AECD-3832

-1-
Contract No. W-7405, eng. 26

TECHNICAL DIVISION

Section IV, Engineering Materials

F. Kerze, Section Chief

Date Declassified: December 7, 1955

PREPARATION OF SAMPLES OF 42 WEIGHT
PERCENT U-AL ALLOY FOR PILE IRRADIATION TESTS

by

C. D. Smith

February 15, 1949

Period Covered by this Report - June 14 to Aug. 20, 1948

DATE ISSUED

MAR 8 - 1949

Photostat Price \$ 3-30

Microfilm Price \$ 2-40

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

This document is
PUBLICLY RELEASABLE

H. Kinsler
Authorizing Official

Date: 5/8/07

639-1

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.

639-3

EXPERIMENTAL WORK

Since the material was to be rolled, the question was raised by the writer as to what effect, if any, hot rolling would have on grain size. It was hypothesized that two possible mechanisms might apply:

- 1) Since the chill-cast metal was in an unstable condition, heating might promote grain growth.⁴
- 2) Since the massive Al-U grains are hard and brittle, working might tend to break them up.

Before processing of the enriched material was started, trial runs were made with natural uranium alloy to determine the effect of hot working on grain size of ingots cast under conditions which gave various cooling rates. Graphite molds were prepared which gave ingots 3" high, 1" wide and ranging from 3/4" to 1/8" in thickness. Six of the ingots were cast in cold molds and, for further variation in cooling rate, two ingots were cast in molds heated to approximately 200°C. One cold steel mold was also used.

Several hundred grams of 42 wt.% U-Al alloy were melted in a graphite crucible by means of an induction furnace, heated to 1200°C and nine ingots were cast as indicated in the tabulation below.

639-4

<u>Ingot</u>	<u>Ingot Thickness, Inches</u>	<u>Mold</u>	<u>Analysis, wt.% U</u>	<u>Approx. Al-U Compound Grain Size, Inches</u>
1	3/4	Cold Graphite	42.04	0.0025
2	1/2	" "	42.70	0.0020
3	3/8	" "	42.30	0.0005
4	1/4	" "	41.90	"
5	1/8	" "	---*	"
6	1/8	" "	42.14	"
7	1/8	Hot Graphite	41.40	0.0015
8	1/4	Hot Graphite	41.42	0.0015
9	3/8	Cold Steel	41.71	0.0020

The thinner ingots 4, 5, 6, 7 and 8 had poor surfaces due to the rapid cooling rate. Samples were cut from the top and bottom of each ingot for metallographic examination. The ingots were then annealed for one hour at 1000°F and additional metallographic samples were prepared.

The ingots were jacketed with aluminum frames and covers and hot-rolled at 1000°F with total reductions of about 50% and 70%. During this rolling, ingot 4 broke into small pieces. Jackets were removed and the thicker ingots 1, 2, 3, 8, and 9 further hot-rolled to 0.060". The surface of the alloys after this rolling was far superior to that of the alloys rolled with the cover sheets. Samples for chemical analysis and metallographic examination were cut after rolling.

* Sample not obtained because the aluminum cladding could not be removed.

639-5

METALLOGRAPHY

The samples were mounted in bakelite and polished using standard techniques applicable to aluminum alloys. These were examined at 2X and 100X. Measurements were made of the grain size and are tabulated above. Photomicrographs of ingots 1, 3, and 6 in the as-cast and hot-rolled condition are appended. Results are as follows:

As-Cast: All ingots showed a dendritic structure characteristic of castings (upper photomicrographs). The ingot tops showed considerable porosity as expected.

The progressive decrease in grain size with decrease in ingot thickness and subsequent increase in cooling rate, is shown in ingots 1 thru 6, ranging from 0.0025" to 0.0005".

Ingots 7 and 8 (cast in hot graphite molds) show larger grain size than the corresponding ingots 4 and 5 which were cast in cold graphite molds.

Ingot 9 (cast in a steel mold) shows a larger grain size than the corresponding ingot 3 (cast in cold graphite), indicating a more rapid rate of cooling in the latter case.

Annealed: No difference between these and the as-cast samples.

Hot-Rolled 50%, 70%: Voids were eliminated and dendritic structure broken up but grain size remained the same as in the as-cast samples (lower photomicrographs).

Hot-Rolled to 0.060": Only ingots 1, 2, 3, 8 and 9 were thick enough for further rolling. Again, examination failed to disclose any change in grain size from that obtained in the cast samples.

639-6

From the above work with natural uranium the following conclusions can be drawn:

- 1) Minimum grain size is achieved with castings 3/8" or less in thickness.
- 2) Heating to 1000°F and hot-rolling breaks up the dendritic structure of the casting but does not affect the massive Al-U grain size.
- 3) It is not feasible to cast sections less than 1/4" thick using the above technique.

PROCESSING OF ENRICHED URANIUM

A total of 76.254 grams of U_3O_8 (uranium enriched with U^{235}) was received. Impurities in ppm. were as follows:

Ni	-	400
Si	-	20
Mg	-	10
Hg	-	<10
Ca	-	40
Cu	-	300
B	-	< 0.1
Fe	-	400

A sample submitted to the Analytical Section showed the material to contain 84.39% U.

The uranium was reduced and alloyed as follows:

Charge:

<u>Material</u>	<u>Weight, gm.</u>
U_3O_8	75.766
Al for reduction	12.87
Al for alloying	88.43
Cryolite	303.0

639-7

Procedure:

1. Mix U_3O_8 and cryolite in can made of aluminum foil.
2. Melt aluminum in graphite crucible.
3. Add can of U_3O_8 - cryolite mix.
4. Heat to $1200^{\circ}C$.
5. Hold 30 minutes; stir every 5 minutes with graphite rod.
6. Remove graphite rod; shut off power; allow crucible to cool for 15 minutes.
7. Remove crucible from furnace; cool to room temperature.
8. Break crucible; separate slag and metal.

The metal pig was then remelted in air and cast from $1200^{\circ}C$ in a graphite mold measuring approximately $2" \times 3" \times 1/4"$. The ingot was sheared to $1" \times 2" \times 1/4"$, fitted in an aluminum frame, coated with a slurry of MgO to prevent sticking and covered with a $1/8"$ sheet of aluminum. This assembly was hot-rolled at $1100^{\circ}F$ with reductions of 10% per pass for a total of 50%. The cover and frame were removed and the slab further hot-rolled to 0.060" with reductions of 10% per pass. A strip measuring $3.2" \times 0.391"$ was sheared from the middle of the hot-rolled slab. An additional small piece was sheared for analysis.

All of the remaining metal was remelted in air, cast from $1200^{\circ}C$ in a cold graphite mold giving an ingot measuring approximately $1-1/4" \times 2-1/2" \times 5/8"$. This was jacketed with aluminum and hot-rolled to 0.060" as was the first ingot (jacket removed after 50% reduction). Again a strip was sheared from the middle of the hot-rolled slab and an additional small sample taken for analysis.

639-8

Analyses were as follows:

1/4" ingot - 39.9% U

5/8" ingot - 41.97% U

Using a micrometer guide set at 0.150" on a small foot-operated shear, six samples were cut from each strip.

Weights and Uranium Contents are listed below as follows:

1/4" Ingot

<u>Sample</u>	<u>Weight, gm.</u>	<u>Uranium, gm.</u>
1	0.2362	0.0942
2	0.2482	0.0990
3	0.2476	0.0988
4	0.2465	0.0983
5	0.2468	0.0985
6	0.2490	0.0993

5/8" Ingot

<u>Sample</u>	<u>Weight, gm.</u>	<u>Uranium, gm.</u>
1	0.2306	0.0968
2	0.2337	0.0981
3	0.2336	0.0981
4	0.2339	0.0982
5	0.2295	0.0964
6	0.2320	0.0974

Since all the samples were within specification both dimensionally and chemically they were transferred to the Atomic Energy Commission for shipment to Knolls Atomic Power Laboratory.

Approved:

Frank Kerze
Frank Kerze

C.D. Smith
C. D. Smith

639-9

ACKNOWLEDGEMENT

Acknowledgement is made for assistance in this work as follows:

Melting and Casting: G. M. Adamson, C. F. Cutcher
Rolling : H. J. Wallace, F. M. Blacksher
Photomicrography : G. M. Carlton
Shearing : J. N. Hix

REFERENCES

- (1) BMI-P-5 Progress Report on Work at Battelle During Month of May 1948.
- (2) BMI-HRN-No. 5 Memo on Work at Battelle During Month of February 1947.
- (3) Journal of Metallurgy and Ceramics, (TID-65), No. 1, July 1948, p. 10.
- (4) BMI-P-6 Progress Report on Work at Battelle During Month of June 1948.

639-10

As-Cast

Hot-Rolled 70%

Ingot 1, 100X, no etch

639-11

As-Cast

Hot-Rolled 50%

Ingot 3, 100X, no etch

639-12

As-Cast

Hot-Rolled 50%
Ingot 6, 100X, no etch

639-13