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2. O.H. Greager - W.K. Woods
3. F.W. Albaugh
4. J.J. Cadwell
5. O.J. Wick
6. V.R. Cooper
7. T. Prudich
8. W.N. Mobley
9. R. Ward
10. M.J. Sanderson
11. L.D. Turner
12. O.F. Hill - K.M. Harmon
13. O.F. Beaulieu - E.G. Pierick
14. L.F. Kendall
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AUTHORITY AOC [✓ ADC] ADD
NAME: J W JORDAN
ORG: PNNL
2nd REVIEW-DATE: 6/10/81
NAME: A E Baker
ORG: PNNL

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PM Eck 4-24-00
JE Savely 8-27-99

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TECHNICAL ACTIVITIES REPORT

PRODUCT METALLURGY - APPLIED RESEARCH SUB - SECTION

JULY 1953

8-7-53

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SUMMARY

METALLOGRAPHIC PREPARATION

The metallographic hoods and equipment have been completed and operate satisfactorily. Practice runs with non-radioactive materials are being made prior to plutonium work.

The Tukon hardness tester has been realigned, tested with calibration blocks, and is now ready for service.

STRESS-RUPTURE STUDIES

Stress-rupture tests have been carried out on the former RG line bomb casing material. The carbon steel specimens were tested under conditions simulating the standard and off-standard pressures and temperatures which have been observed in actual plant operations. The RG line casing material was found to rupture after about 63 and 7 cycles for standard and off-standard conditions, respectively.

THERMAL ANALYSIS

Since the thermal analysis hood has been sealed and an expanded range added to the recorder-controller, the entire apparatus is now ready for plutonium investigations.

PHYSICAL PROPERTIES OF PLUTONIUM

A plutonium tensile specimen was prepared by pouring molten plutonium into a magnesia mold. The "as-cast" plutonium test piece was pulled in tension until failure which occurred at the location of an oxide inclusion within the reduced area of the specimen. The use of SR-4 strain gages provided sufficient data for the calculation of the Modulus of Elasticity. The value of this constant was found to be about 13.7 x 10^6 psi which indicates that a considerable fraction of the "as-cast" metal contained alpha phase plutonium.

PLUTONIUM SKULL STUDIES

If plutonium oxide is reduced to the metal by molten barium, a method for the continuous recovery of plutonium from skulls might be feasible. Further, this operation may be of the "open-pot" type providing the molten barium is covered by a low melting flux.

A portion of a plutonium skull and its associated oxide was charged into a melt containing barium metal covered with a calcium chloride flux. Since this low density flux was appreciably soluble in plutonium metal, the separation of plutonium metal from the slag was poor. In addition, the dissolution of the protective flux in plutonium permitted the oxidation of barium and plutonium by air.

WET PLUTONIUM FLUORIDE STUDIES

Ammonium plutonium fluoride cannot be used in sealed reduction bombs since ammonium

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fluoride volatilises at elevated temperatures and raises the internal pressure of the bomb beyond safe operating limits. If the plutonium double salt is furnace dried at 250 to 300 C, the evolved ammonium fluoride is found to condense and plug off-gas lines. Further, bomb reductions carried out on plutonium fluorides prepared by wet methods have, thus far, produced metal of low quality. The use of the ammonium plutonium fluoride in plant operation does not appear feasible.

Calcium plutonium fluoride is easily prepared, washed, and filtered. Upon drying, the anhydrous fluoride is found to be composed of hard lumps. The use of this material in the reduction bomb would not, however, introduce any foreign substance into the reaction mass. The first bomb reduction carried out produced plutonium metal of poor quality. Additional bomb reductions are being performed with higher booster contents.

ELECTROLESS NICKEL PLATING STUDIES

Electroless nickel plating of plutonium has been shown by other workers to be impractical since the surface of the metal is rapidly oxidised by the aqueous solvent before the nickel can form a protective layer. The electroless process might prove feasible, however, if the plating solution were organic in nature. Preliminary experiments employing alcohol and dioxane electroless plating baths have shown the nickel coats to be non-adherent.

Experiments are now in progress to determine whether nickel can be deposited from an aqueous electroless plating bath onto plutonium or cerium metal which has been wet by gallium.

ELECTRODEPOSITION OF NICKEL FROM FUSED QUATERNARY SALTS

Since a hydrous oxide film forms on the surface of plutonium or cerium prior to the deposition of nickel from an electroless plating bath, attempts are now being made to electrodeposit a thin layer of nickel upon cerium and plutonium from a fused organic bath prior to the electroless step. Sound nickel coats have been electrodeposited upon copper from a nickel chloride - ethyl pyridinium bromide molten bath. Cerium metal could not be plated from this bath, however, since it was rapidly attacked by the plating bath. Pyridine hydrobromide, an impurity in the ethyl pyridinium bromide, is most likely responsible for the corrosive nature of the molten plating bath.

REPLACEMENT OF HYDROFLUORIC ACID IN 234-5 OPERATIONS BY PHOSGENE

Preliminary corrosion experiments have shown phosgene to rapidly attack most common materials of construction at temperatures of about 600 C. Since phosgene is an excellent chlorinating agent with cerium dioxide at temperatures as low as 450 C, further corrosion testing has been carried out at this lower temperature. Since Hastelloy A, B, and C are found to be attacked at a rate of only 0.002-inches per month, and since plutonium dioxide can most likely be chlorinated at temperatures below 450 C, additional corrosion tests will be performed.

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The plutonium remaining in the slag and crucible fragments after a trichloride reduction can be recovered through Recuplex facilities providing chloride ion is first removed. Although the bulk of the chloride ion can be removed by water washing, about 0.1 g/l Cl is found in the Recuplex solvent extraction feed solution. The presence of chloride ion at this point is attributed to the slow dissolution of partially oxidized plutonium chloride or to the slow leaching of soluble chlorides which had adsorbed into the magnesia crucible fragments during bomb firing. The presence of about 100 ppm of chloride in the dissolver is probably tolerable, but corrosion tests must be carried out to firm up this conclusion.

Cerium trichloride reductions have been carried out in a sealed bomb containing a resistance wound heater and several concentric heat shields. This type of bomb design may permit the elimination of internal boosters. Preliminary bomb reductions using cerium chloride and no booster have resulted in metal yields as high as 93.8 percent.

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PRODUCT METALLURGY

I. METALLOGRAPHIC PREPARATION - L.P. Morgan

The individual items of metallographic preparation equipment have been tested and operate satisfactorily. One or more practice runs using cerium specimens will be made under operating conditions to perfect technique before insertion of plutonium into the hoods.

The accuracy of the Tukon microhardness tester has been determined using the calibrated blocks. Values obtained differed by no more than 6% from the calibrated hardness when loads of 100 grams or larger were used. The difference increased to 15% when the 25 gram load was used. This variance in low-load hardness values has been reported by several workers but has not been attributed to any particular factor.

II. STRESS-RUPTURE STUDIES - L.G. Merker

A stress-rupture testing program to determine a suitable steel for the fabrication of plutonium reduction bombs in the RM Line is being carried out. A reduction bomb that will give satisfactory service and provide a large safety factor is desired. The relation between life to rupture under steady state conditions and life to rupture under cyclic heating and loading has not been well established. The uncertainty in this relationship makes it difficult to determine accurately if a given material will satisfy the above requirements. A testing machine to simulate the stresses and temperatures observed in the plant reduction cycle is being used. This machine was designed by the Product Metallurgy Group and built on plant.

For a normal reduction, the internal pressure of the bomb does not exceed 105 psig at a maximum temperature of 750 C. This corresponds to a calculated hoop stress of 760 psi in the proposed design for the new RM reduction facilities. For an off-standard run with an internal pressure of 450 psig, the calculated hoop stress is 3259 psi. All tests will be run under these two conditions.

Tests have been run on the steel that was formerly used for the RG Line reduction bombs. Analysis of this steel showed it to contain 0.11 percent carbon. Previous calculations had shown that this material could safely be used for 12 cycles. The number of cycles which were required to rupture this bomb can material under standard and off-standard conditions was 63 and about seven, respectively.

III. LABORATORY FACILITIES - L.G. Merker

The lathe hood has now been sealed. A leak rate of three tenths cubic foot per hour has been obtained.

IV. THERMAL ANALYSIS - M.D. Freshley

Hood 18 has been sealed and is now ready for plutonium work. The final leak rate is something less than 0.1 cubic feet per hour at a pressure of 3 inches of water on the hood. This rate is considered more than adequate for the successful operation of the hood.

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A zero to five millivolt range has been added to the Leeds and Northrup Micromax recorder-controller and is now being tested. Oscillations in the temperature difference between the nichrome cylinders in the furnace became apparent due to the increased sensitivity of the recorder, and considerable difficulty has been encountered in attempting to eliminate these variations. This undesirable effect is being eliminated by making compensating adjustments on the duration-adjustment type control. The expanded range works very satisfactorily, however, as it affords a finer adjustment of the setting, and also, the desired setting can be reproduced without difficulty.

V. PHYSICAL PROPERTIES OF PLUTONIUM - T.C. Nelson

A plutonium tensile specimen was prepared by pouring molten plutonium into a magnesia mold. The as-cast piece was pulled in tension until failure. Failure occurred prior to the yield point at the location of an oxide inclusion which was outside the gage area but within the reduced area of the specimen.

Closer scrutiny of the X-Ray photographs indicated a slight scarcity of material at this location. The fracture area was perpendicular to this oxide inclusion. About one-half of the total cross sectional area was occupied by this flaw. The freshley broken area was bright and silky in appearance while the flaw was heavily oxidized.

The modulus of elasticity obtained by the use of SR-4 strain gages adhered directly to the specimen varied between 13.4 and 14.1 x 10^6 psi on several cycles in the low elastic range (see Figure I). Some previous doubt has existed that the material was completely delta stabilized. A value of 14 million psi definitely indicates that considerable alpha plutonium was present. Microscopic examinations and hardness tests should verify this hypothesis.

The strain on the cast test bar was measured by using SR-4 strain gages cemented directly onto the piece. One active and one temperature compensating gage were used in bridge circuit with two fixed resistors. This hookup minimizes the effects of variations in cable and terminal contact resistance upon the strain measurements because both sources of error are then external to the gage bridge. There was some erratic behavior as depicted by a rapidly wavering indicator needle. The use of resistors in the bridge was suspected of causing this unsteadiness.

To check the values of strain obtained on the plutonium specimen, several strain gages were applied to a large steel test bar and loaded with dead weights on the stress rupture machine. The comparison of five different tensile sensitive hookups are shown in Table I.

Run IV was a duplicate of the plutonium tensile setup. The deviation from the mean was fairly low, hence it can be safely concluded that the method used to obtain the modulus values on the plutonium tensile test piece was satisfactory.

These tests being destructive, it is desirable to apply the minimum number of gages to any one specimen. Hence, in future testing of plutonium a hookup as used in Run III will probably become standard. The 4.7% deviation is small enough to warrant its use. When greater sensitivity is desired, two active gages will be used.

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TABLE I

COMPARISON OF VARIOUS STRAIN GAGE BRIDGE CIRCUITS

Type A-5-1 SR-4 Gages cemented to steel bar ½" x 1¼" x 12". Identical loads applied in approximately 120 pound incremements from 0 to 1500 psi for all runs.

Run	Figure	Active Gages	Temperature Compensating Gages	Fixed Resistors	Temperature Compensated?	Modulus of Elasticity x 10⁶ psi	Deviation from Average %
I	A	2	2	0	Yes	29.8	6.3
II	B	2	2	0	Yes	27.2	2.9
III	C	1	1	0	Yes	26.7	4.7
IV	D	1	1	2	Yes	27.0 28.3	3.6 0.9
V	E	2	0	2	No	29.2	4.1

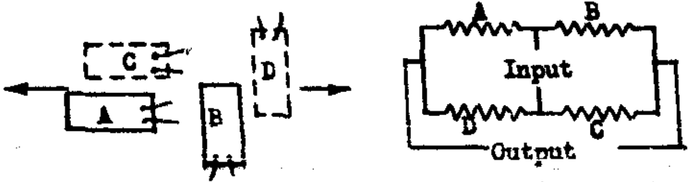

Figure A. Very stable and sensitive due to twice output of single gage - insensitive to bending.

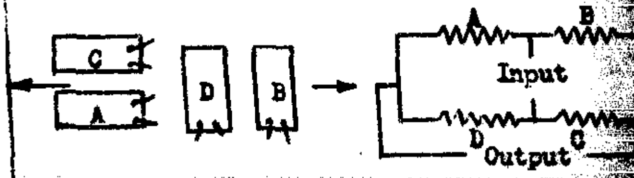

Figure B. Extremely stable - twice output of single gage.

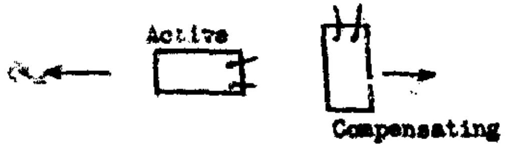

Figure C. No bridge circuit - wired directly to SR-4 indicator.

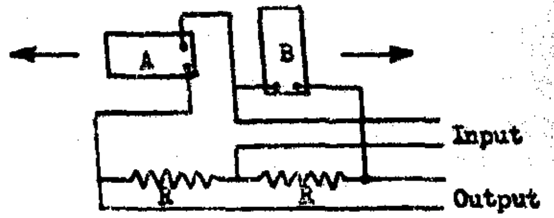

Figure D. Same circuit as used on plutoni
Sensitive to bending.

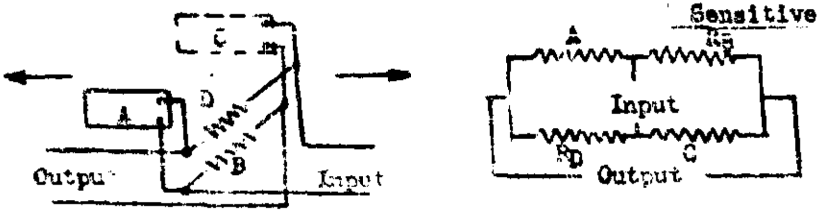

Figure E. Least stable - not temperature compensated. Sensitive to axial tension only.

Note: Arrows indicate direction of strain.

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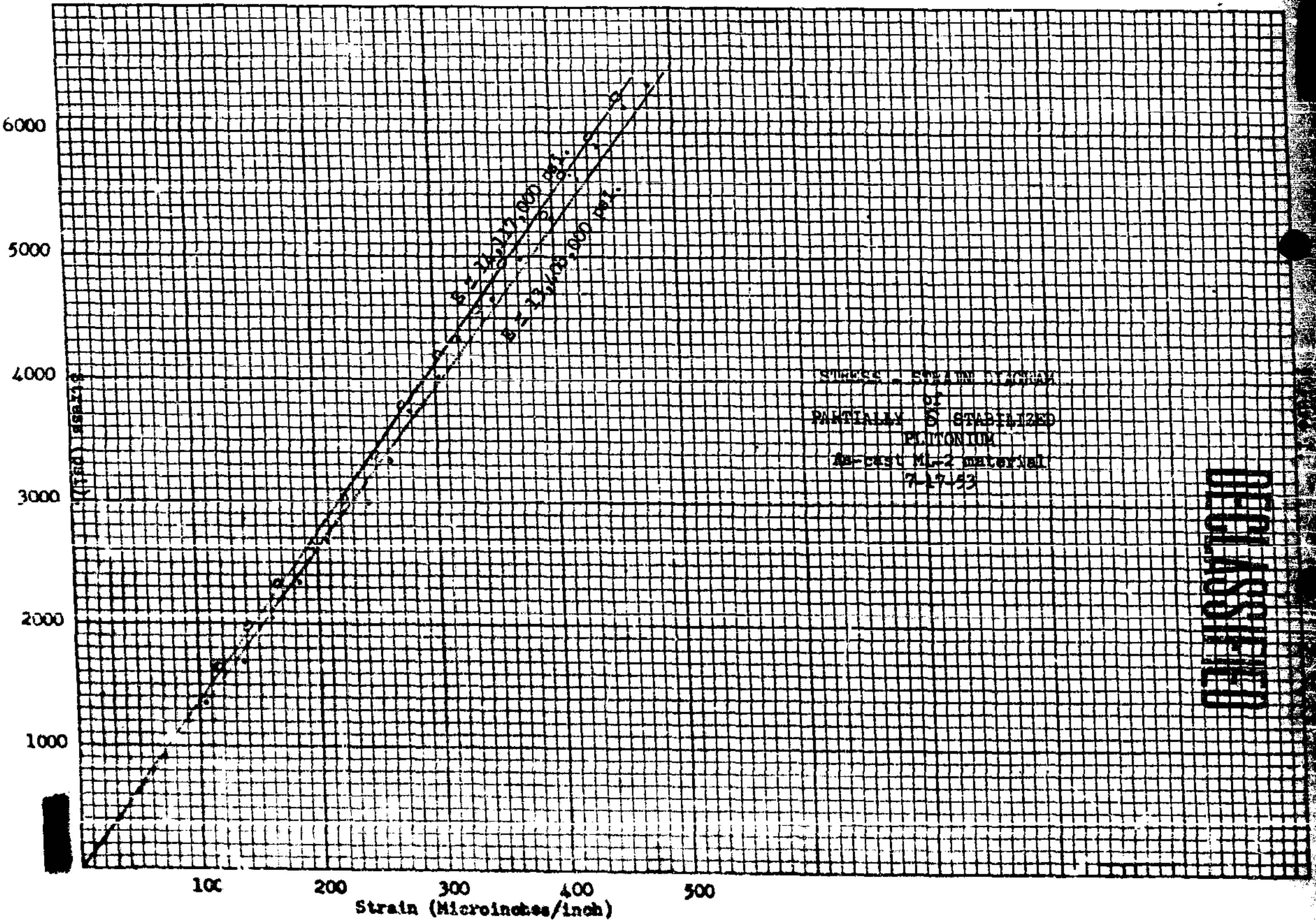

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STRESS - STRAIN DIAGRAM
of
PARTIALLY δ STABILIZED
PLUTONIUM
As-cast ML-2 material
7-17-53

VI. SKULL REMELTING - I.D. Thomas

A number of attempts have been made to obtain a molten pool of barium metal protected by a flux so that the skull material could be charged into the melt with the possible reduction and recovery of the plutonium oxide skins. The low melting fluxes used to date have all contained alkaline earth chlorides which are appreciably soluble in the alkaline earth metals and cerium. A preliminary run with plutonium also exhibited this solubility with calcium chloride.

The solubility of magnesium chloride in magnesium is one of the major difficulties encountered in the electrowinning of magnesium from molten chloride baths. The magnesium chloride is removed in the refining process by use of a viscous flux made up of calcium fluoride, magnesium chloride and magnesium oxide. The metathesis reaction

$$\Delta F_{1000K} = 7{,}000\,\mathrm{Cal}$$

$$CaF_2 + MgCl_2 \longrightarrow CaCl_2 + MgF_2$$

$$\Delta F_{1500K} = 17{,}000\,\mathrm{Cal}$$

converts the magnesium chloride to fluoride, and the composition of the flux is varied so the chlorides and oxides are removed from the metal.

On the basis of preliminary runs the refining principals used in the magnesium industry seem to apply in the case of plutonium. A very fluid slag exhibited appreciable solubility in the metal with poor seperation of slag and metal while a viscous slag gave good button seperation.

VII. WET PLUTONIUM FLUORIDE STUDIES - P.B. .ranin

A. Ammonium Double Salt

One more batch of ammonium plutonium (IV) fluoride was precipitated under the conditions described in last month's report.(1) A vacuum line which provided a pressure of less than one millimeter of mercury was set up in a glove box. An attempt was made to dry the precipitate at 50 to 100 C in this vacuum system, but no change in the appearance of the solid or its weight was observed. The fluoride was finally heated in a stagnant atmosphere of air; water was evolved just above 100 C, and ammonium fluoride which volatilized at 250 to 300 C plugged the lines from the furnace tube. The solid obtained analyzed 23.0 percent fluoride and 73.8 percent plutonium, or F/Pu ratio of 3.93. Reduction of the product was attempted in a 50 g bomb, using 30 percent excess calcium for reduction and 0.6 moles of calcium and iodine per mole of plutonium as booster. The bomb fired at 650 C, but the resulting button was poorly shaped, lumpy, and had occluded slag. Much of the metal formed dissolved when pickled in nitric acid, so no yield could be

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(1) Reas, W.H. "Technical Activities Report - Plant Metallurgy, Applied Research Sub - Section, June 1953", p.7, Document HW-28489 (Secret).

determined. Since no leak occurred during the firing, it was felt that the bomb temperature was not maintained sufficiently high long enough to allow the metal to coalesce and separate from the slag.

B. Calcium Double Salt

Another method of precipitating plutonium fluoride was sought to avoid the problem of having ammonium fluoride plug the process lines. The calcium double salt seemed feasible since it would introduce no new impurities to the system or to the slag during reduction.

A series of experiments was performed to determine the solubility of the salt as a function of nitric acid and hydrofluoric acid concentrations and to observe general behavior of the precipitate with regard to settling, washing, filtering, drying, etc. For each of the experiments, 200 mg of plutonium from AT solution were reduced to Pu (IV) with hydrogen peroxide.

Calcium nitrate was added rapidly. Solubilities were determined by sampling supernatants after the precipitates had settled. The solubility data are presented in Table II.

TABLE II

PLUTONIUM SOLUBILITY AS A FUNCTION OF HYDROFLUORIC ACID AND NITRIC ACID CONCENTRATIONS

Initial Pu conc. = 40 g/l
Initial Ca conc. = 6.7 g.l

	Plutonium Solubility, g/l HNO_3, M				
HF, M	1.0	1.4	2.0	3.0	4.0
1.0	0.34	0.60	0.71	0.16	0.38
2.0	0.17	0.20	0.27	1.0	0.062
3.0	0.22	0.29	0.28	0.17	0.091
4.0	0.33	0.24	0.28	0.16	0.12
5.0	0.25	0.20	0.019	0.028	0.098

In general, settling times increased and bulk densities decreased with increasing nitric acid concentration. Bulk densities ranged from ca. 0.3 g/cm^3 at 1 M HNO_3 to 0.1 g/cm^3 at 4 M HNO_3. No apparent difference in behavior was noticed when the solutions were heated before precipitation. In all cases, the precipitates could be washed with water, and in fact, settled better after being washed than before. They were readily filtered but caked badly when dried at 120 C. A bulk density of the crushed cake was ca. 1.0 g/cm^3.

If the results of a reduction of the precipitated fluoride are encouraging, further experiments will be performed.

VIII. ELECTROLESS NICKEL PLATING INVESTIGATION - R.C. Smith

For a long time high concentrations of hypophosphite have been known to reduce nickel salts to metallic nickel. However, Brenner and Riddell (1) using lower concentrations of hypophosphite found the reaction to be autocatalytic in the presence of certain metals including nickel. The reaction by which nickel is produced may be summarized as:

$$Ni^{++} + H_2PO_2^- + H_2O \longrightarrow Ni^0 + H_2PO_3^- + 2H^+$$

At the same time hydrogen is liberated from hypophosphite oxidation by water.

$$H_2PO_2^- + H_2O \longrightarrow H_2PO_3^- + H_2$$

Hopkins (2) reported as an invention the electroless nickel plating of uranium and plutonium in 1950 and was able to nickel plate uranium successfully; however, the more electropositive plutonium would not take an adherent coating. It is believed that the oxidation of plutonium in an aqueous medium is too rapid to allow a satisfactory coating to form on the plutonium. The emphasis of the present investigation is directed toward overcoming this difficulty.

Thirteen electroless plating runs have been made thus far in an attempt to find a non-aqueous plating bath. Nickel sulfates, chlorides, nitrates and acetates have been tried in solvents such as alcohol and dioxane using iron and cerium as "stand-ins" for plutonium metal. Since no adherent coatings have been obtained and because cerium dissolved in the alcoholic electroless solutions, no further work is planned with either alcohol or dioxane.

The possibility of wetting the surface of plutonium with some suitable material which may be coated in an aqueous electroless bath is being investigated. Experiments are now underway to use gallium for this purpose.

IX. ELECTRODEPOSITION OF NICKEL FROM FUSED QUATERNARY SALTS - L.F. Kendall

One plausible explanation for the failure of the electroless plating method to produce a satisfactory coat on plutonium is that a hydrous oxide forms by reaction of the plutonium with the aqueous solvent. The bonding forces across the plutonium - plutonium oxide - nickel interfaces being small, parting of the nickel occurs readily. One possible remedy for this difficulty is to apply a thin, continuous nickel coat by electrodeposition from an anhydrous medium followed by electroless plating. Because

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(1) Brenner, A. and Riddell, G. "Deposition of Nickel and Cobalt by Chemical Reduction", J. Res. Nat. Bur., 37, 385 - 395 (1947).

(2) Hopkins, H.H., Jr., "Electroless Nickel Plating", Document HW-18894 (1950) (Secret)

[redacted]

of the geometrical complexity of the shapes to be plated, electrolytic deposition alone would not give the uniform thickness required

In the course of their work on electrodeposition of metals from fused quaternary ammonium salts. Hurley and Wier obtained metallic deposits of several metals, including nickel, on a platinum cathode by electrolyzing solutions of the anhydrous chlorides in ethyl pyridinium chloride. (1) Efforts to duplicate these results produced adherent plates of nickel on platinum. Nickel plated on copper by electrolysis at about 25 ma/cm^2 for 15-30 minutes was found to be free of holes when evaluated by spot tests.

Upon substituting pieces of cerium for copper as the cathode, however, it was found that the metal was rapidly attacked, and that this phenomenon also occurred in the molten ethyl pyridinium bromide alone. Hydrolysis products of the organic salt and /or pyridine hydrobromide, the product of a side reaction in the preparation, are currently suspected as being responsible for this behavior. Present work is expected to eliminate these undesirable products.

X. PREPARATION OF PLUTONIUM TRICHLORIDE WITH PHOSGENE - W.P. Tolley

A. Corrosion of Metals by Phosgene

In preliminary investigations the corrosion of metals with phosgene at 600 C was found to be excessive. Since phosgene has proved an excellent chlorinating agent with cerium dioxide at temperatures as low as 450 C, it was felt desirable to test its corrosive action at this lower temperature.

The metals were placed separately in a quartz tube, and phosgene was passed over them for five hours at 450 C. The tube was cooled and then heated a second time to 450 C for three more hours without disturbing the metal sample. The corrosion data obtained in these experiments are presented in Table III.

TABLE III

METAL CORROSION BY PHOSGENE AT 450 C

Metals	Corrosion Rate(a) (in/mo)
Hastelloy A	2.03×10^{-3}
Hastelloy B	1.45×10^{-3}
Hastelloy C	2.69×10^{-3}
Hastelloy D	1.68×10^{-2}
Haynes 25	1.40×10^{-2}
Tantalum	3.07×10^{-2}

(a) Each test was carried out for eight hours. The data was extrapolated to one month.

The corrosion at 450 C is low enough to encourage some additional investigations.

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(1) Hurley, F.H. and Wier, T.P., "Electrodepositions of Metals from Fused Quaternary Ammonium Salts", J. Electrochem. Soc., 98, 203 (1951).

Experiments will be conducted to measure the corrosion occurring during actual chlorination of cerium dioxide.

B. Recovery of Plutonium from Slag and Crucible

The plutonium remaining in the slag and crucible after a trichloride reduction can be returned to process through Recuplex providing the chloride ion can first be removed. To avoid corrosion of stainless steel equipment, the solutions containing chloride must not be made acidic. In the procedure being investigated the slag and crucible from the reduction bomb are leached with water. The pH is adjusted with sulfamic acid and ammonium hydroxide, so the plutonium precipitates as the hydroxide while the calcium remains in solution. The crucible fragments, plutonium hydroxide, and all solid materials are then filtered and washed with dilute ammonium hydroxide to remove chloride ion.

The slag and crucible fragments from a twenty gram plutonium reduction were leached with one liter of water. After standing for one hour with intermittent stirring, the solution was filtered. The filtering and washing were easily accomplished. However, the solution obtained from dissolving the plutonium hydroxide with nitric acid contained 1.3 g/l chloride or 0.117 grams of chloride.

A green film which had not dissolved during the leaching was noticed on some of the crucible fragments. It is believed this may be an oxychloride which does partially dissolve in the concentrated nitric acid. If this is the case, the presence of chloride ion in the acid solution is explained.

C. Reduction of Cerium Trichloride Without Booster

Efforts to find a way of eliminating the need for a booster in reductions have been continued. It will be recalled that the use of either sulfur or iodine as boosters cause difficulty in 234-5 operations and that previous studies (1) have demonstrated that good yields of cerium are obtained from cerous chloride without a booster when the exterior temperature of the bomb is 1100 to 1150 C. Operation on a plant scale at these temperatures would be difficult. Since the temperature of the charge, however, probably does not exceed 950 C, an internally-heated bomb designed to minimize heat losses might prove to be satisfactory.

Such an arrangement has been built and tested. The heating element is resistance wire wound on a stainless steel sleeve which fits over the charge crucible. Three concentric heat shields of shiny platinum are placed around the heating element. A thermocouple well located just above the charge indicates the time of firing and the approximate temperature of the slag. The whole assembly is enclosed in a metal bomb which can be evacuated during the firing.

Four charges of cerium trichloride were fired using 33 percent excess calcium. No button yield was obtained from the first reduction. The second and third reductions gave rough buttons of fair yield. These poor buttons were attributed to insufficient

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(1) Reas, W.H., "Technical Activities Report - Plant Metallurgy, Applied Research Sub-Section, May 1953", p.11, Document HW-28139 (Secret).

[redacted]

heating of the lower portion of the charge crucible where the button is formed. When this was remedied, the fourth reduction produced a smooth button of high yield. The data are presented in Table IV.

TABLE IV

YIELDS OF REDUCTIONS WITHOUT BOOSTER

Scale: 20 g Ce

Run	Thermocouple Temperature	% Yield
1	600 C	0.00
2	800 C	85.8
3	900 C	87.5
4	900 C	93.8

W. H. Reas
Product Metallurgy
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WH Reas:ac

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