UNCLASSIFIED

0
CT-2787

Contract W-7401-eng-37

PROGRESS REPORT

March 17 to March 24, 1945

METALLURGY DIVISION

Section M-I F. Foote
Section M-II E. W. Brugmann
Section M-III J. A. Chapin

Report Received: March 24, 1945
Issued:

MAR 24 1945

Photostat Price \$ 3.30
Microfilm Price \$ 2.40

Available from the
Office of Technical Services
Department of Commerce
Washington 25, D. C.

UNCLASSIFIED

LEGAL NOTICE

This report was prepared as an account of Government sponsored work. Neither the United States, nor the Commission, nor any person acting on behalf of the Commission:

A. Makes any warranty or representation, express or implied, with respect to the accuracy, completeness, or usefulness of the information contained in this report, or that the use of any information, apparatus, method, or process disclosed in this report may not infringe privately owned rights; or

B. Assumes any liabilities with respect to the use of, or for damages resulting from the use of any information, apparatus, method, or process disclosed in this report.

As used in the above, "person acting on behalf of the Commission" includes any employee or contractor of the Commission to the extent that such employee or contractor prepares, handles or distributes, or provides access to, any information pursuant to his employment or contract with the Commission.

CLASSIFICATION CANCELLED

DATE FEB 16 1957
For The Atomic Energy Commission

H. R. Canale
Chief, Declassification Branch

044-1

DISCLAIMER

This report was prepared as an account of work sponsored by an agency of the United States Government. Neither the United States Government nor any agency Thereof, nor any of their employees, makes any warranty, express or implied, or assumes any legal liability or responsibility for the accuracy, completeness, or usefulness of any information, apparatus, product, or process disclosed, or represents that its use would not infringe privately owned rights. Reference herein to any specific commercial product, process, or service by trade name, trademark, manufacturer, or otherwise does not necessarily constitute or imply its endorsement, recommendation, or favoring by the United States Government or any agency thereof. The views and opinions of authors expressed herein do not necessarily state or reflect those of the United States Government or any agency thereof.

DISCLAIMER

Portions of this document may be illegible in electronic image products. Images are produced from the best available original document.

REPORT FOR WEEK ENDING MARCH 22, 1945
METALLURGY SECTION - FRANK FOOTE, SECTION CHIEF

Total Active Personnel as of March 22, 1945 - - - 36
Academic - - - - - 24
Non-Academic - - - - - 12
Transfers In - - - - - 0
Transfers Out - - - - - 0

MM 1.1 - Metallurgy of Tuballoy and Its Alloys

Pure Metal: During this week the first in a series of 8 billets to be cast in the 1 1/2" water-cooled copper mold was made. It was necessary to fit a new copper tube into the mold before further satisfactory casting could be done. Most of the damage to the tube, however, was believed to have been caused by two nichrome castings which had been previously poured into it. The repaired mold has now been returned from the shop and is ready for further use.

Tuballoy-Columbium Alloys: Two series of 1, 2, 4 and 6% samples have been heated one hour at 6950C and 7210C respectively. Metallographic examination is not yet complete, but it can definitely be said that both 1% samples are in a two-phase field.

The 1 and 2.4% alloys, heat treated at 10000C for one week and quenched in brine were started in boiling distilled water corrosion test. No measureable change was detected after 44 hours in test.

Tuballoy-Columbium-Zirconium Alloys: Chemical analyses of the 2% Cb - 4% Zr and 2% Cb - 6% Zr alloys show very small amount of segregation.

Nominal Composition

Reported Analysis

2% Cb - 4% Zr

Top Edge	2.4% Cb	4.0% Zr
	2.4% Cb	4.1% Zr
Top Center	2.4% Cb	4.1% Zr
	2.4% Cb	4.2% Zr

2% Cb - 6% Zr

Top Edge	2.0% Cb	6.4% Zr
	2.1% Cb	6.0% Zr
Bottom Edge	2.0% Cb	6.1% Zr
	2.0% Cb	6.1% Zr

Corrosion Results in boiling distilled water are:

<u>Nominal Composition</u>		<u>Weight Change Rate (mg/sq. cm/hour)</u>		
<u>% Cb</u>	<u>% Zr</u>	<u>A</u>	<u>B</u>	<u>C</u>
2	2	1.2	0.0005-	0.0001+
2	4	0.71	0.0001+	0.0002-
2	6	0.03	0.0007-	0.001-
4	2	0.28	0.0004+	0.0003+
4	4	0.03	0.0002+	0.0002+
6	2	0.02	---	0.0002+

- A - Heat treatment: 256 hours at 6000C and slow water quench; 112 hours in test.
- B - Heat treatment: 256 hours at 7250C and slow water quench; 566 hours in test
- C - Heat treatment: 256 hours at 8500C and slow water quench; 566 hours in test.

With heat treatment A the corrosion resistance improves with total alloy content rather than the Cb or Zr content.

The low corrosion rate of the heat treated 2% Cb - 2% Zr alloy has made it desirable to extend the study of these ternary alloys to lower columbium alloys. Melts of 1% Cb - 1% Zr, 1% Cb - 2% Zr, 1% Cb - 4% Zr have been prepared.

MM 2.1 - Metallurgy of Thorium and Its Alloys

Pure Metal:

Static Hot Wires: Two experiments yielded 3.4 grams of metal. The object of these experiments was to line up a supply of starting wires.

Flow Hot Wire: Revamping of the system was completed and its operation was started. Carbon analyses on metal deposited on a thorium coated tungsten filament was 44 ppm.

Flow Hot Tube: An experiment with a BeO lined aluminum liner was conducted and this yielded about two grams of a metallic appearing substance. X-ray analyses gave a ThO₂ pattern with a few faint extra lines. Evaporation of the iodine was slower and the material deposited in the McDanel tube beyond the liner. The system is being changed to allow for slower evacuation during the experiment and the McDanel tube will be lined over a greater portion of its length.

Three thorium samples extruded at various temperatures were reported last week. The sample extruded at 900°C to 16 to 1 reduction was found to be completely recrystallized to a very fine grain size. The two 4 to 1 reduction samples extruded at 800 and 1000°C were unrecrystallized.

Thorium-Beryllium Alloys: Thermal analyses have been made on alloys of 1, 2, and 3 wt. % beryllium. Results have not been interpreted.

Corrosion results of alloys, as cast, in boiling distilled water are:

<u>Nominal Composition</u> <u>Wt. % Be</u>	<u>Hours in Test</u>	<u>Wt. Change Rate</u> <u>(mg/cm²/hour)</u>
0.4	235	0.003±
0.9	235	0.001±
1.6 (melt prepared in BeO xible)	235	0.0009±
1.6 (melt prepared in graphite xible)	235	0.0005±
2.5	235	0.0006±

044

3

Thorium-Carbon Alloys: Diffraction pattern of the 1.2% Carbon annealed at 850°C for 90 hours and water quenched indicated considerable solution of carbon in thorium. The film was measured and the a_0 value was calculated to be 5.22 as compared to 5.077 for the pure metal. The pattern also indicated fairly strong lines of ThO_2 .

Thorium-Cerium Alloys: A thorium-cerium alloy containing 6.14 wt. % (10 at. %) cerium was prepared and examined. The high temperature reached during melting (1460°C) may have volatilized some of the cerium. The microstructure appeared to be single-phased with low melting impurities at the grain boundaries.

Thorium-Silicon Alloys: Approximate corrosion rate loss of alloys in boiling water test are:

Nominal Composition Wt. % Si	Hours in Test	Wt. Change Rate (mg/cm ² /hour)
1.0	235	0.0004±
2.0	235	—
4.3	235	—

Thorium-Beryllium-Silicon Alloys: Approximate corrosion rate of the 1% Be, 0.6% Si alloy in boiling distilled water was 0.0001± mg/cm²/hour after 234 hours as compared to 0.001± reported last week.

NM 3.1 - Metallurgy of Beryllium and Its Alloys

Hot Compression Tests were made on 0.5" diameter x 1.00" long samples of beryllium cut from part of a billet which has been cast into a water cooled mold in the Site B Foundry. Although the billet contained numerous cracks, it was possible to machine a number of flawless compression specimens from the metal. The data from these tests are now being assembled, and will be included in a report as soon as possible.

The shop is now attempting to machine some tensile specimens from the remainder of the billet, and if this can be done successfully, tensile data on the metal will be obtained.

Beryllium Extrusion: Eight beryllium extrusions were examined. All metal appeared to be very dirty and many of the samples showed longitudinal cracking which was probably due to the presence of this dirt. Some transverse cracks were found which were caused by working the metal. Remarks are presented in the following table:

No.	Metal Temp. °C	Die Temp. °C	Ram Speed in/sec.	Red. Ratio	Extrusion Press. Tons	Remarks
3.4 (10-4)	1000	580	—	16/1	65	Partial recrystallization (just started) Trans. and longi. cracks.
3.5 (10-5)	900	580	18	16/1	65-75	No recrystallization. No strain relief. Longi. cracks.
3.6 (11-2)	800	580	9	4/1	10-15	No recrystallization. Some strain relief. Longi. cracks.

044

4

No.	Metal Temp. °C	Die Temp. °C	Ram Speed in/sec.	Red. Ratio	Extrusion Press, Tons	Remarks
3.7 (11-3)	800	580	3	4/1	10-30	No recrystallization. Less strain relief than 3.6.
3.8 (13-3)	817	580	18	16/1	65	No recrystallization. No strain relief. No cracks.
3.9 (13-1)	900	580	18	16/1	65	Possible started recrystallization. Strain relief. Trans. crack.
3.10 (13-2)	900	580	60	16/1	55	Partial recrystallization (approx. 50%). Longi. cracks.
3.11 (13-4)	800	580	60	16/1	58	No recrystallization. Some strain relief. Very dirty.

MM 4.1 - Metallurgy of Other Metals and Alloys

Liquid Sodium Corrosion: A test to determine the ability of low carbon steel, medium carbon steel, 18-8 stainless steel, and 6% columbium-tuballoy alloy to withstand the static contact of liquid sodium at 500°C has been completed. The duration of the test was 5.7 days. The weight change per unit of area of the specimens after removal of the sodium by dissolution in ethyl alcohol was as follows:

Specimen Material	Weight Change, mg/cm ²	
Low Carbon Steel	1.87	Loss
Medium Carbon Steel	2.62	Loss
Tuballoy plus 6% Columbium	164.4	Loss
18-8 Stainless Steel	13.96	Gain

MM 6.1-Electroplating and Other Coatings

Pretreatment of Thorium: The following attempts were made to deposit zinc by immersion:

The surface of the metal darkened after a 1 minute dip in hot fuming H₂SO₄. There was no evidence of zinc deposition after 1 minute immersion in a boiling solution consisting of 40 grams ZnSO₄, 100 cc H₂O and 20 cc H₂SO₄. A subsequent copper plate failed the solder test.

Another piece of metal was immersed for 30 seconds in boiling H₂SiF₄ containing ZnSO₄. Considerable effervescence occurred and the thorium surface was covered with a dark grey layer, presumably zinc. The specimen was then copper plated. Although the plate failed the solder test, it was not of the egg shell type.

Columbium Plating: Most of the past week was spent in assembling apparatus for the preparation of CbF₅ and CbCl₅.

044 5
DECLASSIFIED

Beryllium Plating: A clear melt was not obtained by heating a mixture of AlF_3 Xl_2O (previously fused) and beryllium oxyfluoride to a dull red heat. The mixture was quite viscous and contained a considerable amount of undissolved material. The addition of SiF lowered the viscosity somewhat.

Corrosion rates were determined for a number of metals in 1:1 NaF - 2BeO · 5BeF_2 melt. All the following data were obtained at the same temperature, approximately 650°C . A platinum crucible was used as container for the melt. The actual time the metals were in contact with the melt varied from 15 to 25 minutes.

	Mgm/cm ² /hr.
Stainless Steel	
(Type 302)	6.5
Tantalum	65
Copper	0
Silver	0
Platinum	0
Columbium	3.6
Monel	0.3
Inconel	0.55

A bath consisting of equal parts by weight of beryllium oxyfluoride and cesium fluoride was prepared. At red heat this bath was quite viscous. The electrical conductivity of the bath was very low. A carbon anode and copper cathode were used. The cathode did not change in weight or appearance during a 20 minute test run. A D.C. potential of 2V was used.

A $\frac{1}{2}$ W-slug was Be plated in the beta range. The plate was spotty with fairly dirty interface and beryllium metal. Thickness of plate found averaged approximately .2 mils.

044

6

DECLASSIFIED

E. W. Brugmann, Section Chief
Report for Week ending March 21, 1945

Corrosion Section

Active Personnel as of March 21, 1945	38
Academic	18
Non-academic	20
Transfers in (Freddie Harding)	1
Transfers out	0

MM 1.2 - Tuballoy and Alloys

Alloy 1.1 (6% Cu, heated 2 hours at 850°C, WQ) was tested in a 1% to 2% solution of sodium chloride in the steam autoclave at 125 pounds per square inch. Results of weighing uncleaned specimens are compared with results of a similar exposure of the same alloy in distilled water:

Time, Days	1% NaCl Wt. change, mg/cm ²	Distilled Water Wt. change, mg/cm ²
1	-0.06	-
2	-0.17	-0.03
4	-0.26	0.00
6	-0.75	+0.05

Although this indicates that corrosion is considerably faster in the sodium chloride solution than in distilled water under these conditions, the indicated rate of corrosion in sodium chloride solution is remarkably low. Data on the cleaned sample are not yet available.

Alloy 1.1 has shown an induction period preliminary to reaction at 180°C with purified hydrogen (passed over Tu at 475°C) which has reached thirty-six hours so far.

The canned slug of alloy E 24 (3.4% Si, epsilon alloy) shows uneven swelling after 46.5 days in water in the steam autoclave at 178°C. The diameter has increased up to 57 mils and the length has increased 147 mils. It has been removed from test and will be opened when a suitable dry box is obtained.

In preparation for corrosion tests, 1×10^{-3} N H₂O₂ solutions were prepared and uranyl sulfate equivalent to 3 and 30 grams of metal per liter was added. It was found that sulfuric acid had to be added to pH about 1.5 and 2.0 respectively, to avoid the presence of precipitated metal peroxide.

MM 2.2 - Thorium and Alloys

Samples of hot-rolled Westinghouse metal (W-30, cold rolled 1.018" to 0.890", rolled at 1140°F from 0.890" to 0.475", rolled at 1470°F from 0.475" to 0.128") have been placed on test in water in the steam autoclave

044 7
DECLASSIFIED

at 178°C. After two days the uncleaned specimens had lost 1.40 and 0.98 grams per square centimeter. This is about the same as an older batch of sintered and extruded Westinghouse metal, where samples lost 1/2 gram per square centimeter in one day and completely disintegrated in three days.

MM 3.2 - Beryllium and Alloys

Beryllium exposed fifteen days in aerated NaCl solution at 70°C and atmospheric pressure undergoes weight change (samples cleaned with 25% HNO₃ at room temperature) as follows:

ppm Cl ⁻	Weight Loss (mg/cm ²)
1	0.08 - 0.10
5	0.12 - 0.25
10	0.75 - 1.30

MM 4.2 - Other Metals and Alloys

Further exposures of 2S aluminum rod in distilled water in a pyrex lined bomb at elevated temperature, confirm that the "disintegration temperature" is near 270°C. A sample exposed thirty-seven hours at 249°C gained 0.35 milligram per square centimeter in weight and a sample exposed fourteen hours at 254°C was slightly attacked. A third sample exposed sixteen hours at 276°C was completely disintegrated.

Potential measurements are being made on 2S and 72S aluminum in solution containing electrolytes. The effect of hydrogen peroxide is being investigated.

MM 9.2 - Survey Volume

First drafts of contributions to the Survey Volume are due April 15th, 1945.

Analytical

The following solution constituent determinations were performed by the analytical laboratory during the week:

Tuballoy and Alloys, MM 1.2	63
Other Metals and Alloys, MM 4.2	124
Others	1
Total	188

0.44

8

Fabrication Section

Report for Week Ending 3/23/45
John H. Chapin, Section Chief

Active Personnel as of 3/23/45

Total - - - - -	10
Academic - - - - -	8
Non-academic - - - - -	2
Transfers in - - - - -	0
Transfers out - - - - -	0

MM 1.3 - Fabrication of Tuballoy and Alloys

.06" thick plate of tuballoy was welded successfully with both argon shielded arc and helium shielded arc methods. Manually operated torches were used in both cases. ✓

.125" thick plates were also tried using controlled conditions as follows:

Type Shield	Current	Welding Speed	Results
11 liters/min.	85 amps.	3.5"/min.	Incomplete penetration. Uneven flow of metal and severe arc blow.
24 liters/min.	40 amps.	5"/min.	Burned thru.

A V-weld instead of a butt-weld will probably give better results.

MM 2.3 - Fabrication of Thorium and Alloys

Extrusion

2 half inch rods of thorium were extruded thru a shear die having no radius. Extrusion was accomplished without appreciable rise in pressure. Billets were copper jacketed. Severe enfolding was found on the rear end of the extruded rod. Examination of the die showed that the extruded metal tended to form its own radius on the die opening.

Cold Rolling

Three experiments were run to compare the rolling characteristics of Ames metal and Westinghouse metal.

- (1) Cold rolling-10% reduction/pass-no anneal.
 - (2) Cold rolling-5% reduction/pass-no anneal.
 - (3) Cold rolling-10% reduction/pass-annealing 1 hr. at 750°C. after each pass.
- 044 9

The Westinghouse metal showed slight lamination after 60% total reduction in each case. After the metal had been rolled to 80% reduction, 90% of the material was still in sound condition.

The Ames metal showed edge cracking after the first pass and continued up to the 30% reduction after which it ceased. The cracks only spread open on subsequent passes. The final strips after 80% reduction was only 50% usable sound metal.

Ames metal requires 50% to 100% more power for rolling than the Westinghouse metal.

Hot Rolling

Hot rolling at 1400° F. was done on billets of Ames and Westinghouse metal. Two other billets are being rolled at 1650° F. Edge cracking occurs at 1400° F. in the Ames metal at less than 10% reduction and continues up to the 30% reduction. 90% of the material is good after 80% total reduction. Ames material requires only 20% more power than Westinghouse when rolled at this temperature.

No visual improvement of the Westinghouse material was noticeable when rolled at 1400° F.

Reduction of 30%/pass can be made by hot rolling at 1400° F.

Less edge cracking is obtained on Ames metal when rolled at 1650° F. This experiment is incomplete.

Swaging

A .5" bar of extruded thorium was swaged to .375" (27.8% reduction) giving a smooth surface and no apparent cracks.

Hardness R_E	Before	After
	96.5	107

Coating of Thorium

Thorium of various thicknesses ranging from .050" to .075" is being rolled for coating experiments and corrosion testing after coating. The varied thicknesses of thorium are to be coated with 2S aluminum then rolled to a total thickness of .080"-.085".

Annealing of Thorium

Small samples of thorium are being annealed in a 50-50 mixture of KCl and NaCl at 700° C. This method is simpler than vacuum or inert gas furnace methods. The salt is readily soluble in water. A very thin black film is found on the samples but is easily removed. A sample of Westinghouse thorium cold rolled 40% reduction was annealed in this manner for 15 minutes.

R_E before annealing - 85
 R_E after annealing - 45
50

044-10

MM 3.3 - Fabrication of Beryllium and Alloys

Extrusion

Experiments have been performed in order to determine the minimum thickness of copper jackets which can be used on extrusion billets.

Billets extruded at 900° C. and 16-1 reduction having 10 and 15 mil wall copper jackets alloyed when run at fast extrusion speeds of 250 ft/min. No alloying occurred on 20 mil jackets. When run at 75 ft/min. 5, 10, 15, and 20 mil jackets did not alloy. No alloying occurs on any thickness jacket when the extrusion temperature is 800° C.

Other lubricants are to be investigated to determine the possibility of extruding the bare metal.

Swaging

A .750" extruded rod made at Wolverine Tube Division, Detroit, Michigan, was swaged at 1600° F. to .474" in 10% area reduction steps. No cracking was apparent. Further swaging to .375" (a 31.7% reduction) was too severe and the piece cracked in several places.

MM 7.3 - Service

Inspection of the tuballoy slugs from the Bureau of Standards is continuing. A separate report covering this investigation will be made.

MM 9.3 - Survey Reports

The Zinc Process has been rewritten and expanded and awaits approval. The Chicago half of the Aluminum-Silicon Process has been outlined and partially completed, while the Cleveland half will probably be held up until April 1, when the final report from that laboratory is expected.

DECLASSIFIED

844-11