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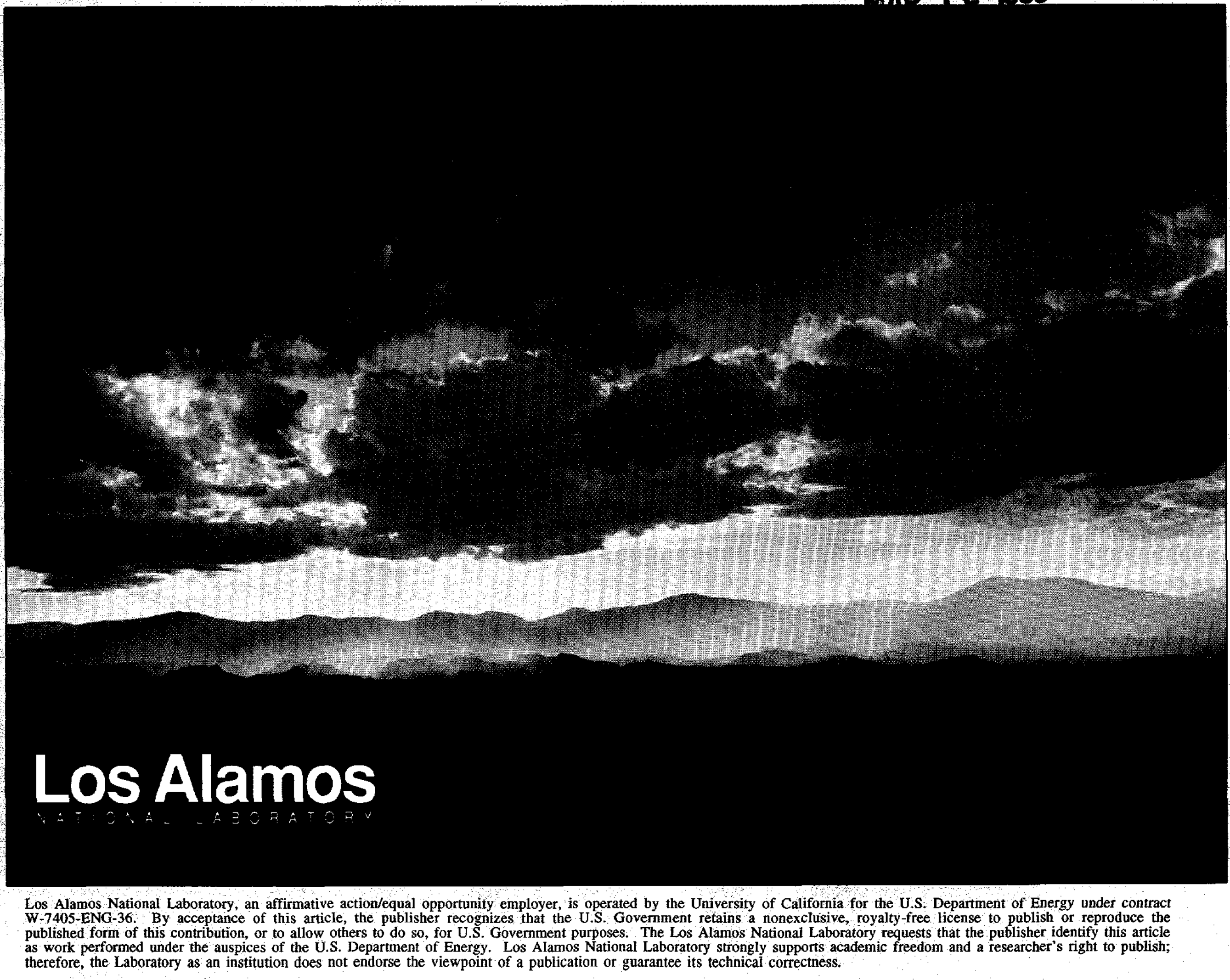
NUCLEAR FUELS TECHNOLOGIES FISCAL YEAR 1998 FUEL FABRICATION PROCESS DEVELOPMENT FEED MATERIALS BASELINE DEVELOPMENT SUMMARY REPORT

by

Stacey L. Eaton, Angelique D. Neuman, Jonathon G. Teague, Stacey A. Talachy, and Kenneth Chidester



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1. INTRODUCTION

This document summarizes a portion of the work performed for the fuel fabrication process development task for the fiscal year 1998 (FY98) research and development (R&D) activities at Los Alamos National Laboratory (LANL). The efforts described here are under the heading of Feed Materials Baseline Development and were performed by the Nuclear Fuels Technologies team at LANL in support of the Fissile Materials Disposition (FMD) Program. It was determined through previous efforts that development work is necessary when new feed materials are introduced into an established fabrication process. The FMD Program decided to select a new UO_2 source in FY98 for use in fuel fabrication R&D activities. The new source identified was UO_2 powder derived from the Ammonium Uranyl Carbonate (AUC) process. Fuel fabrication activities to date have used Cameco UO_2 obtained from Canada. The properties of Cameco UO_2 differ significantly from those of AUC-derived UO_2. Although the AUC-derived UO_2 material was used previously to fabricate the majority of the European reactor-grade mixed oxide (MOX) fuel, it is important to establish how it will interact with weapons-grade plutonium in terms of fuel fabricability. Furthermore, plutonium feed materials can be quite different (from a ceramics perspective) depending on the conversion process and processing parameters, and it is important to quantify the effects these differences may have on the fuel fabrication process. There were two main tasks included in this effort:

1. Develop baseline MOX fuel fabrication processing parameters for the AUC-derived source of UO_2 feed material, using both surrogate CeO_2 and prototypic PuO_2 powders.

2. Fabricate MOX fuel using the baseline fabrication processing parameters, the new source of UO_2 feed material, and an alternative source of PuO_2 feed material.

The experiments performed and results obtained from these Feed Materials Baseline Development activities are described in the following sections.

2. BACKGROUND

LANL acquired a new source of depleted, AUC-derived UO_2 feed material as part of a separate activity in FY98. This material is available commercially through Asea Brown Boveri (ABB) in Sweden, and was converted from UF_6 to UO_2 by the AUC process. The AUC conversion process has been used to supply feed for fabrication of more than 90% of the world's MOX fuel supply. This process produces an oxide with superior ceramic characteristics, including particle size, surface area, and resistance to stoichiometry [oxygen-to-metal (O/M) ratio] shifts under storage conditions. Approximately 125 kg of this feed material was purchased and received at LANL.

In the Test Plan[*] written for this effort, UO_2 development activities were described that would establish a process by which MOX fuel could be successfully fabricated using the AUC-derived material. Samples were to be taken of the powder and characterized for particle size, surface area, O/M ratio, and powder morphology. Sinterability tests were to be performed on UO_2 powder alone and MOX mixtures simultaneously to utilize resources most effectively. Sintered pellets were then to be characterized for such properties as density, microstructure, and shrinkage. Further sinterability tests would be continued until the sintered pellets met a predetermined standard. The immediate goal of these efforts was to obtain sufficient processing information to allow other materials disposition (MD) fabrication activities (test fuel for the Advanced Test Reactor) to begin. Completion of the remainder of the UO_2 development work would then be performed in parallel with the other activities, as schedule and resources permitted.

The second specific task under the Baseline Development effort is the alternative PuO_2 fabrication task. The Test Plan specified that the UO_2 development activities involving MOX powders described above were to be performed using PuO_2 powder created by Lawrence Livermore National Laboratory (LLNL) by the 3-step (hydride-nitride-oxidation) method. For the alternative PuO_2 fabrication task, a different source of PuO_2 powder was to have been identified. This new source was then to be used to fabricate fuel with the AUC-derived material and the processing parameters used in the UO_2 development task. The resulting sintered pellets were to be characterized and directly compared with the results obtained from the 3-step MOX pellets. Possible sources of PuO_2 material for this task were identified in the Test Plan as LLNL 2-step (hydride-oxidation), material created by direct oxidation, or as-yet-unidentified sources resulting from other MD activities.

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[*] S. L. Eaton et al., "Nuclear Fuels Technologies Fiscal Year 1998 Fuel Fabrication Development Feed Materials Baseline Development Plan," letter report from D. Alberstein to J. Johnson (DOE-MD), NMD-98-003, October 31, 1997.

3. UO_2 DEVELOPMENT RESULTS

Approximately 125 kg of depleted, AUC-derived UO_2 powder was received at LANL in early March 1998. The original estimated date of receipt was mid-January, but the schedule slipped as a result of a six-week delay in receiving the cost estimate for the material from the supplier. Once received, the powder was repackaged into containers suitable for use in the glovebox line and transferred into the plutonium facility (PF-4). ABB-Sweden provided a complete characterization of the material, and it was decided that the only additional analyses needed were for particle size and surface area. A comparison of the particle size and surface area results from the ABB data package and LANL analyses is shown in Table I. Results also are shown for a characterization performed at a different point in the fabrication process (after mixing). These results are presented here together to facilitate comparison but will be discussed further in the appropriate section.

Several experiments were then performed to gain understanding of the AUC-derived powder's ability to make MOX fuel according to predetermined specifications. Figure 1 shows a generic process flow diagram for the fabrication steps used in these experiments. A draft test matrix, shown in Table II, was developed for the experiments to describe the full set of parameters to be examined. The test matrix criteria can be adjusted, if needed, as testing proceeds. The full matrix of tests shown in Table II has not yet been completed, but the variables examined and the parameters used for the experiments performed to date are shown in Table III. Each batch utilized 200 g of material, which yielded on the order of 14 pellets (and 14 corresponding data points). However, the additional experiments described in Section 5.0 were on the order of only seven pellets to increase throughput. Detailed descriptions of each experiment and the results obtained are provided below.

The first experiment, labeled 1697-8ST1, was performed to obtain powder flowability and pellet shrinkage information. This first test was performed by pressing the as-received UO_2 powder at various pressing pressures without additives, the additive removal phase, or pre-compaction. The order of the pellets was inadvertently mixed up after sintering of the fuel, so only flowability was determined from this test. In general, it was determined that the powder had good flow properties. The

TABLE I. CHARACTERIZATION RESULTS FOR AUC UO_2 AND MOX POWDERS

Analysis	ABB UO_2	LANL As-Received UO_2	LANL Turbula Mixed UO_2	LANL Turbula Mixed MOX
Particle Size (μ)	Not available	12.5 10.6	15.5 9.7	10.7 15.4
Surface Area (m^2/g)	5.31	5.1402 5.6118 5.2436	1.8265 2.1493	Not available

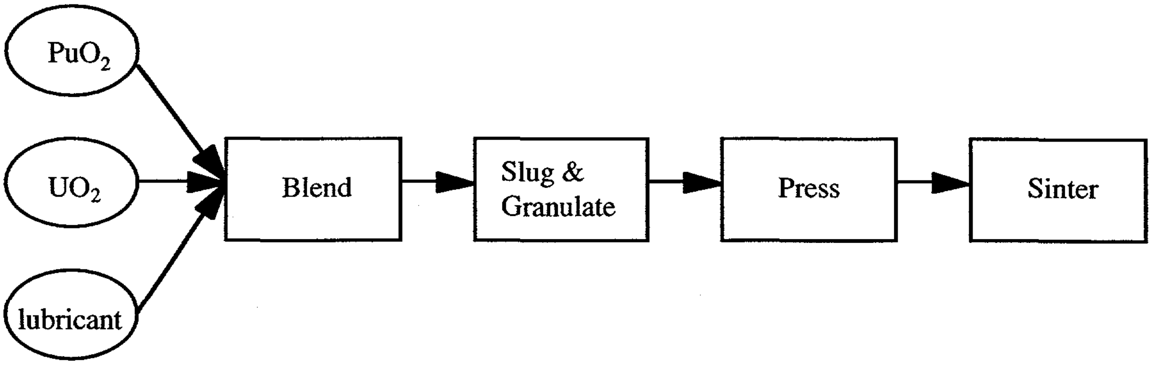

Figure 1. Process diagram for Baseline Development fabrication experiments.

TABLE II. BASELINE TEST PLAN VARIABLES/ANALYSES

Activity	Variable	Range	Variable Action	Number of Analyses per Variable
1.Master blend/mill	load (g)	200	constant	
	atm.	Ar (<100 ppm O_2, H_2O)	constant	
	hard scrap (%)	0	constant	
	time (min)	15, 30, 45, 60	blend at each of 4 times	bulk density (1) surface area (3) flowability (1) particle/granule size (3) pellet density (10)
2.Final blend	load (g)	200	constant	
	atm.	Ar (<100 ppm O_2, H_2O)	constant	
	hard scrap (%)	0, 5, 10	blend at each of 3%	
	time (min)	15, 30, 45, 60	blend at each of 4 times	bulk density (1) surface area (3) flowability (1) particle/granule size (3) pellet density (10)
	lube (w/o) (if no prepress/ granulate)	0, 0.2 zinc stearate	constant	
	pore former	0	constant	
3.Prepressing	die ID (in.)	0.75	constant	
	die ID taper			
	pellet L/D	< 0.5	constant	
	pressure (ksi)	5, 10, 15	prepress at each of 3 pressures	pellet density (3+1 every 50) pellet uniformity (all)
	dwell time (s)	3	constant	
	die lube	0	constant	

TABLE II (cont). BASELINE TEST PLAN VARIABLES/ANALYSES

Activity	Variable	Range	Variable Action	Number of Analyses per Variable
4. Granulator	prepressed pellet density (% TD)	40, 45, 50, 55	granulate at each of 4 pellet densities	granule sizes (1) flowability (1) friability (1)
	lube (w/o)	0.2 zinc stearate	constant	
5. Pressing	die ID (in.)	0.43	constant	
	pellet L/D	1.2–1.5	constant	
	pressure (ksi)	20, 25, 30, 35, 40, 45, 50, 55, 60	press at each pressure	pellet density (all) pellet uniformity (all)
	dwell time (s)	3	constant	
	die lube			
6. Sintering (batch furnace)	load (g)	200	constant	
	ramp up		constant	
	time at temp.	3.5, 4.0, 4.5 h	sinter at three times	
	ramp down		constant	
	temp.	1650,1700,1750°C	constant	
	gas introduced at heat up		constant	
7.Grinding	atm.	Ar (< 100 ppm O_2,H_2O)	constant	
	rate		constant	O/M (3) gas content (3) grain size (3) pellet dimensions pellet density pellet uniformity impurities (3)

Notes: (1) If the resultant curve for a given variable is not well established with the above, typically three points, additional points, either intermediate or end points, need to be evaluated.

(2) Basically all variables, except the one being tested, should be held constant at an optimum value. Fractional factorial testing should reduce the total number of tests required. Blending and pressing may be treated as separate tests to narrow the number of variables.

TABLE III. BASELINE DEVELOPMENT EXPERIMENTAL VARIABLES AND PROCESSING PARAMETERS

Experiment	PuO_2 (g)	UO_2 (g)	Lubricant	Blending	Slug & Granulation	Pressing (ksi)	Sintering
1697-8ST1	none	200	none	none	none	28.8–86.5	1750°C for 7 h
1697-8ST2	none	200	none	none	none	28.8–57.7	1750°C for 7 h
1697-8ST3	none	199.6	0.4 g zinc stearate	Turbula 5 min	none	40.4–69.2	1750°C for 7 h
1697-8ST4	none	190+10	none	Turbula 15 min	none	40.2–54.8	1750°C for 7 h
1697-8ST5	10g 3-step	190	none	Turbula 15 min	none	40.4–57.7	1750°C for 7 h

sintered density was not determined because the identity of the pellets with respect to the pressing variables was lost after sintering.

The second experiment, labeled 1697-8ST2, was performed to obtain the shrinkage data not available from the first experiment. Again the powder was pressed as received, without additives. Figure 2 shows the percent of theoretical density obtained for the green pellets as a function of the pressing pressure. Figure 3 shows the percent of theoretical density obtained for the sintered pellets as a function of pressing pressure.[1] The pellets fabricated in the second experiment showed increasing density with increasing pressure. Several of the pellets (pressed at > ~43 ksi[2]) are within the predetermined specification of 95% (± 1%) of theoretical density. One pellet (pressed at 52 ksi) actually exceeded the specification at 96.8% of theoretical density. The shrinkage values shown in Figure 4 exhibit a nearly linear behavior, decreasing with increasing pressing pressure and ranging from ~19–23%.

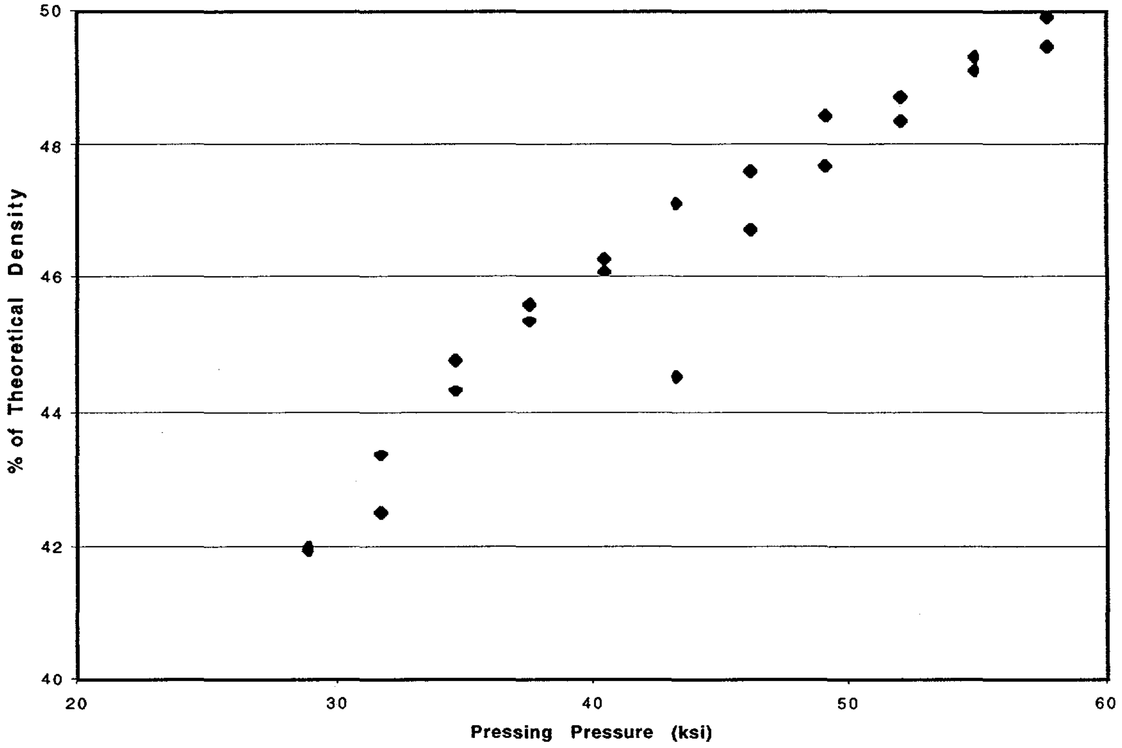

Figure 2. 1697-8ST2 green density as a function of pressure.

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[1] The sintered pellet density results reported in this document were obtained using an immersion method.

[2] ksi = 1000 psi.

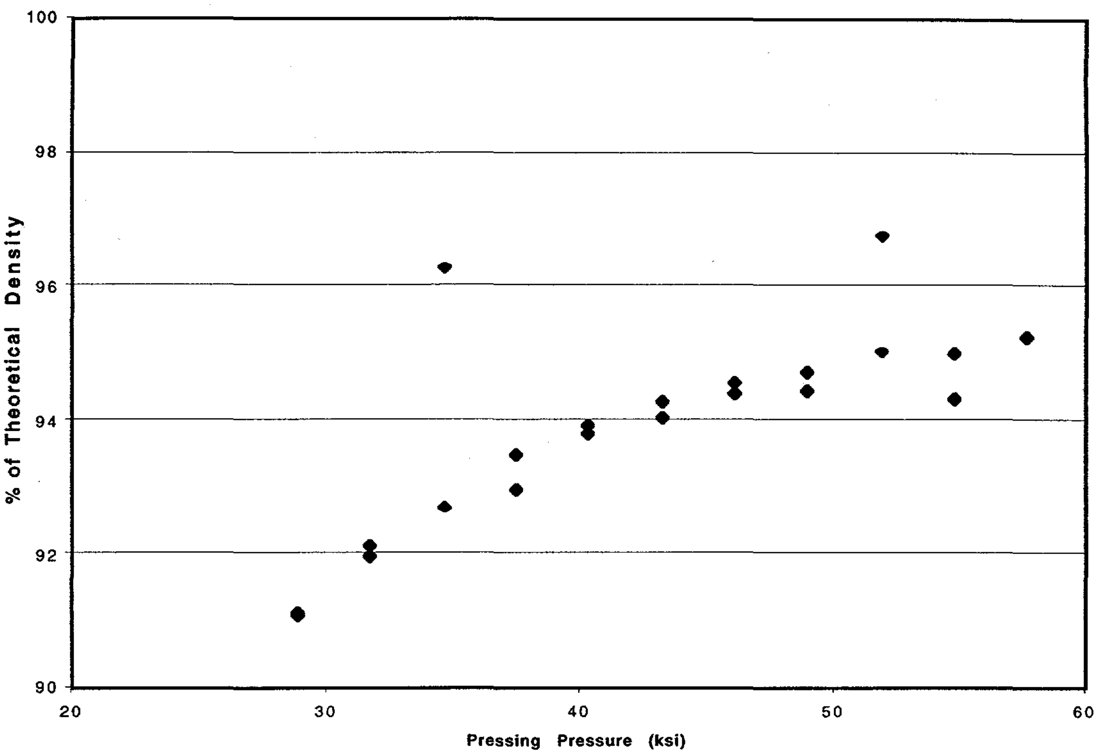

Figure 3. 1697-8ST2 sintered density as a function of pressure.

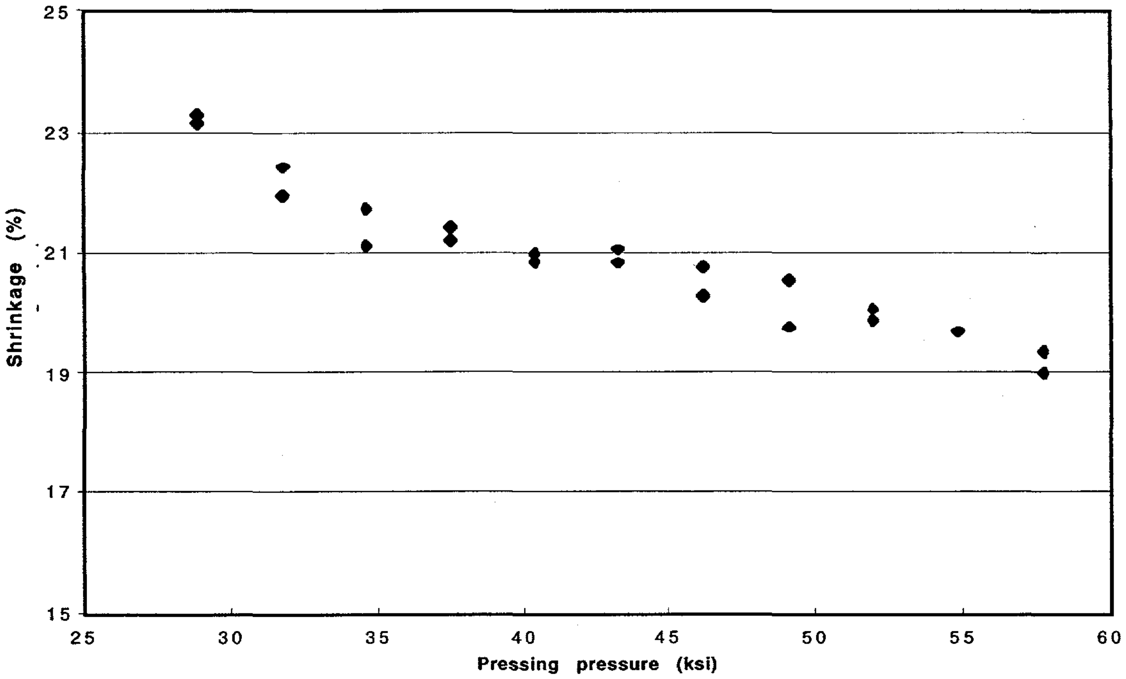

Figure 4. 1697-8ST2 pellet shrinkage as a function of pressure.

The third experiment, labeled 1697-8ST3, was performed to determine the effects of the addition of a lubricant. Zinc stearate (0.2 wt %) was added to the UO_2 powder before pressing. The remaining processing parameters were held constant. Figure 5 shows the percent of theoretical density obtained for the green pellets as a function of the pressing pressure, and Figure 6 shows the sintered pellet results. The pellets had almost linear sintered densities with increasing pressure. The pellets pressed at < ~50 ksi are within the predetermined specification of 95% (±1%) of theoretical density. The shrinkage values shown in Figure 7 also exhibit a nearly linear behavior, decreasing with increasing pressing pressure. The shrinkage of the pellets ranges from ~17–20%, with the data point at 55 ksi being disregarded as an obvious outlier.

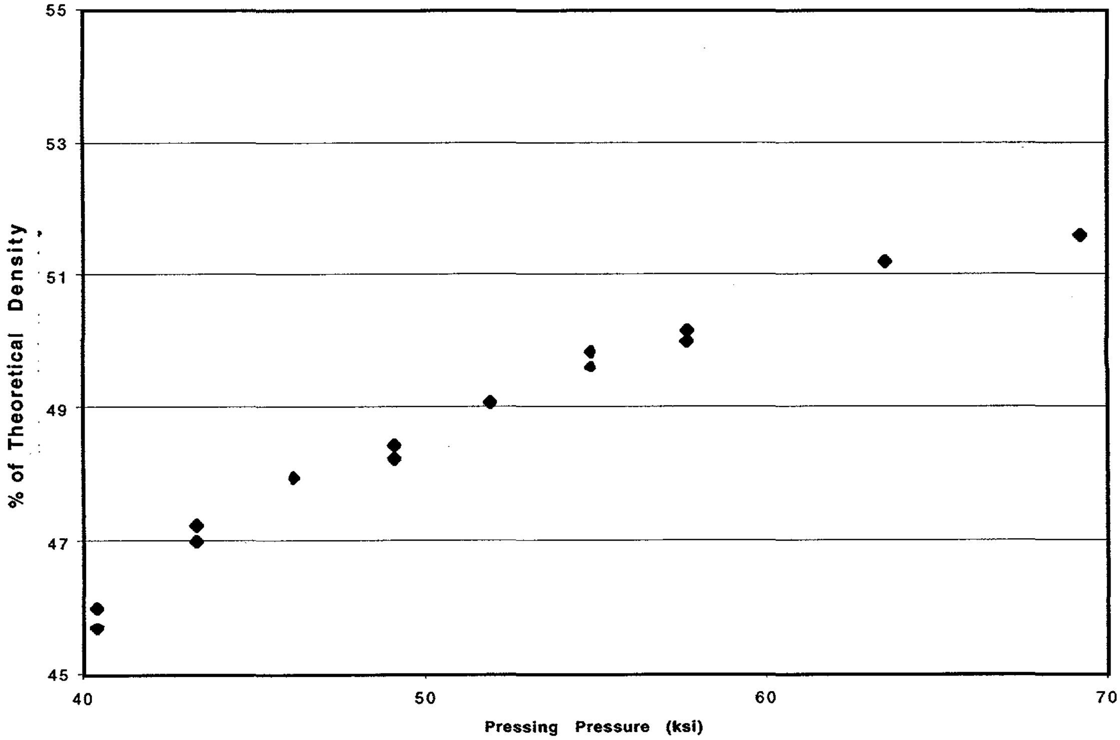

Figure 5. 1697-8ST3 green density as a function of pressure.

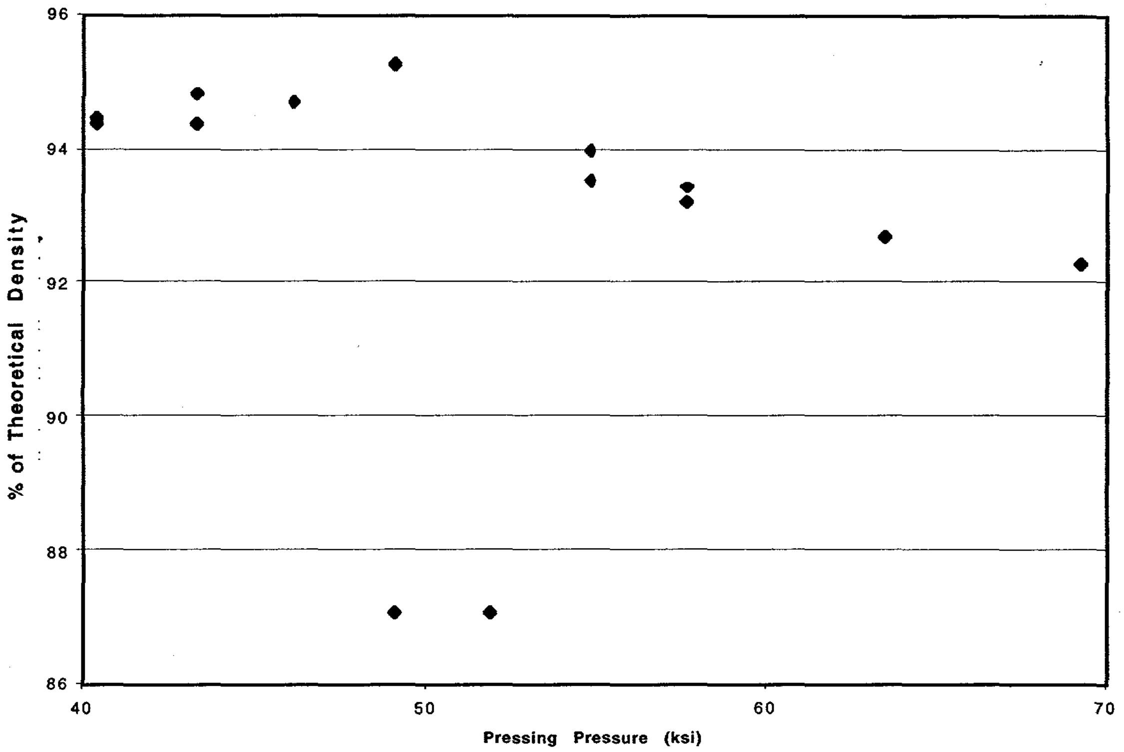

Figure 6. 1697-8ST3 sintered density as a function of pressure.

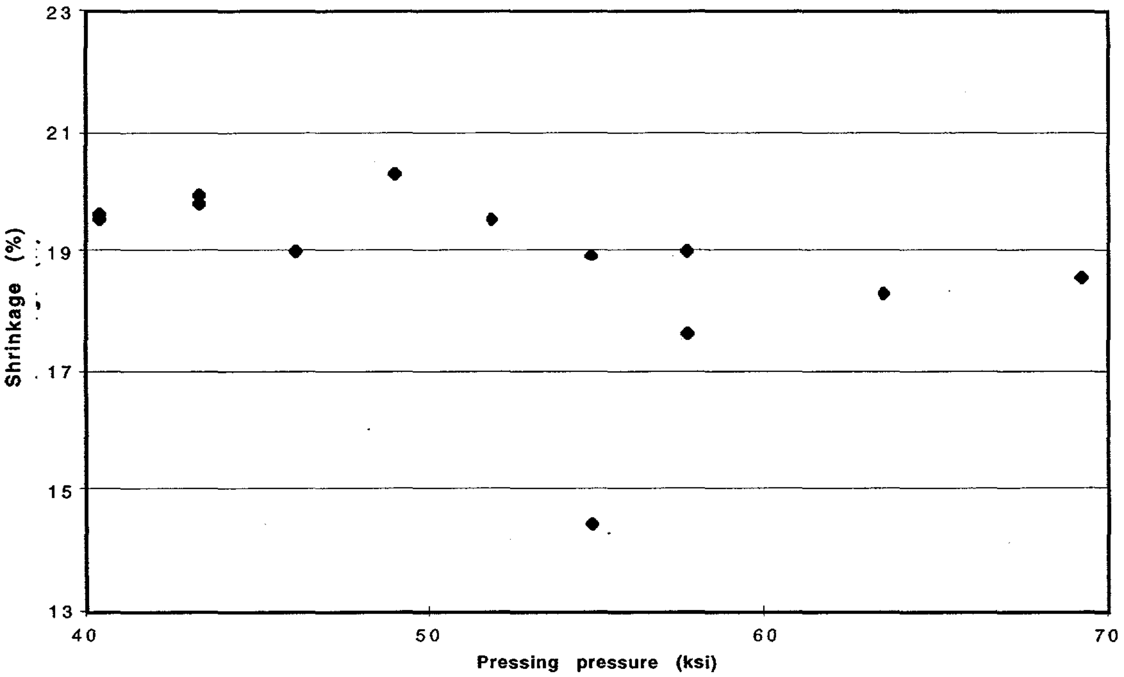

Figure 7. 1697-8ST3 pellet shrinkage as a function of pressure.

The fourth experiment, 1697-8ST4, was performed to determine the effects of making a primary blend with a Turbula® mixer, but using only UO_2. The master blend consisted of 95 wt % UO_2 and 5 wt % UO_2 (as a substitute for PuO_2) to simulate a MOX fuel master blend. The powder was blended with the Turbula® for 15 min and was pressed as blended without any additives. A sample of the Turbula® mixed powder was analyzed for particle size and surface area characterization, and the results are shown in Table I in Section 3. The particle size remains roughly the same after mixing, but the surface area appears to decrease. This decrease is possibly caused by agglomeration during mixing; however, this has not been fully determined yet. The mixed powder showed good flowability properties and pressed without problems. Figure 8 shows the percent of theoretical density obtained for the green pellets as a function of the pressing pressure, and Figure 9 shows the sintered pellet results. The pellets show a slightly increased density with increased pressure. Only the pellets pressed at > ~52 ksi are within the predetermined specification of 95% of theoretical density (± 1%). The shrinkage values shown in Figure 10 exhibit a nearly linear behavior, slightly decreased with increased pressing pressure. The shrinkage of the pellets ranges from ~19–21%. The pellets exhibited a rough, "blistered" diametral surface, and some pellets were "hourglassed." This is probably caused by insufficient die lubrication and could be an indication of a rough die surface. Prepressing and granulation, although not normally required for AUC-derived material, could potentially alleviate these problems and improve the sintered pellet densities.

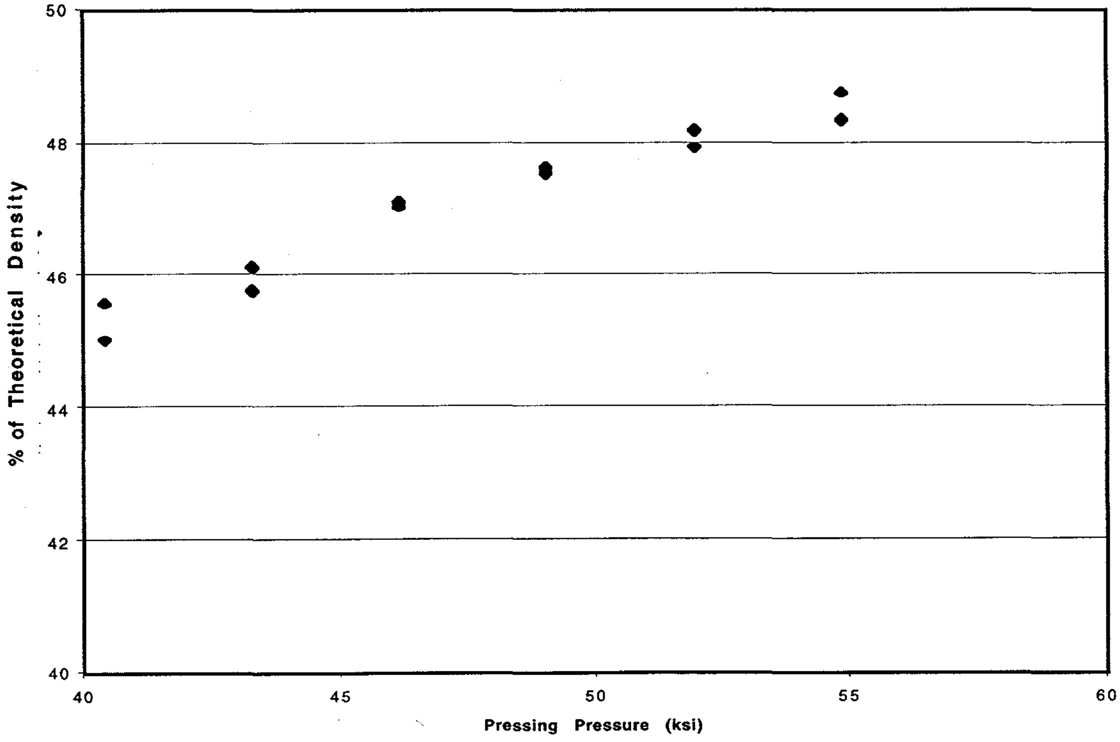

Figure 8. 1697-8ST4 green density as a function of pressure.

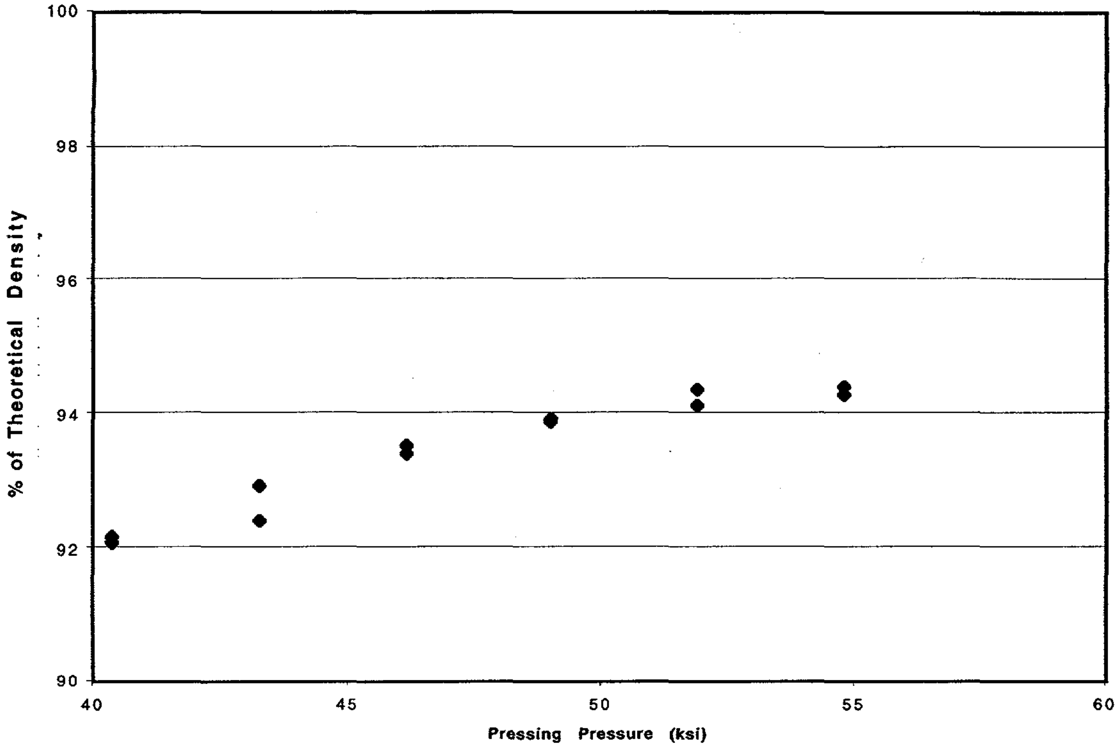

Figure 9. 1697-8ST4 sintered density as a function of pressure.

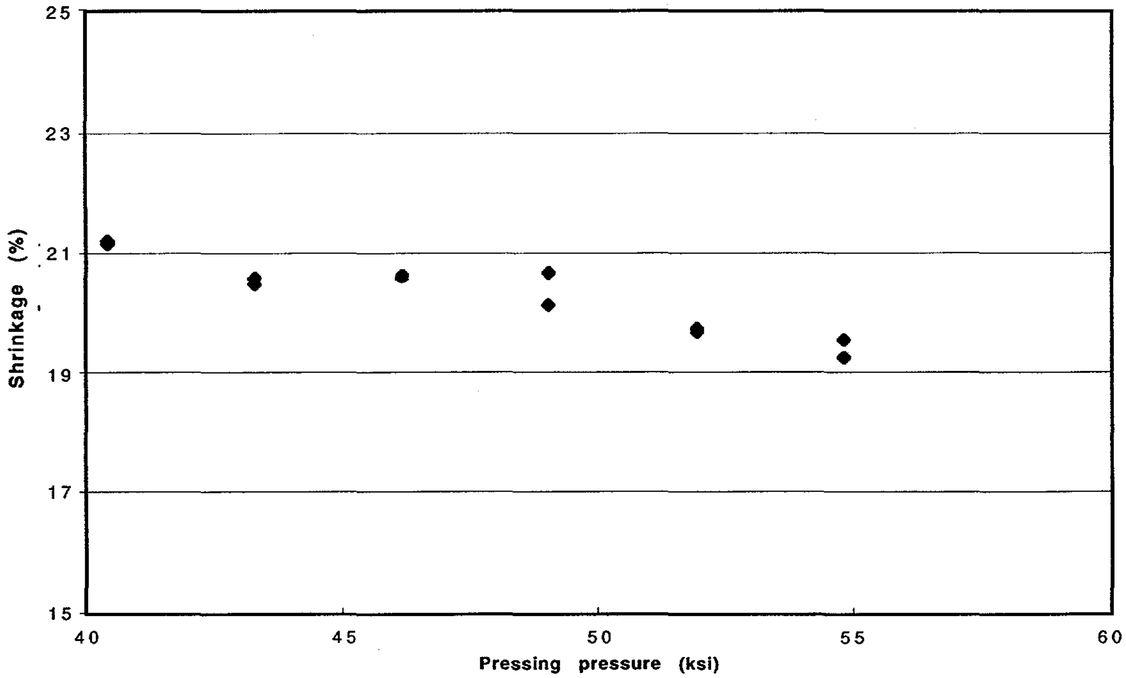

Figure 10. 1697-8ST4 pellet shrinkage as a function of pressure.

The fifth experiment, labeled 1697-8ST5, was the first MOX batch made using the AUC-derived UO_2 and PuO_2 powders. The 3-step PuO_2 powder processed by LLNL was used for this experiment. This experiment was performed exactly as the fourth (1697-8ST4), except that 5 wt % PuO_2 powder was used to create a true MOX blend. Samples of the blended powders were sent for particle size and surface area characterization. Table I shows the particle size characterization results for this MOX blend. The surface area results, however, were unavailable at the time of preparation of this report. The MOX powder showed good flowability properties and pressed without problems, indicating that the addition of the PuO_2 has no detrimental effect on pressing behavior. Figure 11 shows the percent of theoretical density obtained for the green pellets as a function of the pressing pressure, and Figure 12 shows the sintered pellet results. The pellets show an increased density with increased pressure. None of the pellets are within the predetermined specification of 95% of theoretical density (± 1%), but all are > 91% of theoretical density with the point at ~49 ksi being disregarded as an obvious outlier. The shrinkage values shown in Figure 13 exhibit a nearly linear behavior, slightly decreased with increased pressing pressure. The shrinkage of the pellets ranges from ~19–20%. A pellet sample has been submitted for scanning electron microscope (SEM) and autoradiograph analyses, but results were unavailable at the time this report was prepared. Again, the pellets exhibited a rough, "blistered" diametral surface, and some pellets were "hourglassed."

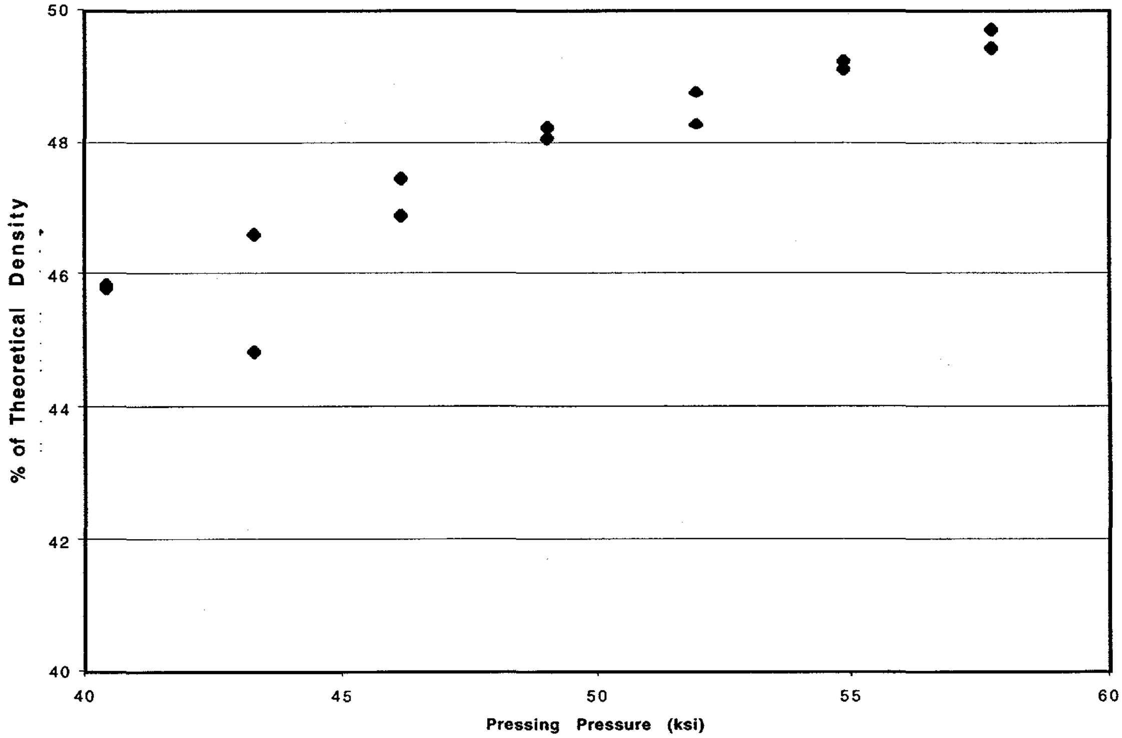

Figure 11. 1697-8ST5 green density as a function of pressure.

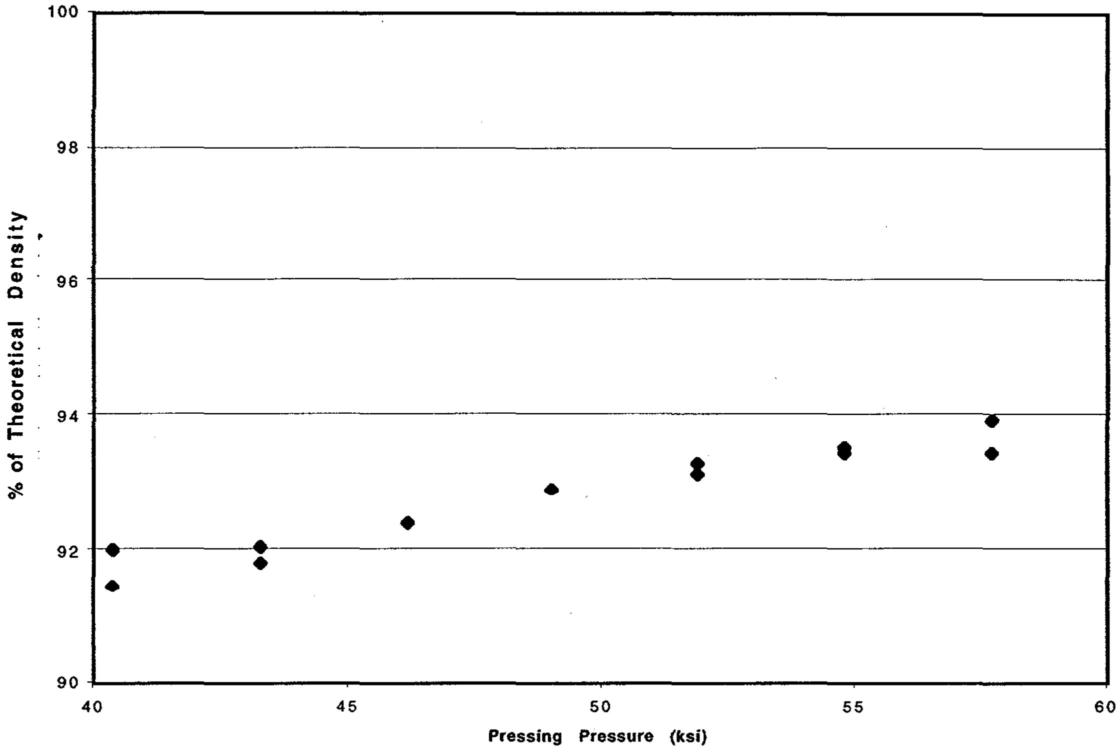

Figure 12. 1697-8ST5 sintered density as a function of pressure.

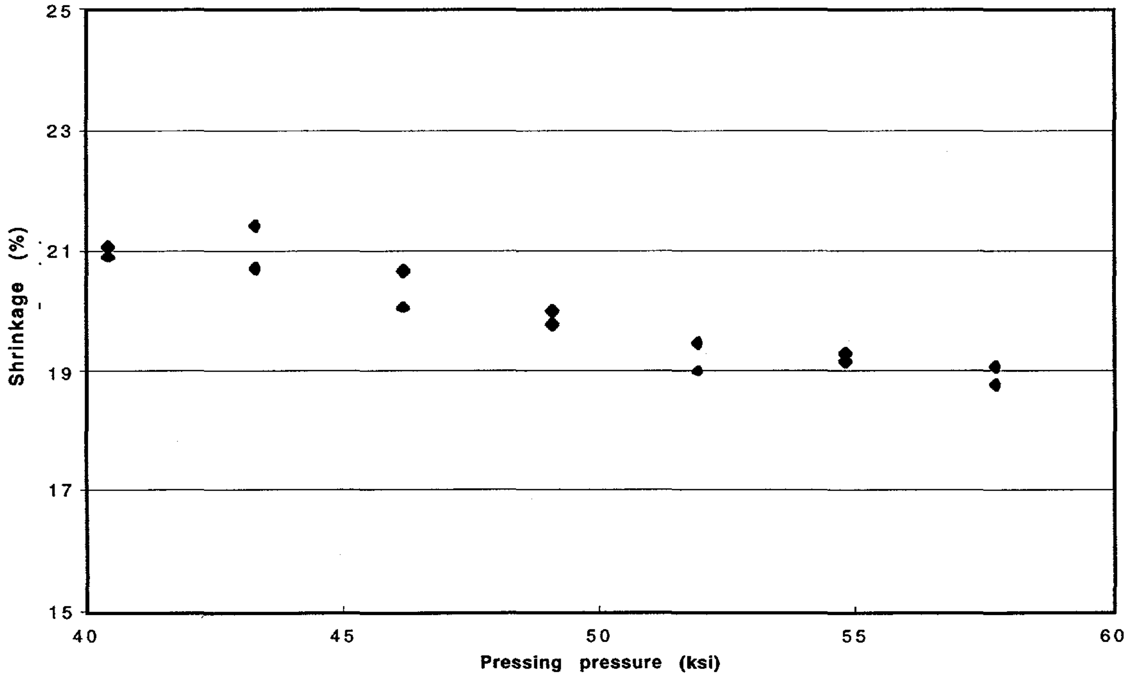

Figure 13. 1697-8ST5 pellet shrinkage as a function of pressure.

4. ALTERNATIVE PUO$_2$ FABRICATION RESULTS

The second specific task under the Baseline Development effort was the alternative PuO$_2$ fabrication task. Two additional sources of PuO$_2$ powder were available for this task in FY98: the 2-step hydride-oxidation material created by LLNL and an aqueously derived source processed at LANL. Although it is not currently anticipated that the 2-step process will be selected as the method for PuO$_2$ conversion, it was hoped that the material would behave like the 3-step material. This would allow its use as an additional source of feed for future experiments because there is little inventory of the 3-step material currently left available. MOX pellets were fabricated using these two sources and the same parameters used for experiment 1697-8ST5 (see Section 3.0). For each batch, the AUC powder was blended with 5 wt % PuO$_2$ (with no additives) in a Turbula® mill for 15 min. Batch number 1697-8ST7 refers to the pellets made with the 2-step material, and batch number 1731-T1 refers to the pellets made with the aqueously derived source.

The intention of this activity was to compare directly these results with those obtained using the 3-step PuO$_2$ (experiment 1697-8ST5). Figure 14 shows the comparison of the green densities for the three batches, whereas Figure 15 shows the comparison of the final sintered densities. None of these sintered densities for the 3-step and 2-step batches met the 95% (±1%) specification. The aqueous batch, however, met the density specification at ~52 and 55 ksi pressing pressures. Figure 16 compares the shrinkage values for the three batches.

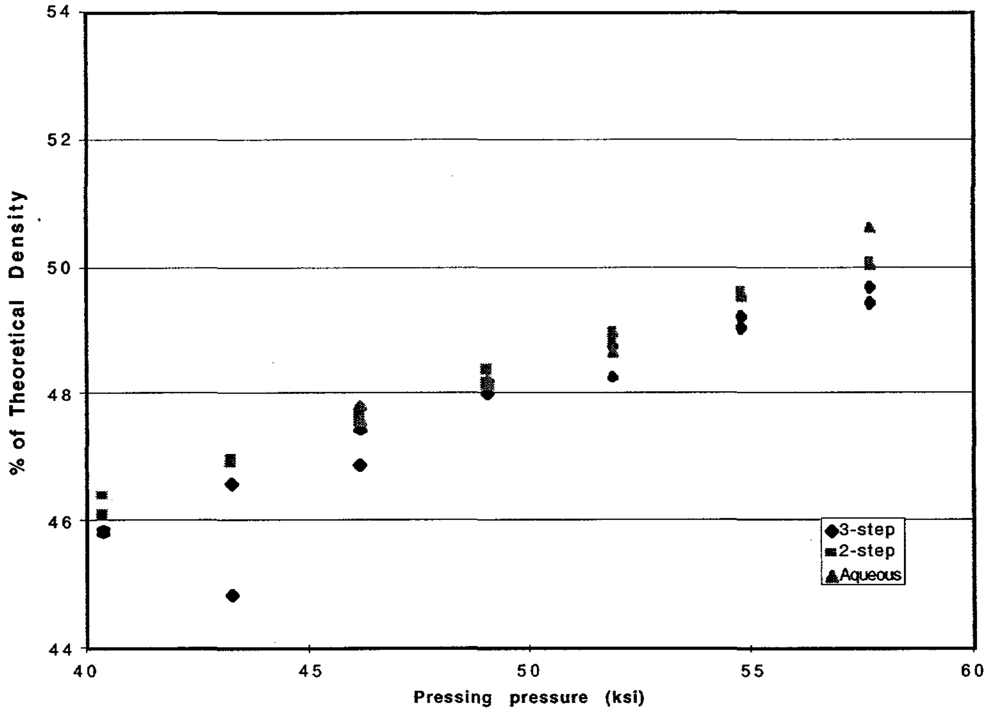

Figure 14. Comparison of green densities as a function of pressure.

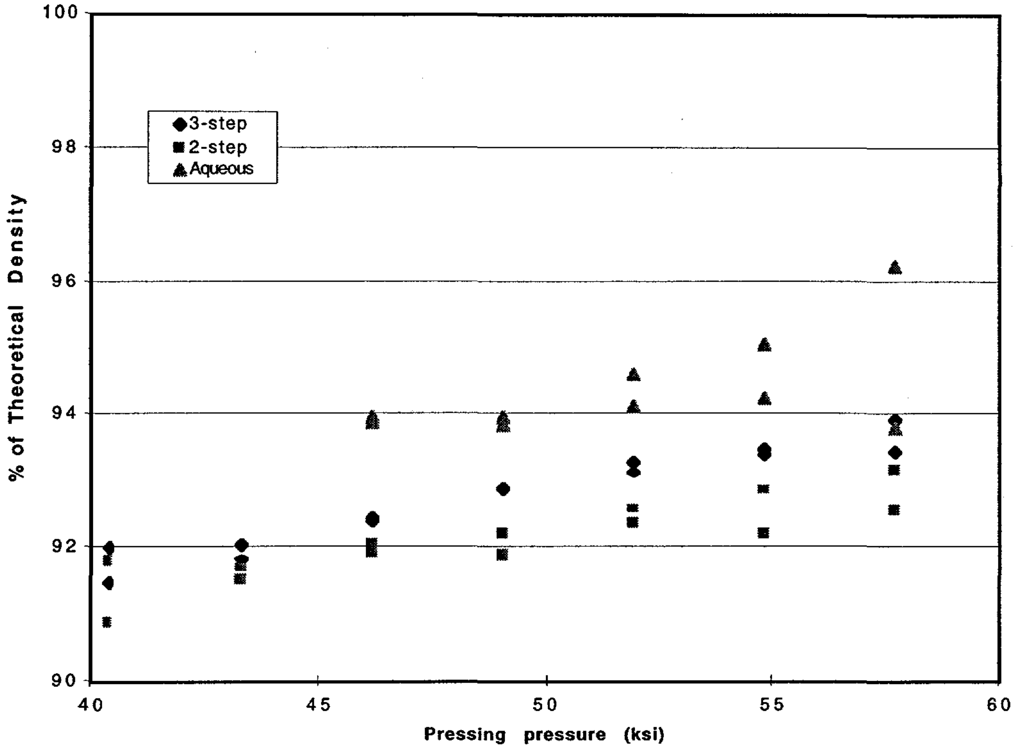

Figure 15. Comparison of sintered densities as a function of pressure.

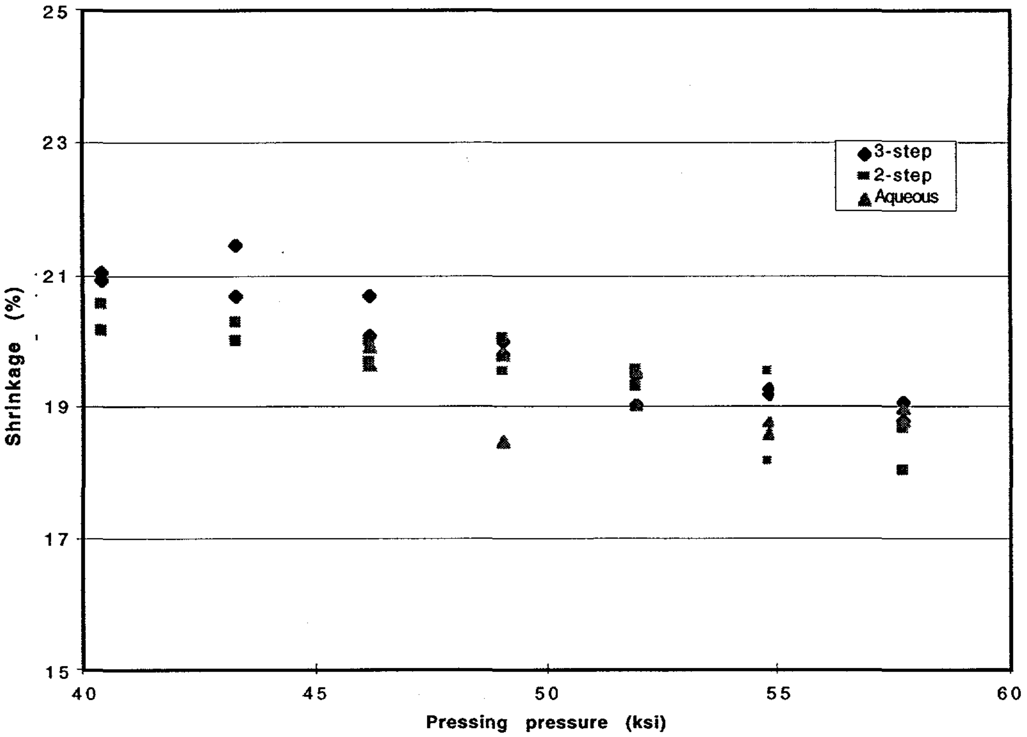

Figure 16. Comparison of shrinkage values as a function of pressure.

Because so few of the pellets actually met the predetermined specification, new batches using the three sources of PuO_2 powder (3-step, 2-step, and aqueously derived) were fabricated in an attempt to increase the sintered density results. The new batches are designated by 1697-8ST9 for the 3-step material, 1697-8ST10 for the 2-step material, and 1731-T3 for the aqueously derived material. A single pressing pressure of 52 ksi was used for all pellets in all batches, as that pressure created higher quality pellets in the initial batches. The main difference in the original and new batches was the addition of zinc stearate to the new batches. Table IV provides a summary of the green and sintered density and shrinkage data from the original batches and compares that summary with the results obtained from the new batches. The new 3-step batch had approximately the same green density as the original 3-step batch, but lower sintered densities and shrinkage values. The same trends were also seen with the 2-step and aqueously derived batches. Overall, the aqueous batches have the highest sintered densities although they are still below the desired specification. The addition of the zinc stearate, therefore, did not have the desired effect of increasing the sintered densities.

5. ADDITIONAL EXPERIMENTS

A few additional experiments not specified in the original test plan were performed in the course of the development activities. Fabrication tests were performed at constant pressures to determine if constant pellet lengths and sintered densities could be achieved (Batch 1697-8ST6). Tests were also initiated to determine the effect that varying the sintering temperature had on the final pellets (Batch 1697-8ST8). Table V shows the parameters used and results obtained for each experiment. Each of the three experiments shown in Table V used a single pressing pressure, and

TABLE IV. COMPARISON OF ORIGINAL AND NEW PUO_2 VARIABILITY STUDY RESULTS

PuO_2 Source	Parameter	Original Value(s)	Original Values at 52 ksi	New Value(s) (at 52 ksi)
3-STEP	Green density (% of TD)	45.9–49.7	48.3, 48.8	48.0–48.8
	Sintered density (% of TD)	91.4–93.9	93.1, 93.3	90.1–91.6
	Shrinkage (%)	18.8–21.4	19.5, 19.0	17.7–18.9
2-STEP	Green density (% of TD)	46.1–50.1	48.9, 49.0	48.6–49.3
	Sintered density (% of TD)	90.9–93.2	92.4, 92.6	90.8–91.5
	Shrinkage (%)	18.0–20.6	19.6, 19.3	17.4–18.8
AQUEOUS	Green density (% of TD)	47.6–50.6	48.7, 49.0	48.7–49.8
	Sintered density (% of TD)	93.8–96.2	94.6, 94.1	91.8–92.8
	Shrinkage (%)	18.5–19.9	19.5, 19.0	17.6–19.3

TABLE V. ADDITIONAL EXPERIMENTAL VARIABLES AND PROCESSING RESULTS

Batch Number	Feed	Blending	Pressing (ksi)	Sintering	%TD	Length (in.)
1697-8ST6	UO_2 + zinc stearate	Turbula 5 min	52	1750°C for 7 h	94.1 ± 0.2	0.404 ± 0.010
1697-8ST8A	UO_2 + zinc stearate	Turbula 5 min	58	1750°C for 7 h	91.9 ± 1.2	0.405 ± 0.011
1697-8ST8B	UO_2 + zinc stearate	Turbula 5 min	58	1600°C for 7 h	92.4 ± 0.2	0.414 ± 0.039

the desired results of constant pellet lengths and sintered densities were obtained. Only Batch 1697-8ST6 met the sintered density specification at 94.1%. Decreasing the sintering temperature (1697-8ST8B) had the effect of increasing the sintered density by half a percent.

6. SUMMARY

The experiments performed to date have completed much of the development work needed to perform other FMD activities, including providing shrinkage data to determine appropriate punch and die sizes for test fuel fabrication. Samples were taken of the as received AUC UO_2 powder, and characterization results were obtained for particle size and surface area analyses. O/M analysis capabilities are currently unavailable at LANL, however, and the morphology data have not been completed. Sinterability tests were performed with both UO_2 and MOX powders, and the sintered pellets were characterized for density and shrinkage. The microstructure results, however, are as yet unavailable. Further sinterability tests were performed in an attempt to increase sintered density, but they have not been successful.

For the PuO_2 variability studies, two different sources of PuO_2 feed material were identified: LLNL 2-step and aqueously derived. Pellets were fabricated, sintered, and characterized for density and shrinkage. The results were directly compared with those obtained in the baseline development efforts. Experiments not specified in the Test Plan were performed in an attempt to increase the sintered densities for the three sources of PuO_2 powder. Additional experiments were also performed to determine if constant densities and lengths could be obtained from constant pressing pressures and to determine the effect that varying the sintering temperature would have on sintered densities.

Overall, these experiments showed that the AUC powder has good flow properties. Pellets were fabricated to almost 94% of theoretical density, which was just outside of the required specification. Future experiments are still planned to develop more fully the ability to make quality MOX fuel using the AUC powder. These experiments will be geared towards obtaining specification densities and examining other variables in the set of parameters described in the draft test matrix. Most of the tests will be performed using only UO_2 powder because the small amount of PuO_2 powder currently available to this program is required for other tasks. Further PuO_2 variability experiments will be performed as new sources of PuO_2 powders become available. Experiments not completed this fiscal year should be completed early next fiscal year to finish the complete characterization of the AUC feed material.