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**PREPARATION OF NEPTUNIUM-ALUMINUM
ALLOY TARGET ELEMENTS**

C. H. BLOOMSTER

OCTOBER, 1966

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BNWL-311

C-44a Nuclear Technology-
Materials

PREPARATION OF NEPTUNIUM-ALUMINUM
ALLOY TARGET ELEMENTS

By

C. H. Bloomster
Plutonium Fuels Engineering Section
Materials Department

October 1966

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PREPARATION OF NEPTUNIUM-ALUMINUM ALLOY TARGET ELEMENTS

C. H. Bloomster

INTRODUCTION

This report describes the preparation of neptunium-aluminum alloy target elements, starting with the oxide and ending with the finished element. Battelle-Northwest was requested to design and develop a fabrication process for a neptunium target element for N-Reactor. Neptunium is irradiated to form Pu^{238} , an important isotopic power source. General Electric N-Reactor Department (GE-NRD) is conducting a Pu^{238} demonstration program to evaluate N-Reactor capability for the production of Pu^{238} . Design criteria and schedule requirements were established by GE-NRD. The neptunium was to be supplied as neptunium oxide, and only a slight excess of neptunium was to be available. Neptunium is only slightly less toxic than plutonium and must be handled in glove boxes. The schedule allotted 8 wk for fabrication between receipt of the oxide and charging of the target elements in N-Reactor.

The objectives of the work were to design a target element and to develop a fabrication process within the design criteria, fabrication schedule, and material and budgetary limitations. Of the many alternative processes considered, an Al-Np alloy casting process was selected for the fabrication. The advantages of the casting process include ease of scrap recycle, uniformity of neptunium concentration,

ease of assembly, ability to fabricate large pieces, and structural stability.

Since previous work on neptunium targets, both at Hanford and Savannah River Laboratory, had been concentrated on the NpO_2 -Al powder compaction processes, little information was available on Al-Np alloys. The neptunium-aluminum phase diagram has not been developed, but Al-Np compounds similar to Al-U and Al-Pu compounds were reported.⁽¹⁾ In 1958, Lyon⁽²⁾ formed small quantities of Al-Np alloys by reducing the oxide with aluminum under a cryolite flux. In addition, the author^(3,4,5) had extensive experience in the preparation, casting, and fabrication of aluminum-plutonium alloys which would be applicable to Al-Np alloys.

SUMMARY

A casting process was developed to fabricate neptunium target elements for irradiation in N-Reactor. Forty-nine targets were fabricated for a Pu^{238} demonstration program. Aluminum-neptunium alloys were prepared by reducing neptunium oxide with aluminum under a cryolite flux. The Al-Np alloy was cast into tubes by use of graphite mandrels and molds. The tubes were machined to specified lengths and diameters, and inserted into aluminum can assemblies which, in turn, were inserted into zirconium cans. Process yields were excellent:

100% in the reduction step, 95% in the casting step, and 98% in the canning step. Scrap from rejected castings, machining chips, and the one rejected element was recovered by remelting. Overall, 5.0 Kg of neptunium entered the process; 4.3 Kg neptunium went into the targets; and 0.7 Kg of neptunium scrap was left for recovery at the end of the fabrication.

DESIGN CRITERIA

The following design criteria were established by GE-NRD for the target elements.

- The reactor coolant and irradiation conditions required an outer cladding of zirconium.
- Chemical processing of the irradiated target required mechanical removal of the zirconium cladding, but a soluble secondary cladding of aluminum was required to prevent contamination spread during transport and storage after removal of the zirconium cladding.
- For greater neutron efficiency, a tubular shape of the neptunium target material was desired to concentrate the neptunium atoms the periphery of the elements.
- The center of the tube was to be filled with graphite to add mass and internal support to the element to prevent collapse and chattering or floating in the process tube.
- The finished element was to be a solid right cylinder, 2.4 in. diam by 21 in. long.
- The neptunium concentration was specified at 70 g/ft for the

first test column and 45 g/ft for the second and third columns.

PROCESS DESCRIPTION

The neptunium, received as the oxide (Figure 1), was reduced by aluminum (Figure 2) in the presence of cryolite to form an Al-12 wt% Np master alloy (Figure 3). The master alloy was diluted with aluminum to 9.2 wt% Np for the first test column elements and to 6.3 wt% Np for the second and third test column elements. The alloy was cast in 1/4 in. thick wall tubes (Figure 4), 11 in. long, by using graphite molds and mandrels. Prior to casting, an aluminum sleeve was shrunk-fit onto the graphite mandrel. The purpose of the inner aluminum sleeve (on the graphite mandrel) was to decrease the loss of neptunium during postirradiation recovery in which the graphite mandrel will be removed by machining. The castings were machined to specified length and diameter. The hollow graphite mandrels were left in the castings, and two castings were assembled into each aluminum can assembly. A full-length graphite rod was inserted in the hollow graphite mandrels to add structural integrity. The aluminum-clad elements were welded shut and inserted into the zirconium cladding, which was then welded shut (Figure 5). The component parts are shown in Figure 6. Supports were welded to the zirconium clad elements which were then etched, autoclaved, and inspected according to conventional processes. The dimensions and design of the target elements are

FIGURE 1. Neptunium Oxide is Received in Lead Casks, and Reduced and Alloyed with . . .

FIGURE 2. Aluminum to Form an . . .

FIGURE 3. Al-12 wt% Np Master Alloy, Which is Diluted with Aluminum and Cast into . . .

FIGURE 4. Al-Np Alloy Tubular Castings. (Pure Aluminum is Substituted for Photograph.)

FIGURE 5. Completed Neptunium Target
Element

FIGURE 6. Components of Np Target
Elements. (NOTE: The Graphite
Mandrels are Normally Inside and
Machined to Length Integrally with
the Np-Al Casting.)

shown in Figure 7. Diametral gaps were kept to a minimum to reduce the strain of the cladding which was not free-standing.

REDUCTION OF NpO_2

In the only available reference⁽⁶⁾ to the preparation of Np-Al alloys, small quantities of NpO_2 were successfully reduced by aluminum in the presence of cryolite to form an Al-Np alloy. The reaction is:

The neptunium alloys with the excess aluminum, and the Al_2O_3 product is dissolved by the cryolite flux. Without the removal of the Al_2O_3 to the

cryolite, the reduction is greatly inhibited by the Al_2O_3 which forms a reaction barrier. We have used this method successfully to prepare large quantities of Al-Pu alloys from plutonium oxide, and the method was originally used by Saller⁽⁷⁾ in the preparation of U-Al alloys.

Earlier experience with reduction of plutonium oxide by aluminum indicated that complete reductions were obtained at 1200 °C. The reaction was judged complete 30 min after disappearance of the dark olive plutonium oxide color from the white cryolite flux. The same criteria were applied to the NpO_2 reductions. Reaction

FIGURE 7. Neptunium Target Element

times usually range between 60 to 90 min. After the reduction is complete, the cryolite is skimmed from the melt, and the melt is cast into billets for sampling and eventual remelting.

Five separate reductions of NpO_2 were made (Table I). The melt charges were calculated to obtain a master alloy of 12 to 13 wt% Np near the expected eutectic composition. Material balances from the last two reductions indicated complete recovery of the neptunium. It was not possible to obtain accurate material balances for the first three reductions because of intermixing during development and initial processing, but overall material balances indicated Np recovery was nearly complete for the entire series of reductions.

CASTING

The Al-Np alloy was cast into 1/4 in. thick wall tubes in a graphite mold around graphite mandrels, which were clad with shrunk-fit aluminum sleeves. To reduce scrap generation in later machining operations, the mold was made to yield a casting only 0.04 in. oversize in diam, which required that the casting have a good surface. The

melt was made in an induction melting crucible and poured into a casting ladle preheated in a resistance furnace. The casting temperature was 720 to 730 °C, the molds were preheated to 290 °C, and casting was by hand pouring. Proper casting technique was important. The metal was initially poured rapidly, slowing to a trickle near end of pour. The mold was supported on a copper block to promote directional solidification. The mold was rotated slowly to prevent local melting of the aluminum sleeves. After casting, the billets were radiographed to detect unsoundness and segregation. The casting technique was identical for the two concentrations of neptunium, 9.2 wt% and 6.3 wt%. High purity, 99.99%, aluminum was used for alloying. Trace amounts, 46 ppm, of lithium were added to one melt of each Np concentration for later neutron flux measurements.

We sought to develop a technique that would produce castings having low porosity, good surfaces, uniform neptunium concentrations, and high yields. The available casting facility comprised two tilt-pour, induction melting furnaces housed in an air-

TABLE I. Reduction of NpO_2 with Aluminum

Melt Number	Charge (Grams)			Yield	
	NpO_2 as Np	Aluminum	Cryolite	Np-Al Alloy (g)	%Np Recovery
1	277	2,046	800	2,243	>97
2	705	5,042	1,600	5,550	>97
3	1,118	7,997	3,000	8,665	>97
4	1,497	10,669	3,000	11,797	~100
5	1,398	9,997	3,000	10,967	~100

atmosphere glove box. The capacity of the two furnaces were 12 and 20 Kg of aluminum. Since the tilt-pour mechanism was not sufficiently sensitive to permit the thick-walled tubes to be cast directly from the melting furnace, a ladle for hand pouring was used. The smaller induction furnace was used to make the reductions and dilutions from the master alloy; the larger furnace was lined with resistance heating elements to preheat the mandrels. The metal was poured from the melting furnace into the ladle, which was a No. 4 clay-graphite crucible preheated in a resistance-wound crucible furnace.

Casting defects, encountered during development, included cracking through the top part of the casting, excessive solidification shrinkage near the middle of the casting, cold shuts, porosity, and local melting of the aluminum sleeves. These were largely eliminated by adjusting the pouring technique, mold temperature, and casting temperature. The casting yield was 95%; the casting rejects, entirely because of trapped gases, were detected by radiography.

MACHINING

Cast billets were rough-cut to length and followed by a finishing cut. The outside diameter was machined to size in one pass by using live centers in the graphite mandrels. No lubricants or cutting fluids were used in machining, and the machined pieces were handled carefully with tongs to prevent surface contamination. The machined castings were placed in plastic bags, weighed, and transferred to

the assembly area, and chips, billet ends, and rejected billets were recycled through the casting operation. No rejects occurred in machining.

ASSEMBLY

Assembly was performed in a fume hood. Two castings and the inner graphite rod were carefully inserted into each aluminum can, and flux detectors, Al-Co or Al-Li wires, were inserted into three elements of each column. The weld zone was wiped free of contamination; the end cap inserted and welded in place. Aluminum welding was performed in the open laboratory by using alternating current, a tungsten electrode, and an argon purge.

The finished aluminum-clad pieces were free from contamination and were handled in the open laboratory. They were cleaned with acetone, coated with a graphite-alcohol slurry, dried, and assembled into a zirconium tube. Both zirconium end caps were TIG welded to the tubing in a helium filled welding chamber.

FINISHING

The assembled elements were helium-leak checked. Zircaloy-2 suitcase handle supports were later spot-welded to the exterior by GE-NRD; and carbon steel shoe covers were crimped on the supports to minimize fretting corrosion. The finished elements were etched, leak checked, and auto-claved in 400 °C, steam for 15 to 66 hr. In general, an adequate oxide film is developed during the shorter cycle, but a longer cycle helps to discriminate between marginal cases.

The elements were inspected and shipped to the reactor. One element was recanned after a helium leak was detected. Another element was rejected because of an improper support weld; this element was disassembled and the target alloy remelted.

COMPONENTS

All aluminum components were extruded tubing of 5052 alloy. The outer aluminum cladding was drawn to 2.33 in. OD from 2.5 in. OD x 0.065 in. wall, while the inner aluminum sleeve was stock extruded tubing, 1.75 in. OD x 0.035 in. wall. Rapid availability dictated the choice of alloy and tubing size. The aluminum end caps were machined from 0.25 in. 5052 aluminum plate.

The zirconium tubing was extruded by GE-NRD, and the zirconium end caps were machined from 0.25 in. zirconium plate. All zirconium components were Zircaloy II.

The graphite mandrels and rods were machined from high density, nuclear grade graphite.

All metal components were degreased, washed thoroughly in hot water and detergent, and rinsed and dried before use. Also, all graphite components were heated in air to 300 °C prior to use.

TEST COLUMN DATA

Element identification, weight, and analytical data for each test column are summarized in Tables II to IV.*

* For additional information see:
D.A. Constable. Production Test
PT-NR-68 Pu 238 Demonstration Program,
RL-NRD-793. March 1, 1966.
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In the listings under core number, the numeral 1, 2, 3, etc. refers to a single alloy melt, and the letter designation A, B, C, etc. refers to the casting order within a melt. Elements 8, 12, and 15 in each column contained flux detector wires and trace additions of lithium.

AUTOCLAVING TESTS

Tests to determine the effect of thermal cycling, differential thermal expansion, and pressure were performed in an autoclave on mock-up elements. Since the cladding was not free-standing, the aluminum components collapsed (plastically) onto the graphite mandrel. The zirconium cladding gradually decreased in diameter with time at

TABLE II. First Test Column - 70 g/ft Np

Element Number	Core Number	Core wt Grams	wt% Np	Grams Np	Total Np wt(g) per Element
1A	6C	640	9.20	58.9	118.6
	6B	649	9.20	59.7	
2A	5C	633	9.30	58.9	118.1
	5D	637	9.30	59.2	
3A	6Q	630	9.20	58.0	116.7
	6P	638	9.20	58.7	
4A	7A	632	8.98	56.8	113.5
	7B	631	8.98	56.7	
5A	6A	623	9.20	57.3	116.7
	6I	646	9.20	59.4	
6A	6R	645	9.20	59.3	118.5
	6D	643	9.20	59.2	
7A	6F	635	9.20	58.4	116.9
	6N	636	9.20	58.5	
8A	8A	620	8.72	54.1	107.8
	8B	616	8.72	53.7	
9A	6K	653	9.20	60.1	118.3
	6L	633	9.20	58.2	
10A	6E	583	9.20	53.6	117.8
	6M	698	9.20	64.2	
11A	6O	628	9.20	57.8	116.8
	6H	641	9.20	59.0	
12A	8C	616	8.72	53.7	107.0
	8E	611	8.72	53.3	
13A	5E	632	9.30	58.8	118.6
	5B	643	9.30	59.8	
14A	5F	627	9.30	58.3	116.3
	5A	624	9.30	58.0	
15A	8F	609	8.72	53.1	107.2
	8G	620	8.72	54.1	

TABLE III. Second Test Column - 45 g/ft Np

Element Number	Core Number	Core wt Grams	wt% Np	Grams Np	Total Np wt(g) per Element
1B	1A	593.6	6.55	38.88	79.46
	1D	619.6	6.55	40.58	
2B	1B	606.6	6.55	39.73	79.99
	1C	614.6	6.55	40.26	
3B	1E	614.6	6.55	40.26	80.45
	1F	613.6	6.55	40.19	
4B	1I	624.6	6.55	40.91	81.76
	1J	623.6	6.55	40.85	
5B	1K	617.6	6.55	40.45	80.97
	1L	618.6	6.55	40.52	
6B	2A	625.6	6.55	40.98	81.17
	2B	613.6	6.55	40.19	
7B	2C	623.6	6.55	40.85	81.30
	2D	617.6	6.55	40.45	
8B	5D	621.6	6.12	38.04	76.20
	5O	623.6	6.12	38.16	
9B	2M	620.6	6.55	40.65	81.43
	2N	622.6	6.55	40.78	
10B	3F	604.6	6.28	37.97	77.07
	3G	622.6	6.28	39.10	
11B	3H	612.6	6.28	38.47	77.32
	3I	618.6	6.28	38.85	
12B	5E	614.6	6.12	37.61	75.22
	5J	614.6	6.12	37.61	
13B	2O	617.6	6.55	40.45	80.44
	2P	610.6	6.55	39.99	
14B	2Q	624.6	6.55	40.91	81.43
	1H	618.6	6.55	40.52	
15B	5H	618.6	6.12	37.86	75.53
	5L	615.6	6.12	37.67	
16B	3A	615.6	6.28	38.66	76.75
	3B	606.6	6.28	38.09	

TABLE IV. Third Test Column - 45 g/ft Np

Element Number	Core Number	Core wt Grams	wt% Np	Grams Np	Total Np wt(g) per Element
1C	2H	623.6	6.55	40.85	81.63
	2I	622.6	6.55	40.78	
2C	2L	627.6	6.55	41.11	82.55
	2K	632.6	6.55	41.44	
3C	2E	628.6	6.55	41.17	82.28
	2F	627.6	6.55	41.11	
4C	2G	630.6	6.55	41.30	82.34
	2J	626.6	6.55	41.04	
5C	3C	622.6	6.28	39.10	77.95
	3D	618.6	6.28	38.85	
6C	3J	560.6	6.28	35.21	77.26
	3K	669.6	6.28	42.05	
7C	4B	611.6	6.37	38.96	78.05
	4C	613.6	6.37	39.09	
8C	5B	622.6	6.12	38.10	75.29
	5N	607.6	6.12	37.19	
9C	3E	620.6	6.28	38.97	77.57
	3L	614.6	6.28	38.60	
10C	4I	607.6	6.37	38.71	78.37
	4J	622.6	6.37	39.66	
11C	4E	614.6	6.37	39.15	78.43
	4G	616.6	6.37	39.28	
12C	5C	616.6	6.12	37.74	76.27
	5P	629.6	6.12	38.53	
13C	4A	613.6	6.37	39.09	77.92
	4F	609.6	6.37	38.83	
14C	4H	615.6	6.37	39.21	78.61
	4L	618.6	6.37	39.40	
15C	5F	619.6	6.12	37.92	75.23
	5K	609.6	6.12	37.31	
16C	4M	619.6	6.37	39.47	79.13
	4N	622.6	6.37	39.66	
17C	1G	613.6	6.37	40.19	79.79
	4K	621.6	6.37	39.60	
18C	5A	622.6	6.12	38.10	75.53
	5M	611.6	6.12	37.43	

temperature and pressure. The zirconium length did not change, nor did the dimension of the graphite components. The aluminum components decreased in length during the first few cycles, but the decrease was not progressive and apparently reversed itself. The autoclave tests (Table V) were terminated after eleven cycles because the dimensional changes were not sufficient to pose a problem during the short irradiation with few cycles originally scheduled for the elements.

RADIATION EXPOSURE

Radiation levels of the neptunium alloys were higher than for similar plutonium alloys. Larger scale fabri-

cation may require installation of some shielding (particularly in the machining operation) and modification of some handling techniques to reduce personnel exposure. Adoption of remote handling techniques should not become necessary. Neptunium-237, the only isotope produced in abundance, decays by alpha emission to protactinium-233. Protactinium-233 emits a 0.31 MeV photon which produces an appreciable radiation field. Radiation levels from newly separated neptunium increase with time as the Pa^{233} content increases until equilibrium is reached in ~160 days. Radiation measurements, taken in the field, from the finished target elements are shown in Table VI. At the

TABLE V. Autoclaving Tests

No. of Thermal Cycles	Dimensional Decrease (%)			Max. Temperature Max. Pressure Accumulative Time of Max. Temp and Pressure
	Zirconium Components Diam	Aluminum Components Diam	Length	
1	0-0.2%	*	*	325 °C, 1350 psi 4 hr
4	0.1-0.4%	*	*	325 °C, 1350 psi 28 hr
8	0.1-0.4%	0.1-0.4%	0.8%	325 °C, 1350 psi 60 hr
11	0.1-0.4%	0.8-1.0%	0.4-0.5%	325 °C, 1350 psi 84 hr
1	0-0.2%	*	*	350 °C, 1500 psi 2 hr
4	0.1-0.4%	0.2-0.4%	0.8%	350 °C, 1500 psi 8 hr
1	0.05-0.4%	*	*	400 °C, 1000 psi 48 hr
1	0.1-0.6%	*	*	400 °C, 1000 psi 66 hr

* No measurement

TABLE VI. Radiation Measurements

Distance From Source	Radiation Level		
	1 Element From First Column	11 Elements From First Column	34 Elements From Second and Third Columns
Contact	1600 mrad/hr	2800 mrad/hr	*
1 ft	75 mrad/hr	400 mrad/hr	110 mrad/hr
2 ft	22 mrad/hr	90 mrad/hr	*
3 ft	10 mrad/hr	*	30 mrad/hr
9	*	*	6 mrad/hr
10	*	7 mrad/hr	*

* No measurement

time of the radiation measurements, the Pa^{233} buildup had reached ~75% of equilibrium.

ACKNOWLEDGEMENT

The author gratefully acknowledges the cooperation and assistance of all those who worked on this project. In particular, the advice and liaison of R. Cooperstein and D. W. Constable,

GE-NRD, was essential. Casting, machining, and assembly were performed by Joe Jensen, Floyd Brecto, Chet Smith, and Bob Borgmier. Ray Reid was responsible for welding. Radiation Monitoring information was provided by Dan McConnon; and Analytical Services by Wayne Delvin and John Michaelson.

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