DPST-78-128-11/12

SAVANNAH RIVER LABORATORY BIMONTHLY REPORT

^{238}Pu FUEL FORM PROCESSES

NOVEMBER/DECEMBER 1978

MASTER

DU PONT

SAVANNAH RIVER LABORATORY

AIKEN, SOUTH CAROLINA 29801

PREPARED FOR THE U.S. DEPARTMENT OF ENERGY UNDER CONTRACT AT(07-2)-1

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Approved by:

M. L. Hyder, Research Manager
Actinide Technology Division

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FOREWORD

This report is one of a series to summarize progress in the Savannah River Laboratory ^{238}Pu Fuel Form Program. This program is supported by the DOE Division of Advanced Nuclear Systems and Projects (DANSP).

Goals of the Savannah River Laboratory (SRL) program are to provide technical support for the transfer of DANSP ^{238}Pu fuel form fabrication operations from Mound Laboratory to new facilities at the Savannah River Plant (SRP), to provide the technical basis for ^{238}Pu scrap recovery at SRP, and to assist in sustaining plant operations. The program includes:

Demonstration and adaptation of processes and techniques, developed by the Los Alamos Scientific Laboratory (LASL) and Monsanto Research Corporation (MRC), for production at SRP. Information from the demonstration will provide the technical data for technical standards and operating procedures.

Technical Support to assist plant startup of new processes and to ensure continuation of safe and efficient production of high-quality heat-source fuel.

Technical Assistance after startup to accommodate changes in product and product specifications, to assist user agencies in improving product performance, to assist SRP in making process improvements that increase process efficiency and safety and product reliability, and to adapt plant facilities for new products.

PuFF PARAMETRIC STUDY

Parametric tests of the fabrication of 103-watt $^{238}PuO_2$ spheres in the Plutonium Fuel Fabrication (PuFF) facility were completed. Results of these full-scale fabrication tests indicated that increasing the shard-sintering temperature improves dimensional stability of the spheres and verified that moderate to low hot pressing load and ramp conditions are needed to minimize fracture. The parametric test results along with earlier preproduction experience were used to select centerline process conditions for the start of Multi-Hundred Watt (MHW) sphere production in the PuFF facility.

The sphere tests (Phase II of the PuFF parametric study[1,2]) consisted of a seven-sphere statistical matrix experiment (Table 1) developed to investigate the effects of four hot pressing variables. The variables included shard-sintering temperature, the temperature, load (pressure), and rate of load application of hot pressing. The immediate objectives of the test were to better define centerline conditions and limits on these variables for acceptable sphere production, and to evaluate the change from 100-watt to 103.5-watt spheres (by increase of ^{238}Pu concentration to 83.5%). Earlier parametric tests with 18-g pellets (Phase I)[1-3] identified the most sensitive or key process variables and defined suitable limits for those variables that are not scale-sensitive. Data from the pellet tests and from preproduction spheres (Nos. 11-15)[4] made in early 1978 with modified LASL conditions were used to select centerline conditions for subsequent 100-watt preproduction spheres and process limits used in Technical Standards and Operating Procedures. Subsequent preproduction spheres (Nos. 16-24), however, suffered from poor dimensional control; the yield of integral spheres meeting dimensional specifications was less than 50%. The Phase II parametric tests were performed to optimize hot pressing conditions to improve dimensional control and fracture resistance of the spheres.

Description of Experiment

The hot pressing variables and levels investigated in the sphere tests are given in Table 1. Conditions used for all other process variables were nominal values defined in Table 2. The fabrication tests were designed using a response-surface method developed by the Applied Statistics Group of the Du Pont Engineering Department. Each variable was tested at three levels to evaluate interaction (or synergistic) effects among the variables. The levels were selected to give a wide range of effects, and to correlate with the variable levels used in the pellet parametric tests.

The values of shard-sintering temperature were 1150, 1300, and 1440°C. The two highest values were selected to make the shards harder and more resistant to breakup during compaction and less sinterable than those in earlier preproduction spheres. Harder, less sinterable shards should decrease carbothermic reduction during hot pressing, improve sphere microstructure (retain shard identity and coarse porosity) and reduce the amount of sphere shrinkage during hot pressing and post-hot pressing heat treatment. All of these effects are beneficial in improving dimensional control and integrity of spheres.

The hot pressing load values were 1500, 2500, and 5050 lb (ram force). Preproduction Spheres 13-24 were pressed with the intermediate load of 2500 lb (∿1500 psi pressure). The lowest load value was chosen to determine whether spheres could be successfully pressed with a much lower force to prevent shard crushing during compaction and to reduce residual stresses in as-pressed spheres. Both effects would reduce cracking in spheres during reoxidation and heat treatment. The highest load, 5050 lb, was chosen to be significantly above any foreseeable operating loads, and represents an extreme upper limit. Preproduction Spheres 1-10 were hot pressed with this load.[1]

The values of hot pressing temperature were 1540, 1440, and 1390°C. Previous preproduction spheres were hot pressed at about 1530°C. The two lowest values were chosen to reduce carbothermic reduction during hot pressing, and thus, subsequent cracking in spheres from reoxidation-induced stresses and possible CO gas effects.

The hot pressing ramp, or load application, conditions were: 10 seconds from initial to final load, a linear ramp over 3 minutes, and a step-ramp function from initial to final load. The 10-second ramp tested the effect of instantaneous loading, as applied in pressing Spheres 1-10. The 3-minute linear ramp was used in the pellet parametric tests, and was chosen for the sphere parametric experiment as a possible alternative to the 10-minute ramp used for Spheres 13-24. The 3-minute ramp was expected to produce effects (reduction of shard crushing and improved uniformity of microstructure) comparable to the 10-minute ramp. The step-ramp function involved loading the shards immediately with a force of 1500 lb (probably not enough to break up shards) followed by a linear ramp of ∿250 lb/min up to final load. The objective of this ramp was to partially compact the shards before sintering and then gradually close the die punches during sintering.

TABLE 1

Sphere Parametric Experiment

Sphere Run No.	*Shard Sintering Temp, °C*	*Hot Press Load, lb*	*Hot Press Temp, °C*	*Ramp*	*Sphere Condition*		*Die Closure*	*Dimensions, in.*			
								As Pressed		*Heat Treated*	
					As Pressed	*Heat Treated*		*Pole*	*Eq*	*Pole*	*Eq*
25	1300	2500	1540[a]	Step[b]	Cracked	Integral	Yes	1.472	1.467	1.472	1.473
26	1300	2500	1440	Step[b]	Uncracked	Integral	Yes	1.472	1.483	1.459	1.460
27	1300	2500	1390	10 sec	Uncracked	Fractured	Yes	1.464	1.453	1.461	1.461
28	1440	5050	1440	Step[b]	Cracked	Fractured	Yes	1.469	1.470	-	-
29	1440	5050	1390	3 min[c]	Cracked	Fractured	No	1.488	1.488	-	-
30	1150	1500	1390	Step	Cracked	Fractured	No	1.535	1.453	-	-
31[d]	1440	2500	1540	3 min[c]	Fractured	-	No	-	-	-	-

a. Load stepped to 1500 lb immediately and then ramped at linear rate of ∿250 lb/min to final load.

b. Linear load application rate from initial to final load in 3 min.

c. Actual temperature achieved 1500°C.

d. Hot press malfunctioned. Pressing rate was <10 sec; maximum load unknown.

TABLE 2

Nominal Process Conditions Not Varied in Sphere Parametric Experiment

- Feed Particle Size

  5-μm mode

- Oxygen Exchange

  5 hr at 800°C
  400 cc/min $^{16}O_2$ flow

- Outgas

  1 hr at 1000°C
  40 cc/min $^{16}O_2$ flow

- Ball Mill

  SRL design
  12 hr at 100 rpm

- Cold Press

  58,000 psi reached in increments

- Breakup and Size

  Granule size, <125 μm

- Presinter and Hot Pressing Varied in Experiment (Table 1)

- Heat Treatment

  12 hr at 1440°C
  Max ΔT, ±5°C/min

All of the fabrication tests of Table 1 were completed except Sphere Run No. 31, which was aborted because of a malfunction of the hot press. This last test could not be repeated because of PuFF production commitments.

Results

Results of the sphere parametric tests are summarized in Tables 1 and 3. Comparing these data with preproduction Spheres 11-15[2,4] verifies that moderate hot pressing loads in combination with slow load application rate reduces the extent of cracking and the probability of fracture. Also, use of shards sintered at 1300°C appears to stabilize dimensions without significantly aggravating fracture.

TABLE 3

Description of Parametric Spheres

Sphere No.	*Condition of As-Pressed Sphere*	*Condition of Heat-Treated Sphere*
25	Hairline polar crack	Additional cracks, but remained integral during gaging and storage.
26	Uncracked	Several cracks but remained integral during gaging and storage.
27	Uncracked	Quadrant of one hemisphere broke out during gaging.
28	Hairline crack across belly band	Fractured prior to heat treatment. Sphere dropped ~4 in. during loading into heat-treatment furnace, but remained integral. Furnace malfunction caused sphere to be thermally cycled to unknown temperature above 500°C in $^{16}O_2$. Sphere was removed from furnace during repairs and fractured in storage.
29	Cracked - hairline polar	Fractured after heat treatment prior to gaging.
30	Cracked - hairline polar	Fractured after heat treatment prior to gaging.

Cracking of heat-treated spheres, as shown in Figure 1, appears to be markedly affected by the rate of load application during hot pressing and by final load. Spheres 25 and 26, although pressed with hot pressing load and temperature conditions similar to those of reference Sphere 13,[2,4] contained more cracks than did Sphere 13 and most other preproduction spheres pressed with a slower (10-min) force ramp. Spheres 25 and 26, however, were both still integral after hot pressing and gaging. Spheres hot pressed with ramps of about 10 sec [Spheres 1-10 (Reference 1) and parametric Sphere 27] generally contained more cracks and fractured (cleaved) during heat treatment. As indicated from microstructural analyses of earlier spheres,[1] a load ramp that is too fast tends to crush the shards and eliminate the coarse, intershard pores that are essential to density and fracture control.

Control of sphere density and dimensions by increasing shard sintering temperature was successfully demonstrated in Sphere 25. Close-to-nominal sphere dimensions were obtained with no shrinkage on heat treatment by using shards sintered at about 1300°C instead of 1175°C used for Spheres 13-24. As indicated above, the greater cracking in Sphere 25 compared to Sphere 13 may be attributed to the faster ramp used to press Sphere 25. The lower shrinkage achieved with 1300°C shards in Sphere 25 was apparently offset in Spheres 26 and 27 by low hot pressing temperature (Spheres 26 and 27) and by fast hot pressing ramp (Sphere 27).

Future Work

Compared to previous PuFF-test spheres, Spheres 28-30 provide better definition of the lower limits for hot pressing load and temperature and for the upper limits for load and shard-sintering temperature within which acceptable density and dimensions can be obtained. A statistical analysis of the properties of the parametric spheres and other preproduction spheres will be performed to develop a model of key fabrication variables which, if successful, will allow interpolation or extrapolation of the variable levels given in Table 1 to estimate limits for acceptable sphere production. Microstructural analyses of the parametric spheres are planned. The data will be analyzed with the objective of specifying optimum conditions for fracture and dimensional control.

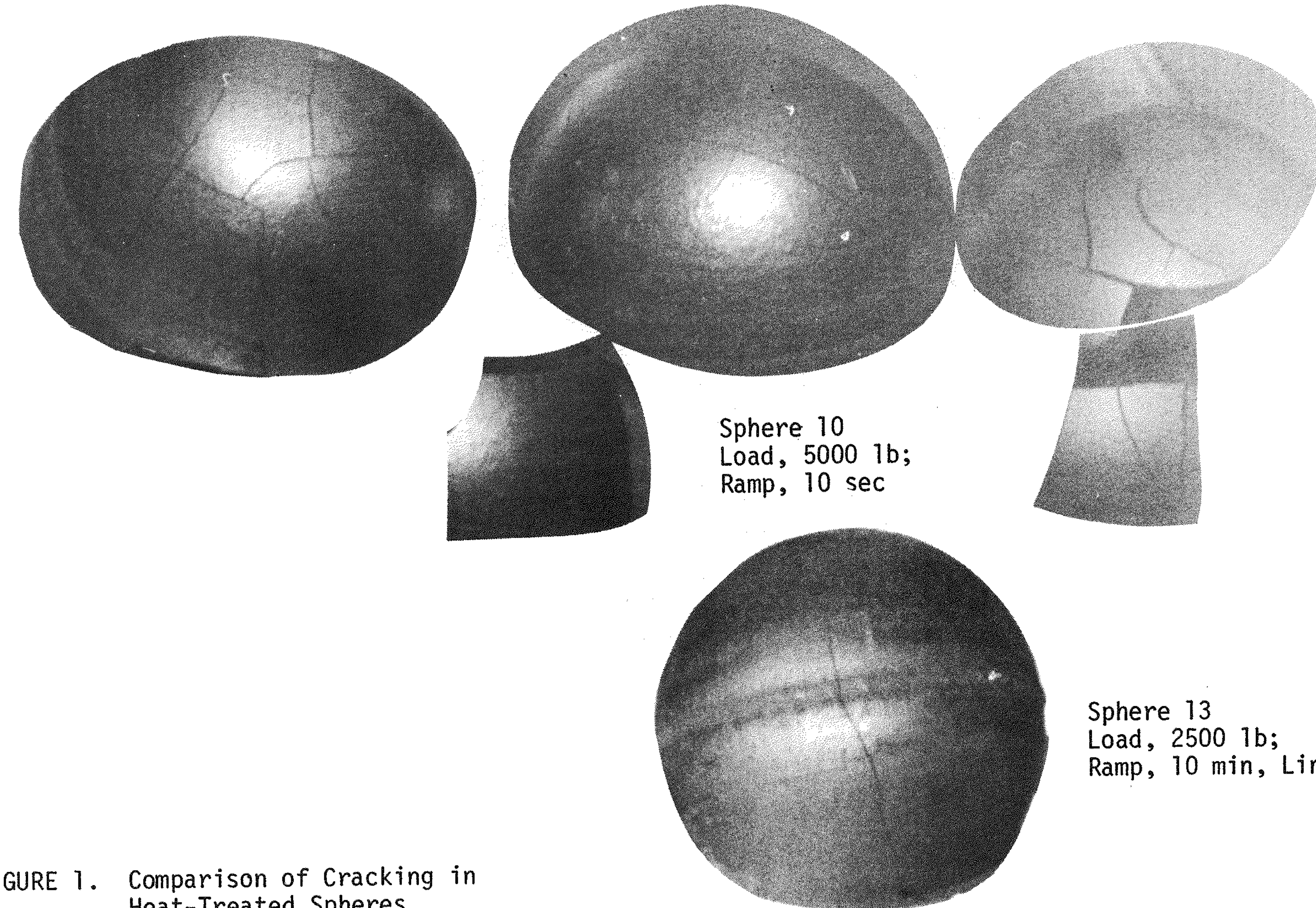
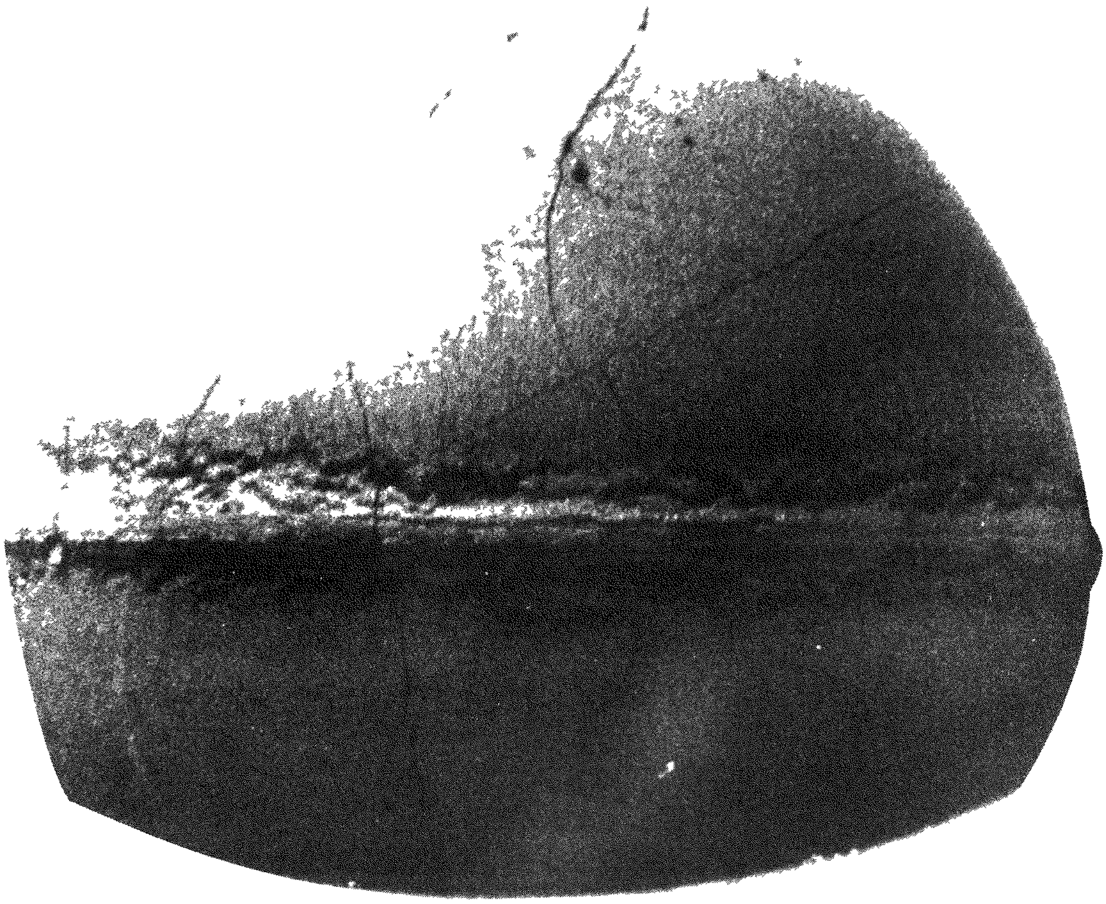


FIGURE 1. Comparison of Cracking in Heat-Treated Spheres



Sphere 25
Load, 2500 lb;
Ramp – Step Function and
Linear Rate Over ∿4 min

FIGURE 1 (Continued)

PROCESS CONDITIONS RECOMMENDED FOR MHW SPHERES

Process conditions that were recommended for initial production spheres for the Galileo Mission are summarized in Table 4. These conditions differ from the conditions used for the second generation of preproduction spheres (Nos. 13-24) in three major ways: (1) a higher shard presintering temperature (1300 versus 1175°C); (2) different preload and preheat conditions of hot pressing; and (3) a slow, graduated application of load during hot pressing (Figure 2). The objective of these process changes was to improve the mechanical integrity and dimensional stability of MHW spheres. These conditions are based on results of MHW fabrication tests in the SRP PuFF Facility and SRL Actinide Materials Facility (AMF).

Higher presintering temperature of shards and slow application of load during hot pressing have been shown in PuFF tests, including the sphere parametric tests described above, to lead to a desirable microstructure (integral shards and stable, coarse intershard porosity), and improved fuel integrity.[2,4] Significant reduction of hot press load and its rate of application for Spheres 11-13 resulted in a significant improvement in the quality of microstructure and fuel integrity over earlier spheres pressed with original LASL conditions. The success of these three fabrication tests, especially Sphere 13, is the primary basis for the recommended reduction of load application rate. Microstructural analyses of Spheres 4, 5, 13, and 15, and pellets made in the parametric test (Phase I)[3] indicate that rate of load application affects homogeneity of microstructure, with improved homogeneity occurring for slower rates. The nonlinear application of pressing load (Figure 2) was designed to increase intershard forces proportionately with sintered neck growth between shards.

Higher shard presintering temperature (1300 versus 1175°C) should also lead to an improvement in fuel integrity and dimensional control, although the data are not as conclusive as for the effects of load and load application rate. Sphere 3, even though hot pressed at the LASL conditions, was extremely rugged as-pressed and after reoxidation; it had a microstructure intermediate between the integral shards and coarse intershard porosity of Sphere 13 and the "crushed" shards and fine porosity of Spheres 4 and 5.[2] These improved properties of Sphere 3 compared to other early spheres hot pressed using LASL conditions are most likely attributable to "deadening" of the shards that occurred from presintering at higher temperatures and longer times than normal. As described above for the parametric tests, the denser, harder shards obtained from presintering at 1300°C seemed to improve the dimensional stability of Sphere 25, over that of previous second-generation preproduction spheres pressed with otherwise comparable conditions.

TABLE 4

Recommended Process Parameters for MHW Production Startup

- Feed Powder

  4 to 6-μm mode size

- Oxygen Exchange

  5 hr at 800°C
  400 cc/min $^{16}O_2$ flow

- Outgas

  1 hr at 1000°C
  40 cc/min $^{16}O_2$ flow

- Ball Mill

  12 hr at 100 rpm
  SRL design

- Cold Press

  58,000 psi reached in increments

- Breakup and Size

  Granule size <125 μm

- Presinter

  6 hr at 1300°C in $^{16}O_2$ atmosphere

- Hot Press

  1540°C maximum temperature
  Force Application (Figure 2)

  - Apply 250-lb preload prior to heating
  - Hold preload during heating and for 5 min after maximum temperature is reached
  - Increase load from 250 to 500 lb at a rate of 50 lb/min
  - Increase load 500 to 1100 lb at a rate of 200 lb/min
  - Increase load from 1100 to 3000 lb[a] at a rate of 700 lb/min
  - Hold load at 2500 lb[a]
  - Release force and begin cooling 5 min after die closure

- Heat Treatment

  12 hr at 1440°C
  Max ΔT, ±5°C/min

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a. Changed to 3000 lb subsequent to hot pressing of Sphere 33.

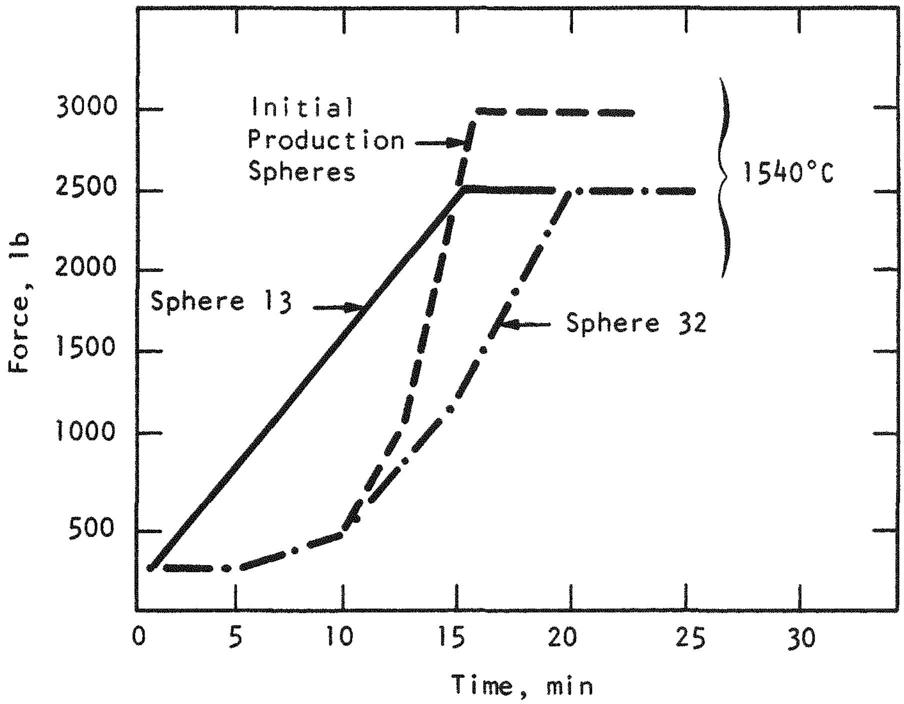

FIGURE 2. Force Conditions for Hot Pressing

A potential additional advantage of denser shards is the possible decrease in carbothermic reduction of PuO_2 during hot pressing. The reduction of PuO_2 during hot pressing in graphite dies produces CO gas which may cause or aggravate cracking and results in a phase (volume) change on reoxidation which has been shown to cause cracking in hot-pressed PuO_2.

Preheating the shards before initiating the pressing ramp will allow carbothermic reduction and the evolution of CO to occur before consolidation of the shards. This may prevent possible cracking due to CO gas evolution.

FINAL MHW TEST SPHERES

The final MHW test spheres (Nos. 32 and 33) were successfully fabricated in the PuFF facility in preparation for production startup in December. These spheres were well formed, dimensionally stable during heat treatment, and contained fewer or less severe visible cracks than previous spheres. The improved quality of these spheres was attributed to (1) a graduated and protracted force ramp during hot pressing and (2) the relatively high shard presintering temperature of >1300°C. These spheres were fabricated to test the process conditions recommended by SRL for production of 103.5-watt MHW spheres for the Galileo Mission (Table 4).

The recommended centerline conditions given in Table 4 were selected to be within the Technical Standards for PuFF facility operation, which were written in May 1978 before all of the PuFF test data were available. An evaluation of the existing data, however, indicated that possibly a longer hot pressing ramp than allowed by the Technical Standards and recommended in Table 4 could improve sphere integrity. Therefore, two final test spheres were fabricated using the conditions of Table 4, except for different hot pressing load conditions: Sphere 32 was pressed with a longer ($\sim$15 min) graduated force ramp than given in Table 4, while Sphere 33 was fabricated with conditions as close as possible to the recommended conditions.

Test Sphere 32

Sphere 32 was hot pressed according to the planned procedure (Table 5) except for the deviations noted in Table 6. The final load had to be increased from the planned maximum of 2500 to 3000 lb to ensure die closure. The recorded hot pressing conditions are shown in Figure 3.

Sphere 32 was integral and well formed with no apparent cracks as-pressed and with only one minor crack (polar) after final heat treatment. This crack widened somewhat and additional hairline cracks formed during gaging and subsequent storage (Figure 4). This sphere also had excellent dimensional stability as very little dimensional change occurred during final heat treatment. The dimensions were within MHW specifications as-pressed and after final heat treatment (Table 6). Sphere 32 also appeared to be the most rugged of spheres made to date. It survived a 4-inch drop test without cleaving. Previous heat-treated spheres when drop-tested intentionally (Sphere 13) or accidentally, fractured into pieces.

TABLE 5

Recommended Process Conditions for Sphere 32

- Same as Table 4 except for force application during hot pressing

- Force Application (Figure 3)

  Apply 250-lb preload prior to heating

  Hold preload during heating and for 5 min after maximum temperature of 1540°C is reached

  Increase load from 250 to 500 lb at a rate of 50 lb/min

  Increase load from 500 to 1250 lb at a rate of 150 lb/min

  Increase load from 1250 to 2500 lb at a rate of 250 lb/min

  Hold load at 2500 lb

  Release force and begin cooling 5 min after die closure

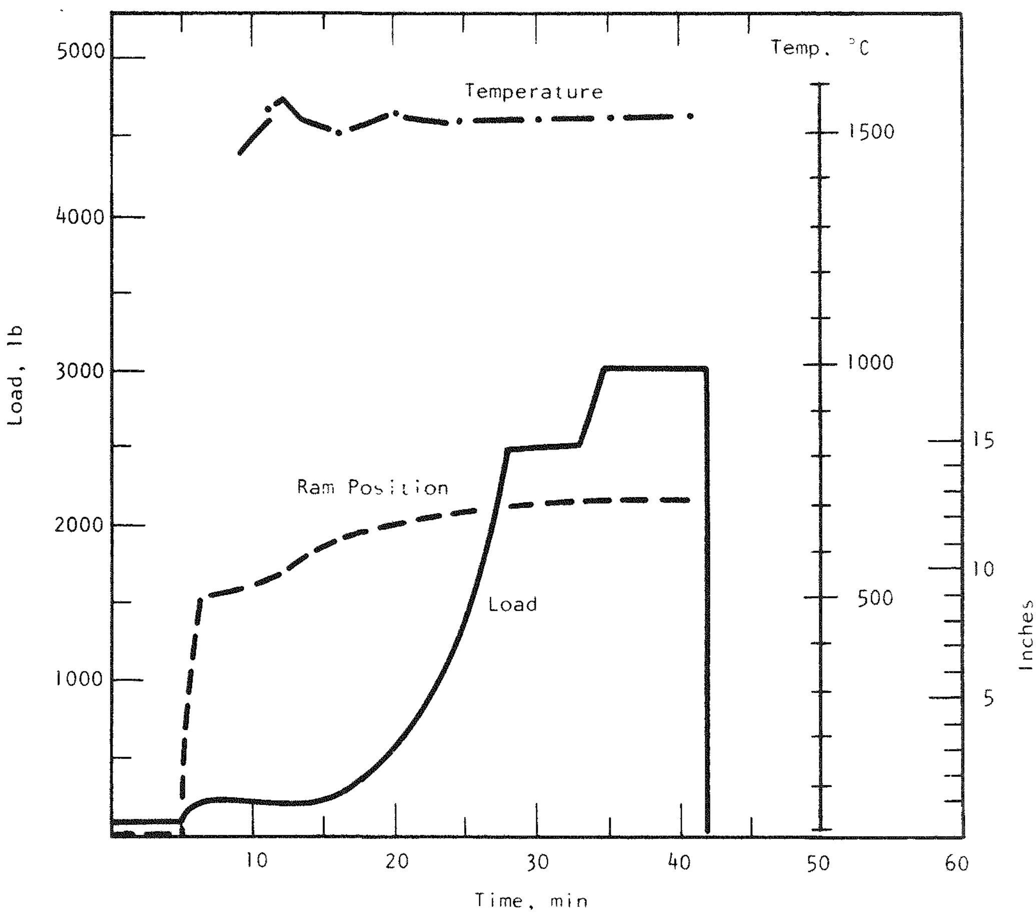

FIGURE 3. Hot Pressing Conditions for Sphere 32

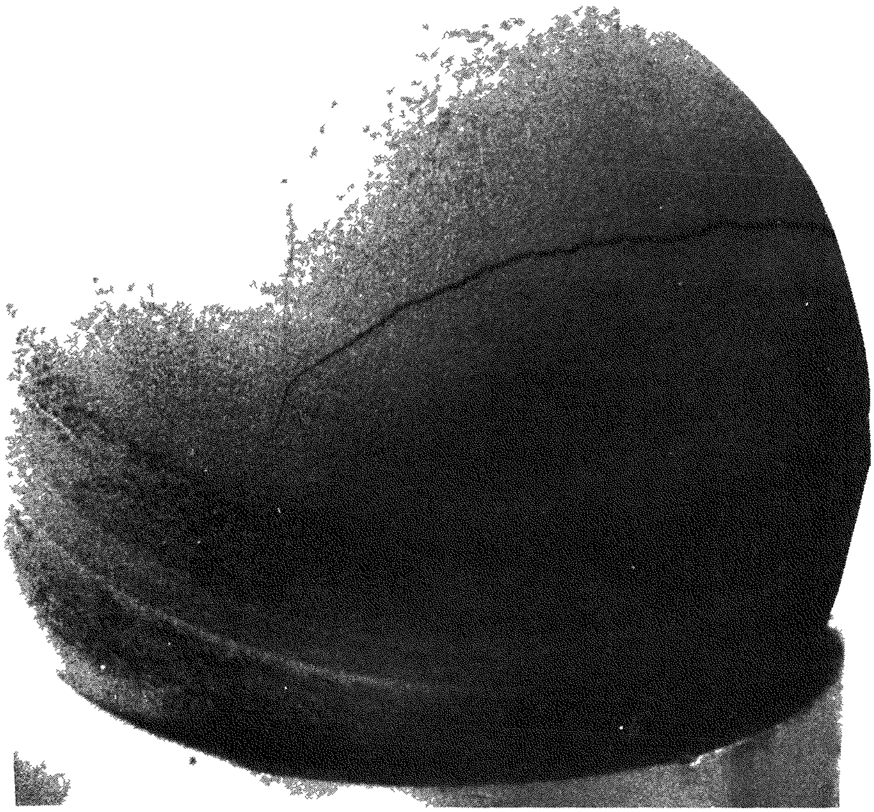

FIGURE 4. Sphere 32 After Heat Treatment, Gaging, and Storage

TABLE 6

Summary of Spheres 32 and 33

Process Conditions[a]

Sphere No.	*Shard Sintering Temp, °C*	*Maximum Load, lb*	*Temperature, °C*	*Ramp*	*Die Closure*
32	1300 (1355)	2500 (3000)	1540 (1535)	*b*	Yes
33	1300 (1360)	2500 (2800)	1540 (1590)	*b*	Yes

Sphere Quality

Sphere No.	*Condition*	*Appearance*	*Dimensions, in.*	
			Pole	*Equator*
32	As-Pressed	Uncracked	1 477	1 466
	Heat-Treated	Integral with minor crack	1 477	1 462
33	As-Pressed	Uncracked	1 477	1 465
	Heat-Treated	Integral with minor cracks	1 475	1 469

a. Both goal conditions and measured conditions (in parentheses) are given

b. See Figures 2, 3, and 5

Test Sphere 33

Sphere 33 was hot pressed according to the procedure given in Table 4 except for the deviations noted in Table 6. The final load had to be increased from 2500 to 2800 lb (Figure 2) to ensure die closure, which extended the ramp time beyond the 12-min limit given in the Technical Standards. The recorded hot pressing conditions are shown in Figure 5.

Sphere 33 was similar in appearance to Sphere 32. Sphere 33 was integral with only minor cracking (Figure 6) and experienced minimal dimensional change during final heat treatment. The polar and equatorial dimensions were within specification as-pressed and after final heat treatment (Table 6).

Test Authorization

On the basis of the excellent results of final test Spheres 32 and 33, the procedure of Table 4 was accepted for production. Initial production spheres, however, had to be pressed with a slightly shorter ramp (<12 min) to be within the Technical Standard limit.

To permit the use of the recommended load conditions and to provide greater production flexibility, Test Authorization (TA) 2-952 was issued in January 1979. This TA authorizes the shipment of nominal 103.5-watt project Galileo fuel spheres produced within increased limits on three hot-pressing variables (Table 7). These variables are: (1) time from start of the force ramp until final load is reached, (2) final load, and (3) maximum temperature. The appropriate process limits in the Technical Standards for the PuFF facility will be revised to reflect these changes. The new limits on final load and maximum temperature provide adequate separation between current operating limits and process limits in the Technical Standards. These broader limits on the hot-pressing variables provide the flexibility needed to compensate for processing variations and ensure die closure without jeopardizing the desired density and microstructure of the spheres.

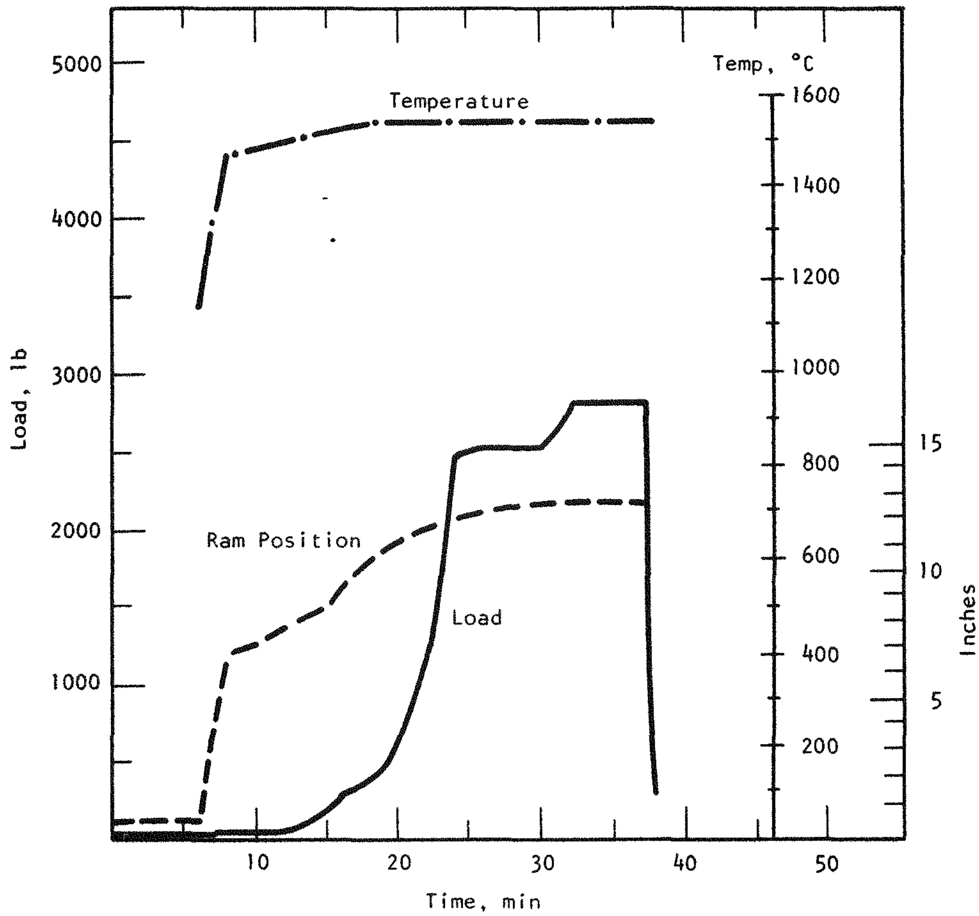

FIGURE 5. Hot Pressing Conditions for Sphere 33

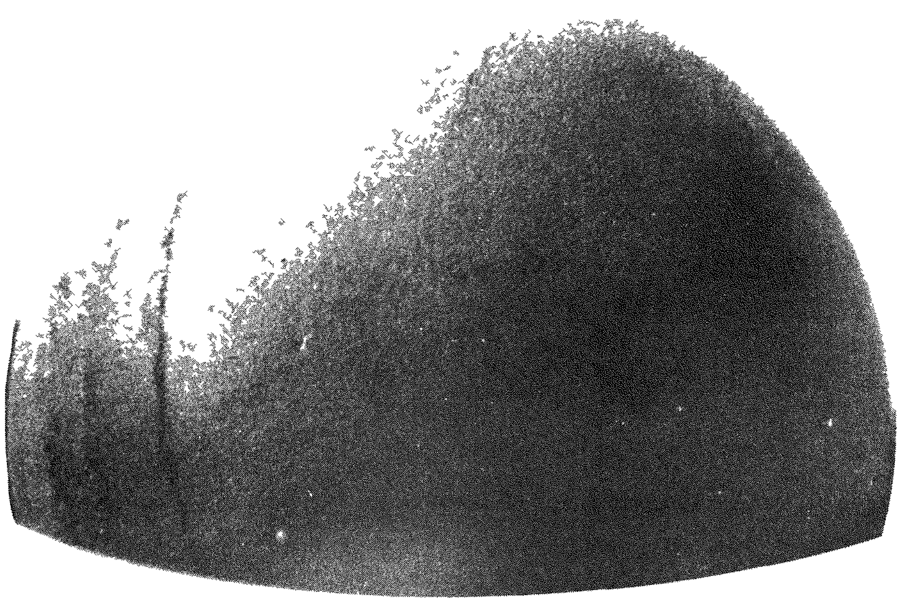

FIGURE 6. Sphere 33 After Heat Treatment

TABLE 7

Process Limits Affected by Test Authorization

	Technical Standard Limits		*Test Authorization Limits*	
	Min	*Max*	*Min*	*Max*
Time from start of force ramp to reach final load on control ram, min[a]	3	12	3	30
Final load on control (bottom) ram, lb	1500	3500	1500	5000
Hot pressing temp, °C	1400	1575	1400	1675

a. At 500 lb, the force preload is defined to end and force ramp is defined to start.

GENERAL-PURPOSE HEAT SOURCE PROCESS DEMONSTRATION

TRANSFER OF TECHNOLOGY

The transfer of technology from LASL to SR for the General-Purpose Heat Source (GPHS) project has been initiated. A preliminary process flowsheet and preliminary product specifications for GPHS fuel forms were presented by LASL during a review meeting at DOE Headquarters in July 1978. A revised flowsheet was transmitted to SRL in December. SRL has begun small-scale fuel fabrication tests and microstructural analyses in the Alpha Materials Facility (AMF) to evaluate the effects of non-scale sensitive process variables on GPHS fuel structure. Full-scale fabrication tests will be initiated in the Plutonium Experimental Facility (PEF) in January 1979, to examine scale-sensitive process variables and to adapt the LASL process for production of GPHS fuel in the PuFF facility for the Solar Polar Mission.

The GPHS fuel form will (1) be a right circular cylinder of $^{238}PuO_2$ (83.5% ^{238}Pu) with rounded edges (Figure 7), (2) have dimensions of about 1 in. × 1 in., (3) be about 85% dense, and (4) generate about 62.5 watts of thermal power. The preliminary process flowsheet, shown in Figure 8, contains a few modifications of the preliminary LASL flowsheet to accommodate differences in equipment between LASL and SR. These modifications are similar to those made to adapt the MHW process for production in the PuFF facility. The primary difference between the MHW and GPHS processes is the granule feed (shards) used to hot press the fuel form. GPHS granule feed consists of a "grog" mixture of shards presintered at 1600°C (40%) and 1100°C (60%). This "grog" feed material may produce a more-stable and more-crack-resistant microstructure than the MHW feed.

The initial small-scale tests are designed to determine the effects of density, shard presintering temperature, and reoxidation temperature on the microstructure and fracture behavior of GPHS fuel. These experiments should help establish the threshold density for reduction-reoxidation-induced cracking for the GPHS structure. These small-scale tests and preliminary full-scale tests in PEF will also investigate the possible reduction of shard-sintering temperature from 1600 to 1500°C. This reduction may be necessary if the new ZGS Pt - 10% Rh furnace racks, presently being designed, are not capable of extended operation at 1600°C. Centerline conditions and process limits for variables such as shard-sintering temperature, hot pressing load, rate of load application and temperature, and final heat treatment temperature will be established based on full-scale tests in PEF.

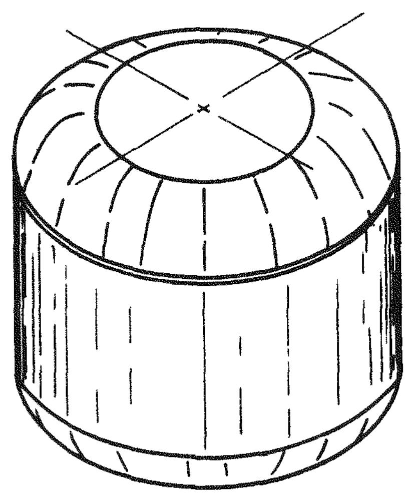

FIGURE 7. GPHS Fuel Pellet Design Geometry

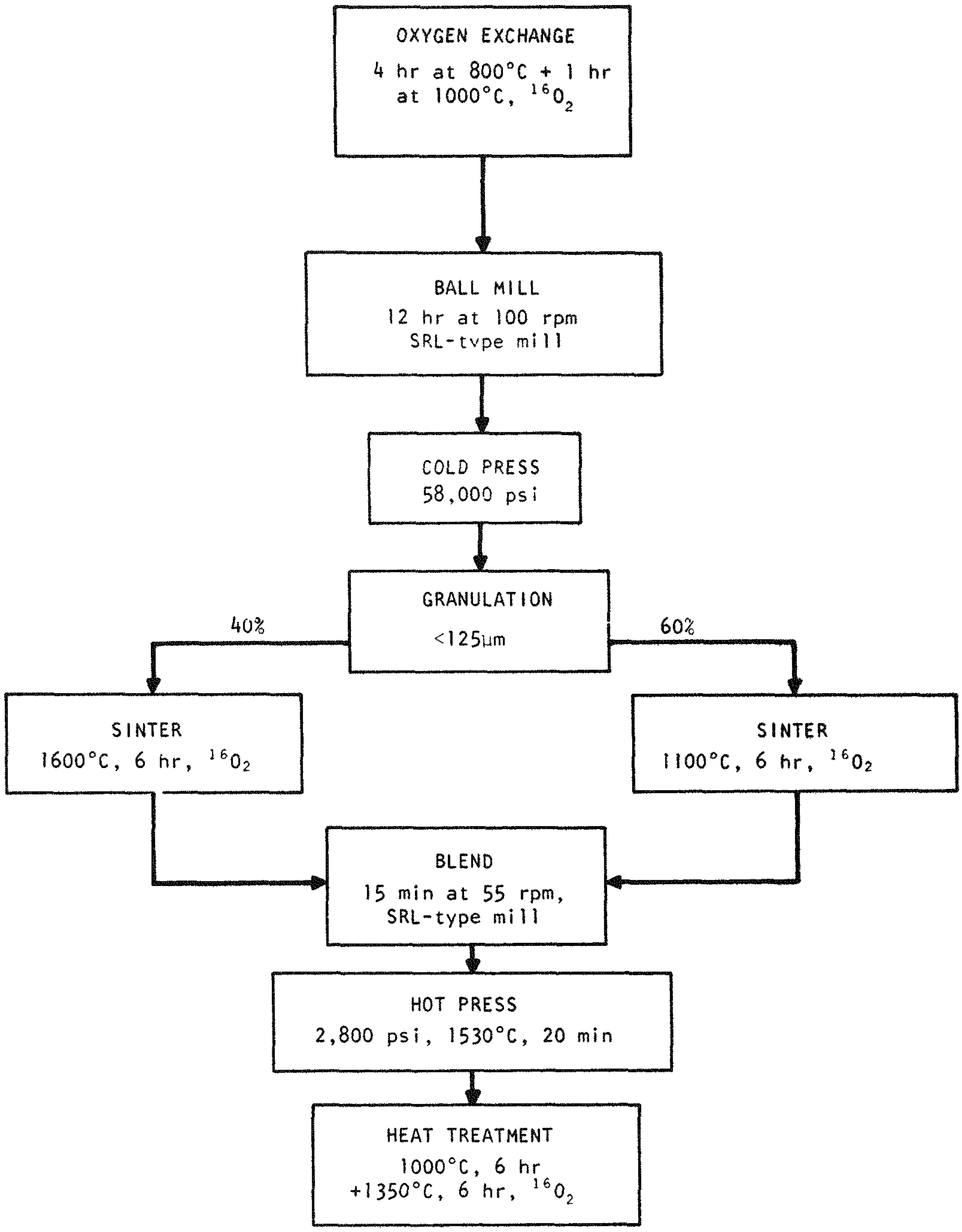

FIGURE 8. GPHS Process Flowsheet

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