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**THE PRODUCTION OF PLUTONIUM METAL
BY DIRECT REDUCTION OF THE OXIDE**

W. Z. Wade

T. Wolf

February 22, 1968

UNIVERSITY of CALIFORNIA
LIVERMORE

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Lawrence Radiation Laboratory
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THE PRODUCTION OF PLUTONIUM METAL BY DIRECT REDUCTION OF THE OXIDE

Abstract

A single-unit process was developed for the rapid production of massive plutonium metal directly from its oxides. The process consists of the following steps: the reduction of plutonium oxides with calcium metal, the extraction of the calcium oxide reaction product with molten calcium chloride, and the quantitative separation of the two immiscible molten-

metal phases by the immiscible salt solution.

Reduction, separation, and massive metal yield efficiencies are in excess of 99.96%; batch cycle time (completely independent of batch scale) is 15 min; and the single waste product is far below recovery level and is discardable as a small-volume solid waste.

Introduction

Innumerable papers have been published on the current processes used for plutonium metal production and shall not be referenced herein. They have in common a transitional aqueous-to-anhydrous process which entails product losses in multiunit operations and, in turn, necessitates many more unit operations to recover that material contained in aqueous and solid waste streams. Figure 1 shows a generalized flowsheet of these current processes.

Successful preparation of massive plutonium metal has been dependent upon the requirements that the reaction constituents be capable of evolving sufficient heat to melt the resulting slag and keeping it molten long enough to allow the plutonium

metal to coalesce into a massive regulus. Thus, the melting point of the slag formed has necessitated the conversion of plutonium dioxide into a halide prior to reduction (see Table I).

It is obvious, therefore, that the PuO_2 reduction (also shown on Table I for comparison) evoked little interest, due to the refractory nature of the sludge (CaO) which imprisoned the finely divided plutonium metal. Jenkins¹ et al. reported the results of experiments wherein PuO_2 was reacted using a CaCl_2 flux. They were unsuccessful in obtaining massive plutonium metal over various scales ranging from 25 mg to 60 g. Cleveland² recently commented that (based on literature published to date) the direct PuO_2 reduction,

Table I. Reduction reactions for the preparation of plutonium metal.

Reaction	H at 291°K (kcal/mole plutonium)	Melting point of slag (°C)
Current processes		
1. $\text{PuF}_4 + 2\text{Ca} = \text{Pu} + 2\text{CaF}_2$	-149.5	1414
2. $\text{PuF}_4 + 0.3\text{I}_2 + 2.3\text{Ca} = \text{Pu} + 0.3\text{CaI}_2 + \text{CaF}_2$	-196.0	1320
3. $\text{PuF}_3 + \frac{3}{2}\text{Ca} = \text{Pu} + \frac{3}{2}\text{CaF}_2$	-54.8	1414
4. $\text{PuCl}_3 + \frac{3}{2}\text{Ca} = \text{Pu} + \frac{3}{2}\text{CaCl}_2$	-56.0	772
Proposed process		
$\text{PuO}_2 + \text{Ca} = \text{Pu} + 2\text{CaO}$	-52.4	2572

using a CaF_2 flux, was apparently not a suitable preparative route.

However, the solubility of CaO in CaCl_2 (as well as the solubility of other alkaline and alkaline earth metal oxides in their respective salts) warranted a more exhaustive investigation. Its potential, as incorporated into a successful plutonium reduction process, would eliminate explosive and neutron hazards and extensive, uneconomical recovery processes.

Consequently, two years have been spent in developing three successive models of reaction vessels, with their design

parameters determined by over sixty experimental reductions.

The results have been successful and a single-unit batch-type process has been developed. This process demonstrates an overall plutonium yield efficiency in excess of 99.96%; an absence of any recoverable plutonium in waste products since the products were far below permissible residual activity (and, therefore, discardable); a 15-min cycle time per batch (independent of scale); and no increase in total impurity level than was already existing in the PuO_2 feed material.

Experimental

A description of the units evolved in these development studies are given below.

EQUIPMENT

The Mark I reaction vessel is shown in Fig. 2. It consisted of a 525-cc MgO crucible, packed into a Hastelloy bomb with

MgO sand. Thermite firing temperatures were attained within an 8-in. induction coil powered by a 30-kW Tocco motor-generator.

The Mark II reaction vessel is shown in Fig. 3. It consisted of a magnesia crucible within a stainless steel secondary container. A variable-speed electrically

Fig. 1. Generalized flowsheet of current plutonium reduction processes (including necessary recovery units). Flowsheet in upper left corner is of the proposed process (no recovery units required).

Fig. 2. Mark I reaction vessel used in experimental runs Nos. 1 through 8.

driven stirrer with tantalum blades and shaft provided agitation. Heating was accomplished with a 1850-W resistance tube furnace which encircled a quartz sleeve. Temperature was controlled with a Wheelco controller and monitored with a Westronics multipoint recorder. The reaction vessel was moved in and out of the furnace area by a standard laboratory jack and a lift rod.

The Mark III reaction vessel is shown in Fig. 4. It consisted of a welded tantalum permanent reaction vessel rotated by a Boston Ratiotrol motor-reducer system which provided precise control of revolutions in the range of 0 to 180 rpm. The entire furnace and rotational device were mounted on a pivoting platform to permit the tilting of the apparatus from the normal 45° angle used in reaction and separa-

Fig. 3. Mark II plutonium reduction apparatus used in experimental runs Nos. 9 through 31.

tion to a negative angle for the top-pouring of the reactor contents.

A final production prototype unit, the Mark IV reaction vessel, has been designed and is shown in Fig. 5. It consists of a permanent reaction-separation vessel fabricated of tungsten. The vessel has cylindrical walls, a conical bottom, and a bottom-pour casting plug. Heating, temperature control, and rotation are accomplished with equipment that is larger, but similar to that described in the Mark III unit.

Fig. 4. Mark III plutonium reduction apparatus used in experimental runs Nos. 32 through 62.

Mold design used in these castings is shown in Fig. 5. Machined from graphite, they were used throughout these experiments without significant degeneration. All mold preheaters were constructed from muffle furnace resistance heating elements, suitably insulated, and controlled by a Wheelco temperature controller.

The CaCl_2 melter is a bottom-pour tungsten vessel with custom-fitted heating elements and insulation; the temperature is controlled by a Wheelco temperature controller in series with an autotransformer. This salt preheater is also shown in Fig. 5.

All equipment was located and operated within an inert atmosphere (argon) gloved box because of chemical reactivity as well as the radiation contamination hazard.

REAGENTS

The PuO_2 feed material (in 1130-g batches) was obtained from the Dow Chem-

ical Company's Rocky Flats Division, Golden, Colorado. One batch was of exceptionally pure material (~500 ppm total impurities) for use in the study of impurity pickup during reduction to the metal. The remaining batches were of production grade PuO_2 for direct simulation of existing feed materials. Some of the batch analyses are incorporated into Tables VII and VIII where they are presented for comparative purposes with analyses of daughter metal produced.

The CaCl_2 for the extractive and separative medium in this process was obtained from Semi-elements, Inc., Saxonburg, Pennsylvania. The CaCl_2 (obtained in 100-lb lots) was of their "optical" grade, which is normally used in the production of single crystals. An analysis of this material is shown in Table II.

The CaF_2 for the mold coating (excellent release properties) was also obtained from Semi-elements, Inc. and was of the

Fig. 5. Mark IV plutonium reduction apparatus for semiproduction runs. The apparatus is shown in the charging, reaction, and casting position. Unit is tilted 45° and reaction vessel is rotated for the extraction step.

Table II. Analyses of calcium halides used in plutonium reductions.^a

Element	Impurities (ppm)		Element	Impurities (ppm)		Element	Impurities (ppm)	
	CaF ₂	CaCl ₂		CaF ₂	CaCl ₂		CaF ₂	CaCl ₂
Ag	5	1	K	10	ND	Si	25	10
Al	5	10	Li	10	ND	Sn	5	<10
Ba	10	10	Mg	25	<10	Sr	25	100
Cr	1	1	Mn	5	<10	Ti	5	<10
Cu	5	<5	Na	500	—			
Fe	10	<10	Pb	5	ND			

^aElements not detected in either compound were: As, B, Be, Bi, Cd, Co, Hg, Mo, Ni, Pd, Pr, Rb, Sb, Ta, V, Zn.

same optical grade. Analysis of the CaF_2 is shown also in Table II.

The calcium metal used in these studies was supplied by the United Mineral and Chemical Corporation, New York. Detectable impurities were Mg, 3000 ppm; N_2 , 18 ppm; Mn, 13 ppm; Al, 39 ppm; and Fe, 13 ppm.

PROCEDURES

No attempt shall be made to detail the evolution of optimum operating parameters; instead, the final procedure developed for each apparatus shall be listed.

Maximum efficiencies were obtained with the Mark II apparatus using the following procedure:

1. $\text{Ca} + \text{PuO}_2$ charge is added at 700°C , fires at $\sim 800^\circ\text{C}$.
2. CaCl_2 is added at 900°C .
3. Contents are stirred at 300 rpm for 15 min; temperature is raised to 1000°C during agitation.
4. Stirrer is turned off, contents are allowed to settle, and furnace is turned off.
5. When the temperature drops to 950°C , the charge is lowered and cooled rapidly.
6. MgO crucible is broken open; the button is etched with dilute HNO_3 , weighed, and sampled.
7. Salt is sampled and discarded.

Mark III reaction apparatus procedure:

1. Reactor is maintained at 900°C .
2. $\text{Ca} + \text{PuO}_2$ charge is added, fires at 740°C .
3. CaCl_2 is added at 950°C .

4. Reactor is rotated at 30 rpm for 12 to 15 min, temperature $\sim 950^\circ\text{C}$.
5. Rotation is stopped, and contents settle for several minutes.
6. Reactor contents are tilted and poured into CaF_2 -coated mold heated to 700°C .
7. Mold is cooled rapidly, inverted, and tapped on a hard surface. The solid salt and massive plutonium metal ingot fall free and independent of each other.
8. Metal and salt are handled as with Mark II procedure.

Optimum procedure designed to be used in the Mark IV:

1. Reactor and salt melter are maintained at 950°C .
2. A charge of PuO_2 and a stoichiometric amount of Ca necessary to react with the oxide + a 50% excess is added. Charge fires immediately.
3. Molten CaCl_2 (an amount 33% in excess of that amount needed to take the CaO product into solution) is added.
4. Reactor is rotated at 30 rpm ($188 \text{ in./min} = 15.7 \text{ ft/min}$ tangential velocity) for 14 min at 950°C .
5. Plutonium metal plus a small amount of CaCl_2 is bottom-poured into mold, (mold starting temperature is 700°C).
6. Bulk of CaCl_2 is bottom-poured into discardable metal can.
7. Metal and salt are handled as with Mark II, No. 7 procedure.
8. Charging and casting consumes ~ 1 min and rotation, ~ 14 min to give a total time of ~ 15 min.

Results and Discussion

Initially, a study of the solubility of CaO in molten CaCl_2 was conducted to expand and confirm the value reported by Seidell³. A second study investigated the solubility of Ca in CaCl_2 . The results of these determinations are summarized in Figs. 6 and 7, respectively. Figure 8 illustrates the relative free energies of

Fig. 6. Solubility of CaO in CaCl_2 .

Fig. 7. Solubility of Ca in CaCl_2 .

formation and the driving energy available for the reaction

to go to completion.

A series of eight static thermite experiments, using CaCl_2 as a flux, were con-

Fig. 8. Free energy of formation.

ducted to confirm the results of Jenkins *et al.*¹ variations of blended and unblended layer structures within the bomb were investigated by using 80-g PuO_2 charges (69.2-g Pu content), 300-g charges of CaCl_2 and 60-g charges of Ca.

One extreme arrangement of charge packing was the placement of a blend of the two reactants (to insure maximum contact) above, in the middle, or below the salt charge. The packed bomb was then fired at 1000°C and held at that temperature for 1 hr.

The other extreme was a uniform blend of all three components (Ca, CaCl_2 , and PuO_2). The remainder of the runs were variations and combinations of these two extremes.

In the first example, when the two reactants, Ca and PuO_2 , are charged as a mass with intimate contact, the ultimate in reduction efficiency occurred but the

minimum in metal yield efficiency. With no agitation, very little of the CaO went into solution and no massive plutonium metal could form.

Conversely, when the two reactants were blended with the diluting salt, the results indicated that while the CaO formed as small particles and went into solution easily, only a small amount of the PuO₂ made contact with the calcium reactant. Upon reaching liquidity the calcium droplets would float to the top of the melt and the insoluble PuO₂ would sink to the bottom. Thus in this other extreme of charging, the minimum of reduction efficiency and the maximum of massive metal yield efficiency was obtained.

The results of the experiments, summarized in Table III, substantiated our theory that agitation is necessary after the reduction reaction is complete to effect the dissolution of the CaO by the CaCl₂, thereby releasing the finely entrapped plutonium metal. The results of this statically fluxed system corroborate Jenkin's¹ results, and Cleveland's² observations.

The Mark II reactor was fabricated to provide this needed agitation. A total of

23 experimental runs were made before its data production potential was exhausted and its deficiencies completely determined. The PuO₂ charges in these runs ranged from 50- to 200-g plutonium content.

The initial procedure with the Mark II reactor entailed utilizing a molten, immiscible, two-phase system consisting of CaCl₂ with a layer of calcium floating on top. The PuO₂ powder was introduced, and it reacted immediately with the agitated calcium reductant. The heavy plutonium metal entrapped in the solid reaction product drifted down through the agitated CaCl₂, which dissolved the CaO, allowing the Pu metal to collect on the bottom of the vessel. Typical results of this technique are shown in Table IV.

The final procedure for the Mark II reactor evolved was to charge the Ca and PuO₂ as a blend (reduces at ~800°C), add the CaCl₂, and begin agitation. This proved far superior to the initial procedure. Typical results of this technique are shown in Table V.

In either case, it was demonstrated that fair yields were obtainable by this agitation technique, and that the final

Table III. Results of static-flux plutonium reductions.

Static layer structure	Yield efficiency, massive metal obtained (%)	Estimated reduction efficiency by PuO ₂ remaining (%)
Charge on top of salt	0	~90
Charge in middle of salt	0	~96
Charge on bottom of salt	0	~99+
Uniform blend	16	84
Highest yield from combination mixes	43	57

Table IV. Results of PuO_2 reductions using calcium float.

Run number	PuO ₂ charge (g)	Pu content (g)	Ca charge (g)	CaCl ₂ solvent (g)	Operating temp (°C)	Pu metal yield		Time stirred (min)
						g	%	
11	77.0	66.6	32.0	500	1000	50.9	76.5	30
12	77.0	66.6	32.0	500	1000	39.5	60.0	20
13	57.7	49.9	32.0	500	900	22.3	44.6	5
14	57.7	49.9	32.0	500	950	38.7	77.6	20
15	57.7	49.9	32.0	500	1000	30.2	60.5	5
16	57.7	49.9	32.0	500	1000	32.8	67.0	30
Total		332.8				214.4	64.4	

Table V. Results of PuO_2 reductions using prefired Ca/ PuO_2 charge.

Run number	PuO ₂ charge (g)	Pu content (g)	Ca charge (g)	CaCl ₂ solvent (g)	Operating temp (°C)	Pu metal yield		Time stirred (min)
						g	%	
26	55.4	48.0	32.0	650	1000	44.9	93.5	30
27	55.7	48.2	32.0	650	950	45.5	94.4	30
28	56.8	49.1	32.0	500	900	43.2	88.0	22
29	56.8	49.1	32.0	550	900	48.2	98.0	30
30	56.8	49.1	32.0	550	900	47.7	97.0	45
31	56.8	49.1	24.0	600	900	46.8	95.5	30
Total		292.6				276.3	94.4	

material content of the vessel was divided into three immiscible strata.

The design deficiencies found in the Mark II equipment never permitted an exact quantitative yield.

In the initial procedure, the necessary agitation caused a variable percentage of the PuO_2 added to the two-phase Ca/CaCl₂ system to contact the salt phase without being reduced and to settle immediately to the bottom. This PuO_2 , in essence, remained isolated from further contact with the reductant calcium float. These losses are evident in Table IV, wherein PuO_2 with a total plutonium content of 332.8 g

was charged, and yet produced only a 214.4-g metal yield (64.4% overall yield efficiency).

In the final procedure, the PuO_2 was quantitatively reduced, and, as the stirrer swept the surface of the CaO/Pu/Ca sludge, the Ca was separated and floated to the top of the melt. Simultaneously, the CaO was taken into solution until the entire surface of the dense bottom phase consisted of pure molten plutonium metal. However, when this occurred, a small amount of the CaO/Pu sludge remained on the crucible bottom and was protected by this covering layer of plutonium metal

from the dissolving action of the agitated CaCl_2 solution. This small loss each run is evident in Table V, wherein PuO_2 with a total plutonium content of 292.6 g was charged, and yielded 276.3 g of plutonium metal (94.4% overall yield efficiency).

These deficiencies led directly to the remedial design of the Mark III reaction apparatus. In this reactor, during the extraction cycle, the gentle rotation of the inclined vessel allows the undissolved CaO/Pu sludge to be bathed alternately in the dissolving salt and then in the collecting pool of plutonium metal. These one-second alternations continue for 12 to 15 min at 950°C . At the end of this time, all of the CaO has been dissolved by the molten salt and the contents have assumed a true three-phase immiscible liquid system and are ready for casting.

Once the operating conditions had been determined, an overall yield of essentially 100% was realized. A consecutive series of runs are shown in Table VI as typical results of reductions made once optimum conditions were evolved. One deficiency of this apparatus was the variable product holdup experienced from run to run; this

produced no overall losses, however the holdup was caused primarily by top-pouring over the cool projecting lip of the vessel. While the yield efficiencies varied from run to run, the overall efficiency of the runs tabulated in Table VI indicate that when PuO_2 with a 300.52-g plutonium content was charged, a yield of 300.72 g of plutonium metal was obtained (100.3% yield efficiency).

The only true losses occurring in these runs is that which is discarded with the fused CaO/CaCl_2 solution. These are negligible (below discard level) and are shown in Table VI. All salt wastes varied from 10^{-5} to 10^{-6} g Pu/g CaCl_2 . The excess calcium metal from each run was normally recycled to the next reduction. However, it may be discarded without significant loss occurring. In three of the four calcium residues assayed for plutonium by alpha counting, the alpha emission was below background level of the analytical counters. The one detectable sample indicated a content of 1.1×10^{-6} g Pu/g Ca .

The Mark III process neither contributes any impurities to the total of that

Table VI. Typical reduction results with the Mark III apparatus.

Run number	PuO_2 charge (g)	Pu content (g)	CaCl_2 solvent (g)	Pu metal yield		Pu losses (g Pu/g CaCl_2)	Extraction temp ($^\circ\text{C}$)
				g	%		
35	57.71	49.52	775	55.82	112.3	6.49×10^{-5}	950
36	57.65	49.66	575	46.64	94.0	2.01×10^{-6}	950
37	58.00	49.97	600	52.77	105.6	3.12×10^{-5}	950
38	57.86	49.85	570	36.96	74.2	2.50×10^{-5}	900
39	57.77	49.77	550	58.15	116.8	1.34×10^{-5}	950
40	57.75	49.75	600	49.38	99.3	2.48×10^{-5}	950
Total		298.52		299.72	100.3		

already existing in the feed material, nor does it purify the plutonium content in any significant manner. Analytical results from three consecutive reductions, which were made from the same batch of PuO_2 feed, are shown in Table VII.

The total detectable impurity level in the produced metal retains the same general value as that of the feed stock. The only apparent changes are a reduction of the chromium content in the plutonium metal vs that in the starting PuO_2 , and an increase in the aluminum content in the metal vs that in the oxide.

Another series of four runs were made to study the distribution behavior of the americium content. One reduction was a control, wherein no AmO_2 was added to the PuO_2 feed stock; three other reductions had AmO_2 added to the PuO_2 in consecutive order-of-magnitude concentrations. The results of these four reductions are shown in Table VIII.

As may have been expected under these extreme reducing conditions of excess calcium, the americium reduced and remained in the high density plutonium metal

phase. No appreciable decontamination occurred at any americium concentration level shown in Table VIII.

Since the Mark III reactor/separator was an interim model, the permanent reaction vessel used in its construction was fabricated of tantalum, because of quick access and easy fabrication. Thus, the second deficiency of the Mark III design was expected and did evidence itself as tantalum impurities introduced into the product metal.

Consequently, the Mark IV model, now under construction, shall have its permanent reactor/separator vessel fabricated from deposited tungsten. Past experience has proven tungsten to be the best material for constant use in this type of environmental application. The tungsten impurity contributed to the product should be nominal. The other deficiency, the varying product holdup due to the top-pouring should be corrected by the standard bottom-pour casting design.

Thus, the Mark IV unit is a final prototype unit and will be used for semiproduction studies in conjunction with studies of a direct calciner to be constructed shortly.

Table VII. Typical impurity levels in PuO_2 feed vs reduced metal.^a

Element	Impurities (ppm)				Element	Impurities (ppm)			
	Oxide feed	Run A1	Run A2	Run A3		Oxide feed	Run A1	Run A2	Run A3
Ag	<0.5	1	<0.5	<0.5	Fe	200	160	250	125
Al	75	150	120	220	Mg	10	200	50	50
B	30	30	30	30	Mn	25	25	25	25
Ca	200	200	200	220	Pb	15	2	<1	<1
Cr	100	5	5	25	Si	450	440	450	430
Cu	5	10	5	3	Totals	1110	1223	1135	1128

^aThe elements below limits of detection were: As, Au, Ba, Be, Bi, Cd, Ga, In, K, Mo, Ni, P, Pd, Sb, Sn, Sr, Ti, V, and Zn.

Table VIII. Distribution of americium in reduction products.

Location	Run Number 58		Run Number 59		Run Number 60		Run Number 61	
	ppm	g	ppm	g	ppm	g	ppm	g
Am present ^a in PuO ₂	58	0.0014	58	0.0014	58	0.0014	58	0.0014
Am added (as AmO ₂)	0	0	76	0.0039	720	0.0369	7288	0.3741
Total Am in feed	58	0.0014	134	0.0053	778	0.0383	7346	0.3755
Am in Pu metal	62		162		704		5700	
Am in excess Ca metal	2.2		b		9.3		17.6	
Am in CaCl ₂ CaO solution	3.6 × 10 ⁻³		5.4 × 10 ⁻³		2.9 × 10 ⁻²		8.8 × 10 ⁻²	

^aThe PuO₂ used in these reductions had a 208.5 g plutonium content and yielded 210.3 g plutonium metal (100.8% efficiency).

^bNo calcium sample for this run.

Conclusions

A simple one-unit chemical process for the direct reduction of PuO₂ to plutonium metal and the quantitative separation of reaction products has been demonstrated.

A prime economic advantage of this process is its complete lack of any waste recovery requirements.

No current process attains the yield efficiency of this process.

Because of the great rapidity and simplicity of this process, all costs (reagents, units, manhours, space, production time/kilogram, etc.) are several orders of magnitude less than that required by presently used processes.

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