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Subject Category: Metallurgy
and Ceramics.

ATTEMPTS TO MANUFACTURE URANIUM -
ZIRCONIUM ALLOYS BY CO-REDUCTION
OF THEIR TETRAFLUORIDES.

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Title: ATTEMPTS TO MANUFACTURE
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SUMMARY

A series of tests were made to determine whether alloys of uranium and zirconium could be produced by the co-reduction of uranium and zirconium tetrafluorides with magnesium in normal uranium production bombs using magnesium fluoride as a liner. No operational difficulties were encountered but the metal produced showed stratification in the low ranges of zirconium content (0.2-0.4%) and was heterogeneous in the higher ranges (up to 5%) of zirconium content.

Operational and analytical data are included.

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EXPERIMENTAL RESULTS

Several runs were made in an effort to achieve a uniform alloy of uranium and zirconium by co-reduction of their respective tetrafluorides with magnesium in bombs lined with ground slag. This slag was essentially magnesium fluoride with magnesium oxide, calcium fluoride and uranium oxides present as impurities.

The reaction by which zirconium tetrafluoride is reduced to the metal is not much more highly exothermic than the reduction of uranium tetrafluoride

For this reason it was considered that the reaction did not constitute a greater safety hazard than normal uranium reduction bombs⁽¹⁾ considering the zirconium concentration necessary in the alloys. Therefore, standard reduction conditions used in normal uranium reductions were used.

The following table outlines the operational results in the firing of these alloy bombs.

- (1) Complete physical description of the uranium reduction facilities in the pilot plant was presented in NYO Report No. 1334 by L. G. Weber, issued June 24, 1952.

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CO-REDUCTION OF URANIUM AND ZIRCONIUM TETRAFLUORIDES

Run No.	Alloy Desired	Lbs. UF_4 ⁽¹⁾ Charged	Lbs. ZrF_4 Charged	Theoretical Yield, Lbs.	Actual ⁽²⁾ Yield, %	Firing ⁽⁴⁾ Time
430	0.2% Zr	140.0	0.38	106.4	94.9	2 hrs:10 min.
431	0.4% Zr	140.0	0.77	106.6	87.6	2:23
432	0.6% Zr	140.0	1.15	106.8	94.2	2:13
433	0.8% Zr	140.8	1.53	107.0	93.9	1:58
434	1.0% Zr	140.0	1.92	107.2	94.7	2:03
441	1.5% Zr	135.0	2.88	104.0	91.2	2:05
435	2.0% Zr	135.0	3.83	104.5	94.7	2:04
429	2.0% Zr	135.0	3.83	104.5	94.4	4:09 ⁽³⁾
436	5.0% Zr	125.0	8.87	99.6	98.4	2:20
437	5.0% Zr	125.0	8.87	99.6	100.	2:25

- (1) Four percent excess magnesium used on all bombs (stoichiometric excess).
- (2) The percentage yield assumes that the "derby" is 100% metal with no slag mixed in it.
- (3) This bomb was lined with a 3/4" thick side liner at the top tapering to a 1" thick side liner at the bottom while all the rest were 1/2" thick at the top and 3/4" thick at the bottom.
- (4) The furnace control temperature was 1190°F. on all bombs.

A visible stratification of material in the derby metal was noted in all of these runs and was most evident in the low zirconium alloys where a pronounced layer, slightly yellow in color, was seen at the top of the derby when a saw cut was made. In the higher percent zirconium range, this stratification was not evident but the metal did have a heterogeneous appearance. Samples for zirconium analysis were taken of the metal at several spots in the derby by drilling the metal with a steel drill. Results of these are shown below.

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ZIRCONIUM ANALYSES IN ALLOY DERBIES

Run No.	Desired Alloy	% Zirconium		
		Top of Derby	1/2" Below Top of Derby	Bottom of Derby
430	0.2% Zr	1.34	0.11	0.09
431	0.4% Zr	1.61	0.21	0.23
432	0.6% Zr	0.47	--	0.52
433	0.8% Zr	0.63	--	0.64
434	1.0% Zr	0.85	--	0.78
441	1.5% Zr	1.75	--	1.29
435	2.0% Zr	1.79	--	1.84
429	2.0% Zr	2.04	--	1.71
436	5.0% Zr	5.08	--	4.13
437	5.0% Zr	5.84	--	4.21

The extent of stratification in the 0.2% and 0.4% Zr derbies is evident from these figures. Incomplete reduction of the zirconium tetrafluoride or failure of the zirconium metal to separate into the derby mass is evident from the analyses of the 0.6%, 0.8% and 1.0% Zr runs.

Other analyses performed on this metal are listed in the table below.

ANALYTICAL DATA ON ZIRCONIUM-URANIUM ALLOYS

Run	N ₂ %	Spectographic Results, ppm							Density gm/cc	
		Fe	B	Mn	Mg	Ag	Ni	Co	Top	Bottom
430	0.0020	28	<0.20	8.9	1000	<1	10	<10	19.00	19.00
431	0.0021	42	"	12	1000	"	10	"	18.92	18.97
432	0.0020	82	"	13	300	"	10	"	18.58	18.84
433	0.0012	38	"	19	1000	"	10	"	18.06	18.78
434	0.0015	47	"	25	500	"	10	"	18.21	18.74
441	0.0032	300	"	35	500	"	100	"	18.19	18.17
435	0.0018	43	"	8.7	50	"	10	"	---	---
429	0.0014	400	"	55	500	"	45	"	---	---
436	0.0033	39	"	75	1000	"	10	"	---	---
437	0.0031	35	"	45	1000	"	10	"	---	---

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