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FABRICATION REPORT
FFTF PROTOTYPIC FUEL PELLETS
FOR ANALYTICAL CHEMISTRY PROGRAM

H. T. Blair

April 1970

AEC RESEARCH &
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FFTF Fuel Department
FFTF Division

April 1970

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BY

H. T. Blair

ABSTRACT

Approximately 4000 sintered, 25 wt% PuO_2 -75 wt% UO_2 , FFTF type fuel pellets were fabricated in the initial test of the FFTF Fuel Pin Fabrication Development and Demonstration Facility. The materials, equipment, techniques, and sampling used in the fabrication are described. Complete characterization of the sintered pellets including dimensions, density, chemical composition, and homogeneity is reported. The sintered pellets have an average density of 91.35% TD and an average oxygen-to-metal ratio of 1.99. The pellets are homogeneous, and no evidence of the existence of more than one phase was detected. The O/M of 200 of the sintered pellets was reduced to 1.96. One hundred of the pellets were centerless ground for use in determining pellet density by geometrical methods.

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INTRODUCTION

This report contains the fabrication history and characterization of the mixed oxide pellets produced in the initial test of the FFTF Fuel Pin Fabrication Development and Demonstration Facility located in the Plutonium Fuels Laboratory, 308 Building, of Battelle-Northwest. These studies were conducted to evaluate the fabrication facilities, procedures, and sampling methods used for producing and characterizing these pellets.

The pellets were fabricated in support of the second phase of the FFTF Analytical Chemistry Development Program.⁽¹⁾ For this program a special batch of 3000 to 4000, 25 wt% PuO₂-75 wt% UO₂, sintered, fuel pellets of high uniformity was required. The pellets were fabricated in accordance with the then applicable revision of the manufacturing process specification,⁽²⁾ except that a sintered pellet density of 91 to 92% of theoretical (TD = 11.09 g/cm³) and an oxygen-to-metal ratio between 1.95 and 1.97 were requested. One hundred sintered pellets centerless ground to be cylindrical were also requested for use in determination of densities by the geometrical method.

SUMMARY

Approximately 4000 sintered, mixed oxide fuel pellets were fabricated in the initial test of the FFTF Pin Fabrication Development and Demonstration Facility during the months of February and March of 1969. These pellets were produced for use in the second phase of the Analytical Chemistry Program.⁽¹⁾ The powder preparation and pellet fabrication processes and the sampling plan followed during these processes are shown in Figure 1 and Table 1, respectively. Two deviations from the process specification⁽²⁾ were made during the fabrication:

- 1) the PuO_2 source material was calcined 25 °C below the temperature specified because the calcining was completed prior to the issue of this revision of the specifications, and
- 2) two cross-blend steps were introduced in the process to level individual batch variations and to assure homogeneity of the oxide powder mixture. These cross-blends were necessary since the criticality mass safety limits in effect in the facility at that time permitted preparation of only one-third of the feed material at one time. The pressed pellets were debound as a single batch and sintered as a single batch.

The importance of glovebox atmosphere control for mixed oxide processing was emphasized on several occasions during the fabrication of the pellets. The milled, mixed oxide powder was removed from the demonstration facility to a glovebox where mass safety limits would permit the cross-blend of all prepared powder. The powder gained weight during the cross-blend by absorbing water from the glovebox atmosphere, determined to have a relative humidity of approximately 14%. The necessity of atmosphere control was again emphasized when the green pellets were debound in a furnace outside the demonstration facility. The debound pellets were removed from the furnace into a glovebox having a nitrogen atmosphere. The pellets began to gain

FIGURE 1. Flow Diagram of Powder Preparation and Fuel Pellet Fabrication

TABLE 1. Sampling and Testing Plan Followed During Fabrication of the Analytical Chemistry Fuel Pellets

Process Step	Size of Sample	Purpose of Sample	Remarks
Source PuO ₂	----- 35 g	Lot Characterization History	Performed at ARHCO 25 g Can 17, 10 g Can 18
Source UO ₂	25 g 10 g	Lot Characterization History	Can 7
Calcined PuO ₂	25 g	Characterization	Composite of Cans 17 and 18
Ball Milling	24 g	Surface Area Carbon Content	12 g from PL-1 and 12 g from PL-2
First Cross Blend	17 g 10 g 68 g	% Pu, Impurity Levels Surface Area History	5 g each PL-8, PL-9, and PL-10 Composite of PL-8, PL-9, and PL-10
Pressing	12 pellets	Green Density and Dimensions	Random Sample
Sintering	10 pellets 183 pellets 53 pellets 19 pellets 9 pellets 3 pellets	History Dimensions and Density Analytical Chemistry O/M Ceramography, α - autoradiography, microprobe, X-ray diffraction	Equal number of pellets taken from each tray From trays 1, 4, 6
O/M Adjust	30 pellets 5 pellets 2 pellets	Dimensions and Density O/M Ceramography	
Centerless Grinding	35 pellets	Dimensions and Density	Random Sample

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weight and heat up immediately upon exposure to the glovebox atmosphere. The glovebox atmosphere was found to contain traces of oxygen.

The green pellets had an average density of 48.61% of theoretical ($TD = 11.09 \text{ g/cm}^3$). Some laminating of the pellets during pressing was encountered when the feed material was not well dried.

The as-sintered pellets exhibited the following properties:

Dimensions

- An average density of 91.35% TD.
- An average taper of 0.0012 in.
- No tendency to assume either an hourglass or barrel shape (both shapes were equally present with the average effect being 0.0004 in. for each shape).

Chemical Composition

- Impurity levels, oxygen-to-metal ratio, gas content, and moisture content within the limits set by the process specification.⁽²⁾
- Plutonium and uranium contents that varied less than 0.5% from the nominal values of 22.10 wt% Pu and 66.05 wt% U.

Homogeneity

- A homogeneous, undeveloped microstructure with generally good PuO_2 dispersion (some areas greater than 50 μ across were found which contained more than 25 wt% PuO_2).
- No evidence of the existence of more than one phase was detected.

One hundred sintered pellets were centerless ground to an average diameter of 0.1970 in. This grinding resulted in a reduction from 0.664 to 0.592 of the estimated standard deviation of the pellet densities as determined by geometrical methods, but did not significantly change the mean.

The oxygen-to-metal ratio of two hundred pellets was reduced from 1.99 to 1.9⁶/₈ as requested for the Analytical Chemistry Program.

An investigation of the effect of position in the furnace on pellet properties indicates a slight tendency of the pellet dimensions and O/M to decrease with positions from the top to the bottom of the furnace while pellet densities tend to increase with position from the top to the bottom.

FUEL PELLET FABRICATION

A flow diagram depicting powder preparation and pellet fabrication is presented in Figure 1. The sampling plan followed during the fabrication is outlined in Table 1.

SOURCE MATERIAL

The plutonium oxide powder used in the fabrication of the mixed oxide fuel pellets was part of an 18 kg lot supplied to Battelle-Northwest by Atlantic Richfield Hanford Company. This material was produced at ARHCO by a continuous process of oxalate precipitation from a plutonium nitrate solution followed by calcination of the oxalate at 450 °C. Tables 2, 3, and 4 present the results of analyses performed by ARHCO on this lot of plutonium oxide. Cans 17 and 18 of Lot 2 were used in this fabrication test run.

The uranium oxide powder used in the fabrication of these fuel pellets was ceramic grade UO_2^* of natural enrichment. Tables 2 and 3 present the results of analyses performed at BNW on the as-received UO_2 powder.

CALCINING

The PuO_2 powder was calcined in two separate batches (Table 5). Each batch of powder was calcined in a Magnorite

* *Procured from Nuclear Fuel Services Corporation (manufactured by Eldorado of Canada).*

TABLE 2. Impurity Levels in the Oxide Feed Materials

Impurity ppm By Weight	ARHCO Lot 2 PuO ₂			Eldorado UO ₂	Calcined PuO ₂	Milled Mixed Oxide		Milled and Blended Mixed Oxide		
	Can 16	Can 19	Can 22	Can 7	Cans 17 and 18	PL-1	PL-2	PL-8	PL-9	PL-10
Ag				<.1	2			<.1	<.1	<.1
Al	<5	<5	<5	5	10			50	20	50
As				**	**			**	**	**
B				<.1	.1			**	**	**
Ba				**	**			**	**	**
Be				**	**			**	**	**
C	2106	485	655	170	280	1000	1080			
						1030	1130			
Ca	100	100	100		>1000			500	500	500
Cd	<1	<1	<1	**	**			**	**	**
Cl	*	*	*	<10	14-					
					19					
Cr	<10	<10	<10	<5	<25			<25	<25	<25
Cu				2	2			5	5	5
F	<20	<20	<20	<5	125-					
					138					
Fe	<25	<25	62	50	20			<10	<10	<10
K				**	**			**	**	**
Li				**	**			**	**	**
Mg	<1	<1	<1	<1	50			10	<5	5
Mn				<1	5			2	2	2
Mo				**	**			**	**	**
N	30	30	33							
Na	5	5	5	**	10			2	2	2
Ni	<5	<5	<5	<5	<10			<10	<10	<10
P				**	<50			**	**	**
Pb				<.5	100			10	10	10
Sb				**	**			**	**	**
Si				5	<5			10	10	10
Sn				1	5			10	10	5
Tl				**	<.2			**	**	**
V	<100	<100	<100	**	**			<20	<20	<20
W								**	**	**
Zn				<50	<50					
The sum of Cu, Zn, Si, Ti	<36	<89	201							
The sum of Ag, Mn, Mo Pb, Sn	<22	<22	<22							

* Original analysis were <20 ppm. Later samples were contaminated by chloride from sample containers.

** Not detected.

TABLE 3. Chemical Composition of the Oxide Feed Materials

	ARHCO Lot 2 PuO ₂			Eldorado UO ₂		Calcined PuO ₂		Milled Mixed Oxide		Milled and Blended Mixed Oxide	
	Can 16	Can 19	Can 22	Can 7		Cans 17,18		PL-1	PL-2	PL-8	PL-9 PL-10
% Pu	87.56	87.96	87.72			87.8				21.9	21.9 22.0
U Content	<20ppm	<20ppm	<20ppm	86.6%							
H ₂ O Content		1.48%*		3700ppm		5950ppm					
Gas Content		2.36%*				5.3 cm ³ /g					
Surface Area (m ² /g)		39.4*		8.5		15		11	11		6.2**
O/M	<2.0	<2.0	<2.0	2.155							
Average Particle Size (microns)			11.8 to 12.0*								
Bulk Density g/cm ³			1.60*								
Tap Density g/cm ³			2.02*								

* A single sample taken for Lot 2

** Composite of three batches: PL-8, PL-9, and PL-10

TABLE 4. Plutonium Isotopic Composition
of PuO₂ Source Material

Isotope wt%	ARHCO Lot 2 PuO ₂			Calcined PuO ₂
	Can 16	Can 19	Can 22	Cans 17, 18
238	0.095	0.087	0.099	0.086
239	86.373	86.393	86.369	86.56
240	10.789	10.902	10.910	10.83
241	2.498	2.390	2.404	2.30
242	0.245	0.228	0.218	0.222

TABLE 5. PuO₂ Source Material Calcining Data

	<u>Can No. 17</u>	<u>Can No. 18</u>
Initial Powder wt, g	992.3	989.9
Rate of Rise, °C/hr	650	660
Soak Temp, °C	650-680	650-670
Soak Time, hr	1	1
Rate of Cooling, °C/hr	140	137
Calcined wt, g	984.1	982.3
Weight Lost, g	8.2	7.6

crucible placed in an open, resistance heated, pot furnace. The powder was stirred every 15 to 20 min during the heat-up and soak phases of the calcining cycle. The crucible was covered with a loose fitting cap whenever the powder was not being stirred. The atmosphere in the glovebox was dry air.

The PuO_2 source material was calcined by using a rate of rise greater than that specified in the process specification and a soak temperature 25 °C less than that specified because the calcining was completed before process requirements for calcining of PuO_2 powder had been included in the process specification.

Tables 2, 3, and 4 present results of analyses of samples taken from the calcined PuO_2 powder. An average of 0.8 wt% was lost as volatiles in calcining the PuO_2 powder because of a change in the moisture content of the powder. The moisture content of the calcined powder was 0.89 wt% less than that of the PuO_2 as received from ARHCO. The carbon content was reduced several hundred parts per million, while fluoride, magnesium, and calcium were picked up in the calcining process. The fluoride may have originated from the sample vials, and the magnesium and calcium came from the calcining crucible and lid. The calcining process reduced the specific surface area of the PuO_2 powder from 39.4 to 15 m²/g.

SCREENING

Before the PuO_2 was batched for mixing with the UO_2 , it was passed through a 325-mesh stainless steel screen to break up any large agglomerates and to remove any foreign objects picked up from the shipping containers or in the calcining process. The UO_2 powder was also screened before batching by passing it through a 100 mesh stainless steel screen.

POWDER BATCHING AND BLENDING

At the time these fuel pellets were being fabricated in the new Process Development and Demonstration Facility, the Criticality

Safety Specifications for the facility had not been completed. Thus, the facility was operating under conservatively restrictive mass safety limits. These limits allowed preparation of only 2 kg of mixed oxide at one time within the facility. A total of 7 kg of mixed oxide powder was prepared for the program in 1 kg batches (Table 6).

Two 1 kg batches were prepared at one time and processed together through the milling stage of powder preparation. The screened PuO_2 was weighed into 250 g batches on a Mettler Model P10 balance, weighing to the nearest gram. The screened UO_2 was weighed into 750 g batches on the same balance. These batches were then mixed by layering them on a 20-mesh stainless steel screen and passing them through the screen five times. After the fifth pass through the screen, the mixed oxide powder batch was further blended 10 min in a 2 qt capacity, Patterson-Kelly Model CB-S-2, Cantilever Type, "V" Blender equipped with an intensifier.

MILLING

Each 1000 g batch was dry milled for 20 hr in a Size 0 ball mill jar at a speed of 80 rpm. The jars used were U. S. Stoneware brand, made of steel, and lined with low ash rubber. Each jar contained 10 lb of U. S. Stoneware brand tungsten carbide, radius end, 3/8 in. size cylinders. The milled powder was separated from the cylinders by emptying the contents of a jar onto a 20-mesh screen. The mixed oxide powder forms a heavy coat on the milling cylinders which must be knocked off. Each milled batch was again blended in the "V" blender with the intensifier for 5 min before being canned for movement to the next process step.

After seven batches of mixed oxide had been milled, samples were taken from batches PL-1 and PL-2 for surface area and carbon content analyses to determine the influence of the milling.

TABLE 6. Mixed Oxide Powder Preparation Batches

Batch Number	PuO ₂ Source		UO ₂ Source		Batch Weight, g	Milling Time, hr
	Can Number		Drum Number			
PL-1	17		7		1000	20
PL-2	17		5**		1000	20
PL-3	18		7		1000	22
PL-4	18		7		1000	20
PL-5	17 + 18*		7		1000	20
PL-6	17 + 18*		7		1000	20
PL-7	17		7		1000	20

* 55.3 g of PuO₂ from Can 17 were added to 469.7 g of PuO₂ from Can 18.

** Drum 5 was part of the same lot as Drum 7.

The results of these analyses are reported in Tables 2 and 3. The average specific surface calculated for the mixed source material was $10.1 \text{ m}^2/\text{g}$. The specific surface area of the milled powder as determined by BET methods was $11 \text{ m}^2/\text{g}$. Thus, the milling process had little effect on the surface area. The carbon content of the milled powder was increased by about 800 ppm. This carbon was assumed to have come from the rubber mill linings and not the tungsten carbide cylinders as no trace of tungsten was detected in the sample of cross-blended powder analyzed.

A test batch, designated PL-2, was chosen to be put through the remaining fabrication steps ahead of the bulk material to evaluate those steps and the new equipment.

FIRST CROSS-BLEND

Mixed oxide batches PL-1, PL-3, PL-4, PL-5, PL-6 and PL-7 were removed from the Process Development and Demonstration Facility to an area where they could be brought together and cross-blended to level any individual batch variations and to assure the homogeneity of the mixed oxide powder. The plan followed in cross-blending is shown in Figure 2. The batches were split using a Jones sample splitter. They were then blended by layering on a 20-mesh screen. After Blend 3 (Figure 2), the three resulting 2 kg batches were split into thirds by weighing 666 g quantities. These quantities were then cross-blended (Figure 2) in a Patterson-Kelly "V" Blender for 2 min using the intensifier for 5 sec of every 30 sec during the 2 min. The resulting three batches of powder were designated PL-8, PL-9 and PL-10.

During the cross-blending operation, the mixed oxide powder was observed to be gaining weight. By the time the cross-blending was completed, the powder had increased in

FIGURE 2. Diagram for First Cross-Blend

weight by 1.3 wt%. This powder weight increase was due to moisture absorbed from the glovebox atmosphere which was determined to have a relative humidity of approximately 14%.

After the cross-blending was completed, the mixed oxide powder was sampled and analyzed to determine the percent plutonium, specific surface area, and impurity content of the powder. The results are reported in Tables 2 and 3. The results of the percent plutonium and impurity analyses show that the powder batches were well leveled and that the PuO_2 and UO_2 had been batched properly. (the calculated plutonium content of 25 wt% PuO_2 -75 wt% UO_2 is 22.10 wt%.) The only impurity picked up was aluminum which increased by 40 ppm. The source of this aluminum is unknown. The iron, magnesium, and lead content all appear to have decreased. The specific surface area decreased from $11 \text{ m}^2/\text{g}$ to $6.2 \text{ m}^2/\text{g}$. The cause of this decrease has not been determined.

BINDER ADDITION

A 2 wt% Carbowax 20M binder was added to the powder to act as a lubricant during pressing and to increase the green strength of the pressed pellets. A 20 wt% Carbowax 20M solution was prepared by dissolving 200 g of Carbowax 20M in 800 g of deionized water. ^{from} ~~For~~ this solution 100 g quantities were removed and combined with 1000 g quantities of mixed oxide powder and from 140 to 200 g of deionized water as needed to make a stiff slurry. The slurry was mixed by hand in a stainless steel pan with a stainless steel spatula and a mild steel putty knife. The pan of slurry was vibrated several minutes after mixing to remove air bubbles.

The slurry was dried in a Gruenberg Electric Company laboratory drying oven at 70 to 80 °C in an air atmosphere until a firm but not completely dry cake had formed.

GRANULATION

The cake was then granulated by gently working the material through a 20-mesh stainless steel screen over a 150-mesh stainless steel screen. The -150 mesh material (15 to 20 wt% of the batch) was saved from each batch and reprocessed as a single batch at the end of the binder addition and granulation of all other powder. This was done by adding only deionized water to the -150 mesh material to make a slurry again, and processing the slurry as described above.

SECOND CROSS-BLEND

After all the mixed oxide material was granulated, it was again moved to an area where it could all be cross-blended. The powder was cross-blended the second time by simply passing all the granulated material through a Jones sample splitter several times.

Following the second cross-blend, the granulated material was dried two days in a Thelco drying oven at 70 °C in an air atmosphere. The powder was then stored in the oven under an air atmosphere and maintained at 50 °C until it was removed as needed to feed the press. This powder was considered one batch and was labeled PL-8910.

PRESSING

The granulated mixed oxide with binder was pressed into pellets at 20,000 psi using a dwell time of one second. The press used was a Western Sintering Pellet Press, Model H-10-2, having a pressing rate of 320 pellets per hour at the given pressure and dwell time. The die and punches were made of D-2 type tool steel hardened to approximately 58 Rockwell C. The die cavity diameter was 0.246 in. and the punch diameter was 0.245 in. The die size was chosen to make a pellet having a diameter near prototypic of FTR fuel pellet size. The relatively low pressing pressure was chosen to aid in achieving

the desired low sintered density of 91 to 92% of theoretical. This choice of pressure was based on the results of tests conducted using batch PL-2 material processed through the same press and furnaces to be used in fabricating the final pellet batch. The relatively long dwell time was used to reduce some of the pronounced axial shrinkage gradient which typically occurs with sintered pellets pressed at low pressures, resulting in an hourglass effect. The extended dwell permits more time for the forces and material within the die to become distributed.

Three thousand six hundred pellets were pressed. The green pellets were randomly sampled as they came off the press. Dimensions and green densities from this sampling are summarized in Table 7. Each dimension was measured once with a micrometer reading to 0.001 in. and weighed on a Mettler Model P120 balance to 0.01 g. The range reported for green length includes those pellets measured while the press was being adjusted for length control. Green pellet lengths were held to 0.313 ± 0.005 in. after the press was set up.

Some pellet laminating and powder build-up on the punch was encountered during pressing when the moisture level of the feed powder was too high.

TABLE 7. Green Pellet Dimensions and Density

	<u>Average</u>	<u>Range</u>
Diameter, in.	0.2478	0.2480 0.2476
Length, in.	0.3118	0.3164 0.3090
% TD	48.61	48.38 48.74

BINDER REMOVAL

The total mass of green pellets was debound as a single batch at 675 °C for 3 hr in a Harper muffle furnace using the firing cycle shown in Figure 3. The soak at 375 °C for 8 hr was an overnight hold so that the final rise and soak would be under constant observation and manual control.

The pellets were loaded into a single large molybdenum boat. The furnace was evacuated to 60 μ and backfilled with dry helium before the heating cycle started. The helium flow rate through the furnace was maintained at 7.5 scfh throughout the debinding cycle.

Following debinding, the pellets were removed from the furnace into a nitrogen atmosphere glovebox where they were weighed and prepared for movement to the sintering glovebox. During the weighing, the pellets were observed to gain weight and to heat up. This phenomenon had been observed earlier with debound mixed oxide pellets exposed to air. The glovebox atmosphere was determined to contain traces of oxygen which are believed to be the cause of the exothermic reaction.⁽³⁾

SINTERING

The debound pellets were sintered as one batch at 1550 °C for 4 hr in a Richard D. Brew and Company, High Temperature, High Vacuum Furnace, Model 426 Modified, using the firing cycle shown in Figure 4. A 200 °C/hr rate of rise and a 300 °C/hr cooling rate were used.

The pellets were distributed in six molybdenum trays and loaded into the furnace (Figure 5). The furnace was evacuated to 1.4×10^{-4} Torr twice and backfilled with argon-8% hydrogen gas after each evacuation. An Ar-8% H₂ gas flow rate through the furnace was maintained at 8 scfh during the sintering cycle.

FIGURE 3. Binder Removal Firing Cycle

FIGURE 4. Sintering Cycle

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FIGURE 5. Sintering Furnace, Showing Pellet Load Arrangement

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The Ar-8% H₂ supplied to the furnace was passed through a United States Dynamics automatic, catalytic dryer and purifier before entering the furnace. This reduced both the water content and the oxygen content of the mixture to less than 1 ppm by volume. The sintered batch of pellets was designated PL-10-S11.

CENTERLESS GRINDING

One hundred and two (17 from each sintering tray) sintered pellets were centerless ground on a miniature, dry wheel, centerless grinder.⁽⁴⁾ Approximately 1.5 to 2 mills were removed from each pellet diameter. The effects of this grinding on the dimensions of the pellets and on the density as determined by the geometrical method is discussed under Characterization of Sintered Pellets.

OXYGEN-TO-METAL RATIO ADJUSTMENT

All of the sintering tests made with PL-2 material produced pellets having an oxygen-to-metal ratio between 1.93 and 1.95. However, all of those tests had been made with furnace load masses of between 50 and 150 g of mixed oxide pellets and 2 kg of previously sintered, depleted UO₂ pellets. PL-10 was the first large mass of mixed oxide, green pellets to be sintered in the new facility, and the relationships among O/M, load mass, and gas flow had not been established for the new furnace. Thus, the average O/M of the PL-10-S11 pellets was 1.99. Since a ratio between 1.95 and 1.97 was requested, a series of firings using Ar-8% H₂ gas was conducted in the sintering furnace to reduce the O/M of 200 PL-10-S11 pellets to 1.96. The results of these firings are given in Table 8. The same firing procedure as used to sinter the pellets was used in the O/M adjustment firings. PL-10-S12 and PL-10-S13 were the same 200 pellets. PL-10-S14 was a second 200 pellet batch.

TABLE 8. Oxygen-to-Metal Ratio Adjustment

<u>Batch Number</u>	<u>Load Mass, g</u>	<u>Gas Flow, scfh</u>	<u>Temperature, °C</u>	<u>Time at Temp, hr</u>	<u>Average O/M</u>
PL-10-S11	4000	8	1500	4	1.990
PL-10-S12	260	8	1200	3	1.989
PL-10-S13	254	8	1500	3	1.972
PL-10-S14	260	8	1650	3	1.962

CHARACTERIZATION OF SINTERED PELLETS

A summary of the tests conducted on samples of the sintered pellets is contained in Table 1, and the results of these tests are discussed below.

DIMENSIONS AND DENSITY

The results of the dimensional analyses made of samples taken from the sintered pellets are given in Table 9. Statistical distribution of the dimensions and densities are shown in Figures 6 through 11. The dimension measurements were made with a flat anvil micrometer reading in 0.0001 in. increments or with a blade micrometer reading in 0.001 in. increments. The pellets were weighed on a Mettler Model P120 balance to 0.001 g. The diameter of each pellet was measured at least in three places; once at each end and once at the center of the pellet length. The diameters of twenty pellets were measured in six places; twice at each end and twice at the center of the pellet length. Each diameter measurement on a pellet was made after a 90° rotation of the pellet from the position of the previous measurement. The lengths of fifty of the pellets were measured twice. The lengths of all other pellet samples were measured once. No difference greater than several tenths of a mill was observed when two measurements were made of a pellet diameter at the same axial position with a 90° rotation between measurements or when two measurements were made of a pellet length.

TABLE 9. Sintered Pellet Dimensional Characteristics and Density

Dimension	Number of Samples	Average	Minimum	Maximum	Estimated Standard Deviation	Upper Tolerance Limit	Lower Tolerance Limit
Diameter as Sintered, in.	100	0.1987	0.1970	0.2015	0.00065	0.2009	0.1965
Diameter Ground, in.	30	0.1970	0.1968	0.1971	0.00008	0.1973	0.1968
Length, in.	130	0.2514	0.2452	0.2604	0.00320	0.2626	0.2411
Taper, in.	100	0.0012	0.0001	0.0033	0.00053	0.0030	0.0006
Hourglass, in.	100	-0.0004	0.0000	-0.0010			
Barrel, in.	100	+0.0004	0.0000	+0.0011			
% TD as Sintered	50	91.35	89.40	92.88	0.66367	93.58	89.13
% TD Ground	30	91.39	89.70	92.39	0.59183	93.36	89.40

95% confident that the tolerance intervals cover 99% of the population.

FIGURE 6. Statistical Distribution of As-Sintered Pellet Diameters

FIGURE 7. Statistical Distribution of Pellet Lengths

FIGURE 8. Statistical Distribution of As-Sintered Pellet Densities

FIGURE 9. Statistical Distribution of Ground Pellet Densities

FIGURE 10. Statistical Distribution of Pellet Tapers

FIGURE 11. Statistical Distribution of Pellet Barrel and Hourglass Effects

Taper

Pellet taper was determined by using the following formula:

$$\text{Taper} = \bar{d}_1 - \bar{d}_2$$

where $\bar{d}_1$ is the average of all diameter measurements made at one end of a pellet and $\bar{d}_2$ is the average of all diameter measurements made at the other end. All pellets sampled had some taper.

Hourglass Effect

The barrel or hourglass effect was determined by using the following formula:

$$\text{Effect} = \bar{d}_3 - \frac{\bar{d}_1 + \bar{d}_2}{2}$$

where $\bar{d}_3$ is the average of all diameter measurements made at the center of the pellet length. Ten percent of the sample pellets showed no effect. Forty-eight percent were hourglass-shaped, and 42% were barrel-shaped. Thus, the pellets exhibited no tendency to assume either shape as both were equally present.

Special Dimensional Characteristics

Three pellets were used in the development of a method of evaluating perpendicularity, flatness, surface texture, chamfer, and end parallelism. The results reported in Table 10 are not satisfactory because the process was not perfected. The measurements were taken from 30X enlargements of polished pellet sections ground approximately to the midplane of the pellets. Perpendicularity is reported as the maximum angle between the pellet side and end. Flatness is reported as the maximum deviation from a flat pellet end. Surface texture is reported as the maximum depth of any defects observed in the polished section. End parallelism is reported as the maximum difference in length between the two sides of a pellet shown in the enlargements. Taper is reported as the maximum difference

TABLE 10. Sintered Pellet Special Dimensional Characteristics

Pellet Number	Perpendicularity	Flatness, in.	Surface Texture, in.	End Parallelism, in.	Taper, in.	Hourglass, in.
1	90° 05'	-0.0012	0.0017	0.0003	0.0006	0.0012
2	90° 10'	-0.0014	0.0017	0.0006	None	0.0026
3	90° 45'	-0.0003	0.0023	0.0026	0.0017	0.0005

Perpendicularity - Maximum angle between pellet side and end

Flatness - Maximum deviation from a flat end in inches

Surface Texture - Maximum depth of defect in inches

End Parallelism - Maximum difference in length of pellet in inches

Taper - Maximum difference in width of pellet in inches

Hourglass - Maximum deviation from straight sides

All measurements above are taken from 30X enlargements of polished pellet sections

in diameters of the pellets as measured at each end of the pellet. Hourglass is reported as the total maximum deviation from straight sides as measured on both sides of the pellet shown in the enlargements. No chamfer was observed in the enlargements.

Density

The percent theoretical density of each pellet was calculated by using the following formula:

$$\% \text{ TD} = \frac{0.70 (\text{wt})}{(\bar{d})^2 (\bar{l})}$$

where the constant 0.70 contains the theoretical density of 11.09 g/cm³, $\bar{d}$ is the average of all diameter measurements made on the pellet, and $\bar{l}$ is the average of all length measurements made on the pellet. Approximately 53% of the as-sintered pellets were within the density of 91 to 92% theoretical requested for the Analytical Chemistry Program. Approximately 70% were within the 93 ± 2% theoretical density range established by the process specification.

Centerless Grinding

Centerless grinding the sides of one hundred of the sintered pellets reduced the range of measured diameters to 0.0003 in. and eliminated all measurable taper, hourglass, and barrel. The calculated mean percent theoretical density for the ground pellets was 0.04% less than that for the as-sintered pellets, but the estimated standard deviation of percent theoretical density for the ground pellets was 0.07184 less than that for the as-sintered pellets, and the tolerance limits were closed by 0.49. Thus, grinding the sides of the pellets does not greatly alter the mean value of density as determined by geometric methods, but it does reduce the variance. This indicates that the assumption that the pellets are cylindrical results in a true average density but leads to a high range of values and a resulting estimated standard deviation for the overall population.

Position in Furnace

An investigation was conducted to see if tray position within the furnace had an effect upon the dimensions and density of the pellets. Sixteen to eighteen pellets were sampled from each tray for length and diameter measurements. The densities of 7 to 9 pellets sampled from each tray were calculated. The results of this investigation are shown in Figures 12 to 14. The trays were numbered from the top of the furnace to the bottom, starting with Tray 1 at the top (Figure 5). The pellet dimensions and density appear to be a function of the trays the pellets were in. The pellets in the tray at the top of the furnace have lengths 0.0023 in. and diameters 0.0005 in. greater than the average batch length and diameter. Also, the pellets in the top tray have densities 0.33% of theoretical less than the average batch density. However, a direct relationship between these variations and position within the furnace cannot be established. As Figure 15 shows, there was a variation in

FIGURE 12. Average Pellet Diameter for Each Tray

FIGURE 13. Average Pellet Length for Each Tray

FIGURE 14. Average Percent Theoretical Density for Each Tray

FIGURE 15. Average Pellet Weight for Each Tray

the size of the pellets originally put into the trays. This is indicated by their average weight, which corresponds to the noted variations in dimensions of the sintered pellets, particularly the pellet length. Thus, the variations noted may be more a result of what was put into the trays than where the trays were placed in the furnace.

The suggestion has been made that knowledge of either the length or weight of ground pellets may be sufficient to determine the correct pellet density since such a close relationship exists between pellet length and weight, and since the ground diameters are so consistent.

CHEMICAL COMPOSITION

Table 11 contains the results of analyses for impurity content of the as-sintered pellets, and Table 12 contains other chemical analyses results. These results were obtained following the methods outlined for analytical chemistry in support of FFTF driver fuel fabrication,⁽⁵⁾ and are compared with the requirements of the FFTF manufacturing process specifications.⁽²⁾

Impurity Levels

All the impurity levels are well below the maximum limits established by the specifications. A comparison of the as-sintered impurity levels with the impurity levels of the blended powder shows that the binder removal and sintering processes reduced the calcium content by 1000 ppm and the carbon content by 400 ppm. The aluminum, chloride, fluoride, and lead contents were also decreased. No significant impurity level increases during pellet fabrication were detected. The water and gas content of the as-sintered pellets are also well within the process specification limits.

TABLE 11. Impurity Levels in Sintered Pellets

Impurity	1st Results		2nd Results		Process Specification
	Number of Samples	ppm by Weight	Number of Samples	ppm by Weight	Limits ⁽²⁾
Ag	3	<.1	4	<.5	**
Al	3	10	4	=	500
As	3	=	4	=	
B	3	=	4	=	20
Ba	3	=	4	=	
Be	3	=	4	=	
C			4	10 - 45	150
Ca	3	100	4	2+	250
Cd	3	=	4	=	20
Cl			5	<5	25
Cr	3	<25	4	=	250
Cu	3	1 - 2	4	2+	*
F			5	<5	25
Fe	3	20	4	<50	500
K	3	=	4	=	
Li	3	=	4	=	
Mg	3	<5	4	10+	25
Mn	3	1	4	<5 - 10+	**
Mo	3	=	4	=	**
N	3	3 - 4			200
Na	3	<2 - 5	4	<10	500
Ni	3	<10	4	=	500
P	3	=	4	=	100
Pb	3	1	4	<10	**
Sb	3	=	4	=	
Si	3	5	4	<10	*
Sn	3	<5	4	=	**
Ti			4	=	*
Tl	3	=	4	=	
V	3	<20	4	=	500
Zn	3	<50	4	=	*

* The sum of Cu, Zn, Si, and Ti $\leq$ 800

** The sum of Ag, Mn, Mo, Pb, and Sn $\leq$ 200

= Not Detected

TABLE 12. Chemical Composition of Sintered Pellets

	Number of Samples	PL-10 Pellets	Process Specification Limit ⁽²⁾
H ₂ O (200 °C)	3	<5 ppm	30 ppm
H ₂ O (300 °C)	3	9 - 12 ppm	
Gas Content	5	0.02 - 0.04 cm ³ /g	0.05 cm ³ (STP)/g
% Pu	5	22.00 - 22.14	22.10***
% U	5	66.03 - 66.38	66.05***
O/M as Sintered*	15	1.987 - 1.997	1.97 ± 0.02
O/M Adjusted*	5	1.960 - 1.965	
O/M Adjusted**	3	1.969 - 1.975	

* Determined using a whole pellet by a modified thermogravimetry method⁽⁶⁾

** Determined using 100 mg samples from each pellet by thermogravimetry

*** Not specified in Reference (2). Customer requested
25 wt% PuO₂ - 75 wt% UO₂

Plutonium and Uranium Content

The percent plutonium and percent uranium are not specified in the process specifications. The calculated nominal weight percent of each metal is 22.10% Pu and 66.05% U for a 25 wt% PuO₂-75 wt% UO₂ mixture, using an atomic weight of 239.00 for plutonium and 238.04 for uranium. The deviation from the nominal plutonium content of the sample pellets was no more than ± 0.10 wt%. The deviation from the nominal uranium content of the sample pellets was no more than ± 0.33 wt%. This is less than 0.5% deviation from the nominal values of each metal.

Oxygen-to-Metal Ratio

The oxygen-to-metal ratio results have been discussed under the heading Oxygen-to-Metal Ratio Adjustment. The effect of position in the furnace on the oxygen-to-metal ratio of the as-sintered pellets was considered, and the results are shown in Figure 16. These results are based on the average O/M of 2 or 3 pellets from each tray. Similar results were obtained for the pellets from the O/M adjustment sintering cycles. The increase in O/M of the pellets from the bottom to the top of the furnace is related to the gas flow through the furnace which affects the temperature profile and atmosphere profile within the furnace. The effect of position within tray (center versus edge) on the O/M was also considered. No effect was detected, but the sampling was small.

HOMOGENEITY

Evaluation of the homogeneity and dispersion of plutonium and uranium in the pellets is based on the results of ceramography, alpha-autoradiography, microprobe scans, and X-ray diffraction patterns.

FIGURE 16. Average Pellet O/M for Each Tray

Microstructure

The photomicrographs, Figures 17 and 18, show an undeveloped grain structure with grains in the range of 3 to 5 μ across being most common. This lack of grain growth is attributed to the low pressing pressure and low sintering temperature used to attain the desired density. Porosity is uniformly distributed and closed. No major pressing defects are observed although some laminar microcracks are observed in longitudinal section microphotographs (Figure 17). Voids with a continuous shell, ranging in size from 10 to 20 μ across (Figure 17), appear frequently in the microstructure. Although their origin is not known, the results of other studies indicate that they do not represent another phase.

Neg 49561

Etched - 1000X

FIGURE 17. Longitudinal Section of Pellet

Neg 4957J

Etched - 1000X

FIGURE 18. Transverse Section of Pellet

Alpha-Autoradiographs

Figures 19 and 20 are photographs developed directly from alpha-autoradiographs made from the polished sections prepared for ceramography. Thus, the plutonium concentrations are shown as white spots on the photographs. No standards for evaluating alpha-autoradiographs have been established, but comparison of these alpha-autoradiographs to similar prints from other fuel lots fabricated at BNW show equal or better dispersion.

Microprobe Scans

Microprobe scans using a $1\text{ }\mu$ wide beam were made along a radius of the transverse polished sections of three pellets and along the central axis of the longitudinal polished sections of three pellets. A total of 0.459 in. was covered by the scans and in the areas covered, no areas of 100% PuO_2 were found. The largest concentration detected was 97% PuO_2 in an area $21\text{ }\mu$ across. Twelve areas were found that contained more than 25 wt% PuO_2 and that were wider than $50\text{ }\mu$ --the maximum limit set by the process specifications. The width of these areas measured at the 25% PuO_2 level and the maximum PuO_2 concentration within each area are reported in Table 13. A plot of the number of areas of PuO_2 concentration greater than 25% as a function of the distance along the radius scans from the edge to the center of the pellet section is shown in Figure 21. This plot indicates that the pellets are more homogeneous in the center than near the surfaces. This condition is also observed in the alpha-autoradiographs.

X-Ray Diffraction

X-ray diffraction results from using counter-diffractometer and powder mounts indicate an undeveloped structure having a lattice parameter of $5.453\text{ }\text{\AA}$. No evidence of the existence of more than one phase was detected.

Neg 4957A

FIGURE 19. Alpha-Autoradiograph of Fuel Pellet

Neg 4956A

FIGURE 20. Alpha-Autoradiograph of Fuel Pellet

TABLE 13. Concentration of PuO_2 in Areas Wider than $50\ \mu$

<u>Width, μ</u>	<u>% PuO_2</u>
50	45
53	45
56	46
57	38
57	39
58	43
62	41
72	40
73	38
79	62
82	36

FIGURE 21. Results of Radius Microprobe Scans Across Three Transverse Pellet Sections

SURFACE CHARACTERISTICS

The sintered pellets had a uniform color and texture as shown in Figure 22. No surface cracks could be observed when the as-sintered pellets were examined without the aid of magnification. Grinding of the pellets did not reveal any cracks outside the limits set by the process specification. Some pellets had end chips which resulted from transferring the green pellets from the press to the binder removal boat, and from the boat to the sintering trays after debinding. The necessity for these transfers has now been eliminated from the process.

REFERENCES

1. F. M. Smith, B. R. Hayward, C. F. Metz and J. F. Williams. Analytical Chemistry Program for LMFBR/FFTF Fuels, BNWL-SA-2748. Battelle-Northwest, Richland, Washington, December 4, 1969.
2. C. A. Strand. Unpublished Data, February 1969.
3. D. W. Brite and R. J. Anicetti. Uranium Dioxide Fabrication, HW-64684, p. 28, Available from Clearing-house for Federal Scientific and Technical Information, Springfield, Virginia, December 5, 1960.
4. J. L. DeMeyer. Dry Wheel Centerless Grinder for Development Laboratory Use, BNWL-1052. Battelle-Northwest, Richland, Washington, April 1969.
5. W. L. Delvin. Fast Flux Test Facility, Analytical Chemistry Methods in Support of FFTF Driver Fuel Fabrication, BNWL-1024. Battelle-Northwest, Richland, Washington, October 25, 1968.
6. T. D. Chikalla and C. E. McNeilly. Determination of Oxygen/Metal Ratios for Uranium, Plutonium, and (U, Pu) Mixed Oxides, BNWL-SA-2493. Battelle-Northwest, Richland, Washington, April 8, 1969.

Neg 0691427-1

FIGURE 22. As-Sintered PL-10 Fuel Pellets

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- Page 6 In the first sentence on the page, change 1.95 to 1.96.
- Page 7 Under the column labeled Calcined PuO₂ and opposite the impurity B, delete the extraneous number 1.
- Page 15 In the third sentence of the second paragraph, a period and a parenthesis were omitted. The sentence should read:
- The results . . . batched properly. (The calculated . . . is 22.10 wt%.)
- Page 15 In the third sentence of the third paragraph, change "For this solution . . ." to read "From this solution . . ."