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HW-77040 RD

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March 22, 1963

GENERAL ELECTRIC

HANFORD ATOMIC PRODUCTS OPERATION - RICHLAND, WASHINGTON

TITLE

THE PLUTONIUM NITRIDE PROBLEM

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AUTHOR

R. H. Bond

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THE PLUTONIUM NITRIDE PROBLEM

By

R. H. Bond

Plutonium Chemistry Laboratory
Research and Engineering Operation
Chemical Processing Department

March 22, 1963

HANFORD ATOMIC PRODUCTS OPERATION
Richland, Washington

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THE PLUTONIUM NITRIDE PROBLEM

This is a summary and documentation of the "Nitride Problem" and related subjects concerning fabrication metal. It contains the background information leading to details published in the Weapons Process Engineering Progress Reports. This summary may provide a better understanding of the problem, since little of the background was suitable for publication in the WPE progress reports.

BACKGROUND

The WPE progress report for September, 1962 (HW-75070, pps. 8, 9, 10, and 17) documents reasons for suspecting nitrogen as a possible cause of weapons fabrication difficulties and loss of production caused by radiographic rejects.

Prior to that report a British source indicated that difficulties had been encountered when molten plutonium was exposed to a nitrogen atmosphere (personal discussions during the information exchange meeting with AWRE personnel, September 15 - 17, 1962). Also prior to that report, one HLO-PMO metallurgist had observed surface nitriding of plutonium castings exposed to a nitrogen atmosphere while hot (personal communication). All other sources of information available to me at that time--at HAPO and LASL--stated firmly that nitrogen in plutonium was not and could not be a problem. There was very little physical property data on plutonium nitride and no phase diagram data for nitrogen in plutonium because no one had ever had (or recognized) a plutonium nitride problem.

Subsequent to the September report, an investigation was begun to determine whether nitrogen in plutonium could cause fabrication difficulties.

ANALYTICAL EVALUATION

A few attempts have been made at HAPO in recent years to determine nitrogen in plutonium metal by fusion and gas measurement methods. The results were of little value because of sampling and handling problems.

Wet chemical methods for nitrogen were set up in the WMO analytical operation during October and November, 1962, based on techniques in use at LASL and being evaluated by HL in 300 Area.

INGOT METAL FINDINGS

The first reliable analytical values for nitrogen by wet chemical methods were reported in November (WPE progress report, HW-75667, pp. 9). Zone-cooled metal from FPT 62-7 (extensively-recycled ingot material) was found to contain from 2 ppm to about 75 ppm nitrogen. Comparison of these results

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with photomicrographs of the same metal (HW-75070, pp. 17) showed that nitrogen in excess of 15 to 20 ppm could possibly account for radiographic defects, such as are found in fabricated parts.

Nitrogen analyses of shape-casting boomerang samples and button metal samples were begun in December.

BOOMERANG SAMPLE FINDINGS

The first analyses of boomerang samples showed 100 to 200 ppm nitrogen. These results, compared with earlier ingot metal analyses, indicated possible sampling problems or process problems in the foundry (see attached letter to A. E. Smith dated January 4, 1963).

Comparative sampling of boomerang samples (as-cast vs. cleaned surface) was suggested to evaluate the possibility of surface nitride on the boomerang samples. This might result from cooling the casting--and boomerang--from 300 C in a nitrogen atmosphere. Comparative analysis has not been achieved to date because of communications difficulties with the analytical laboratory, analytical equipment breakdown, and the urgency of other nitrogen analysis. Recent partial comparisons are discussed below. Comparative sampling is still desirable, but will be more meaningful at a later date.

Evaluation of nitride pickup from the silicon nitride crucibles by statistical methods was suggested. Statistical comparison of boomerang samples for this purpose proved inconclusive, possibly because of surface nitriding mentioned above.

Evaluation of silicon nitride crucible effects in the Plutonium Chemistry Laboratory was also suggested. This has not been done to date because of greater urgency of other problems, but still appears worthwhile. (This evaluation at a later date will also include possible silicon pickup.)

BUTTON METAL FINDINGS

The first nitrogen analyses for button samples showed 200 to 700 ppm nitrogen. These results indicated need for evaluation of the reduction process or procedure and evaluation of sampling techniques (see attached letter to R. J. Sloat dated January 8, 1963).

A button was obtained for comparison of drill-sampling in a dry air atmosphere and saw-sampling in an inert atmosphere. Comparisons were made on the button, as received, and on the metal from the button after pouring and zone-cooling. These comparisons (HW-76372, pps. 6 and 7 and HW-76842, pps. 6 and 7) showed that drill sampling as done on the Button Line is probably as satisfactory a technique as saw-sampling suggested by LASL. Results also showed that the nitrogen content of the button was excessive and that the nitrogen in the button was severely segregated.

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Subsequent analysis of 16 buttons showed nitrogen content ranging from 160 to 2725 ppm with an average of 800 ppm. (Results tabulated in letter to R. J. Sloat dated March 5, 1963.) The extreme range probably indicates low values for samples obtained from the bottom (pole) of the button and high values for samples obtained from the center of the top (plane) of the button, where the nitrogen would be most concentrated by segregation.

IMPROVING BOMB REDUCTION

Extensive calculations indicated that pressure purging of the reduction bomb, as presently done, could probably account for the nitrogen content found in the buttons (see attached letter to R. J. Sloat dated January 30, 1963). Vacuum purging of the bomb was recommended. An interim process test was proposed to verify the improvements predicted for vacuum purging by means of more thorough pressure purging.

One button was specially prepared by super-purging (11 purges vs. normal 3 purges). This button showed radically lower nitrogen content than previously reported buttons (160 ppm top-center, 40 ppm elsewhere across top, 75 to 80 ppm along vertical central axis)(HW-76842, pps. 7 and 8). This super-purge button was subsequently ingotted by a two-step, chill-cast procedure discussed below. A sample from the top-center of this ingot contained 5 ppm nitrogen (bottom sample analysis not yet available).

An extensive process test is now under way to further evaluate super-purging (Process Test 63-2-T, HW-76812).

A recent report from the Savannah River Plant revealed that unintentional purging of the reduction bomb with nitrogen resulted in poor reduction yield (49.4 to 83.7 percent) and low button density (13.4 to 18.7)(see attached letter to R. J. Sloat dated March 7, 1963). This further verifies the contention that residual nitrogen in the bomb is a source of production difficulties.

REDUCTION PROCESS IMPROVEMENTS

The super-purging process test presently under way should show a decrease of nitrogen content of the button metal proportional to the purging effectiveness. These special buttons will be processed through fabrication to evaluate radiographic improvement. If the hoped-for radiographic improvement is realized, vacuum purging of the bomb should be undertaken as soon as possible.

With vacuum purging of the bomb, the average nitrogen content of the buttons should decrease to 10 ppm or less. The actual nitrogen level attained will depend on the effectiveness of the gasket seal, the effectiveness of purging, and will also depend on the nitrogen content of the charge.

Evaluation of the nitrogen content of the calcium metal and fluoride

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powder have been suggested (see letter to R. J. Sloat, January 30, 1963).

With vacuum purging of the bomb, other benefits should be obtained. Decrease of nitrogen in the bomb atmosphere should result in lower viscosity of the metal, hence better separation from slag, higher button density and possibly slightly higher yield. Elimination of oxygen from the bomb atmosphere should eliminate the incidence of "burned lids", decrease the iron content of the buttons, and possibly contribute to improved density and yield (see letter to R. J. Sloat dated March 5, 1963).

IRON-NITROGEN CORRELATION

Few reliable nitrogen analyses are available for fabrication metal. Some representative values are as follows:

1. A single specimen of radiographically porous metal from a machined part showed 155 ppm nitrogen. The same metal contained 490 ppm carbon and 3900 ppm iron (see letter to R.J. Sloat dated March 5, 1963, for nitrogen-iron correlation).
2. Low density ingots after zone-cooling show nitrogen gradients from a minimum of 2 ppm at the bottom to a maximum of 225 ppm at the top (28 analyses of five batches of metal processed in various ways; no meaningful correlation as yet).
3. An ingot from a "normal purge" button after poorly-controlled zone cooling (RX-22-12-307) showed 34 to 90 ppm nitrogen (five analyses; see HW-70842, pps. 6 and 7). This ingot was the major part of a charge resulting in a radiographically acceptable part.
4. Boomerang samples cut and filed to remove surface metal showed 15 to 35 ppm nitrogen (three analyses; no corresponding analyses for as-cast boomerang samples or for source metal). This compares with a general level of 180 to 350 ppm nitrogen reported for as-cast boomerang samples (26 analyses to 1/28/63--one reported as 45 ppm went into part that was rejected for crack-like defect).
5. The ingot from the first super-purged button after two-stage chill casting (RX-23-1-29) showed 5 ppm nitrogen (one analysis). (The part from this metal was rejected in Final Inspection because of a visible crack.)

The above values represent almost the entire information available for nitrogen in production metal.

At present, button nitrogen analyses for "normal" buttons appear to be high by about a factor of 10 due to sampling in segregation zones. This factor is a pure guess based on an assumed average nitrogen content of 3 ppm for button ingots. The error may be larger. There is no way of sampling a button for "average" nitrogen content. Much of the nitrogen in

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the button may not pass through to the button ingot. Sampling of a few button ingots for nitrogen content could shed some light on these problems.

At present, nitrogen analyses for as-cast boomerang samples appear to be high by about a factor of 10 due to surface nitride. Comparison of as-cast and cleaned samples from boomerang samples could shed some light on this problem.

Based on the original information suggesting the existence of a nitrogen problem (HW-75070, pps. 8, 9, 10, and 17), casting skull has been found to be high in nitrogen. It is reasonable to assume that the nitrogen content of buttons would eventually be lost in the skull during extensive recycling of the metal. Failure to deplete nitrogen from the recycle metal leads to the equally reasonable assumption that nitrogen is entering the metal during one or more of the recycle steps.

Possible sources of nitrogen pickup by the recycle metal in order of probably decreasing importance are the following:

1. Moving of castings, while hot, from the casting furnace into a nitrogen atmosphere. Nitriding is probably a slow process, but exposure time is appreciable.
2. Pickup from silicon nitride crucibles.
3. Pickup during machining in a nitrogen atmosphere. Exposure time at high temperature is quite short.
4. Pickup during briquet melting because of bell jar leakage (reasonably long exposure time), or pickup from residual lard oil (protein content).

The first item can be evaluated readily by comparative sampling suggested above. The remedy, if found necessary, would be conversion from nitrogen to argon atmosphere at the cooling station.

The second item can probably be evaluated best in the PCL by before-and-after comparison of a batch of metal subjected to long exposure to a new silicon nitride crucible at high temperature (see letter to A. E. Smith dated January 4, 1963). This will be done as time and opportunity permit.

The remaining items probably cannot be evaluated effectively until the general nitride content of the recycle metal is lowered.

FABRICATION IMPROVEMENTS

Nitrogen in button metal is probably a significant source of fabrication difficulties. Conversion of the reduction bomb to vacuum purging should

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provide a major improvement in radiographic acceptance of parts. This conversion may also increase yield in the button ingotting step (WM-75042, pgs. 7 and 8). Elimination of the nitrogen atmosphere cooling of castings should provide further improvement.

IMPROVING PROPOSAL

It is reasonable that certain impurities may be present in the metal from the charge or equipment as trapped tramp impurities, but not dissolved or disbursed in the metal. Melting of the button at a low temperature (700 - 750 C) and pouring after minimum hold time may separate the metal from such tramp impurities. Conversely, melting of the button at a high temperature (850 - 900 C) and holding for a long time to remove volatile metals may cause dissolution of the tramp impurities in the metal.

The possibilities for metal quality improvement by this two-step process should be investigated. The single super-purge button (X-23-1-29) was processed in this manner.

This two-step process may not require an additional casting step. The initial, low-temperature pour could take the place of the present button ingotting pour. The high-temperature pour could possibly be a part of the first shape-casting operation.

ANOTHER PROBLEM

The presence of relatively large amounts of silicon in recycle metal (as much as 10 to 50 ppm) relative to button metal (seldom more than 1 to 2 ppm) indicates need for improvement of analysis or investigation of the source of silicon.

FURTHER IMPROVEMENTS

As the most obvious sources of nitrogen indicated above are eliminated, evaluation and elimination of the lesser sources, if necessary, should be more easily accomplished.

R. H. Bond

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28-22-12-196 BOOKING
28-22-12-197 BOOKING
28-22-11-313 piece of porous wall seen
195
196
197

Metropolitan Police
Metropolitan Police

The following analytical results for nitrites in FAD metal have
been obtained to date:

Mr. A. B. Smith, Manager
Metropolitan Police

January 4, 1963

It appears likely that the booming samples are showing high nitride for this reason.

To eliminate this possibility, a limited number of booming samples should be sampled for nitride in duplicate. The first sample should be analyzed as cut or broken from the end of the boom. The second sample should be cleaned by filing on

2. Reaction with Crucible

Silicon nitride appears unreactive with molten plutonium, at least at low temperature (<1000 C). Thermodynamic properties also indicate that silicon nitride should be highly stable. The crucibles used by PAB are coated with CaF_2 which should further limit reaction between metal and crucible. In spite of these facts, it is possible that the metal may pick up some

silicon nitride from the general nitride
or from the old,
storage pit-up,
of the

and will be used from a new silicon nitride crucible
which will not, and

77040

January 8, 1963

Mr. E. J. Sloat, Manager
CINCOALUM STEELS ENGINEERING
Research and Development Division
CINCOALUM STEELS ENGINEERING

ANALYSIS OF THE EFFECTS OF BURNING MATERIAL

Some of the samples of the material have been analyzed for nitrogen. These analyses were made using a button drill sample of one gram or more (as much sample as was available).

Long standing investigations in this facility on low-density metal materials are showing nitrogen values in the range of 10 to 20 ppm, with only a few values in excess of 70 ppm. The samples for these materials are saved from the inside of the metal and from a clean, bright surface. Samples are two to three grams in weight. Sampling is done in an inert atmosphere (dry argon containing 1 to 5% nitrogen).

The samples are sent to Bill Knicker for analysis. The results are reported to them.

A button drill sample of the material is acceptable spectroscopic analysis should be used. It can be broken with the button breaker; however, a portion can be obtained allowing sampling near the vertical axis of the button.

These are sent to Bill Knicker for analysis. This center axis sample should be used. The sample should be sampled, weighed, and analyzed. The results are reported to them. The samples are sent to the analytical laboratory for nitrogen analysis.

The piece of button from which these drill samples were taken will then be sent to the FCL for comparative sampling. Samples will be saved and analyzed in the same manner as the button samples. These will be two to three-gram massive samples taken as nearly as possible from the same location as the drill samples.

77040

January 8, 1963

ANALYSIS AND TESTING METHODS OF BUTTON MATERIAL (Continued)

with a comparison of results for drilled samples by button line techniques with massive samples by PCI techniques. It will also provide some information on nitride distribution as a function of sample location. It should also give some

The button metal can be transferred to the PCI under allotment 15-12104-01-2. It is probably desirable to keep all of the metal of this button in one place. The metal will be returned to PAB after completion of testing.

R. H. Bond
Metallurgy Chemistry Laboratory
Engineering Division

77040

January 30, 1963

R. J. Sliet, Manager
Platinum Process Engineering
2704-S Building, 200-West Area

Summary And Recommendation

Excessive nitrogen content in button material now appears confirmed. This is probably a major factor in rejection of castings and finished components.

Substitution of vacuum purging of the reduction bath for pressure purging now used should significantly reduce the nitrogen content of button material.

A program work is proposed to partially define the merits of vacuum purging and to tentatively estimate nitrogen in the fluoride powder.

Vacuum purging of the reduction bath may provide benefits in addition to decreased nitrogen content. The thickness of "burnt lids" and "high

edges" of castings may be reduced. The composition of the castings may be improved.

Button Analysis

One button (X-22-12-307) was broken to permit sampling at three positions along the vertical central axis by two sampling methods. These results are shown in Table I. The PCL samples were taken from the button and filed to clean massive chunks (~2 g each) in the inert atmosphere Platinum

the same location in the button (same height, within 1/4" lateral center-to-center distance). The massive metal samples conform to recommended (LAL) sampling techniques.

Sample Location	Bottom Line	PHL
Drilled Sample	Drilled Sample	Drilled Sample

Average	140	90
---------	-----	----

This bottom metal was poured in the PHL facility and processed through
 some cooling. (The processing was far from ideal because of thermo-
 couple failure.) Nitrogen analysis of several samples from this resulted
 in the following values:

Drilling
 1st lot
 2nd lot
 3rd lot
 4th lot
 5th lot
 6th lot
 7th lot
 8th lot
 9th lot
 10th lot
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 13th lot
 14th lot
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 99th lot
 100th lot

100-443887-100

Abstract

Calculations for a "normal" charge indicate that 7 to 8 percent of the original nitrogen remains in the bomb, most of it within the charge. This is equivalent to 0.2 g nitrogen and can account for about 100 ppm residual nitrogen in the button. Large charge, lower argon pressure, or short-cutting of the purge cycle would permit more residual nitrogen than this.

Production of 100,000 copies of 1955-56 Family Information to about 200 libraries

...to reconstruction
...change involved
...This would
...not, I am sure, be
...the only way to
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...the only way to

Recent mapping of the East coast collectively suggests the major course of

elimination of residual oxygen (or nitrogen) would be reduced to nitrides in the bath. These gases are particularly suspect in this regard (A.E.).

Calcium metal should be removed from the bath. The calcium would probably react with the nitrogen for each 100 ppm nitride in the calcium metal.

Elimination of Residual Oxygen

Elimination of residual oxygen from the bath should permit better scaling of the finely divided metal. Elimination of nitride should produce a less viscous metal. These factors combined should provide better maintenance of the bath and segregation of slag or impurities, thus providing a purer, better bath.

The elimination of residual oxygen will be due to residual oxygen

the elimination of residual oxygen will be due to residual oxygen

the elimination of residual oxygen will be due to residual oxygen

the elimination of residual oxygen will be due to residual oxygen

the elimination of residual oxygen will be due to residual oxygen

the elimination of residual oxygen will be due to residual oxygen

The buttons produced in these two special tests will be examined for nitrate (also yield and carbon content in the off-standard fluoride batch) by special sampling and processing as was done with button X-22-12-307.

RE Butte

cc: Mr Hopkins, Jr.

W. H. Hatcher

File

A. J. Clark, Manager
Photonix Process Engineering

High Temperature, High Pressure, and High Velocity Processes

EXPLANATION OF RESULTS FROM SPECIAL PURGE BUTTONS

Special purging of the reaction zone for alternative button runs may produce worthwhile results in addition to less nitrogen and possibly better particle size. The following explanation should be made to statistically compare special-purge buttons with normal-purge buttons.

1. Button yield may be slightly higher for special-purge buttons because of better separation of metal from slag, hence capture yield.
2. Special purge buttons should have higher density because of better

...more through purging
...increase the iron content
of the buttons.

...nitrogen content for all

...concentrate toward the
top and bottom ends of the button. The special-purge button (X-23-2-29)
showed much lower and more uniform nitrogen content than most previous

In the case of a "surface-ice" incident, the button should have high
...concentration near the top and available from concentration near
the bottom. However, "icing" should occur only after prolonged heating
in the lad, before the ice is removed.

...to other

$$R_1 = a + \frac{1}{2}x_2 - u_2$$

$$P^1 = 1 + 0.1x + 0.3x^2 - 0.01x^3 - 0.001x^4$$

$$P^2 = 1.25 + 0.1x + 0.3x^2 - 0.01x^3 - 0.001x^4$$

$$P(x,y) = 0 - 0.01x - 0.001y - 0.0001xy$$

$$P(x,y) = 0.01x^2 + 0.001y^2 - 0.0001xy + \dots$$

Calculus - Taylor's Series

$$f(x,y) = 1 + 0.1x + 0.3x^2 - 0.01x^3 - 0.001x^4$$

$$f(x,y) = 1.25 + 0.1x + 0.3x^2 - 0.01x^3 - 0.001x^4$$

Handwritten notes

Handwritten notes

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					6340	1
					2601	2
1120				108	14641	3
320	144	102400	4,050	109	20736	4
725	48	305625	34,750	111	2304	5
740	25	547600	48,100	121	4225	6
					2100	7
720	144	102400	4,050	123	32400	8
635	70	403225	7,250	125	8100	9
415	150	172225	20,750	125	2500	10
160	40	25600	1,360	125	9216	11
200	60	40000	1,900	125	1000	12
120	144	14400	2,160	125	1000	13
1500	225	2250000	25,000	051	50625	14
739	12	545961	9,760	125	12544	15
					5476	16
12890	1646	16,381,900	1,615,950	22-12-307	212618	17
145	55	21025	7,405	22-12-307	3025	18
150	60	22500	7,500	22-12-307	2500	19
150	60	22500	7,500	23-1-24	5625	20

1.266 541 - 8,877,781.5 + 16,440,175 = 1,643,200

7,562,344

1,049,899

(from - 2) = 61.81 + 0.04989 (ppm N)

March 7, 1953

R. J. Slout, Manager
Fibronium Process Engineering
Research and Engineering Operation

Document LPHF-63-1-18 (SRP) contains an account of accidental cross connection of argon and nitrogen sources to reduction beams.

Eight of fourteen reductions for the stated period gave poor yields (49.4 to 83.7 percent) and produced buttons of low density (12.4 to 18.7). Two analyses of the purge gas from the supply main during this period showed 19 percent and 99.9 percent nitrogen.

Appreciable quantities also were identified as the major constituents of the gas which was present in the beam furnace during the stated period. The gas was identified as nitrogen and the source of the gas was traced to the nitrogen supply main.

This gas effect was caused by appreciable air being present in the beam furnace at the time of the stated period. The gas was present in the beam furnace as a result of insufficient purging or failure to purge the beam. No mention is made of "burning" of the beam lid, only spattering to the lid. If air was present, the lid would burn in the manner observed in the stated period.

Fibronium Chemistry Laboratory
Research and Engineering Operation

SR 20012

cc: Mr. Hopton, Jr.

702

**DATE
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5/27/94

END