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AEC RESEARCH AND DEVELOPMENT REPORT

Contract W-7401-eng-37

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PROGRESS REPORT

January 20 to January 27, 1945

METALLURGY DIVISION

CLASSIFICATION CHANGED TO
CONFIDENTIAL
DATE - 5-56
-R- [Signature]
Chief, Declassification Branch Jd.

Section M-1 F. Foote

Section M-2 E. W. Brugmann

Section M-3 J. H. Chapin

Report Received: January 27, 1945
Issued: JAN 31 1945

CLASSIFICATION CANCELLED

DATE FEB 19 1957

For The Atomic Energy Commission

-H.R. Canale
Chief, Declassification Branch

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REPORT FOR WEEK ENDING JANUARY 25, 1945

METALLURGY SECTION - FRANK FOOTE, SECTION CHIEF

Total Active Personnel as of January 25, 1945 - 36
Academic - - - - - 24
Non-Academic - - - - - 12
Transfers in - - - - - 0
Transfers out - - - - - 0

MM 1.1 - Metallurgy of Tuballoy and its Alloys

Pure Metal: A yield of 2.85 grams of tuballoy dripped from a thorium coated tungsten filament was obtained using tank hydrogen, purified only by passing through a CaSO_4 drying tube. Another run in which the hydrogen will be purified and then passed into the apparatus through a palladium thimble is to be made. The metal from these runs and from the hot tube and slow cool methods of obtaining pure metal are to be analyzed for carbon, hydrogen and oxygen. The object of the investigation is to determine the influence of these impurities on the microstructure of tuballoy prepared by these methods.

Tuballoy-Columbium Alloys: A series of 4, 9, and 21% Cb alloys was heated to 850°C slowly cooled to 655°C , soaked for three days and quenched out. All of these samples showed two-phase structures very similar to, but a little coarser than, the ones observed in the corresponding alloys treated at 600°C . However, the 21% sample had only partially transformed from single to the duplex structure. Measurements were made to judge the rate of transformation of the 21% alloy at 600°C . It was found that soaking a homogenized sample 2 hours at 600°C had no appreciable effect on the structure. After 18 hours soaking transformation had begun at grain boundaries and around inclusions. After 15 days it was complete and the second phase was spheroidized. A sample compressed 50% before annealing was almost completely transformed after 16 hours at 600°C . A series of 9, 14, and 21% Cb alloys was soaked 3 days at 725°C and quenched. All of the specimens showed single-phase structures.

The $\sin^2\theta$ values were computed for the lines of the 21% Cb alloy heat-treated 24 hours at 850°C and quenched in water. Three separate and distinct phases appeared, all of them body-centered cubic in structure. The intensities of these were relatively very strong, weak and very weak. The values for the lattice constants, respectively, are (in Angstroms): 3.413, 3.352, 3.312. These values are only approximate, particularly in the last case in which only one line with a $\sin^2\theta$ value above 0.5 was measured. It is considered probably that this substance is columbium or an impurity phase.

A Debye photogram of the 9% Cb alloy heat-treated at 850°C for 40 hours, water quenched, showed the presence of at least two body centered cubic phases, the lines of which are displaced in either direction from the two strongest patterns of the 21% Cb alloy just reported. Equilibrium was not obtained in this sample, however, and further heat treatment must be given it to sharpen the lines sufficiently for accurate measuring.

The photogram of the 48% Cb alloy heat-treated at 850°C for 40 hours and water quenched showed a body centered cubic phase which may be either the predominant phase or one of the lesser phases in the 21% Cb alloy noted above. This phase appeared to be the only body centered cubic phase present although some extra lines in this photogram may be one of the other B.C.C. patterns greatly displaced. Here again the heat treatment was not long enough to attain equilibrium.

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3.

Status of preliminary corrosion tests in boiling distilled water on the 2, 4, 9, and 21% homogenized specimens has not changed since last week.

A series of compression tests has been started on samples from a billet of tuballoy plus 6% columbium alloy. The specimen size was 0.505" diameter x 1" long. The following results were obtained:

Temp. °C	Scale, lbs.	Yield Point lbs/in ²	Load, lbs/in ² for 10% Red.	Load, lbs/in ² for 20% Red.
Room	60,000	190,000	245,000	285,000
609	30,000	28,750	25,000	21,500
604	12,000	35,000	32,000	22,000
660	12,000	6,300	6,000	6,000
670	6,000	6,150	6,100	6,150

The 6% columbium alloy appears to be very strong at room temperature, although its hardness is low enough to allow for easy machining. The yield strength dropped considerably above 600°C. Since the tests in the high alpha region showed that no greater load was needed for deformations beyond the yield point, it was assumed that annealing took place on the specimens tested at these temperatures.

Tuballoy-Aluminum Alloys: Three rolled Al-Tu samples were examined, the fabrication history of which is contained in a memo from A. B. Greninger to Foote, Chapin and Brugmann dated January 8, 1945. These samples contained 1.88, 4.95 and 9.0 wt. % tuballoy. Sections both parallel and transverse to the rolling direction showed the amount of tuballoy to be very small, making it difficult to distinguish ordinary dirt in 28 aluminum from the tuballoy. However, it was possible to see that there was a considerable amount of tuballoy in the center of the cross-section, that is, that most of the tuballoy had not been rolled to the surface.

Three as-cast Al-Tu samples of nominal 2, 4 and 15% Tu were examined. The cast structured showed primary aluminum with increasing amounts of eutectic in the grain boundaries.

Tuballoy-Antimony Alloys: Approximate weight loss rate of 0.5% alloy in boiling distilled water after 39 hours was 2.8 mg/sq.cm/hour which is half the value reported last week.

Tuballoy-Cerium Alloys: Approximate weight loss rate of 0.17% alloy in boiling water is now 0.43 mg/sq.cm/hour after 350 hours as compared to a value of 0.35 reported last week. The 0.6% alloy has an approximate weight loss rate of 1.6 mg/sq.cm/hour after 207 hours in test.

Tuballoy-Indium Alloys: A 5% In alloy, after two successive meltings, still showed two layers.

Tuballoy-Tantalum: Preliminary corrosion test results in boiling distilled water shows that all compositions have decomposed or are decomposing except the 4 and 8% Ta alloys at the end of 250 hours.

Identification of the needle phase separated from the matrix of 12% Ta alloy is continuing.

Tuballoy-Tellurium Alloys: A eutectic and an eutectoid were observed in an alloy of unknown composition. It is doubtful whether the eutectoid is part of the Tu-Te system.

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Tuballoy-Rhodium Alloys: Approximate weight loss rate of 4.6% alloy heat-treated at 850°C and water quenched was 8.1 mg/sq.cm/hour after 162 hours in test.

Tuballoy-Vanadium Alloys: Approximate weight loss rate of 0.2, 1, and 2.5% alloys are:

<u>Nominal wt. % V</u>	<u>No. Hours in Test</u>	<u>Corrosion Loss mg/sq.cm/hour</u>
0.2	206	0.59
1	208	0.29
2.5	164	0.33

Tuballoy-Zirconium Alloys: Approximate weight loss rate of 4 and 8% as cast and heat-treated at 850°C and water quenched are:

<u>Nominal wt. % Zr</u>	<u>No. Hours in Test</u>	<u>Corrosion Loss mg/sq.cm/hour</u>
4% (as cast)	350	2.1
4% (H.T.)	48	0.09
8% (as cast)	302	0.62
8% (H.T.)	46	0.01

Tuballoy-Columbium-Zirconium Alloys: Heat treated tuballoy-columbium and tuballoy-zirconium alloys have good resistance to boiling water corrosion. A series of ternary Tu-Cb-Zr alloys containing up to 8% of alloying elements are being prepared for corrosion testing.

MM 2.1 - Metallurgy of Thorium and its Alloys:

Pure Metal: Enough metal was produced by the static hot wire method in two experiments (1 gm.) to submit samples for general spectrograph and carbon analysis. Metal produced on a thorium coated tungsten filament was examined metallographically and found to be purer than metal from other sources. A small amount of a cross shaped second phase was noted.

Examination of wire rolled and swaged from a 3/16" square of Westinghouse sinter prepared from electrolytic powder to determine the recrystallization temperature was continued. As previously reported, back reflection X-ray pattern of specimens annealed at 478 to 485°C for 12 hours showed that this treatment removed the lattice distortion. Similar patterns were obtained for specimens annealed at 575-585 and 697-707°C for 12 hours, and at 810-823°C there was evidence of grain growth. Incomplete stress relief was noted for a specimen annealed 5½ hours at 300°C. Further work on metal annealed for less than twelve hours and at temperatures of 500°C and below is under way. Microscopic examination and hardness measurements are still to be completed.

Thorium-Aluminum Alloys: Thorium-aluminum alloys containing 10, 14.5 and 21% aluminum were made for metallographic examination. It was observed that these high thorium alloys disintegrated to a fine powder on standing in air.

MM 3.1 - Metallurgy of Beryllium and its Alloys

Hot Compression Tests: The series of hot compression tests started previously on an extruded beryllium rod were completed this week. The test specimens used in the earlier tests were cut from the leading end of the rod. These earlier specimens either failed in shear or showed some ductility. The samples tested this week were cut from the back end of the rod and all failed by longitudinal cracking. This effect made correlation of the new data with the former results impossible. The latest results obtained are tabulated on the following page:

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Temp. °C	Scale, lbs.	Yield Point lbs/in ²	Load, lbs/in ² for 10% Red.	Load, lbs/in ² for 20% Red.
425	30,000	10,000	20,000	25,000
500	6,000	12,000	17,500	20,000
560	6,000	12,000	19,500	25,500
605	12,000	9,000	19,000	24,500
655	6,000	8,000	15,500	19,250
700	6,000	8,000	11,750	13,500
750	6,000	3,500	6,500	8,000

MM 6.1 - Electroplating and Other Coatings

Pretreatment of Thorium: There is little or no reaction of the metal with cold, concentrated H_2SO_4 . Heat and/or anodic treatment do not increase the rate of attack appreciably. Anodic treatment of the metal in 1:1 H_2SO_4 results in considerable gassing at the metal surface, however, the rate of attack on the metal is quite low. Very little of the dark surface film is formed during this treatment, probably because the amount of metal dissolved is quite low. Crystal structure of the metal is not revealed by this treatment.

The metal promising procedure investigated to date involves anodic treatment of the metal in trichloroacetic acid followed by a dip in hot concentrated HNO_3 containing a small amount of HF to remove the dark surface film formed during the anodizing step. The metal surface appears to be quite clean after this treatment but the crystal structure of the metal is not revealed. The HF concentration was varied from 20 to 60 cc/liter. Lower HF concentrations are being investigated. The visual appearance of copper plates deposited on surfaces thus prepared is good but none have passed the solder test.

Columbium Plating: Several fused melts of Lithium and Potassium Halides containing columbium in the form of the double fluoride or the oxide were investigated with little success. A bath of the following composition has been patented for the deposition of tantalum: KCl-720 parts; KF-280 parts; K_2TaF_7 -100 parts; Ta_2O_5 -100 parts. An analogous bath was prepared using columbium compounds. Some columbium metal was obtained, however, it was very finely divided and not adherent. Higher bath temperatures are being investigated.

Beryllium Platings: Beryllium was deposited on Copper, Platinum and Tuballoy. The deposits were dull and rather brittle. Alloying of the plate and base metal occurred in each instance. The melt was contained in a platinum crucible and heat was supplied by a gas burner. Graphite anodes were used. A furnace with a graphite pot will be used in the future, the pot serving as anode. Efficiency based on weight gain of the cathode is about 30%.

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Corrosion Section

E. W. Brugmann, Section Chief
Report for week ending Jan. 26, 1945

Active Personnel as of January 26, 1945	39
Academic	18
Non-academic	21
Transfers in	None
Transfers out (Edwin Poole)	1

MM 1.2 - Tuballoy and Alloys

Unbonded tuballoy slugs with pinholed aluminum (2S) jackets have failed in the internally heated test after 38-40 hours operation at 100% heat input. Although the daily operation schedule was approximately 8 hours with heat on, followed by 16 hours with heat off, the accumulated hours operation at 100% heat input up to time of failure is the same as for continuous operation with 100% heat input. Cooling water pressure of 225-140 lbs/sq.in. gave the same result as when the cooling water pressure was 95-35 lbs/sq.in., with the possible exception that the final rate of swelling was higher for the higher water pressure. Pimple growth started at about 15 hours operation at 100% heat input, but failure was caused by pimples which appeared at a later time.

The reaction of tuballoy with purified hydrogen (over Tu turnings at atmospheric pressure and 475°C) proceeded at 100°C and constant pressure of one atmosphere at rates up to about 1260 mg of Tu/cm²/hr. An induction period of 18 hours preceded the reaction. This might be due to air introduced through a small leak found in the system later.

The reaction of tuballoy with hydrogen (purified at atmospheric pressure by contact with platinized carbon and a liquid nitrogen trap) in a constant volume system has been reported previously to be 848 mg/cm²/hr at 100°C. In this case, the sample was vibrated to remove hydride as it formed from the metal surface. An attempt to detect further reaction between the corrosion product, presumably TuH₃, and hydrogen produced negative results. This indicates that the corrosion product does not contain free metal.

Boiling distilled water tests continue to show low corrosion rates for alloys of silicon (epsilon) and columbium, with the 6% alloy of the latter looking the best (.001 to .002 mg/cm²/hr in a 189 hour test). The corrosion product was removed from samples of columbium alloys with 25% HNO₃. Nitric acid removes the corrosion product from all columbium alloys if the correct concentration and temperature are used. For the 6% alloy, a 10 minute dip in 25% HNO₃ at 75°C was used. Lower temperatures at the same concentration are generally effective for alloys of lower columbium content. Correction is made in all cases for the clean metal attacked by the acid. It is usually small compared to the weight of the corrosion product.

Two tests on alloy 1,4 (5% Mo, 2 hour heat treatment at 850°C, then water quenched) have given average rates of metal loss of 0.48 mg/cm²/hr for an 8-1/2 day run and 0.21 mg/cm²/hr for a 4 day run in boiling distilled water. Samples were cleaned with 25% HNO₃ at room temperature, correcting for the metal dissolved by the acid.

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MM 2.2 - Thorium and Alloys

Tests in beakers have given the following results:

Temp. °C	Solution	Sample	Wt. loss after cleaning, mg	Time, Hours	Average Metal Loss from start mg/cm ² /hr
100	Boiling distilled water	W-0	20.3	4	0.57
			48.8	24	.23
			42.6	72	.07
70	Water, aerated 1×10^{-3} N H ₂ O ₂ , pH 7.4, 20 ppm Ca ⁺⁺ , 6 ppm Mg ⁺⁺ , 80 ppm SO ₄ ⁼ , 1 ppm Cl ⁻	W-1	360 & 400	397	0.17 & .18
		A-1	400 & 640	397	0.14 & .20

W-0 is extruded Westinghouse metal from an old batch; W-1, extruded Westinghouse metal from the batch of that number, and A-1 extruded Ames metal from the batch of that number. The cleaning procedure used was not too good, consisting of cleaning in concentrated nitric acid containing a small amount of NaF.

A specimen of Westinghouse metal (old batch) alloyed with 0.6% Tu was tested in water in the steam autoclave at 125 lbs/sq.in. The weight changes are as follows:

1-1/2 days ... +18.8 mg/cm² (wt. of specimen)
4 days +163.5 mg/cm² (wt. of corrosion product)

The specimen had completely disintegrated in four days to a black and white powder. The increase in weight of the corrosion product over the initial weight of the specimen was sufficient to account for conversion of only 63% of the metal to dioxide.

MM 3.2 - Beryllium and Alloys

The experiments are under way in distilled water and in NaCl solutions both saturated with oxygen and oxygen free. Due to the shortage of equipment, the only data obtained during this week correspond to the change of weight occurring in distilled water saturated with oxygen after 15 days of exposure. Together with the previously reported result these data seem to indicate a self-stopping reaction.

The change in weight in air saturated distilled water (pH=7.2) at 90° was found to be -

after 1 day	7.5 . 10 ⁻⁴ mg/cm ²	loss of weight
" 3 days	3.0 . 10 ⁻⁴ "	" "
" 7 days	3.2 . 10 ⁻⁴ "	gain in weight
" 15 days	3.6 . 10 ⁻⁴ "	" "

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MM 4.2 - Other Metals and Alloys

Data on the corrosion of aluminum-Tu sheets from Battelle in water under steam at 125 lbs/sq.in. are given for 2, 4 and 8 days. The specimens were cleaned after 2 days, but were not cleaned after the four and eight day periods because the chromic-phosphoric acid stripping solution did not work very well.

Alloy Sample	Wt.% Tu	Sample Weight Change, mg/cm ²		
		2 days	4 days	8 days
1-1 A	1.88	+ 0.24	+ 0.33	+ 0.33
1-1 B		+ .28	+ .38	+ .38
4-2 A	4.95	+ .23	+ .33	+ .29
4-2 B		+ .26	+ .31	+ .30
6-2 A	9.00	+ .20	+ .30	+ .29
6-2 B		+ .21	+ .31	+ .29

It appears that a film has built up on the alloys and that the corrosion has slowed down. These alloys were rolled and annealed.

It has been reported previously that in stirred beakers, 72S aluminum is anodic to 2S aluminum at hydrogen peroxide concentrations below about 1×10^{-4} N and cathodic when the peroxide concentration is greater than that figure. The solution used was water at 70°C and pH 7.5, containing 10 ppm SiO_2 , 2 ppm $\text{Na}_2\text{Cr}_2\text{O}_7 \cdot 2\text{H}_2\text{O}$, 20 ppm Ca^{++} , 6 ppm Mg^{++} , 80 ppm SO_4^{--} , 1 ppm Cl^- . Recent runs at peroxide concentrations from 5×10^{-4} to 1×10^{-2} N have checked the old conclusion. In recirculating systems where the solution velocity was about 15 ft/sec, however, the 72S aluminum remained anodic to the 2S aluminum at all peroxide concentrations used.

Analytical

The number of solution constituent determinations performed by the analytical laboratory during the past week follows:

Tuballoy and Alloys, MM 1.2	9
Thorium and Alloys, mm 2.2	10
Other Metals, mm 4.2	80
Others	4
Total	103

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Fabrication Section

Report for Week Ending 1/26/45
John H. Chapin, Section Chief

Active Personnel as of 1/26/45

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

MM 1.3 - Fabrication of Tuballoy and Alloys

No work was done on the fabrication of Tuballoy or its alloys during this period.

MM 2.3 - Fabrication of Thorium and Alloys

Two billets of thorium have been prepared for hot extrusion. The experimental extrusion press was operated this past week using brass billets for testing the equipment. A 1" diameter 2" long billet was extruded to .25" x 28" long at a temperature of 1350°F. No pressure registered on the gauge. On the second brass billet the container broke and no further work was done. New tools will be ready for extrusion experiments on 1/27/45, at which time the thorium will be extruded.

The tooling for the 950 ton horizontal extrusion press at Alcoa have been designed and are being sent to R. W. Andrews for criticisms and suggestions.

Ames billet A2 is being sectioned for rolling experiments, hot compression testings, and tensile tests.

MM 3.3 - Fabrication of Beryllium and Alloys

In preliminary forging of Beryllium a 1 1/8" round rod was flattened to 1/2" along 4" of the longitudinal section without cracking. The rod was heated to 800° C. before forging.

Another rod was given a reduction from 1 1/8" diameter to 1/2" dia. by hot swaging at 800° C. Only the point was swaged because of the severe reduction and the low capacity of the swaging machine. Only one die size was available and the experiment is to be continued when tools are available.

1/8" thick Beryllium plates have been welded using a 1/8" diameter Tungsten electrode in an Argon atmosphere. Three welds were made using the atomic hydrogen transformer. 91 amperes current were used at 16 volts. The Argon flow was 8 liters per minute. Two welds were made using a GE transformer (6 WK 18 A-9) at 130 amperes by machine setting. Excessive cratering was evident due to high

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heat input. By increasing the rate of welding the cratering decreased in amount. Oxidation occurred when only 8 liters/min. of Argon were used. 10 liters or more are required.

MM 4.3 - Fabrication of Other Metals and Alloys

Experimental work is being continued on the welding of the long tube cartridge.

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