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M-4585

CORROSION OF THORIUM AND RELATED MATERIALS

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ABSTRACT

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A study of the corrosion of thorium and related materials in both air and water at various temperatures was made. By varying the method of treating the sample after exposure to water, it was found that values obtained by polishing the sample with either a non-metal-cutting cloth or an abrasive wheel were more reliable than those obtained by other methods.

The effects of cooling, acid and salt solutions, and temperature were investigated. The cooling of aluminum and thorium had little effect on the corrosion rate of thorium but elevation of the temperature caused an increase in the rate of corrosion. Partial disintegration of the samples occurred in solutions containing acid and dissolved salts.

Alloys of thorium were found to be less resistant to corrosion than was the unalloyed metal. All alloys of uranium partially disintegrated during the tests.

* Based on the following reports:

CC-2470
CC-2715

March 26, 1945
April 25, 1945

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By exposing thorium and a thorium-beryllium alloy to air at various temperatures it was observed that both metals corroded appreciably but the alloy was corroded to a lesser extent than was the unalloyed thorium. A preferential corrosion of one phase of the alloy took place but the phase most seriously attacked could not be identified.

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1. WATER CORROSION OF ALLOYS OF THORIUM AND OF URANIUM

1.1 Introduction. Future development and design involving the use of uranium and thorium and also their alloys presupposes that a knowledge of the corrosion rate of these materials is available. The most pertinent data can be obtained only by subjecting the test samples to conditions duplicating those which will be encountered in normal operations. Since it was known that these conditions could not be attained during the tests, it was decided to use water as the corroding medium and thus obtain at least relative values for the corrosion rate of these metals.

These investigations are concerned mainly with the effects of water corrosion tests, but the results of a series of tests employing other corroding media are also included.

1.2 Description and Preparation of Samples. The samples of unalloyed metal used in these corrosion studies were prepared by the reduction of the fluoride⁽¹⁾. The uranium-tantalum and the thorium-iron alloys were prepared by mixing the metals in the molten state⁽²⁾. All other alloys were produced by co-reduction of the fluorides⁽³⁾. Unless otherwise specified, all samples are recast metal and none of the samples had been heat treated. The maximum oxide content of the thorium-beryllium alloys was 0.5 per cent while the unalloyed thorium^{contained} two per cent oxide, maximum.

The shape of the test pieces is not critical but samples approximately one centimeter in length and 0.5 centimeters square

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were found to be most convenient for the apparatus used in the tests. Each sample, before testing, was rough ground to remove surface defects and then polished with a No. 00 emery polishing paper. The surface of the sample exposed to the corroding medium was calculated from micrometer measurements. After weighing, the sample was ready for testing.

1.3 Experimental.

(a) Thorium and Thorium Alloys. Originally the tests were conducted by placing the sample in water refluxed at the desired temperature. After a suitable period of exposure, usually several days, the sample was removed, washed, dried, and weighed. The corrosion factor was calculated as the change in weight in milligrams per square centimeter of original surface area per hour of exposure. A negative corrosion factor indicates a loss of weight.

Table 1

Corrosion of Th and Th-Be Alloys in Water at 100°C

Sample No.	Composition of Sample % by wt.	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ²
1	Th	19.75	9.20	1.70×10^{-2}
2	Th	95.25	6.38	-1.98×10^{-3}
3	99% Th-1% Be	95.25	6.44	-7.51×10^{-3}
4	99% Th-1% Be	113.0	6.15	3.34×10^{-2}

Other thorium beryllium alloys were prepared and further corrosion studies were made on these materials. The results

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of these tests are given in Table 2.

Table 2
Corrosion of Th and Th-Be Alloys in Water at 100°C

Sample No.	Composition of Sample % Be by wt.	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ²
1	0	193.3	6.46	13.65×10^{-4}
2	0.5	214.2	7.21	-5.83×10^{-4}
3	1.0	213.9	7.08	-0.66×10^{-4}
4	2.0	215.9	5.72	0.81×10^{-4}
5	4.0	225.0	6.30	2.82×10^{-4}
6	7.0	213.6	4.88	3.22×10^{-4}

To determine the effect of increased temperature on the corrosion of thorium and thorium-beryllium alloys, a series of alloys of the same composition as used above were selected and tested at 140°C. Each sample was placed in a separate glass tube filled with distilled water and the series of tubes mounted in a frame and placed inside a steel bomb partly full of distilled water. The bomb was heated by a gas burner and some of the generated steam exhausted to free the bomb of entrapped air. During the test, the pressure within the bomb varied between twenty and fifty p.s.i. (gage) with an approximate average pressure of thirty p.s.i. (gage). This steam pressure is equivalent to a temperature of 140°C.

At the end of the test the massive metal was removed, washed, dried, and weighed. The following table summarizes the results obtained.

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Table 3

Corrosion of Th and Th-Be Alloys in Water at 140°C

Sample No.	Composition of Sample % Be by Wt.	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ²
1	0	64	6.45	5.08×10^{-3}
2	0.5	64	7.13	1.97×10^{-3}
3	1.0	64	6.66	3.29×10^{-3}
4	2.0	64	5.03	4.04×10^{-3}
5	4.0	64	6.46	3.64×10^{-3}
6	7.0	64	4.22	2.67×10^{-3}

The inconsistency of the results obtained by the above method of treating the sample after exposure, particularly as shown by comparison of Tables 1 and 2 disclosed a need for a change in technique if reliable and reproducible results were to be acquired. The ideal method of treatment would be the use of a reagent which dissolved the oxide film formed during the test but which did not dissolve the metal. Unfortunately, no such solvent is known. Any reagent that will dissolve the oxide will also attack the metal. The only methods deemed suitable were those involving the polishing of the sample by mechanical means.

Four different methods of treatment after exposure were outlined and they are described as follows:

METHOD I: Sample washed, dried, and weighed.

METHOD II: Sample dried, brushed off with camel hair brush and weighed.

METHOD III: Sample dried, polished with a non-metal-cutting wool cloth (AB Miracloth) and weighed.

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METHOD IV: Sample dried, polished to a bright metallic luster with a No. 000 emery polishing paper and weighed.

Using these methods, the foregoing tests were repeated. To avoid temperature variations during the 140°C test, the bomb was placed in a paraffin bath. Tables 4 and 5 contain the results of these tests.

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Table 4

Corrosion of Th and Th-Be Alloys in Water at 100°C

Sample No.	Composition of Sample % Be by Wt.	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ²			
				Method I	Method II	Method III	Method IV
1	0	69.9	3.24	1.08×10^{-5}	-1.71×10^{-5}	-3.09×10^{-5}	-22.5×10^{-5}
2	0.5	90.2	3.94	0.85×10^{-5}	-1.51×10^{-5}	-1.87×10^{-5}	-44.8×10^{-5}
3	1.0	90.6	3.50	-----	11.10×10^{-5}	1.72×10^{-5}	-62.0×10^{-5}
4	2.0	91.3	2.79	No Change in Wt.	No Change in Wt.	No Change in Wt.	-55.8×10^{-5}

Table 5

Corrosion of Th and Th-Be Alloys in Water at 140°C

Sample No.	Composition of Sample % Be by Wt.	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ²			
				Method I	Method II	Method III	Method IV
1	0	138.1	2.21	1.64×10^{-5}	-0.99×10^{-5}	-2.82×10^{-5}	-42.9×10^{-5}
2	0.5	138.1	3.38	-746×10^{-5}	-787×10^{-5}	-817×10^{-5}	-1110×10^{-5}
3	1.0	138.1	7.45	5.45×10^{-5}	3.89×10^{-5}	1.17×10^{-5}	-104×10^{-5}
4	2.0	138.1	6.14	10.75×10^{-5}	9.1×10^{-5}	7.48×10^{-5}	66.0×10^{-5}

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The high corrosion rate of Sample No. 2 is explained by the history of the material. This alloy was a sample of metal prepared by recasting scrap pieces of several old castings of the 0.5 per cent beryllium-thorium alloy. An analysis of the material showed an oxide content of ten per cent and the high rate of corrosion exhibited by this sample is typical of samples having an appreciable oxide content. Sample No. 3 contained 5.8 per cent oxide while all the other thorium beryllium alloys used throughout these tests had a maximum oxide content of 0.5 per cent. The samples of unalloyed thorium contained a maximum of two per cent oxide.

On the basis of these tests, it was decided to eliminate the use of Methods No. I and No. II since large errors were introduced if small particles of the corroded metal fell off after exposure and previous to weighing. Since the depth to which the sample has been corroded is unknown and varies over the surface, some of the uncorroded metal is removed during the abrasive polishing operation and the values obtained by Method IV are considered as a maximum value of corrosion.

Increasing the temperature of the corrosion test from 120°C to 140°C seemed to have very little effect upon the corrosion rate. It was therefore of interest to determine the effect of a further increase in temperature on the corrosion rate of these materials. Based on the data of the

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preceding tests, a sample of thorium and thorium-beryllium alloy (2% Be by wt.) were selected for corrosion tests at 180°C. The results are shown in Table 6.

Table 6

Corrosion of Th and Th-Be Alloy in Water at 180°C

Sample No.	Composition of Sample % Be by Wt.	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ²	
				Method III	Method IV
1	0	71.25	7.19	-8.98×10^{-3}	-66.7×10^{-3}
2	2.0	70.3	8.20	-9.52×10^{-2}	-41.5×10^{-2}

No disintegration of the samples took place during these tests. The samples became coated with a dark film which was probably thorium dioxide. This higher temperature slightly increased the corrosion rate of thorium but the rate of corrosion of the alloy was increased by a factor of approximately ten by raising the temperature from 140°C to 180°C.

Alloys of thorium and iron were prepared and two of these were selected for corrosion studies. It was believed that these alloys might exhibit special corrosion resistant properties because the structure indicated a dispersion of thorium in a network of thorium-iron compound. It was thought that the compound may resist corrosion. These samples were tested in water at 180°C.

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Table 7

Corrosion of Th-Fe Alloys in Water at 100°C

Sample No.	Composition of Sample % Fe by Wt.	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ²	
				Method III	Method IV
1	1.32	142.0	2.89	0.98×10^{-3}	-12.2×10^{-3}
2	2.69	51.2	3.16	-3.07×10^{-3}	-43.0×10^{-3}

The corrosion factors obtained for these alloys are of the same order of magnitude as those of other samples tested. No exceptional corrosion properties were exhibited.

The effect of the coupling action of aluminum and thorium on the corrosion rate of both metals was investigated. The results are shown in Tables 8 and 9. No attempt was made to determine the potential produced by the couple or to study its variation during the tests.

Table 8

Corrosion of Al and Al-Th Couple in Water at 100°C

Sample	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ²	
			Method III	Method IV
Couple	Al	1.65	7.9×10^{-3}	-----
	Th	233.3	-1.25×10^{-4}	-1.87×10^{-2}
	Al	234.1	6.83×10^{-3}	-----

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Table 9

Corrosion of Al and Al-Th Couple in Water at 140°C

Sample	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ²	
			Method III	Method IV
Couple	Al	93	1.13	6.0×10^{-2}
	Th	93	7.01	-4.6×10^{-4}
	Al	93	1.13	4.1×10^{-2}

In these tests, the thorium was wrapped in a thin sheet of aluminum. The contact between the thorium and aluminum was good but at the end of the test it was difficult to use the method outlined above in determining the corrosion of the aluminum.

In spite of this difficulty, the test did indicate that when aluminum and thorium are coupled the rate of corrosion of aluminum is increased slightly.

A second test of the coupling effect at 140°C was run by selecting thorium and aluminum samples of approximately the same size and shape. The samples were ground to fit snugly into a glass tube so that contact between the metals could be maintained throughout the test.

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Table 10

Corrosion of Al and Al-Th Couple in Water at 140°

Sample	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ²	
			Method III	Method IV
Couple	Al	98	5.63	1.63×10^{-3}
	Th	98	4.80	-5.08×10^{-3}
			No Change in wt.	-58.7×10^{-3}
	Al	98	6.89	0.75×10^{-3}
				-4.06×10^{-3}

These results verify the previous conclusion that if aluminum and thorium are coupled during the corrosion test, the aluminum corrosion rate is increased but the rate of corrosion of thorium is practically unaffected.

A study of the corrosion effects of various acid solutions on an alloy of thorium containing 0.5 per cent beryllium were studied. The following solutions were employed in the tests.

<u>Solution No.</u>	<u>Description</u>
1	0.3 M in Uranyl nitrate and 0.1 M in Nitric acid
2	0.3 M Thorium nitrate
3	0.5 M Nitric acid
4	15.4 M (concentrated) Nitric acid

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Table 11

Corrosion of Th-Be Alloy (0.5% Be by Wt.) in Acid Solutions

Solution No.	Temperature °C	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ² Method I
1	100	114.0	2.05	-8.55×10^{-2}
2	100	113.8	1.91	-4.14×10^{-2}
3	98	113.4	1.77	-4.83×10^{-2}
4	86	113.8	2.62	-123.8×10^{-2}

Although the sample in Solution No. 1 was attacked, it retained a high polish throughout the test. When the alloy was placed in Solution No. 4, a reaction began immediately. At the end of the test, this sample was black and deeply etched. A comparison of these results with those obtained for the same alloy in water as given in Table 2 discloses that the alloy is much less resistant to corrosion in these solutions than in water at approximately the same temperature.

(b) Uranium and Uranium Alloys. A study of the corrosion of uranium and two of its alloys was made. Uranium was not expected to withstand the attack of water under the conditions of the test but it was hoped that by alloying it with some metal like beryllium or tantalum, it would exhibit corrosion resistant properties. Samples No. 2 and 3 of Table 13 and the uranium tantalum alloy were crude (uncast or bisquit) metal. All the other alloys were recast metal.

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A large part of each of the samples tested disintegrated into a black powder. Undoubtedly the excess uranium in these alloys was attacked by the corroding medium. Because of this disintegration, the corrosion factors given in the following tables are of little significance since they are based on the original area of the samples.

A comparison of the corrosion rate of recast and crude material as shown by Samples No. 1 and 2 of Table 13 clearly indicates the effects of impurities on the corrosion rate of the metal.

Table 13

Corrosion of U and U-Be Alloys in Water at 100°C

Sample No.	Composition of Sample % Be by Wt.	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ²
1	0	25.0	5.35	-2.51
2	0.75	47.3	4.57	-1.20
3	7.0	19.5	7.52	-0.82

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Table 13

Corrosion of U-Be Alloys in Water at 140°C

Sample No.	Composition of Sample % Be by Wt.	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ² Method I
1	0.75	64.0	6.14	-4.45
2*	0.75	64.0	8.01	-10.92
3*	1.0 (atomic %)	64.0	4.28	-10.14

* Crude metal

Table 14

Corrosion of U-Ta Alloy in Water at 170°C

Sample No.	Composition of Sample % Ta by Wt.	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ² Method I	Method III	Method IV
1*	4.5	43.5	3.63	-1.21	-1.36	-1.63

* Crude metal

(c) Beryllium. Beryllium is known to be very resistant to water corrosion and having available some of this metal prepared by the reduction of the fluoride⁽⁴⁾, it was subjected to the type of corrosion test employed throughout these experiments to determine what effects these conditions might have.

The first samples of this metal that were tested were porous and pitted. Later a better sample of this metal was obtained and the test at 140°C repeated. This latter sample is designated as Sample No. 3 in the table below.

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Table 15
Corrosion of Beryllium in Water

Sample No.	Temperature of Test °C	Length of Test Hr.	Area of Sample cm ²	Corrosion Factor mg/hr/cm ²	
				Method III	Method IV
1	100	96.7	4.38	4.48×10^{-3}	-----
2	140	93.0	4.02	2.44×10^{-2}	1.74×10^{-2}
3	140	96.7	5.06	2.22×10^{-3}	-1.62×10^{-3}

Sample No. 1 remained polished throughout the test. Samples No. 2 and 3 which were run at 140°C were covered with a dark film at the completion of the test.

2. AIR CORROSION OF THORIUM

2.1 Introduction. The fabrication of thorium and thorium-beryllium alloys will be accomplished by such operations as rolling, swaging, extruding, etc. It is therefore necessary to know something about the rate of corrosion of these metals in air at various temperatures. The work undertaken here is in the nature of obtaining data which will be of interest during these fabrication operations or in other related problems.

2.2 Description and Preparation of Samples. The materials selected for study in these tests were samples of thorium and of a thorium-beryllium alloy containing ^{two percent} 2% beryllium by weight. The thorium contained ^{one to two percent} ~~1% to 2%~~ thorium dioxide while the alloys contained a maximum of ^{percent} 0.2% oxide. These metals were prepared by reduction of the fluorides ⁽¹⁾ and all samples of each metal were taken from the same casting.

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Usually samples approximately ^{one} $\frac{1}{2}$ cm. in length and 0.5 cm. square were selected for the corrosion tests. The sample was rough ground to remove surface defects and then polished with a No. 00 emery polishing paper. The dimensions of the specimen were obtained by micrometer measurements and from these data the surface area was calculated.

2.3 Experimental. The sample was placed in a porcelain crucible and after weighing was transferred to the furnace shown in Figure 1. Furnace temperatures were measured with a Chromel-Alumel thermocouple and a potentiometer; the temperature was controlled by means of a variable transformer. In each test, the first ten minutes were spent in establishing equilibrium conditions of temperature within the furnace. At the end of each hour, the crucible and contents were cooled and weighed and the corrosion calculated as the gain in weight in milligrams per square centimeter of original surface area.

Tables 16 to 20 contain the results of air corrosion tests on thorium. A test at 200°C was made but the change in weight after six hours of exposure was negligible; the only visible effect was the formation of a light gray film on the surface.

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Table 16

Air Corrosion of Thorium at 300°C
Area of Sample: 6.72 cm²

Exposure Time Hr.	Change in Wt. mg.	Corrosion mg/cm ²
1	0.3	4.46×10^{-2}
2	-	-----
3	0.9	13.40×10^{-2}
4	0.8	11.90×10^{-2}

Table 17

Air Corrosion of Thorium at 400°C
Area of Sample: 5.86 cm²

Exposure Time Hr.	Change in Wt. mg.	Corrosion mg/cm ²
1	-	---
2	2.4	0.41
3	5.5	0.94
4	10.1	1.72
5	13.1	2.23

Table 18

Air Corrosion of Thorium at 425°C
Area of Sample: 2.62 cm²

Exposure Time Hr.	Change in Wt. mg.	Corrosion mg/cm ²
1	5.8	2.21
2	-	--
3	18.2	6.95
4	24.8	9.48

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Table 19

Air Corrosion of Thorium at 450°C
Area of Sample: 2.93 cm²

Exposure Time Hr.	Change in Wt. mg.	Corrosion mg/cm ²
1	15.4	5.27
2	32.9	11.23
3	47.8	16.31
4	62.8	21.40

Table 20

Air Corrosion of Thorium at 500°C
Area of Sample: 5.58 cm²

Exposure Time Hr.	Change in Wt. mg.	Corrosion mg/cm ²
1	33.5	6.01
2	67.4	15.65
3	141.2	25.30
4	193.8	34.75

These results are plotted in Figure 2. Below 400°C the samples at the end of the four hour tests were coated with a light gray film, but above 400°C a flaking off of the oxide layer occurred during the test. This explains the rapid increase in the slope of the curves above 400°C.

The results of the air corrosion tests of the thorium-^{two percent} beryllium alloy containing 2% beryllium by weight are given in tables 21 to 25.

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Table 21

Air Corrosion of Th-Be Alloy at 400°C
Area of Sample: 3.71 cm²

Exposure Time Hr.	Change in Wt. mg.	Corrosion mg/cm²
1	0.7	0.19
2	2.8	0.76
3	4.8	1.29
4	9.4	2.53

Table 22

Air Corrosion of Th-Be Alloy at 425°C
Area of Sample: 3.79 cm²

Exposure Time Hr.	Change in Wt. mg.	Corrosion mg/cm²
1	3.7	0.98
2	6.6	1.74
3	9.8	2.58
4	13.0	3.43

Table 23

Air Corrosion of Th-Be Alloy at 437°C
Area of Sample: 4.87 cm²

Exposure Time Hr.	Change in Wt. mg.	Corrosion mg/cm²
1	4.0	0.82
2	10.0	2.06
3	15.1	3.10
4	20.5	4.22

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Table 24

Air Corrosion of Th-Be Alloy at 450°C
Area of Sample: 4.75 cm²

Exposure Time Hr.	Change in Wt. mg.	Corrosion mg/cm ²
1	6.7	1.41
2	14.8	3.12
3	20.7	4.37
4	27.8	5.82

Table 25

Air Corrosion of Th-Be Alloy at 500°C
Area of Sample: 4.09 cm²

Exposure Time Hr.	Change in Wt. mg.	Corrosion mg/cm ²
1	13.4	3.28
2	24.4	5.97
3	31.1	7.61
4	38.6	9.43

Figure 3 shows the curves obtained from these data.

These alloys consist of a dendritic formation of thorium and a finely divided eutectic composed of thorium and thorium-beryllium compound. The structure of the metal is clearly visible and it was observed that one of the phases was attacked more than the other. At this time, however, because of lack of information on the structure of these alloys, it is not possible to state which of the phases is preferentially attacked.

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3. SUMMARY AND CONCLUSIONS

3.1 Water Corrosion Tests. It should be remembered that in all but the Air Corrosion tests described, the samples were at all times in contact with the corroding medium in the liquid phase. Radically different results would undoubtedly be obtained if the samples were exposed to the corroding medium in the vapor phase.

Consistent results are not obtainable if the samples after exposure are merely dried and weighed. Some method of removing the oxide film either completely or partially must be resorted to if reliable results are to be achieved. Polishing the sample with a non-metal-cutting cloth does not remove all the oxide film from the sample but using an abrasive material removes some of the uncorroded metal. The true corrosion value for the material lies between the values calculated from these methods and Met od No. IV may be considered a maximum corrosion rate for the metal.

The rate of corrosion of the sample is not greatly affected by a slight increase in temperature. However, if the temperature is increased appreciably, a more rapid rate of corrosion results.

In all these tests the rate of corrosion of the alloyed thorium was greater than that of the unalloyed thorium irrespective of the temperature at which the test was conducted.

The coupling of aluminum and thorium during the corrosion test has no significant effect on the rate of corrosion of thorium although under these conditions the rate of corrosion

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of the aluminum is increased slightly.

The presence of acid and dissolved salts in the corroding medium greatly increases the corrosion rate of the 0.5 per cent beryllium-thorium alloy. High acid concentrations are particularly objectionable because they cause the metal to partially disintegrate.

The factor having the greatest effect on the corrosion rate of thorium alloys and undoubtedly thorium would be affected in a similar manner, is the presence of appreciable amounts of oxide in the metal. This adverse effect is clearly shown by the results in Table 5.

The corrosion rate of uranium is excessive and no suitable corrosion resistant alloy has been developed yet.

3.2 Air Corrosion Tests. While the data and curves obtained in the air corrosion investigation do not and are not intended to represent absolute values, they do, nevertheless, indicate the magnitude and trend of the corrosion of thorium and of the thorium-beryllium alloy at the indicated temperatures. It may be seen from the curves that the rate of corrosion of the thorium-beryllium alloy is much less than that of the unalloyed thorium.

Thorium corrodes by the formation of layers of oxide which then fall off and expose new surface. The alloy corrodes by the oxide flaking off in small pieces. Until more information on the structure of the thorium-beryllium alloy is obtained, no statement can be made as to which phase of the alloy is preferentially attacked.

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Fig. 1. Cross-Section
of Air Corrosion
Furnace

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Fig. 2. Corrosion of
Thorium in Air at ²⁷
Various Temperatures

Fig. 3. Corrosion of
Th-Be Alloy (2% Be by
wt) in Air at 28
Various Temperatures