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MASTER

**A DESCRIPTION OF FUEL AND
OTHER CORE MATERIALS PROCURED
FOR SEFOR CRITICAL EXPERIMENTS
IN ZPR-III**

M.L. JOHNSON

U.S. ATOMIC ENERGY COMMISSION
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ADVANCED PRODUCTS OPERATION
GENERAL ELECTRIC
SAN JOSE, CALIFORNIA

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ABSTRACT

Critical Experiments for the Southwest Experimental Fast Oxide Reactor (SEFOR) are being conducted in the Zero Power Reactor-III (ZPR-III) fast-flux critical assembly at the National Reactor Testing Station, Idaho.

In the ZPR-III, core materials in the form of small blocks are loaded into the compartmented core structure. Stainless steel cans containing metal alloy (19.5 wt% Pu-78.0 wt% U-2.5 wt% Mo) and cans containing sodium carbonate were developed and procured to simulate the oxide fuel and sodium in SEFOR. Sintered beryllia blocks coated with Kel-F were also procured. Nickel blocks were provided for reflector material, and Kel-F-coated Boral blocks simulated the boron carbide radial shield annulus.

Quantities, weights, and design features of these materials are described.

SECTION I

INTRODUCTION

The Southwest Experimental Fast Oxide Reactor (SEFOR) is a 20 MWt fast-spectrum reactor fueled with $\text{PuO}_2\text{-UO}_2$ and cooled with sodium. SEFOR will have characteristics similar to the large, soft-spectrum, fast breeder reactors. Economic studies of these large power reactors indicate the potential for producing low cost power. SEFOR will be used to obtain physics and engineering data at fuel compositions, crystalline states, and temperatures characteristic of the power reactor. SEFOR is particularly designed for the systematic determination of the Doppler coefficient of reactivity at temperatures up to the vicinity of fuel melting.

The SEFOR Project consists of two major parts: (1) The design and construction of the reactor and (2) A related research and development program. Funds for the design and construction of the facility are being provided by the Southwest Atomic Energy Associates (a group of seventeen investor-owned utility companies located in the South and Southwest part of the United States), together with the Karlsruhe Laboratory of West Germany, Euratom, and the General Electric Company.

The United States Atomic Energy Commission is supporting the research and development program. The R & D program consists of two phases:

- Phase I - Pre-Operational Research and Development
- Phase II - Post-Construction Research and Development (the Program)

This report describes work performed in Phase I of the Research and Development Program under Task 2 - Experimental Program in the ZPR-III.

In support of the SEFOR program, critical experiments are being conducted in the Zero Power Reactor-III (ZPR-III) located at the National Reactor Testing Station (NRTS) in Idaho. These experiments include the measurement of the required plutonium concentration in the SEFOR fuel, the Doppler coefficient, and the control strength of the radial reflector. The measurement of the sodium loss coefficient, neutron lifetime, fission rates, and other related parameters are also being performed. Materials used in the critical experiments duplicate as closely as practicable the materials of the SEFOR core.

Some of the materials needed to mock up the SEFOR core were on hand, having been used in previous ZPR-III experiments. Additional materials not on hand were procured to complete the mock-up requirements for SEFOR. These materials included plutonium-uranium-molybdenum alloy, sodium carbonate, nickel, beryllia and Boron. The materials were designed and specified by the Advanced Products Operation (APO) of the General Electric Company in conjunction with the Idaho Division and Metallurgical Division of the Argonne National Laboratory (ANL). Purchase of the material was handled by the San Francisco Operations Office of the Atomic Energy Commission. General Electric and ANL provided technical assistance during the procurement phase.

SECTION II

DESCRIPTION OF THE SEFOR MOCK-UP IN ZPR-III

2.1 ZPR-III FACILITY DESCRIPTION

The ZPR-III core structure is built in two opposed halves mounted on a common carriage. Each half of the core structure is a matrix of stacked tubes which receive drawers loaded with the core material. One half is constrained in position by clamps fastened to the carriage. The other half is moveable along the carriage by a drive system to provide safety and convenience in loading.

Each matrix is constructed of stacked 0.040-inch-thick stainless steel square tubes, each 2.18 inches square and 33 inches long, forming a matrix 67 inches wide by 67 inches high by 33 inches long. Each tube of the matrix receives two drawers which contain the core materials. The drawers are loaded in a separate work room, brought into the assembly room, and placed in the matrix tubes. Each matrix is loaded from both front and back, with a front drawer and rear drawer placed end-to-end extending the length of the matrix.

The drawers are filled with small blocks of the several materials needed to form the reactor core. The blocks are all 2 inches high to match the height of the drawer, and of various widths and lengths. Common widths are 1/8 inch and 1/4 inch, and common heights fall between 1 inch and 7 inches. Partially loaded drawers are shown in Figure 2-1, and the loaded critical assembly machine in Figure 2-2. More detailed descriptions of the ZPR-III may be found in ANL documents.⁽¹⁾

Figure 2-1. Partially Loaded Front and Rear Drawers

Figure 2-2. ZPR-III Critical Assembly Machine

2.2 SEFOR MOCK-UP DESCRIPTION

The SEFOR core structure is formed by 109 hexagonal channels which are tightly clamped in a bundle approximating a cylinder 35 inches in diameter. Each channel contains PuO_2 - UO_2 pelletized fuel in Type-316 stainless steel tubes, sintered beryllia pellets in Type-316 stainless steel tubes, and Type-304 stainless steel structural pieces. Sodium coolant flows through the channels during reactor operation. Nickel is used for both axial and radial reflectors. A boron-carbide-filled cylinder forms the first layer of shielding outside the radial reflector.

The SEFOR core and reflector are simulated as accurately as practicable in the ZPR-III. A cross section of the mock-up is shown in Figure 2-3, and an elevation in Figure 2-4. Although the cylindrical core is upright in SEFOR, it is horizontal in the ZPR-III to simplify the assembly.

Metallic plutonium and uranium are substituted for the oxide fuel in SEFOR (See explanation in Section 3.1). Sodium carbonate wafers are used to mock up the sodium and a part of the oxygen in the SEFOR oxide fuel. The remainder of the oxygen is simulated by appropriate combinations of aluminum and carbon. Beryllia tiles are used in correct amounts for the BeO in SEFOR.

Figure 2-3. Core Mock-Up in ZPR-III (Section)

Figure 2-4. Core Mock-Up in ZPR-III (Elevation)

Type-304 stainless steel is used throughout the mock-up. The small amount of molybdenum in the Type-316 stainless steel fuel cladding used in SEFOR is provided by the molybdenum required in the Pu-U alloy for corrosion resistance (See Section 3.3, Alloy Development).

The boron carbide surrounding the radial reflector in SEFOR is simulated by the use of Boral. As noted in Figure 2-3, the boron carbide is simulated over only a part of the core periphery. The boron carbide is considered to be necessary only in regions where the reflector control rods are to be moved to obtain measurement of the reflector control strength.

The nuclear considerations of these material simulations in the mock-up relative to the SEFOR core will be discussed in a future report which will describe the critical experiments and results.

SECTION III

FUEL WAFERS

3.1 FUEL SELECTION

The SEFOR fuel is in the form of PuO_2 - UO_2 -filled stainless steel tubes. At the time fuel was selected for the ZPR-III mock-up, calculations indicated that the SEFOR core would require approximately 330 kg of plutonium (including all isotopes). Plutonium wafers containing 210 kg of plutonium and a complementary weight of uranium metal blocks were already on hand at the ZPR-III site from previous programs. An additional 180 kg of plutonium for the SEFOR mock-up were procured which increased the total available plutonium to 390 kg. The margin of 60 kg provided a contingency to cover possible future revision to the SEFOR core design and discrepancies between calculated and experimental requirements.

The additional 180 kg were fabricated in the form of a plutonium-uranium metallic alloy. The plutonium-uranium alloy, in contrast to separate wafers of plutonium and uranium, provided a negative Doppler coefficient for the mock-up because of the close heat transfer coupling of the plutonium and uranium.

The metallic alloy was chosen instead of the ceramic PuO_2 - UO_2 for several reasons. Fuel in the oxide form with a lower density would occupy too much of the critical assembly volume, leaving insufficient space for the nonfuel core materials. Furthermore, a mock-up using combinations of the new oxide fuel and the existing metallic fuel was calculated to have a very small negative, or perhaps even positive, Doppler effect. It was considered advisable to have a significantly negative Doppler coefficient for the ZPR-III mock-up.

3.2 DESCRIPTION OF FUEL WAFER

A fuel wafer design developed at Argonne National Laboratory was selected for use in SEFOR experiments.⁽²⁾ In this design, cast blocks of fuel alloy are sealed in a stainless steel jacket by fusion welding. A jacketed wafer is shown in Figure 3-1. The flanged design permits assembly without contaminating the jacket. Flat sheets are formed and trimmed in a die to produce a flanged jacket panel. The core plate is sealed between two identical jacket panels by edge welds along two opposite flanges, and weld castings at the other two edges.

The weld castings, when machined, form a shoe which will butt against the shoe of the adjacent wafer in the same column in the fuel drawer, thereby providing for expansion of a fuel column in the event of fuel temperature rise. The sealed wafer contains a helium-argon mixture in the void space, and was helium leak checked during fabrication to insure integrity. Detailed information on the development is contained in ANL 6995, Development of a Process for Manufacturing ZPR-III Pu-U-Mo Test Fuel Elements.⁽³⁾

1962-7

Figure 3-1. Fuel Wafer

Wafers are either 1/4 or 1/8 inch thick, 2 inches high and in lengths ranging from 1 to 7 inches. In Table 3-1 are shown the sizes and quantities procured and the nominal weight of fuel and jacket for each size of wafer.

TABLE 3-1

SEFOR-ZPR-III FUEL, SIZES AND QUANTITIES

<u>Nominal Size, inches</u>	<u>Quantity</u>	<u>Nominal Weight, grams</u>	
		<u>Fuel</u>	<u>Jacket</u>
1/8 × 2 × 2	216	79.88	14.64
1/4 × 2 × 1	30	76.32	8.74
1/4 × 2 × 3	612	294.89	22.11
1/4 × 2 × 4	306	404.11	28.54
1/4 × 2 × 5	306	511.42	35.42
1/4 × 2 × 6	306	622.43	42.78
1/4 × 2 × 7	306	727.62	49.00

Detailed drawings of the jacket panels, core plate, and completed wafer are shown in Figures 3-2, 3-3, and 3-4. Fuel wafers conform to General Electric Specification 22A1501 and Quality Control Plan FP-129.

UNLESS OTHERWISE SPECIFIED USE THE FOLLOWING.—				
APPLIED PRACTICES	SURFACES	TOLERANCES ON MACHINED DIMENSIONS		
		FRACTIONS	DECIMALS	ANGLES
-----	63√	± 1/64	± .005	± —

STEP 18 USE STRIP						PART NO	NAME	
G7	G6	G5	G4	G3	G2	G1	1	PANEL
						I	2	
				I			3	
			I				4	
		I					5	
	I						6	PANEL
2	2	2	2	2	2		7	STRIP
1							8	PANEL

NOTES

1. ALL DIMENSIONS NOTED APPLY AT A PART & GAGE TEMPERATURE OF 68°F.
2. GEOMETRIC TOLERANCES PER MIL-STD-88.

GR NO	C ⁺ 002	D ⁺ 002 ₀₀₀	"E"	"F"
G1	1.210	.782	.120 ^{+0.002} _{-0.000}	.025R ^{+0.000} _{-0.005}
G2	3.210	2.782	↓	↓
G3	4.210	3.782		
G4	5.210	4.782		
G5	6.210	5.782		
G6	7.210	6.782	.120 ^{+0.002} _{-0.000}	.025R ^{+0.000} _{-0.005}
G7	2.210	1.782	.060 ^{+0.000} _{-0.000}	.025R ^{+0.000} _{-0.005}

Figure 3-2. Jacket Panel

PART NO	NAME
1	CORE PLATE, CSTG
2	
3	
4	
5	
6	CORE PLATE, CSTG
7	CORE PLATE, MACH
8	
9	
10	
11	
12	CORE PLATE, MACH
13	CORE PLATE, CSTG
14	CORE PLATE, MACH

NOTES

1. FOR MATERIAL & FLATNESS REQUIREMENTS SEE SPEC 22A1501.
2. SERIAL NUMBERS WILL BE SCRIBED ON EACH PART PER SPEC. 22A1501.
3. ACCEPTABILITY OF SURFACE FINISH, SURFACE DEPRESSIONS & EDGE CHIPS SHALL BE DETERMINED BY VISUAL COMPARISON WITH STANDARDS APPROVED BY G.E. - APED.
4. BOTH ENDS MUST BE MACHINED AS SHOWN. MACHINING INTO .375 R AT EITHER END MUST NOT EXCEED .030 INCHES
5. ALL DIMENSIONS NOTED APPLY AT A PART & GAGE TEMPERATURE OF 68°F.
6. GEOMETRIC TOLERANCES PER MIL-STD-88.

RNO	C ^{+0.05} _{-0.05}	E ^{+0.01} _{-0.01}	RNO	"D"	"F"
1	.791	.210	7	.780 ⁺⁸⁸ ₋₈₈	.037 ^{+0.016} _{-.005}
2	2.791	.210	8	2.780 ⁺²⁵⁵ ₋₂₅₅	↑
3	3.791	.210	9	3.780 ⁺⁴⁰⁰ ₋₄₀₀	
4	4.791	.210	10	4.780 ⁺⁶⁰⁰ ₋₆₀₀	
5	5.791	.210	11	5.780 ⁺⁸⁰⁰ ₋₈₀₀	↓
6	6.791	.210	12	6.780 ⁺⁹⁰⁰ ₋₉₀₀	.037 ^{+0.016} _{-.005}
13	1.791	.092	14	1.780 ⁺⁸⁸ ₋₈₈	.037 ^{+0.016} _{-.005}

UNLESS OTHERWISE SPECIFIED USE THE FOLLOWING—				
APPLIED PRACTICES	SURFACES	TOLERANCES ON MACHINE DIMENSIONS		
		FRACTIONS	DECIMALS	ANGLES
	—√	± _____	± _____	± 5°

CASTING REQUIREMENTS

TOLERANCE ON CAST DIMS AS SHOWN

TOLERANCE ON CAST ANGLES ± 0°15'

Figure 3-3. Core Plate

3.3 ALLOY DEVELOPMENT

In selecting a metal fuel for the ZPR-III experiments, the following properties and characteristics were prime considerations:

-

When a plutonium-uranium alloy was being considered for the ZPR-III it was known that the alloy in the desired composition is metallurgically unstable, extremely brittle, and has poor air corrosion resistance. In tests at Argonne National Laboratory, a uranium - 30 weight percent plutonium (U-30 wt% Pu) alloy casting 1/4 inch thick, when exposed to air, began gaining weight on the second day and was completely disintegrated on the sixth day. Similar samples ignited spontaneously after a 30-hour exposure in air.⁽⁴⁾ The poor properties are caused by the presence of the zeta phase which is present in alloys containing over about 15 percent plutonium.⁽⁵⁾

An active development program at Argonne National Laboratory to reduce these undesirable characteristics of the alloy was accelerated to meet the needs of the SEFOR schedule. Early efforts were directed toward a plutonium-uranium-iron (Pu-U-Fe) alloy because iron was the least undesirable element for physics reasons. The Pu-U-Fe alloys were found to be sensitive to fabrication methods, with fine-grain chill castings showing the best properties. A chill cast U-27 wt% Pu-1.44 wt% Fe alloy had high self-heating temperatures and showed no weight gain after a 2-month exposure to flowing room air at 24°C and 25 to 56 percent relative humidity. However, the casting began to disintegrate after 11 weeks, and began gaining weight the twelfth week. The ignition temperature after several months storage was much lower, suggesting that the alloy deteriorates with time, making it an undesirable zero-power-reactor fuel.⁽⁴⁾

Other alloying elements which were tried included aluminum, carbon, copper, zirconium, molybdenum, vanadium, and titanium.^(2, 5) Of these alloys, a U-Pu-2.5 wt% Mo alloy appeared to have the most desirable characteristics. It is relatively insensitive to the method of casting and specimens were easily machined with nonlubricated carbide tools. Specimens showed no weight gain after a 4-month exposure to the atmosphere described above for iron.

Ignition tests were also performed on these materials. Tests were conducted in flowing air with the furnace temperature increased 10°C per minute until ignition occurred. The U-Pu-2.5 wt% Mo alloy did not self-ignite, even when heated to 800°C. Based on these tests at ANL, a U-19.5 wt% Pu-2.5 wt% Mo alloy was selected for use in the fuel wafers. Although molybdenum is undesirable in fast reactors because of neutron cross section, its use in the fuel for the SEFOR mock-up served as a useful substitution. Cladding used in SEFOR will be of Type-316 stainless steel containing 2 - 3 wt% molybdenum. Stainless steel used in the mock-up is Type-304, which does not contain molybdenum. The amount of molybdenum in the fuel alloy closely approximates the amount in the SEFOR cladding.

3.4 ALLOY PROPERTIES

The U-Pu-2.5 wt% Mo castings have acceptable expansion behavior for the critical experiments in ZPR-III. Dilatometric studies were made at Argonne National Laboratory in which 1-inch-long cast specimens were heated and cooled through several cycles between 30 and 700°C to assure reproducibility.

Average expansion coefficients for ranges of temperatures on heating are given in Table 3-2. The average coefficient increases from 15.3×10^{-6} cm/cm-°C in the range 30 to 220°C to 24.8×10^{-6} cm/cm-°C in the range 380 to 545°C. A transformation occurs between 545 and 617°C which results in an expansion of 3.95×10^{-3} cm/cm. The over-all expansion coefficient from 30 to 700°C is 24.6×10^{-6} cm/cm-°C.

TABLE 3-2

EXPANSION COEFFICIENT OF U-20 wt% Pu-2.5 wt% Mo
ALLOY CASTING

<u>Temperature Range °C</u>	<u>Average Expansion Coefficient, cm/cm-°C</u>
30-220	15.3×10^{-6}
220-380	23.7×10^{-6}
380-545	24.8×10^{-6}
545-617	Transformation
617-700	23.4×10^{-6}

The alloy is very strong. The compressive strength at room temperature and at 280°C was 190,000 psi and 150,000 psi, respectively. It has enough toughness at room temperature to permit handling and machining, but exhibited brittle failure in the above compression tests. Ductility increases rapidly above 400°C.

Thin castings of the metal have smooth, bright surfaces. Porosity is not a problem. Castings can be drilled and machined with carbide tools.

Density of the cast alloy is 18.28 gm/cc. Specific heat is 0.037 cal/gm-°C in the range from 25°C to 501°C.⁽⁷⁾

3.5 EXPANSION TEST

A requirement of the ZPR-III fuel wafers is that, during heating, the wafers expand along the column to increase the core length and thereby provide a shutdown mechanism. The cladding must avoid restraint of the fuel expansion, and clearance between fuel and cladding must be minimized to provide minimum lag and maximum expansion. Fuel wafers of the ZPR-III design containing depleted uranium were tested by ANL in the TREAT reactor at the National Reactor Testing Station.⁽⁸⁾ The experiments confirmed that expansion could be relied on as a shutdown mechanism.

3.6 STARTING MATERIALS

3.6.1 Plutonium

Plutonium for the fuel wafers, conforming with General Electric Specification 22A1522, was supplied by the Atomic Energy Commission and prepared at Hanford, Washington. The metal was furnished in the form of 2-kilogram ingots which were prepared by vacuum melting plutonium buttons and casting into ingots. Ingots were individually sealed in steel cans in a dry air atmosphere for shipment.

After casting, the ingots were sampled and measured for isotopic content and impurities. Isotopic content was measured in every fifth ingot by mass spectrometry, and Pu-240 content was verified by neutron counting. The Pu-240 content was specified to be between 8 and 9 percent. Data for five ingots, selected at random, are shown in Table 3-3.

TABLE 3-3

PLUTONIUM ISOTOPIC CONTENT

<u>Ingot No.</u>	<u>Isotopic Pu Content by Mass Spectrometry, %</u>				<u>Pu-240 Content by Neutron Counting, %</u>
	<u>239</u>	<u>240</u>	<u>241</u>	<u>242</u>	
24-11-75	90.485	8.570	0.901	0.044	8.526
24-11-90	90.401	8.613	0.940	0.046	8.524
24-12-1	90.425	8.596	0.936	0.043	8.441
24-12-9	90.483	8.552	0.924	0.041	8.290
24-11-15	90.640	8.418	0.898	0.044	8.480

Impurities were measured by emission spectrography in every lot, except for Co, Ta, W, N, O, F, Cl and C, which were measured by other methods in every fifth lot. Total impurities were generally less than 500 ppm. The approximate range of analyses for the entire order is shown in Table 3-4.

TABLE 3-4

TYPICAL RANGE OF IMPURITIES IN PLUTONIUM

<u>Element</u>	<u>Content, ppm</u>		<u>Element</u>	<u>Content, ppm</u>	
	<u>Low</u>	<u>High</u>		<u>Low</u>	<u>High</u>
Ag	1	1	Li	-	-
Al	5	5	Mg	2	100
As	-	-	Mo	-	-
B	1	2	Mn	5	50
Be	-	-	N ₂	1	2
Bi	-	-	Na	5	200
C	77	400	Ni	2	100
Ca	5	20	O ₂	1	101
Cd	-	-	P	-	-
Cl	3	11	Pb	2	20
Co	0	15	Si	15	33
Cr	1	50	Sn	1	20
Cu	2	10	Ta	0	24
F	1	25	Ti	-	20
Fe	40	545	Tl	-	-
Ge	-	-	V	-	-
H ₂	1	8	W	0	10
K	5	10	Zn	0	50
La	-	-	Total	23	868

3.6.2 Uranium

Uranium metal for the alloy was also in the form of ingots. Uranium was specified in G. E. Specification 22A1523. Depleted uranium was specified, with the U-235 content between 0.20 and 0.24 percent. A typical impurity analysis is shown in Table 3-5.

TABLE 3-5

IMPURITIES IN URANIUM

<u>Element</u>	<u>Content, ppm</u>	<u>Element</u>	<u>Content, ppm</u>	<u>Element</u>	<u>Content, ppm</u>
Ag	1	Cu	8	O ₂	82
Al	8	F	1	P	100
As	10	Fe	45	Pb	1
Au	1	Ga	1	Pd	1
B	1	Ge	0.4	Rb	20
Ba	2	Hf	100	Si	10
Be	0.01	In	2	Sb	2
Bi	1	K	6	Sn	1
C	20	Li	2	Sr	20
Ca	10	Mg	10	Ta	10
Cd	0.1	Mn	8	Ti	4
Cl	10	Mo	4	Tl	4
Co	1	N ₂	48	V	1
Cr	2	Na	1	W	2
Cs	100	Ni	12	Zn	10
				Zr	20

Uranium was shipped in wooden boxes with vermiculite packing between ingots.

3.6.3 Molybdenum

Molybdenum was procured as 3/4-inch-diameter round rod. Total metallic impurity content was specified at 1000 ppm maximum. Specific elements were limited as follows:

<u>Element</u>	<u>Maximum Content, ppm</u>	<u>Element</u>	<u>Maximum Content, ppm</u>
B	2	N ₂ + O ₂	10
C	30	Cl + F	25
H ₂	5		

3.7 FUEL WAFER FABRICATION

Fuel wafers were fabricated by Nuclear Materials and Equipment Corporation (NUMEC). Production equipment was designed and built by NUMEC and the fabrication process developed by ANL was modified as necessary to permit mass production. Core plates were made by first preparing a uranium-molybdenum

binary then combining the binary and plutonium in the proper proportion to produce the specified ternary core plate castings.

Uranium metal was received as 1 inch $\times$ 2 inch $\times$ 3 inch ingots and molybdenum was received as 3/4 inch round rod. Both were cleaned by a detailed pickling and rinsing procedure and weighed together to produce a nominal 20 kilogram charge, 3.11 wt% molybdenum and 96.89 wt% uranium. The charge was placed in a zirconia-coated graphite crucible and melted in a vacuum induction furnace. The metal was poured into zirconia-coated graphite molds to produce small ingots.

Plutonium ingots were removed from sealed cans, cleaned by brushing with a fiber brush, and broken in a nitrogen atmosphere. Binary and plutonium were weighed into approximately 8 to 9 kg charges, 19.55 wt% Pu, 2.50 wt% molybdenum, and the balance uranium. The graphite crucible was prepared by coating the pull rod with beryllia, followed by coating both the crucible and pull rod with zirconia.

The charge was melted in a vacuum induction furnace and poured into beryllia-coated graphite molds.

Core plates were notched and fractured to remove the sprue, and machined to length and width using a tungsten carbide milling cutter. All edges were also chamfered by using a milling cutter.

Core plates were adjusted to a specified weight, ± 0.2 percent. Weight was adjusted by machining the width of the plate (within tolerance) and by drilling material from the flat surfaces of the core plate.

All operations except U-Mo alloy preparation were performed in nitrogen-atmosphere glove boxes. Plutonium storage between operations was in gas-tight containers back-filled with nitrogen.

Jacket panels were stamped from Type-304 stainless steel strip and ultrasonically cleaned in acetone, rinsed in alcohol, and serial numbered with an electro-etch tool. Panels were washed in water after electro-etching. Filler strips, which provide shoe-weld material, were spot welded to both ends of the jacket panel as shown in Figure 3-2.

The assembly sequence used is illustrated in Figure 3-5. One jacket panel (at left in picture) was placed in a welding chill block (not shown), recess up. A core plate (simulated by a machined steel piece,

Figure 3-5. Fuel Wafer Assembly Sequence

second from left in picture) was placed in the jacket panel, and a second jacket panel was placed over the core plate, recess down. The upper chill block was placed over the jacket panel. Records were completed to cross reference the wafer number and the contained core plate number. The assembly was transferred into the low current welding box. Five wafers were assembled in this manner, and loaded into a welding jig for edge welding. An edge weld detail is shown in Figure 3-4.

Wafers were removed from the welding box and slotted weld runout rods were attached to the four corners (third from left in Figure 3-5). These runout rods resemble a one-piece clothespin. They provide material beyond the edges of the wafer for starting and stopping the arc, thereby preventing burnout of the jacket panel. Ten assemblies were placed in the shoe-weld box and loaded into a lazy-susan welding jig. The welding box was evacuated, back-filled with welding grade argon, re-evacuated and back-filled to atmospheric pressure with welding grade argon. A shoe weld was made on one end of each of the 10 assemblies. The assemblies were rotated in their fixtures and the welding box was evacuated and back-filled to 6 psi with an argon-helium mixture. A shoe weld was then made on the other end of the 10 assemblies, producing a gas-tight wafer. Figure 3-4 shows the outline of a shoe weld, both as cast and after machining.

Shoe welds were machined with a high-speed-steel end mill, and the weld runout rod was machined off, using care to prevent machining into the edge welds. The completed wafer is shown in Figure 3-5 (right side). Completed wafers were cleaned in pure acetone followed by a rinse in methyl alcohol. The outside surfaces of the completed wafers showed a fixed alpha count of less than 200 counts per minute per 61 square centimeters, as measured with an Eberline PAC-3G gas proportional counter, and a removable alpha contamination of less than 10 disintegrations per minute (above background) per square foot.

The cleaned wafers were leak-checked by two methods. The first test was by mass-spectrometer leak detector, with a maximum permissible leak rate of 1×10^{-8} standard cc/sec. In the second leak test, the wafers were placed in a pressure chamber and the chamber pressurized to 45 psig. The chamber was depressurized and opened; the wafers were removed and immersed in methyl alcohol. The second test was intended to reveal large leaks that may not have been detected in the first test due to complete evacuation of the wafer during the pumpdown operation.

After leak tests, wafers were cleaned by washing in Lab-Sol water solution, stains were removed by buffing with fine steel wool, and wafers were rinsed in alcohol and dried in air. They were again smeared to check for contamination.

A more detailed description of the development and fabrication of the fuel wafers may be found in ANL and NUMEC reports and procedures. (3, 9, 10)

3.8 QUALITY CONTROL

Stringent quality control was necessary because of the nature of the experiments and the material being handled. Characteristics most important are given in Table 3-6, with the tolerance and frequency of measurement.

TABLE 3-6
FUEL QUALITY CONTROL METHODS AND FREQUENCY

<u>Characteristic</u>	<u>Nominal</u>	<u>Tolerance</u>	<u>Specified Accuracy of Measurement</u>	<u>Measurement Method</u>	<u>Frequency</u>
<u>Pu-240</u>					
Total Pu	0.085	±0.005	±0.0015	Neutron Counting	Each ingot
<u>Pu-239 + 241</u>					
Total Pu	Not Specified	Not Specified	±0.0015	Mass Spectrometry	Each ingot
<u>U-235</u>					
Total U	0.0022	±0.0002	±0.25% of measur- ed isotope	Mass Spectrometry	Each lot
Core Plate Dimensions	See Figure 3-3	See Figure 3-3	See Figure 3-3	Micrometer	Each core plate
Core Plate Weight	See Table 3-1	±0.2%	±0.05 gram	Balance	Each core plate
Homogeneity of Pu Dispersion	No sample to deviate more than 1 wt% from the average composition	Not Specified	Not Specified	X-ray fluorescence mini-probe	One piece from each melt.
<u>Pu-239-241</u>					
Total Core Plate	0.1790	±0.0005	±0.05%	Mass spectrometer and balance	Each melt
<u>Mo</u>					
Total Core Plate	0.0250	±0.0006	±0.05%	Balance	Each melt
Jacket Dimensions	See Figure 3-2	See Figure 3-2	See Figure 3-2	Not Specified	Each jacket
Visual Weld Quality	---	---	---	Visual	Each wafer
Weld Current	---	---	---	Recorder	Each wafer
Bead Thickness	See Figure 3-4	See Figure 3-4	See Figure 3-4	Metallography	Each shift
Leak Check	1×10 ⁻⁸ std cc/sec	---	Inst. Sensitive to 10 ⁻⁹ std cc/sec	Mass Spectrometer	Each wafer
Fixed Surface Contamination	200 cpm/ 61 sq cm (max.)	---	---	Eberline PAC-3G	Each wafer
Removable Surface Con- tamination	10 cpm/ft ² (max.)	---	---	NRD Scintillation Counter	Each wafer
Wafer Weight	See Table 3-1	Not Specified	±0.05 gm	Balance	Each wafer

Only the most important quality control requirements are listed in Table 3-6. On most characteristics, there was duplication of measurements, e.g.: (1) Plutonium composition was checked on a sampling basis by the wafer fabricator; (2) All processes required qualification by making preproduction pieces and checking results before production could begin; (3) Finished preproduction pieces of each size were shipped to ANL for examination before production of that size was begun; (4) Production samples were selected at random, one from each 100 wafers produced, and sent to ANL for inspection; and (5) Primary control of the core plate composition was by weight of each material used in the melt, but the composition of core plates from each melt was checked by wet chemistry to eliminate the chance of gross errors. Record forms were prepared for each operation and completed during production. A data book prepared at the completion of production tabulates for each wafer, by serial number, its Pu-239, Pu-241, U-235, U-238, and total weights.

SECTION IV

BERYLLIA4.1 QUANTITIES AND APPLICATION

Beryllia is used in SEFOR to soften the neutron spectrum and thus increase the Doppler coefficient. The weight of beryllia in SEFOR will be duplicated in the critical experiments.

Three-hundred-twenty-five kilograms of beryllia in the form of thin tiles were made by Brush Beryllium Company. The tiles were made in three sizes and in quantities indicated in Table 4-1.

TABLE 4-1
BERYLLIA SIZES AND QUANTITIES

<u>Size, Inches</u>	<u>Pieces</u>	<u>Weight per Tile, grams</u>
$1/4 \times 2 \times 2$	6750	45.24
$1/4 \times 1 \times 2$	850	22.62
$1/8 \times 2 \times 2$	200	22.62

A drawing of the beryllia tile is shown in Figure 4-1.

PT. NO.	C	D
1	1.995	.242
2	.995	.242
3	1.995	.117

NOTES

1. FOR SPECIFICATION SEE 22A1504

Figure 4-1. Beryllia Tile

4.2 DESCRIPTION OF MATERIAL

The tiles were prepared by pressing BeO powder and sintering to a density of 2.91 gm/cc (theoretical density is 3.01 gm/cc). All tiles were prepared from a single powder lot, and were processed in batches of approximately 1000 tiles.

After sintering, tiles were checked for cracks by treating with Fuschin dye in an ethyl alcohol base. They were then ultrasonically cleaned in a commercial cleaning solution, ultrasonically rinsed in deionized water, and dried in a nonvented furnace. During cleaning and rinsing, tiles were separated to prevent abrasion between tiles.

Impurities in the tiles were less than 5000 ppm. No element exceeded the specified limits tabulated in Table 4-2.

TABLE 4-2

BERYLLIA IMPURITY LIMITS

<u>Element</u>	<u>ppm</u>	<u>Element</u>	<u>ppm</u>
Al	6000	Li	20
Ag	10	Mg	4000
B	20	Mn	200
Ba	100	Mo	40
C	600	Na	250
Ca	500	Ni	150
Cd	20	Pb	40
Co	10	Si	1600
Cr	100	Sn	20
Cu	100	Sr	200
F	200	Ti	20
Fe	600	Zn	40
H	50		

Tiles from each batch were checked for the following properties and met the specified limits.

Average Grain Size	20 - 55 microns
Cracks	1/64 inch maximum width, 1/4 inch maximum length, depth less than 1/8 inch
Chips and Pits	1/16 inch deep (maximum) by 3/16 inch diameter (maximum)
Modulus of Rupture	25,000 psi

Finished tiles were weighed to an accuracy of 0.1 gram. Weight of each tile fell within 1 percent of the specified weight.

Tiles were specified as bare tiles, but because of the toxicity of BeO powder, a limit on surface powder of 25 micrograms/ft², as determined by a smear test, was specified. Preproduction tiles prepared by the production process met this specification, but a large percentage of the production tiles exceeded the specified limit. Repeated cleaning failed to reduce the surface powder to an acceptable level, and it was necessary to coat all of the tiles with a Kel-F nonhydrogenous plastic coating. Tiles were coated by dipping in the plastic and drying. Coating thickness was less than 0.001 inch. The coating was effective in reducing the surface powder level.

SECTION V

SODIUM CARBONATE

5.1 SELECTION OF MATERIAL

Metal fuel was provided for ZPR-III critical experiments for reasons previously explained, even though SEFOR will use oxide fuel. A dense material high in oxygen was needed for the ZPR-III loading to provide a simulation of the amount of oxygen present in oxide fuel.

Beryllia was the preliminary selection as a mock-up for oxygen, but further studies revealed the adverse effects of the beryllium (n, 2n) reaction and high energy cross section on physics measurements. (11) Other materials considered included Fe_2O_3 , Na_2O , Na_2O_2 , C, Al_2O_3 , and Na_2CO_3 . Of these, Na_2CO_3 containing sodium equal in weight to the amount in SEFOR was found to provide most of the oxygen needed. Also, the carbon in the Na_2CO_3 served as a reasonable substitute for oxygen. The balance of the oxygen was simulated by Al_2O_3 and graphite. Laboratory tests showed that Na_2CO_3 powder could be melted and cast in blocks at greater than 93 percent of theoretical density and powder could be easily pressed to 85 percent of theoretical density. Sodium carbonate was selected as the mock-up material, and the fabricator, Nuclear Materials and Equipment Corporation, was given the option of casting or pressing 310 kilograms. NUMEC chose to press the powder.

5.2 WAFER DESCRIPTION

Because of the deliquescence of Na_2CO_3 and its low strength, it had to be contained in a stainless steel jacket. The design used by the fabricator was 1/4 inch $\times$ 2 inch rectangular tubing, 0.012 inch wall, filled with Na_2CO_3 blocks and sealed by welded end plugs. A drawing of the wafer is shown in Figure 5-1. The quantities shown in Table 5-1 were provided.

TABLE 5-1

SODIUM CARBONATE WAFER SIZES AND QUANTITIES

<u>Size, inches</u>	<u>Pieces</u>	<u>Na_2CO_3 Weight per Wafer, gm</u>	<u>Container Weight, gm</u>
1/4 $\times$ 2 $\times$ 3	4550	42.73	26.71
1/4 $\times$ 2 $\times$ 4	1300	56.92	33.37
1/4 $\times$ 2 $\times$ 5	650	71.19	38.84

The specified composition of the sodium carbonate was as shown in Table 5-2.

TABLE 5-2

SODIUM CARBONATE COMPOSITION

Constituent	Content, %
Na_2CO_3	99.6 minimum
NaHCO_3	0.10 maximum
NaCl	0.20 maximum
Na_2SO_4	0.02 maximum
B	0.001 maximum
H_2O	0.10 maximum

The density of the sodium carbonate was specified at 14 grams per inch of can length (minimum).

Figure 5-1. Sodium Carbonate Wafer

5.3 WAFER FABRICATION

Rectangular tubing was made from sheet stock by forming around a die and seam welding at one corner of the tube. Sodium carbonate was pressed into small blocks 0.973 inch $\times$ 0.949 inch $\times$ 0.220 inch, at 88 percent of theoretical density. The lower end plug was welded into the tubing and the tubing filled with blocks; six blocks per 3-inch can, eight blocks per 4-inch can, and ten blocks per 5-inch can. Can, blocks, and end plug were weighed and the upper end plug was inserted and welded. The can was

machined lightly on both ends to meet the finished dimension. Sodium carbonate and cans were kept hot until loading, and final welding was done promptly after loading to reduce moisture content.

Finished cans were leak-checked by immersing in 180° F water and watching for bubbles.

Weighing was done on a balance accurate to ± 0.05 gram. An average empty can weight was determined by weighing a large number of empty cans and end plugs. The contained sodium carbonate weight was determined by subtracting the average can weight from the measured wafer weight at the time of loading.

SECTION VI

NICKEL BLOCKS

6.1 DESCRIPTION OF MATERIAL

Two-thousand-two-hundred kilograms of nickel blocks were contracted by the Chicago Development Corporation to supplement the existing 2400 kilograms already on hand at ZPR-III. The blocks were of one size, nominally 1/4 inch $\times$ 2 inches $\times$ 2 inches. The average block weight was 141.45 grams, and 15,750 were procured. A drawing of the nickel block is shown in Figure 6-1.

Chemical composition was specified as shown in Table 6-1:

TABLE 6-1

NICKEL BLOCK COMPOSITION

<u>Element</u>	<u>Content, %</u>	<u>Element</u>	<u>Content, %</u>
Ni	98.9 minimum	Fe	0.40 maximum
B	0.003 maximum	Mn	0.35 maximum
C	0.15 maximum	S	0.01 maximum
Co	0.10 maximum	Si	0.35 maximum
Cu	0.25 maximum		

NOTES

1. FOR SPECIFICATION SEE 22A15B1

2. STAMP "Ni" IN $\frac{1}{8}$ " HIGH LETTERS IN POSITION SHOWN. (BOTH SIDES)

Figure 6-1. Nickel Block

6.2 FABRICATION

Some blocks were fabricated from hot-rolled bar stock and some from 1/4 inch sheet. The stock was cleaned and cold-rolled to thickness, then cut slightly oversize. Blocks were milled to length, then gang milled to width. They were identified as nickel with a metal-stamped mark and cleaned with acetone.

SECTION VII

BORAL BLOCKS

7.1 SELECTION OF MATERIAL

Fifty kilograms of B_4C were procured to supplement existing material at ZPR-III in mocking up a 90-degree sector of the 2-inch-thick shield annulus surrounding the SEFOR reflector. Boron carbide in three forms was considered: hot-pressed and sintered blocks, canned B_4C powder, and Boral. Boral was found to be the most economical source. One-thousand-four-hundred and twenty blocks $1/4$ inch $\times$ 2 inches $\times$ 7 inches were procured. Fabricator was Brooks and Perkins Co. A drawing of the Boral blocks is shown in Figure 7-1.

NOTES

1. FOR SPECIFICATION SEE 22A1586.
2. BREAK ALL EDGES.

Figure 7-1. Boral Block

7.2 FABRICATION

Boral is a trade name for a sheet material made by suspending B_4C in molten aluminum. The molten mixture is cast, rolled, and clad on both sides with aluminum to make a sheet $1/4$ inch thick. The B_4C used in the Boral sheet was specified to be at least 70 percent boron, and uniform (± 1 percent) from lot to lot. Grit size was 20 to 200 mesh. The B_4C content was specified to be between 22 and 25 weight percent of the total block weight.

Boral sheet was sheared to size and coated with Kel-F nonhydrogenous plastic to protect the sheared edges and prevent particles of B_4C powder and aluminum from flaking off.

The average block weight was determined by weighing the entire order and dividing the weight by the number of blocks.

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Development of fuel alloys and the fuel jacket design was performed by ANL. General Electric Company gathered information from many sources to prepare the specifications, drawings, and quality control plans. Sodium carbonate was being developed as a mock-up material by Kernforschungszentrum Karlsruhe when the SEFOR Project was started, and additional work on pressing and casting the material was done by General Electric. Assistance was obtained from ANL in specifying other materials used in the critical assembly.

Procurement of all materials was carried out by the San Francisco Operations Office of the USAEC, with Messrs. J. P. Holliday and J. Duffy coordinating the procurement activities. Technical liaison with the contractors throughout the procurement period was performed by the AEC, assisted by G. E. and ANL. Major contributions to the General Electric effort were made by Messrs. R. Wallace and S. A. Wilson.

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