behavior of zirconium metal in concentrated hydrochloric acid in these two tests may possibly be attributed to differences in the purity of the separate lots of metal.

In further investigation of this problem tests were conducted in concentrated hydrochloric acid using six lots of zirconium metal, two zirconium alloys, and tantalum metal. The lots of zirconium metal designated SA 1107, SA 1109, and SA 1110 were hot rolled from arc melted ingots produced from high purity (Y-12) oxide. Lots S-1145-C, S-1147-C, and S-1151-C

were from the same source material induction melted in graphite. The two zirconium alloys (W-1060 and W-1071) were also induction melted in graphite and contained respectively 1.08 percent tantalum and 3.70 percent columbium. These alloys were in the cold rolled condition. The tantalum metal used was in the annealed condition and was obtained from the Fansteel Metallurgical Corporation.

In concentrated (37 percent) hydrochloric acid at room temperature for thirty days arc melted zirconium showed no signs of corrosion or embrittlement and corrosion rates were all less than 0.01 mils per year. In contrast, the samples of zirconium melted in graphite were evenly etched showing dendrites (presumably of zirconium carbide) throughout the samples. (Figures 1 and 2). Rates for these etched samples were respectively 0.38, 0.45, and 0.49 mils per year and no signs of embrittlement were apparent. The tantalum and columbium alloys showed no visible signs of corrosion or embrittlement, giving rates of 0.02 and 0.01 mils per year, respectively.

Tests were then conducted in concentrated (37 percent) hydrochloric acid at 60°C for six days. The samples were sealed in glass tubes half filled with acid (50 ml.) and tested under the pressure developed at this temperature (about 3 atmospheres). The arc melted zirconium samples showed no signs of corrosion or embrittlement with corrosion rates of 0.09 to 0.30 mils per year. (Table II). However, the zirconium samples melted in graphite were badly embrittled giving rates ranging from 14.0 to 164 mils per year. A sample of ordinary purity zirconium (induction melted in graphite) was completely disintegrated to a fine powder. The tantalum alloy suffered relatively slight surface embrittlement, while the columbium alloy showed no signs of embrittlement. The tantalum metal showed no signs of corrosion or embrittlement.

The above tests were repeated at 100°C at which temperature the pressure developed was calculated to be about 11.5 atmospheres. This

increase in temperature and pressure had no effect on the arc melted zirconium samples since they still showed no visible signs of corrosion or embrittlement (Figure 3). However, the zirconium samples melted in graphite were badly embrittled (Figure 4). Sample S-1147-C was not quite as severly embrittled as samples S-1145-C and S-1151-C. The two samples representing the zirconium-tantalum alloy (W-1060) were, for the most part, reduced to a fine powder, being completely embrittled. The two samples of zirconium-columbium alloy (W-1071) showed only a slight darkening of their surfaces and very small and shallow embrittled areas on their surfaces (Figure 5). The two tantalum samples showed no visible signs of corrosion and no signs of embrittlement (Figure 6). From these results it appears that arc melted zirconium may serve equally as well as tantalum metal in industrial applications requiring resistance to the corrosive action of hydrochloric acid.

Arc melted zirconium versus zirconium induction melted in graphite and zirconium alloys:

Three lots of arc melted zirconium, three of zirconium induction melted in graphite and two zirconium alloys melted in graphite were tested for comparison purposes in red fuming nitric acid, mixed acid, 80 percent sulfuric acid, 20 percent ferric chloride solution, concentrated phosphoric acid, and fuming sulfuric acid. The results in hydrochloric acid have already been described in the preceding section and the description of the two types of zirconium and the two alloys may also be found there.

In red fuming nitric acid at room temperature for 30 days arc melted zirconium was superior in corrosion resistance to zirconium induction melted in graphite. Of the three arc melted zirconium samples tested weight gains ranged from 0.4 to 1.7 milligrams. For the three samples melted in graphite the figures were from 24.7 to 78.9 milligrams and their surfaces were covered with loosely adhering, flaky, gray-black film. Average weight gains for the tantalum and columbium alloys were 0.4 and 1.5 milligrams, respectively, and

like the arc melted zirconium samples showed no signs of corrosion other than varicolored films on the surfaces of the samples.

In mixed acid (84 percent white fuming nitric acid plus 14 percent fuming sulfuric acid) at room temperature for 30 days there was little difference between the corrosion resistance of arc melted zirconium and zirconium melted in graphite. The average rate for the former was 20.5 mils per year compared with 23.2 mils per year for the latter. However, rates were much lower for the tantalum and columbium alloys, being respectively, 1.08 and 4.72 mils per year.

Tests were made in aerated 80 percent sulfuric acid at 35°C for six days on the high purity zirconium samples and the two alloys. At the same time tests were run on ordinary purity zirconium (induction melted in graphite). The arc melted high purity zirconium was far superior in corrosion resistance to both the high purity zirconium induction melted in graphite and the ordinary purity zirconium. The average corrosion rate for the arc melted zirconium was 1.20 mils per year, that for the high purity zirconium melted in graphite was 70.9 mils per year, while that for ordinary purity zirconium melted in graphite was 23.5 mils per year. The tantalum and columbium alloys were not as resistant as the arc melted zirconium, the rates being 29.8 and 5.62 mils per year, respectively.

Tests were also made in aerated 20 percent ferric chloride solution at 35°C for six days. The arc melted high purity zirconium was far superior in corrosion resistance to the high purity zirconium induction melted in graphite and appreciably better than the ordinary purity zirconium induction melted in graphite and the average corrosion rates were respectively, 6.83, 107.6, and 11.6 mils per year. The tantalum and columbium alloys were not as resistant as the arc melted zirconium samples, the rates being 12.6, and 15.3 mils per year, respectively.

In contrast to the results obtained in the other solutions discussed above, high purity zirconium induction melted in graphite was found to be superior in corrosion resistance to both high purity arc melted zirconium and ordinary purity zirconium melted in graphite when tested in aerated, concentrated (85 percent) phosphoric acid at 100°C for six days. Average corrosion rates were respectively, 31.4, 71.2, and 52.6 mils per year. The tantalum and columbium alloys were not as resistant as the high purity zirconium melted in graphite, the rates being 41.1 and 44.5 mils per year, respectively.

Tests in fuming sulfuric acid at 35°C for six days showed that high purity zirconium induction melted in graphite was superior in corrosion resistance to high purity arc melted zirconium and ordinary purity zirconium melted in graphite. Average corrosion rates were respectively, 111, 811, and 646 mils per year. The tantalum and columbium alloys were not as resistant as the high purity zirconium melted in graphite, the rates being 510 and 470 mils per year, respectively.

Titanium, zirconium, zirconium alloys, and stainless steel - concentrated hydrogen peroxide:

Samples of titanium prepared by three different methods (powder metallurgy, induction melted in graphite, and arc melted), were tested in concentrated (90 percent) hydrogen peroxide at 35°C. Three types of zirconium (ordinary purity induction melted in graphite, high purity arc melted, and high purity induction melted in graphite), four different zirconium-titanium alloys, three zirconium-iron alloys, a zirconium-tantalum alloy, and a zirconium-columbium alloy were also tested. All of the zirconium alloys tested were induction melted in graphite. Zirconium samples designated as SA-1109 and SA-1110 were high purity arc melted material, while S-1151-C was high purity material induction melted in graphite.

As seen from the results in table III all types of titanium were only slightly resistant to the corrosive action of the peroxide with the arc melted titanium giving the lowest rate. Moreover, all three samples accelerated the catalytic decomposition of the peroxide with the arc melted metal having the least effect. From the standpoint of corrosion resistance and compatibility, arc melted titanium is but little better than either powder metallurgy titanium or titanium induction melted in graphite.

The attack by the hydrogen peroxide on the titanium metal (all types) was characterized by the formation of an amorphous yellow precipitate of TiO_3 which settled out of the solution. This is in direct contrast to previously reported results (monthly report for April 1948 and semiannual report, December 1948) where titanium was practically unattacked by concentrated hydrogen peroxide. Further tests will be made to determine whether titanium can be made compatible with concentrated peroxide.

All of the zirconium and zirconium alloy samples tested caused less decomposition of the peroxide than was caused by Carpenter No. 20 stainless steel (7.40 percent decomposition per month) and corrosion rates were in all instances either zero or negligible.

Titanium - sulfuric and nitric acid:

Titanium was tested at 100°C for six days in aerated 1, 2, 3, 4, and 5 percent sulfuric acid. These tests were also repeated in the same concentrations of acid but aerated with oxygen-free nitrogen instead of air. Titanium was also tested at 60°C for six days in aerated 1, 2, 3, and 4 percent sulfuric acid. Tests at 100°C in various concentrations of aerated nitric acid were also made. Corrosion rates for these tests are presented in table IV.

Zirconium - boiling phosphoric and sulfuric acid solutions:

Zirconium was tested for six days in boiling phosphoric acid

solutions ranging in concentration from 10 to 85 percent and in sulfuric acid solutions ranging from 60 to 85 percent. Results are recorded in table V.

Zirconium - sulfuric acid:

Zirconium was tested at 35°C in sulfuric acid solutions ranging in concentration from 10 to 96.6 percent. These solutions were kept saturated with oxygen-free nitrogen. In concentrations from 30 to 85 percent rates obtained in nitrogen-aerated solutions were all lower than the corresponding rates in air-aerated solutions. These differences are most clearly shown in the 80, 82.5, and 85 percent acid solutions. (Table VI.)

Stainless steel - sulfuric and nitric acid mixtures:

Carpenter No. 20 stainless steel was tested at 35°C, 60°C, and 100°C for six days in various mixtures of concentrated sulfuric and nitric acids ranging from 99 percent sulfuric plus 1 percent nitric acid to 1 percent sulfuric plus 99 percent nitric acid. Results of these tests are shown in table VII.

Stainless steel - inorganic acids:

Carpenter No. 20 stainless steel was tested at 35°C, 60°C, and 100°C for six days in various concentrations of aerated sulfuric, nitric, hydrochloric, and phosphoric acids and in nitrogen aerated (oxygen-free) solutions of hydrochloric and sulfuric acids. Results of these tests are presented in tables VIII and IX.

Zirconium and stainless steel - boiling phosphoric acid solutions:

Zirconium and Carpenter No. 20 stainless steel were tested for six days in boiling phosphoric acid solutions ranging in concentration from 10 to 85 percent. As seen in table X zirconium gave lower corrosion rates than stainless steel in concentrations of acid up to 50 percent but above this concentration the steel was far superior.

Stainless steel - aqua regia:

Carpenter No. 20 stainless steel was tested in aqua regia (3 parts concentrated hydrochloric acid plus 1 part nitric acid) at room temperature. All of the samples tested were almost completely dissolved in one hour. This is in contrast to titanium which is completely resistant to attack by this mixture.

Titanium, zirconium, and stainless steel - inorganic chlorides:

The results of tests on titanium, zirconium, and Carpenter No. 20 stainless steel in twelve different inorganic chloride solutions at 35°C, 60°C, and 100°C are summarized in table XI.

Neither zirconium nor stainless steel have adequate corrosion resistance to cupric chloride solutions. Zirconium suffered embrittlement in these solutions while the steel samples were badly pitted and perforated with many large holes. Mercuric chloride solutions also badly pitted and perforated stainless steel while having no effect on either titanium or zirconium. Nickel chloride and manganese chloride solutions both caused pitting in stainless steel while titanium was unaffected. Titanium and zirconium were both unaffected by stannic chloride and aluminum chloride solutions. However, stainless steel was corroded in aluminum chloride solutions of high concentration, especially at elevated temperatures. Corrosion was uniform with no pitting. Zirconium showed no signs of corrosion in calcium chloride solutions. When tested in 5 percent calcium chloride solution at 100°C several small blisters formed on the surface of the titanium metal. These solutions caused pitting in the stainless steel samples. Zirconium and stainless steel showed no signs of corrosion in 3 percent sodium chloride solutions. A few small blisters were present on the titanium samples tested at 100°C. One of the stainless steel samples tested at 35°C in 10 percent ammonium chloride solution developed a small, elongated pit on its surface.

Titanium - trisodium phosphate:

Tests at 100°C for six days in 5 percent and saturated solutions of trisodium phosphate gave zero corrosion rates.

Titanium, zirconium, and stainless steel - organic compounds:

Titanium, zirconium, and Carpenter No. 20 stainless steel were tested in various organic compounds. The results of these tests are recorded in table XII. The only appreciable corrosion rates were those obtained for zirconium in boiling dichloro- and trichloro- acetic acids.

Titanium, zirconium, and stainless steel - chlorinated hydrocarbon - water mixture:

Titanium, zirconium, and Carpenter No. 20 stainless steel were tested in six different chlorinated hydrocarbon-water mixtures boiling under reflux for six days. Each metal had a pair of samples exposed with half of their areas in the hydrocarbon layer and the other half in the water layer, while another pair of samples was suspended above the water layer and exposed to the vapors. A blank was run at the same time.

In the carbon tetrachloride-water mixture average corrosion rates for titanium and zirconium were very low while that for the stainless steel was much higher (table XIII). Corrosion rates in the vapor zone were less for titanium and zirconium than in the liquid zone, while the opposite was true for the stainless steel samples. Also the steel samples showed staining and pitting where the carbon tetrachloride-water interface was in contact with the sample surface. The titanium and zirconium samples showed no visible signs of corrosion.

Furthermore, these two samples had no catalytic effect upon the decomposition of carbon tetrachloride since the acidity developed in the water layer during the test was no greater than that developed in the blank. The stainless steel, however, did catalyze the decomposition of the carbon

tetrachloride as was shown by the development of appreciable acidity in the water layer.

Tests using the other five chlorinated hydrocarbons gave very low average corrosion rates for all three metals and there was no appreciable difference between rates in the vapor zone and the liquid zone. There was little or no catalytic effect upon the decomposition of these compounds.

Zirconium alloys - rocket fuels:

Three zirconium-iron alloys containing respectively 2.6, 4.0, and 1.2 percent iron were tested in the following solutions: red fuming nitric acid, white fuming nitric acid, mixed acid (84 percent white fuming nitric acid plus 14 percent fuming sulfuric acid), methyl, ethyl, and furfuryl alcohols, gasoline (both white and leaded), xylidine, a mixture of 70 percent xylidine and 30 percent gasoline, a mixture of 65 percent aniline and 35 percent furfuryl alcohol, and 100 percent aniline. These tests were made at room temperature for 30 days. In red fuming nitric acid these three alloys all showed gains in weight (up to 8.5 mgms.) caused by the formation of a gray-black film on their surfaces. However, in white fuming nitric acid these samples showed no visible signs of corrosion. In mixed acid these alloys were covered with a gray film and gave corrosion rates of 1.11, 1.38, and 1.10 mils per year, respectively. In the organic solutions listed above none of these alloys showed any signs of corrosion.

Forty-one of the eighty-eight zirconium alloys furnished by the Foote Mineral Company were tested in white fuming nitric acid at room temperature for thirty days. The results of these tests are summarized in table XIV together with the composition of these alloys. Because of the irregular shapes of most of the samples tested and because gains in weight were encountered as often as losses, results were expressed in weight gain or loss in mgms. together with a description of the appearance of the samples and

any changes in physical properties noted.

Eighty-five of these alloys were tested in xylidine, 82 in aircraft fuel (JP-3), 76 in aniline, 38 in a mixture of 65 percent aniline plus 35 percent furfuryl alcohol, 30 in methyl alcohol, and 25 in leaded (ethyl) gasoline. None of these organic solutions had any effect on the alloys which were tested at room temperature for 30 days.

Zirconium and zirconium alloys - salt spray tests:

Samples of zirconium, four zirconium-titanium alloys containing respectively 10.3, 32.9, 46.4, and 71.8 percent titanium and three zirconium-iron alloys containing respectively 2.6, 4.0, and 1.2 percent iron were exposed to 3 percent sodium chloride spray and also to a substitute ocean water spray (A.S.T.M. designation D 1141-50 T) in a salt spray cabinet at room temperature for thirty days. Except for very slight gains and losses in weight the tests had no effect whatsoever on any of the samples.

Eighty-four of the zirconium alloys furnished by the Foote Mineral Company were exposed to 3 percent sodium chloride spray and also to substitute ocean water spray in a salt spray cabinet at room temperature for thirty days. With the exception of eight samples in the 3 percent sodium chloride spray test the test had no effect whatsoever on the samples tested other than very slight gains and losses in weight. Weight losses among these eight samples ranged from 5.0 to 33.8 milligrams and were caused by pit formations.

The compositions of these alloys and the weight gains or losses in grams are recorded in table XV. Results were expressed in this manner because of the irregular shapes of most of the samples tested. A description of the samples effected by the 3 percent sodium chloride spray follows: Sample 31 had several small pits filled with salt. Sample 50 had large areas covered with rust spots. There were several large pits filled with gray-black

powder underneath the rust spots. This powder was probably unalloyed silicon and the rust was probably derived from iron in the silicon. Sample 53 had two large pits or cracks filled with salt. Samples 54 and 56 had small pits on one edge. Sample 330 had large areas on one side covered with rust. This coating appeared to be superficial and not derived from the sample. Sample 408, although having an appreciable weight loss, showed no visible signs of corrosion. Sample 423 had one large shallow pit. All other samples showed no visible signs of corrosion.

Future program:

The program for the immediate future will include a continuation of tests on titanium and zirconium metals and their alloys with inorganic and organic compounds at different concentrations and temperatures. Tests will be made on a series of arc-melted zirconium alloys in concentrated hydrochloric acid at elevated temperatures under pressure (embrittlement tests). A comparison of the relative corrosion resistance of arc-melted metals (both titanium and zirconium) and metals induction-melted in graphite will be continued. Zirconium alloys will be tested in materials used for rocket fuels and in simulated marine atmosphere (salt spray) tests.

However, the greatest portion of time and effort for the immediate future will be spent in conducting galvanic corrosion research on titanium and zirconium and their alloys.

Table I. Zirconium - Hydrochloric Acid

Test solution * (percent by weight)	Average corrosion rate, 6-day run, mils per year		
	35°C	60°C	100°C
1	0.02	0.04	0.03
5	< 0.01	0.07	0.01
10	0.06	0.10	0.06
15	0.08	0.11	0.06
20	0.07	0.10	0.05
37	0.32	-	-

*All solutions non-aerated and static.
Specimen configuration - 1/2" x 2" x 0.040".

Table II. Hydrochloric Acid Embrittlement Tests

Sample Number	Composition (percent by weight)	Average corrosion rate, mils per year	
		60°C	100°C
SA-1107	100 Zr (arc melted)	0.30	0.13
SA-1109	100 Zr (arc melted)	0.09	0.08
SA-1110	100 Zr (arc melted)	0.17	0.11
S-1145-C	100 Zr (induction melted)	164	386
S-1147-C	100 Zr (induction melted)	14.0	55.2
S-1151-C	100 Zr (induction melted)	68.8	199
W-1060	1.08 Ta - rem. Zr.	8.15	#
W-1071	3.70 Cb - rem. Zr.	0.64	2.47
CI-37	100 Zr *(induction melted)	#	-
TA	100 Ta	0.03	0.06

Embrittled and disintegrated.
* Ordinary purity zirconium.
Specimen configuration - 1/2" x 2" x 0.040".

Table III. Titanium, Zirconium, Zirconium Alloys, and Stainless Steel - Concentrated (90 percent) Hydrogen Peroxide (at 35°C)

Sample No.	Composition (percent by weight)	Average Corrosion rate (mils per year)	Decomposition of 90% H_2O_2 (% per month)
Ti (P.M.)*	100 Ti	163	45.4#
Ti (I.M.)	100 Ti	216	61.6#
Ti (A.M.)	100 Ti	105	32.1#
Zr(O.P.)	100 Zr	0.00	2.16
20 S.S.	20 Cr-29 Ni-2 Mo-3 Cu	0.00	7.40
1001 B	10.3 Ti - bal Zr	0.04	2.79
1002 A	32.9 Ti - bal Zr	0.06	4.48
1003 B	46.4 Ti - bal Zr	0.06	3.57
1004 A	71.8 Ti - bal Zr	0.01	6.11
W 1009 A	2.6 Fe - bal Zr	0.01	3.11
W 1011 A	4.0 Fe - bal Zr	0.01	2.39
W 1012	1.2 Fe - bal Zr	0.03	2.14
SA 1109	100 Zr	0.03	2.13
SA 1110	100 Zr	0.06	1.56
S 1151 C	100 Zr	0.13	0.60
W 1060	1.08 Ta - bal Zr	0.08	0.00#$_a$
W 1071	3.70 Cb - bal Zr	0.11	0.14

Percent decomposition per week.

#$_a$ Lower rate than blank.

* P.M. = Powder metallurgy.
I.M. = Induction melted in graphite.
A.M. = Arc melted.
O.P. = Ordinary purity induction melted in graphite.
Specimen configuration - 1" x 1" x 0.050"

Table IV. Titanium - Sulfuric and Nitric Acids

Solution (percent by weight)	Average Corrosion Rate Mils per year		
	60°C[1]	100°C[1]	100°C[2]
1 H_2SO_4	0.32	0.19	282
2 H_2SO_4	0.30	735	654
3 H_2SO_4	0.54	920	830
4 H_2SO_4	69.5	840	929
5 H_2SO_4	-	811	1065
10 HNO_3		0.92	
20 HNO_3		1.51	
30 HNO_3		4.05	
40 HNO_3		2.34	
50 HNO_3		7.40	
60 HNO_3		1.91	
69.5 HNO_3		0.74	

[1] Aerated with air - rate 250 ml/min.
[2] Aerated with O_2-free N_2 - rate 100 ml/min.
Specimen configuration - 1" x 1" x 0.050".

Table V. Zirconium - Boiling Phosphoric and Sulfuric Acid Solutions

Solution (percent by weight)	Average Corrosion Rate Mils per year	
	Boiling H_3PO_4	Boiling H_2SO_4
10	0.16	
20	0.32	
30	0.58	
40	1.19	
50	2.67	
60	7.29	0.88
70	23.4	0.56
75	48.6	30.3
80	229	270
82.5	350	570
85	1095	-*

*Completely dissolved in less than one hour.
Specimen configuration - 1/2" x 2" x 0.040".

Table VI. Zirconium - Sulfuric Acid (35°C)

Solution (percent by weight)	Average Corrosion Rate, mils per year	
	Nitrogen Aerated*	Air Aerated*
10	0.11	0.05
20	0.17	0.08
30	0.15	0.34
40	0.12	0.41
50	0.09	0.37
60	0.08	0.42
70	0.07	0.34
80	0.41	50.3
82.5	11.6	242
85	303	864
90	>1450[1]	1620[2]
96.5	771	752

* Rate: Nitrogen - 100 ml. per minute.
Air - 250 ml. per minute.

1/ Completely dissolved in less than 6 days.
2/ Three day run.

Table VII. Stainless Steel - Sulfuric and Nitric Acid Mixtures

Test solution (percent by weight)	Average Corrosion rate, 6-day run, mils per year		
	35°C	60°C	100°C
99 H_2SO_4 + 1 HNO_3	0.19	0.73	1[illegible]
95 H_2SO_4 + 5 HNO_3	0.31	1.36	25.8
90 H_2SO_4 + 10 HNO_3	0.34	2.05	38.7
80 H_2SO_4 + 20 HNO_3	0.48	1.36	24.8
70 H_2SO_4 + 30 HNO_3	0.47	1.30	15.7
60 H_2SO_4 + 40 HNO_3	0.28	1.13	13.3
50 H_2SO_4 + 50 HNO_3	0.19	1.00	7.2
40 H_2SO_4 + 60 HNO_3	0.17	[illegible]	[illegible]
30 H_2SO_4 + 70 HNO_3	0.09	0.70	[illegible]
20 H_2SO_4 + 80 HNO_3	0.04	0.[illegible]3	4.14
10 H_2SO_4 + 90 HNO_3	0.05	0.36	[illegible]
5 H_2SO_4 + 95 HNO_3	0.05	0.43	[illegible]
1 H_2SO_4 + 99 HNO_3	0.04	0.43	[illegible]

All solutions non-aerated and static.
Specimen configuration - 1/2" x 2" x 0.067".

Table VIII. Stainless Steel - Sulfuric Acid

Test solution (percent by weight)	Average corrosion rate, 6-day run, mils per year				
	35°C[1]	35°C[2]	60°C[2]	100°C[2]	Boiling
10	0.91	0.08	-	-	41.2
20	0.99	0.15	-	-	48.6
30	0.68	0.13	-	-	62.1
35	-	-	-	-	75.2
40	0.57	0.24	-	-	113
45	-	-	-	-	135
50	0.46	0.62	-	43.2	117
60	0.34	4.70	-	31.3	2570
65	-	-	-	-	7180
70	0.45	4.50	10.20	48.3	2570
75	0.36	2.72	7.41	43.2	15,700
80	2.14	4.72	6.15	42.5	16,200
82.5	-	3.70	6.59	-	19,500
85	1.56	4.72	6.80	-	22,800
90	1.78	17.60	6.42	-	-
96.5	3.57	16.70	11.30	-	-

[1] Aerated with O_2-free N_2 - rate 100 ml/min.
[2] Aerated with air - rate 250 ml/ min.
Specimen configuration - 1" x 1" x 0.067" (1/2" x 2" x 0.067" for boiling tests).

Table IX. Stainless Steel - Inorganic Acids

Test solution (percent by weight)	Average corrosion rate, 6-day run, mils per year		
	35°C	60°C	100°C
10 HNO_3	0.05	0.04	0.12
20 HNO_3	0.12	0.05	0.29
30 HNO_3	0.16	0.09	0.81
40 HNO_3	0.09	0.14	0.90
50 HNO_3	0.02	0.18	1.26
60 HNO_3	0.03	0.22	1.67
69.4 HNO_3	0.03	0.23	1.96
0.5 HCL	-	-	20.4
1.0 HCL	3.27	4.41	96.5
1.5 HCL	-	-	106
2.0 HCL	-	67.5	167
3.0 HCL	51.0	93.5	-
4.0 HCL	-	92.5	-
5.0 HCL	60.0	103	-
6.0 HCL	52.1	-	-
1.0 HCL#	1.83	-	-
3.0 HCL#	4.12	-	-
5.0 HCL#	4.96	-	-
6.0 HCL#	5.61	-	-
1.0 H_3PO_4	-	-	0.09
3.0 H_3PO_4	-	-	0.12
5.0 H_3PO_4	-	-	0.09
10 H_3PO_4	0.01	-	0.15
15 H_3PO_4	-	-	0.21
20 H_3PO_4	0.00	-	0.26
30 H_3PO_4	0.00	-	0.24
40 H_3PO_4	0.05	-	-
50 H_3PO_4	0.08	0.04	0.34
60 H_3PO_4	0.05	0.08	-
70 H_3PO_4	0.03	-	-
75 H_3PO_4	-	0.07	-
80 H_3PO_4	0.05	-	-
85 H_3PO_4	0.08	0.07	-

Aerated with O_2-free N_2 - rate 100 ml/min.
All other samples aerated with air - rate 250 ml/min.
Specimen configuration - 1" x 1" x 0.062" .

Table X. Zirconium and Stainless Steel - Boiling Phosphoric Acid Solutions

Solution (percent by weight)	Average corrosion rate, mils per year	
	Zr[1]	20 S.S.[2]
10	0.16	10.1
20	0.32	13.7
30	0.58	7.21
40	1.19	1.13
50	2.67	6.45
60	7.29	5.02
70	23.4	2.03
75	48.6	-
80	229	21.3
82.5	350	-
85	1095	54.0

[1] Zirconium.
[2] Carpenter No. 20 stainless steel.
Specimen Configuration - Zirconium - 1/2" x 2" x 0.040".
Stainless steel - 1/2" x 2" x 0.082".

Table XI. Titanium, Zirconium, and Stainless Steel - Inorganic Chlorides (with aeration)

Test solution (percent by weight)	Average corrosion rate, 6-day run, mils per year								
	Titanium			Stainless Steel			Zirconium		
	35°C	60°C	100°C	35°C	60°C	100°C	35°C	60°C	100°C
1.0 $CuCl_2$	-	-	-	-	-	59.3	-	-	17.5
2.5 $CuCl_2$	-	-	-	-	163	368	-	41.8	149
5.0 $CuCl_2$	-	-	-	-	1365	-	18.2	167	-
20.0 $CuCl_2$	0.04	-	-	-	-	-	-	-	-
5.0 $NiCl_2$	0.07	0.13	0.17	-	-	0.16	-	-	-
20.0 $NiCl_2$	-	0.16	0.11	-	-	2.13	-	-	-
30.0 $FeCl_2$	0.02	0.13	0.08	-	-	-	-	-	-
10 NaCl+5 $FeCl_3$	0.04	0.00	0.04	-	-	-	-	-	-
1.0 $HgCl_2$	-	0.02	0.01	-	0.02	0.33	-	0.00	0.00
5.0 $HgCl_2$	-	-	0.42	-	-	625	-	-	0.00
10.0 $HgCl_2$	-	-	-	-	-	-	-	-	0.00
5.0 $MnCl_2$	-	-	0.00	-	-	0.14	-	-	-
20.0 $MnCl_2$	-	-	0.00	-	-	1.78	-	-	-
5.0 $SnCl_4$	-	0.20	0.12	-	-	-	-	-	0.00
4.0 $SnCl_4$	-	0.16	0.41	-	-	-	-	-	0.13
5.0 $AlCl_3$	-	0.16	-	0.08	0.15	2.80	-	0.01	0.02
25.0 $AlCl_3$	0.04	-	-	5.00	15.6	-	0.00	0.00	0.24
5.0 $CaCl_2$	-	0.10	0.02	.03	0.09	7.70		0.03	0.01
25.0 $CaCl_2$	-	-		-	-	0.13	-	-	0.02
3.0 NaCl	-	0.0[illegible]		0.04	0.04	0.08	-	0.03	-
5.0 $BaCl_2$	-	-	0.00	-	-	0.10	-	-	-
20.0 $BaCl_2$	-	-	0.01	-	-	0.23	-	-	-
1.0 NH_4Cl	0.08	-	-	0.06		-	0.03	-	-
10.0 NH_4Cl	0.05	-	-	0.10	-	-	0.01	-	-

Specimen configuration - Titanium - 1" x 1" x 0.062"
Zirconium - 1" x 1" x 0.040"
Stainless Steel - 1" x 1" x 0.082"

Table XII. Titanium, Zirconium and Stainless Steel - Organic Compounds

Test solution (percent by weight)	Temp. °C	Average corrosion rate, 6-day run, mils per year		
		Titanium	Zirconium	Stainless Steel
10 formic acid	35	0.06	0.04	0.13
25 formic acid	35	0.08	0.05	0.13
50 formic acid	35	0.10	0.03	0.15
90 formic acid	35	0.02	0.04	0.13
50 formic acid 1/	35	0.25	0.17	0.19
90 formic acid 1/	35	-	0.24	-
50 formic acid 1/	60	0.27	0.07	0.13
90 formic acid 1/	60	0.00	0.00	0.87
50 formic acid 1/	100	0.10	-	-
90 formic acid 1/	100	0.00	-	-
10 lactic acid	100	-	0.03	-
50 lactic acid	100	-	0.05	-
85 lactic acid	100	-	0.07	-
25 citric acid	100	0.03	-	-
50 citric acid	100	0.05	-	-
50 tartaric acid	100	0.49	-	-
5 aniline • HCL	100	0.00	-	-
20 aniline • HCL	100	0.00	-	-
100 dichloroacetic acid	Boiling	0.29	8.45	-
100 trichloroacetic acid	Boiling	-	455	-

1/ Non-aerated and static.

Specimen configuration: - Titanium - 1" x 1" x 0.062"
Zirconium - 1" x 1" x 0.040"
Stainless Steel - 1" x 1" x 0.032"
(1/2" x 2" x 0.040" for boiling tests)

Table XIII. Titanium, Zirconium and Stainless Steel - Chlorinated Hydrocarbon - Water Mixture

Chlorinated hydrocarbon	Average corrosion rate, 6-day run, mils per year		
	Titanium	Zirconium	Stainless Steel
Carbon tetrachloride	0.18	0.18	2.08
Chloroform	0.01	0.03	0.04
Ethylene dichloride	0.18	0.11	0.12
Trichloroethylene	0.04	0.03	0.01
Tetrachloroethylene	0.02	0.02	0.02
Tetrachloroethane	0.02	0.01	0.06

Specimen configuration: - Titanium - 1/2" x 2" x .062"
Zirconium - 1/2" x 2" x 0.040"
Stainless Steel - 1/2" x 2" x 0.062"

Table XIV. Zirconium Alloys White Fuming Nitric Acid

Specimen No.	Composition (percent by wgt.)	Wgt. of Sample in grams	Loss or gain (+) in wgt. (gms)	Observations
29	5 Ti - 15 W	12.2492	0.0035	No visible signs of corrosion.
115	35 Ti	1.1150	0.0003	" " " " "
116	1 Al - 35 Ti	2.0215	0.0007	" " " " "
117	50 Ti	2.1303	0.0010	Sample covered with iridescent, varicolored film.
118	1 Al - 50 Ti	1.3677	0.0012	" " " " " "
120	1 Al - 65 Ti	2.0214	0.0011	Sample covered with bright blue film.
136	2.5 Cb	2.5767	+0.0004	Sample slightly darkened.
186	4 Al	2.2811	+0.0003	Sample covered with blue-black film.
187	2 Al	3.0185	+0.0059	" " " " " "
191	6.25 Ta - 6.25 Cb	2.1403	+0.0004	Sample partly covered with dark gray film.
192	8.75 Ta - 8.75 Cb	2.7718	+0.0005	" " " " " " "
193	12.5 Cb	2.4473	+0.0187	Sample covered with blue-black film.
194	17.5 Cb	2.3100	+0.0201	" " " " " "
195	22.5 Cb	1.3109	+0.0184	Sample covered with loose gray-black film of embrittled metal.
197	2.5 Cb	2.8784	+0.0017	Sample slightly darkened.
216	17.5 Ta	2.8920	+0.0004	Sample slightly dulled.
217	17.5 Ta	2.3015	+0.0025	" " "
218	17.5 Ta	3.2668	+0.0004	" " "
219	50 Ti	1.1949	0.0003	No visible signs of corrosion.
220	1 Al - 50 Ti	1.1270	0.0007	Sample covered with bright, blue-violet colored film.
222	3 Al - 50 Ti	1.9024	0.0005	Sample covered with light blue colored film.
297 A	5 Mo	1.6075	0.0001	No visible signs of corosion.
298 A	5 W	3.1661	+0.0005	Sample slightly darkened.
299 A	5 Ni	3.4926	0.0352	Sample badly embrittled and cracked thru in many places.
300 A	5 Ta	2.2236	+0.0010	Sample partly covered with blue film and slightly darkened.
302 A	6 Al	2.4973	+0.0007	Sample covered with tightly-adhering blue-gray film.
304 A	4 Al - 50 Ti	1.2656	0.0004	Sample Covered with bright blue film.
305 A	6 Al - 50 Ti	2.7886	0.0014	Sample covered with rose and amber-colored film.
318	2.5 Mo	3.1056	0.0748	Sample partly covered with gray-black film and partly embritld
319	2.5 W	1.9820	+0.0044	Sample covered with very thin, gray-black film.
326	1 Ta	2.0990	0.0000	Sample covered with faint, amber-colored film.
327	10 Mo	5.1897	0.0005	Sample slightly dulled.
330	10 Ta	4.1208	0.0000	No visible signs of corrosion.
403	0.5 Cu	43.1011	0.0001	" " " " "
404	0.5 Mg	18.0684	+0.0002	" " " " "

Table XIV. Zirconium Alloys - White ai· Nitric Acid (Continued)

Specimen No.	Composition (percent by wgt.)	Wgt. of Sample in grams	Loss or gain (+) in wgt. (gms)	Observations
405	0.5 Ni	29.5842	+0.0001	No visible signs of corrosion.
407	0.5 Si	35.4498	+0.0001	" " " " "
415	1 Ni	12.0966	+0.0001	" " " " "
416	2.5 Ni	19.6410	+0.0003	" " " " "
417	1 Cu	9.6350	+0.0010	" " " " "
418	2.5 Cu	16.8931	+0.0011	" " " " "

Notes: (1) Compositions listed are intended compositions. Actual compositions may vary somewhat from those listed.

(2) Alloys numbered 29-222 were prepared in graphite crucibles (induction melted.) Alloys numbered 297A-418 were prepared in water-cooled copper crucibles (arc melted in inert atmosphere).

Test solutions were non-aerated and static.

Table XV. Zirconium Alloys - Salt Spray Test (3% NaCl and Substitute Ocean Water)

Sample No.	Composition (percent by weight)	Weight of sample in grams	Loss or Gain (+) in wgt. (gms.) (3% NaCl)	Loss or Gain (+) in wgt. (gms.) (Subst. Oc. Water)
27	50 Ti	27.3251	0.0004	+0.0001
29	5 Ti - 15 W	11.9109	0.0005	0.0000
31	5 Ti - 15 Cr	34.6313	0.0053	0.0000
32	5 Ti - 15 Ta	18.0350	0.0005	0.0001
33	5 Ti	13.1326	0.0012	0.0002
42	5 Al	14.3895	0.0010	0.0001
46	5 Al	8.9328	0.0002	0.0003
47	5 Be	20.8608	0.0008	+0.0002
48	5 Pt	20.4030	0.0020	+0.0003
49	5 V	17.5321	0.0014	+0.0006
50	5 Si	12.9485	0.0055	0.0000
51	1 Al	9.4480	0.0010	+0.0002
52	100 Zr	9.8041	0.0022	+0.0003
53	2.25 O_2	12.9547	0.0048	0.0006
54	10 Ta	13.6775	0.0054	0.0001
55	20 Ta	8.8425	+0.0001	0.0001
56	5 Cb	13.1163	0.0066	0.0000
115	35 Ti	1.0437	0.0002	+0.0002
116	1 Al - 35 Ti	1.9078	0.0001	0.0000
117	50 Ti	3.3823	0.0002	+0.0001
118	1 Al - 50 Ti	1.1667	0.0002	0.0000
120	1 Al - 65 Ti	1.9527	0.0002	0.0000
121	65 Ti	7.2426	0.0004	0.0000
136	2.5 Cb	1.9530	0.0002	0.0001
168	2.0 Al	15.3825	0.0001	0.0000
169	4.0 Al	13.5315	0.0002	0.0000
170	6 Al	25.6370	0.0003	0.0003
180	3 Al	20.6691	0.0011	+0.0006
181	5 Al	13.0627	0.0001	0.0003
186	4 Al	2.4930	0.0000	0.0003
187	2 Al	1.2723	+0.0001	0.0001
191	6.25 Ta - 6.25 Cb	1.8437	0.0002	0.0001
192	8.75 Ta - 8.75 Cb	2.1341	0.0000	0.0001
193	12.5 Cb	1.7842	0.0000	0.0000
194	17.5 Cb	2.8467	0.0006	0.0000
195	22.5 Cb	2.1600	0.0000	0.0002
196	27.5 Cb	1.7515	0.0002	0.0003
197	2.5 Cb	3.1332	0.0004	0.0000
216	17.5 Ta	4.0398	0.0000	0.0003
217	17.5 Ta	1.8354	0.0001	+0.0004
218	17.5 Ta	2.0117	0.0001	+0.0004
219	50 Ti	1.1654	0.0001	+0.0006
220	1 Al - 50 Ti	1.1120	0.0000	+0.0004
222	3 Al - 50 Ti	0.9851	0.0001	+0.0006
297A	5 Mo	1.5016	0.0001	+0.0004

Table XV. Zirconium Alloys - Salt Spray Test (3% NaCl and Substitute Ocean Water) (Cont.)

Sample No.	Composition (percent by weight)	Weight of samples in grams	Loss or Gain (+) in wgt. (gms.) (3% NaCl)	Loss or Gain (+) in wgt. (gms.) (Subst. Oc. Water)
298A	5 W	1.7752	0.0002	+0.0004
299A	5 Ni	2.7802	0.0005	+0.0006
300	5 Ta	2.7205	0.0002	+0.0007
302	6 Al	2.1621	0.0000	+0.0007
304A	4 Al - 50 Ti	1.1792	0.0002	+0.0007
305A	6 Al - 50 Ti	4.2773	0.0002	+0.0012
318	2.5 Mo	1.4506	0.0002	+0.0007
319	2.5 W	2.7691	0.0003	+0.0012
320	2.5 Ni	1.6166	0.0001	+0.0005
321	2.5 Ta	2.4838	0.0002	+0.0010
324	1 W	2.4096	0.0002	+0.0006
325	1 Ni	3.5873	0.0003	+0.0013
326	1 Ta	1.9617	0.0000	+0.0009
327	10 Mo	4.6462	+0.0001	+0.0008
328	10 W	4.0939	0.0004	+0.0010
329	10 Ni	3.2025	0.0006	+0.0009
330	10 Ta	4.0920	+0.0003	+0.0008
403	0.5 Cu	42.9696	0.0000	+0.0004
404	0.5 Mg	18.0357	+0.0001	+0.0002
405	0.5 Ni	29.5819	+0.0003	+0.0002
407	0.5 Si	35.4095	+0.0004	+0.0003
408	1.0 Si	37.0575	0.0050	+0.0003
409	0.5 B	47.1957	0.0002	0.0002
414	0.25 Si	24.1016	+0.0002	0.0000
415	1.0 Ni	12.0733	0.0000	0.0004
416	2.5 Ni	19.6402	0.0002	0.0000
417	1.0 Cu	9.4048	0.0002	0.0003
418	2.5 Cu	16.8314	0.0014	0.0048
420	0.5 Fe	34.9446	+0.0002	+0.0002
421	1.0 Fe	20.4865	0.0000	0.0002
422	0.5 Be	11.6490	+0.0009	0.0009
423	1.0 Be	21.2388	0.0338	+0.0013
424	1.0 B	16.4369	0.0000	0.0000
425	0.5 V	23.6863	0.0009	0.0000
426	1.0 V	20.5614	+0.0001	+0.0002
427	0.5 Cr	13.0943	0.0001	0.0000
428	1.0 Cr	18.6535	+0.0002	+0.0002
429	0.5 Mn	18.1078	0.0000	+0.0005
430	1.0 Mn	20.3328	+0.0003	+0.0001

Notes:

(1) Compositions listed are intended compositions. Actual compositions may vary somewhat from those listed.

(2) Alloys numbered 27-222 were prepared in graphite crucibles (induction melted). Alloys numbered 297A-430 were prepared in water cooled copper crucibles (arc melted in inert atmosphere).

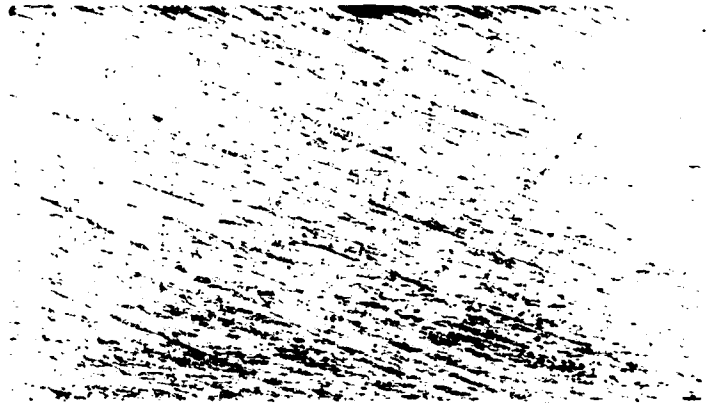

Fig.1 X 25

High purity arc melted zirconium after 30 days immersion in concentrated (37%) hydrochloric acid. Solution non-aerated and static.

Fig.2 X 25

High purity zirconium induction melted in graphite after 30 days immersion in concentrated (37%) hydrochloric acid. Solution non-aerated and static.

Fig. 3 X 1

High purity arc melted zirconium after 6 days immersion in concentrated (37%) hydrochloric acid at 100°C (under pressure). Solution non-aerated and static.

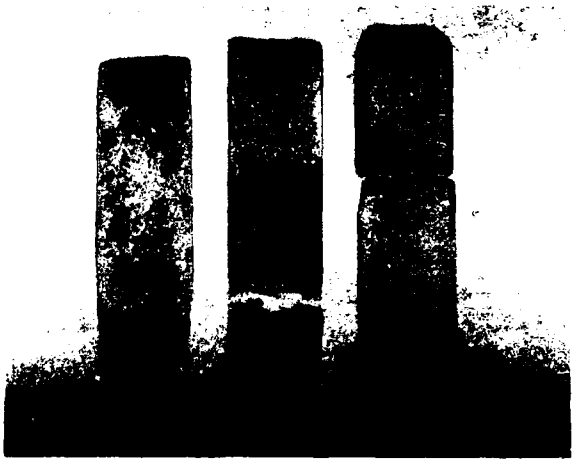

Fig. 4 X 1

High purity zirconium induction melted in graphite after 6 days immersion in concentrated (37%) hydrochloric acid at 100°C (under pressure). Solution non-aerated and static.

Fig. 5 **X 1**

Zirconium-columbium alloy (3.70% Cb) induction melted in graphite after 6 days immersion in concentrated (37%) hydrochloric acid at 100°C (under pressure). Solution non-aerated and static.

Fig. 6 **X 1**

Tantalum metal after 6 days immersion in concentrated (37%) hydrochloric acid at 100°C (under pressure). Solution non-aerated and static.