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^{238}Pu FUEL FORM PROCESSES QUARTERLY REPORT

JANUARY - MARCH 1981

E. I. du Pont de Nemours & Co.
Savannah River Laboratory
Aiken, SC 29808

PREPARED FOR THE U. S. DEPARTMENT OF ENERGY UNDER CONTRACT DE-AC09-76SR00001

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Approved by:

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CONTENTS

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 primarily by the DOE Advanced Nuclear Systems and Projects Division (ANSPD).

Goals of the Savannah River Laboratory (SRL) program include providing technical support for the production of $^{238}PuO_2$ fuel forms in the Savannah River Plant's (SRP) Plutonium Fuel Form (PuFF) Facility. This part of the program includes:

Demonstration of processes and techniques developed by the Los Alamos National Laboratory (LANL) 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 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 efficiency and product reliability, and to adapt plant facilities for new products.

GENERAL-PURPOSE HEAT SOURCE (GPHS) PROCESS SUPPORT

EFFECT OF ATMOSPHERE ON THE SINTERING OF $^{238}PuO_2$ GRANULES

Summary

Recent process development work at Los Alamos National Laboratory (LANL) and Savannah River Laboratory (SRL) indicates that the atmosphere (cover gas) in which oxide granules are sintered is a critical parameter in the production of General-Purpose Heat Source (GPHS) fuel forms. Different atmospheres were originally assumed to have no significant effects on granule sintering or the properties of fuel forms but differences observed between the microstructure of SRL and LANL GPHS pellets[1,2] indicated that this assumption might not be valid. The study discussed in this report was initiated in an attempt to correlate changes in granule sintering atmosphere with differences in the microstructure and integrity of GPHS pellets fabricated by LANL, SRL, and Savannah River Plant (SRP). Results of the study show clearly that the effects of granule sintering atmosphere cannot be neglected.

The solubility of the cover gas in $^{238}PuO_2$ and the effect of the gas on the stoichiometry of the oxide have significant effects on the final density, grain size, and pore size of the granules. Characteristics of the sintered granules, in turn, affect the microstructure, extent of cracking, and integrity of hot-pressed GPHS fuel forms.

GPHS fuel forms are currently produced in the Plutonium Fuel Form (PUFF) Facility in an $Ar/^{16}O_2$ atmosphere because of existing furnace restraints. SRL normally sinters granules in an Argon atmosphere. Early production yield was lower than expected because of pellet cracking problems. The results of these granule sintering and pellet fabrication tests were useful in improving the quality of the production pellets. The low yield was improved by increasing the sintering temperature of the low-fired granules from 1100°C to 1150°C. This increase in temperature helped to compensate for the oxidizing atmosphere used in the production process.

Background

Effects of atmosphere on sintering behavior of $^{238}PuO_2$ were not considered in development of fabrication processes for the MHW and GPHS fuel forms. The original GPHS process development work at LANL used Ar bubbled through $H_2{}^{16}O$ as the cover gas during granule sintering. In additional process development work at SRL in the Plutonium Experimental Facility (PEF) Ar gas was fed directly from a tank.[3] A mixture of Ar and $^{16}O_2$ gas is now being used as the cover gas during granule sintering in the production process in the PuFF Facility at SRP. These sintering atmospheres were chosen because of availability (LANL), cost savings (SRL), and process equipment constraints (SRP).

Experimental Details

Unfired granules (diameter ≤125 mm) fabricated by SRL and LANL were used in this study. Randomly selected samples (~1 g) of these granules were heated in a horizontal platinum tube furnace in the SRL Alpha Materials Facility (AMF). In each test, granules were sintered at 1600°C for 6 hr and heated and cooled at rates of ~300°C/hr. Sintering atmospheres were varied to include combinations of reducing, oxidizing, soluble, and insoluble gases as well as "pure" gases (which probably contained some H_2O and were therefore slightly reducing). Granules were heated in Ar, Ar/5% O_2, Ar/H_2O, O_2, air, He, He/5% O_2, He/H_2O, and He/4% H_2.

Samples of these sintered granules (~0.1 g) were mounted in Bakelite™ (Union Carbide Corp.), carefully ground and polished to 0.5 μm, and examined with a metallograph. Micrographs of as-polished and acid-(grain-boundary) etched granules were used to document the results. Although quantitative image analyses of these micrographs would have been beneficial to this study, the instrumentation was not available. The effects were so obvious, however, that definite trends could be detected by qualitative analyses.

Results and Discussion

LANL vs SRL Granules

The results of this investigation showed that unfired granules made at LANL and sintered at SRL have a lower final density than SRL PEF granules that were sintered at SRL under identical conditions. This density difference is shown in Figure 1 for granules sintered in Ar and in Figure 2 for granules sintered in Ar/5% O_2. The lower sintered density of LANL granules may be a direct result of a lower unfired density. The lower unfired density may be related to the larger height-to-diameter

cold-pressed compact ratio used in the LANL process or by slightly different granulation techniques. A method was not available to measure the density of the unfired granules, therefore the hypthesis stating that LANL granules have a lower unfired density than SRL granules was not confirmed.

As shown in Figure 3, granules fabricated and sintered at LANL had a lower final density than granules fabricated at LANL and sintered at SRL. This difference may be due to bed-depth effects since the LANL granules were sintered in large quantities (~ 150 g) with a bed depth of over one-half inch compared with <1-g quantities (< 1/8-in. layer) used in this granule sintering study.

Differences between LANL and SRL granules do not fully explain all of the microstructural differences between LANL and SRL[2,3] fuel pellets but these granule differences may be partially responsible for these differences.

Cover Gas Solubility Effect

Additional granule sintering experiments with SRL and LANL unfired granules indicated that high-density $^{238}PuO_2$ granules were produced only in a cover gas that dissolves in and reduces the plutonia.

As observed with other ceramic materials, certain gases will readily dissolve in the ceramic and diffuse to the surface at such a rate as to not limit the sintering rate.[4,5] On the other hand, some gases may not be soluble in the ceramic and pores filled with these gases will not disappear.[4] In general, pore elimination requires that gas be absent from the pores (vacuum sintering) or that the granules be fired in a gas which will dissolve and diffuse out of the pore.

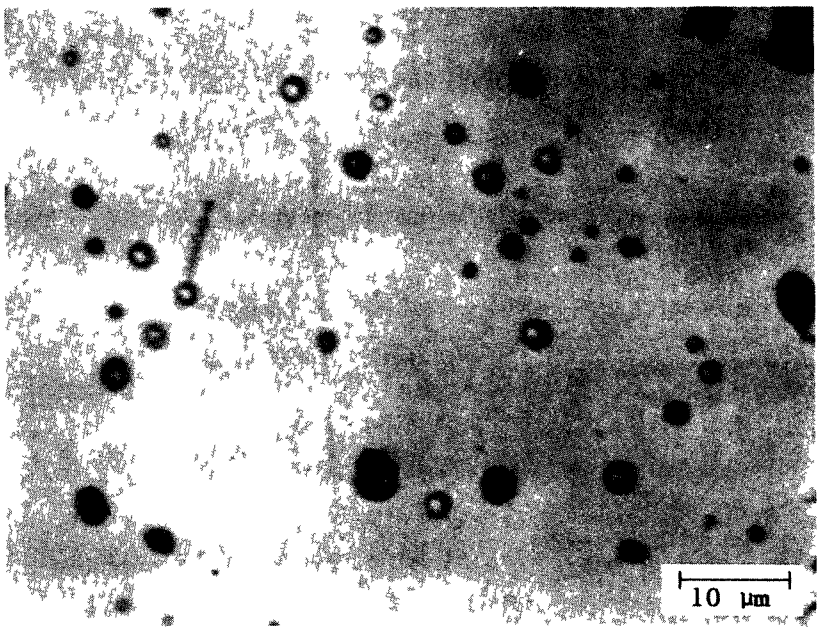
Experimental results of this study indicate that He is much more soluble in PuO_2 than is Ar (Figure 4) The granules sintered with He as the primary cover gas had a significantly higher density than granules sintered with argon. In fact, results of this study indicate that the pores in granules sintered in argon are probably much larger than the pores in unfired granules. Pore growth during sintering (pores normally shrink during sintering) can be explained by a pore coalescence mechanism.[6] As grain growth occurs during sintering of a polycrystalline ceramic, the pores move to the grain boundaries and coalesce. These pores, which initially have an equilibrium pressure of $2\gamma/r$ (γ is surface tension of the gas-filled pore and r is the pore radius) of the insoluble gas, are larger after coalescence and have a lower equilibrium pressure. Consequently, this excess gas pressure

creates a strain field in the matrix which provides a driving force for vacancy diffusion to the pore until the pressure in the pore is again balanced by the surface tension. Therefore, this pore coalescence mechanism can cause pore size to increase as grain growth occurs during sintering with an insoluble gas that is trapped within the pores.

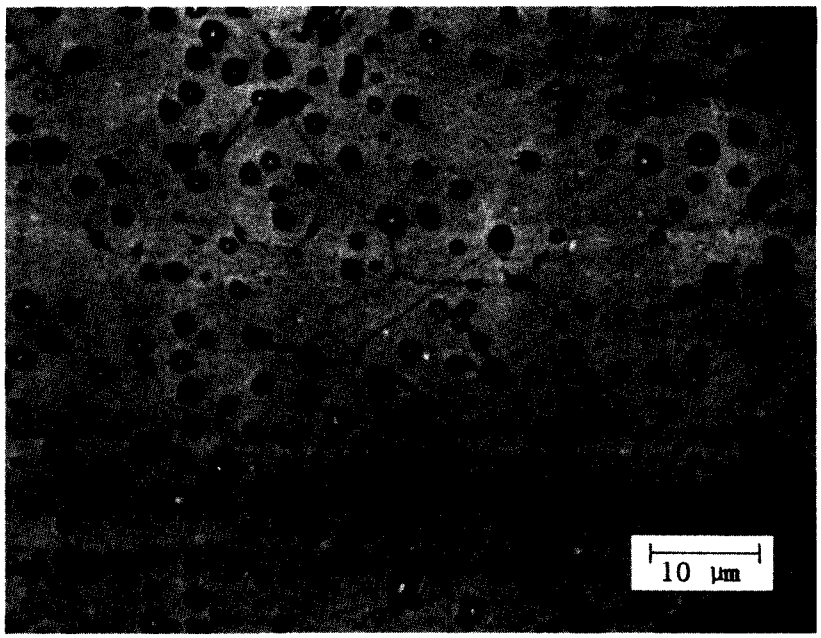
Granule Stoichiometry Effects

Another separate effect, related to that of sintering atmosphere, is the correlation found in this study between sintering rate and oxide stoichiometry. According to calculations by Bickford (unpublished work) PuO_2 reduced to $PuO_{1.98}$ in a H_2O - saturated inert gas atmosphere at 1600°C. In pure O_2 or Ar/5% O_2 plutonia is fully stoichiometric. Figure 5 shows that granules sintered in Ar/H_2O have a much larger grain size than granules sintered in Ar/O_2. This grain size difference implies that substoichiometric plutonia ($\sim PuO_{1.98}$ at 1600°C in Ar/H_2O) has a much higher sintering rate than fully stoichiometric plutonia. Oxygen-deficient PuO_2 is known to exist in a slightly reducing atmosphere.[7,8] This defect structure increases the diffusion rates and, hence, the sintering rates in plutonia. When PuO_2 is reduced below an O/M ratio of 1.98 a two-phase mixture of cubic PuO_2 and cubic Pu_2O_3 exists.[8] Despite the similarity of structure, PuO_2 and cubic Pu_2O_3 are distinct phases with different crystal lattices. The calculated density of cubic Pu_2O_3 is 10.2 g/cm^3,[8] and the density of PuO_2 is 11.46 g/cm^3.[8] Apparently, this lower-density, more-open-structured substoichiometric phase or possibly just oxygen-deficient PuO_2, significantly increases the sintering rate of plutonia granules.

It should be noted here that studies on other ceramic oxides have clearly demonstrated that a variation in stoichiometry can profoundly influence the sintering rate of oxides. A study[9] of the initial sintering kinetics of UO_2 indicated that extremely large changes in the diffusion coefficient of U in UO_2 can occur with very slight changes in stoichiometry; a change in stoichiometry from UO_2 to $UO_{2.02}$ causes an increase in the diffusion coefficient of U by a factor of 10^8.

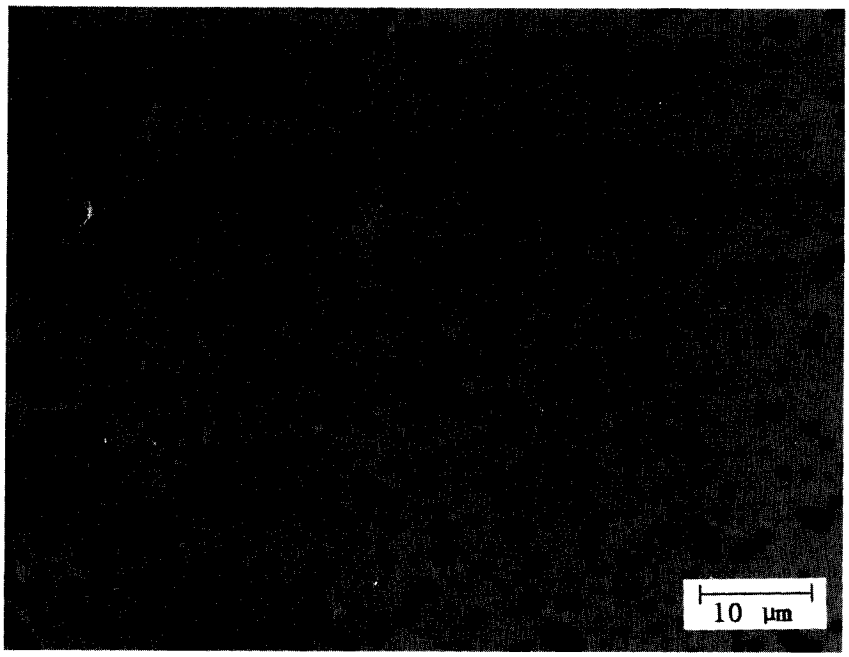


Fabricated at SRL

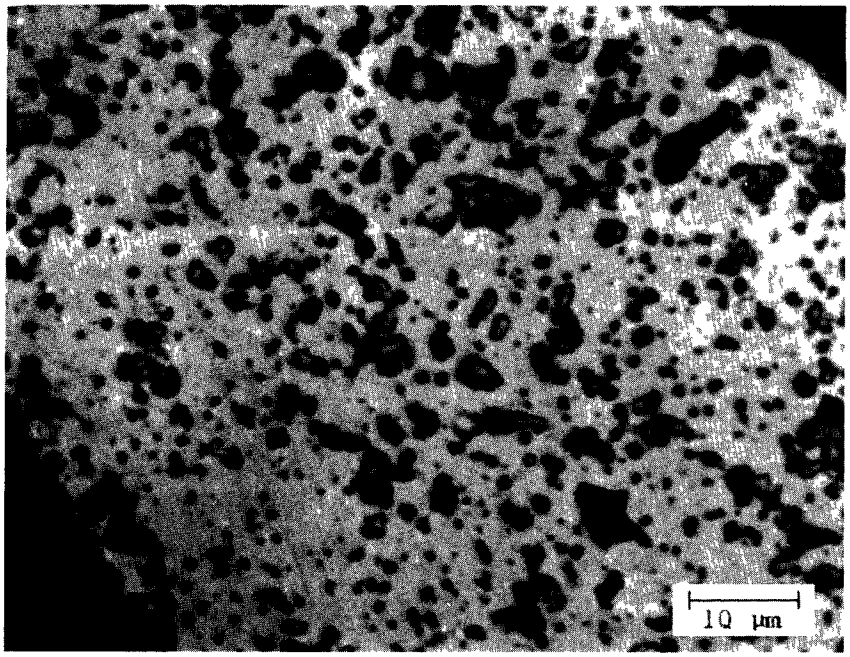


Fabricated at LANL

FIGURE 1. $^{238}PuO_2$ Granules Sintered at SRL in Ar for 6 hr at 1600°C. Note Lower Density of Oxide Fabricated at LANL.

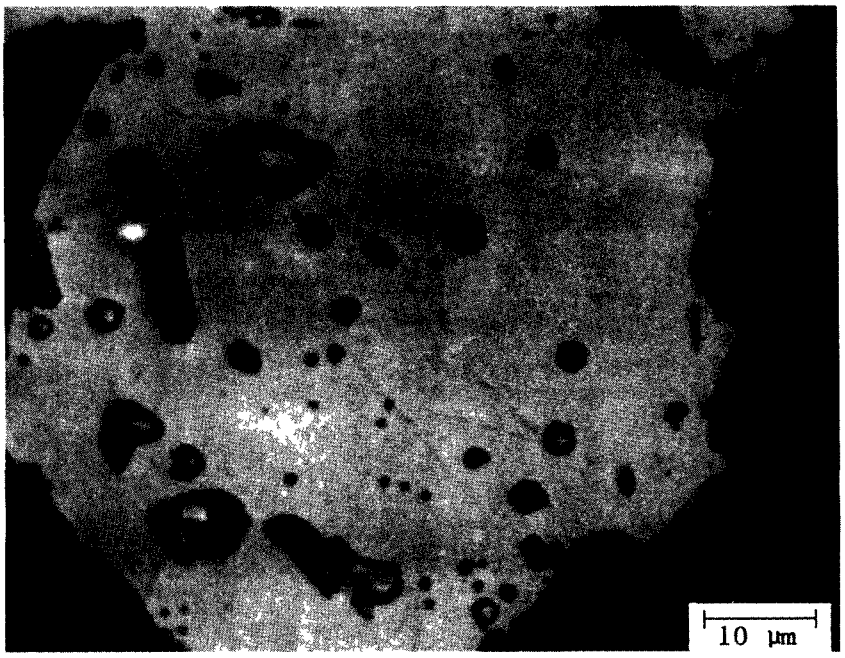


Fabricated at SRL

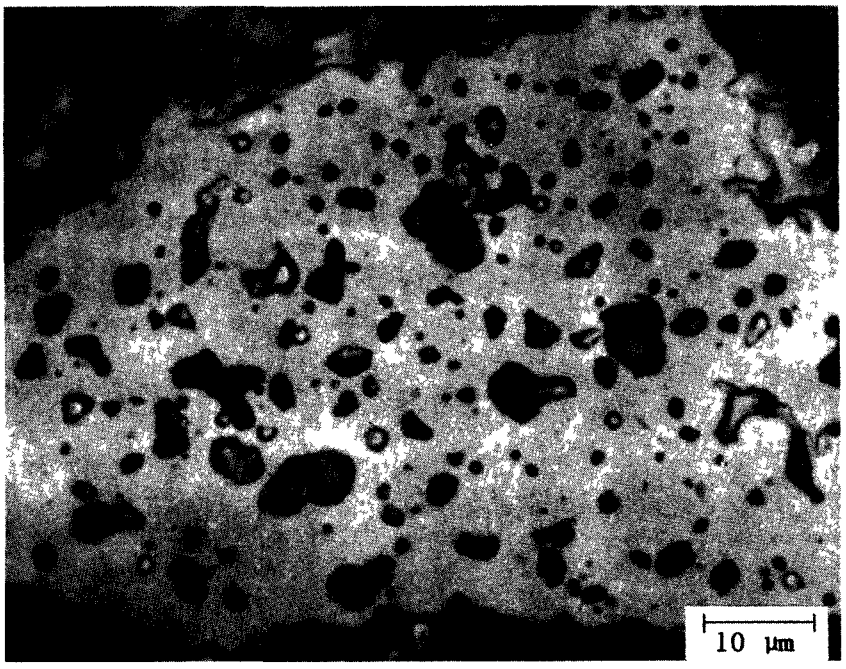


Fabricated at LANL

FIGURE 2. $^{238}PuO_2$ Granules Sintered at SRL in Ar/5% O_2. Note Lower Density of Oxide Fabricated at LANL.

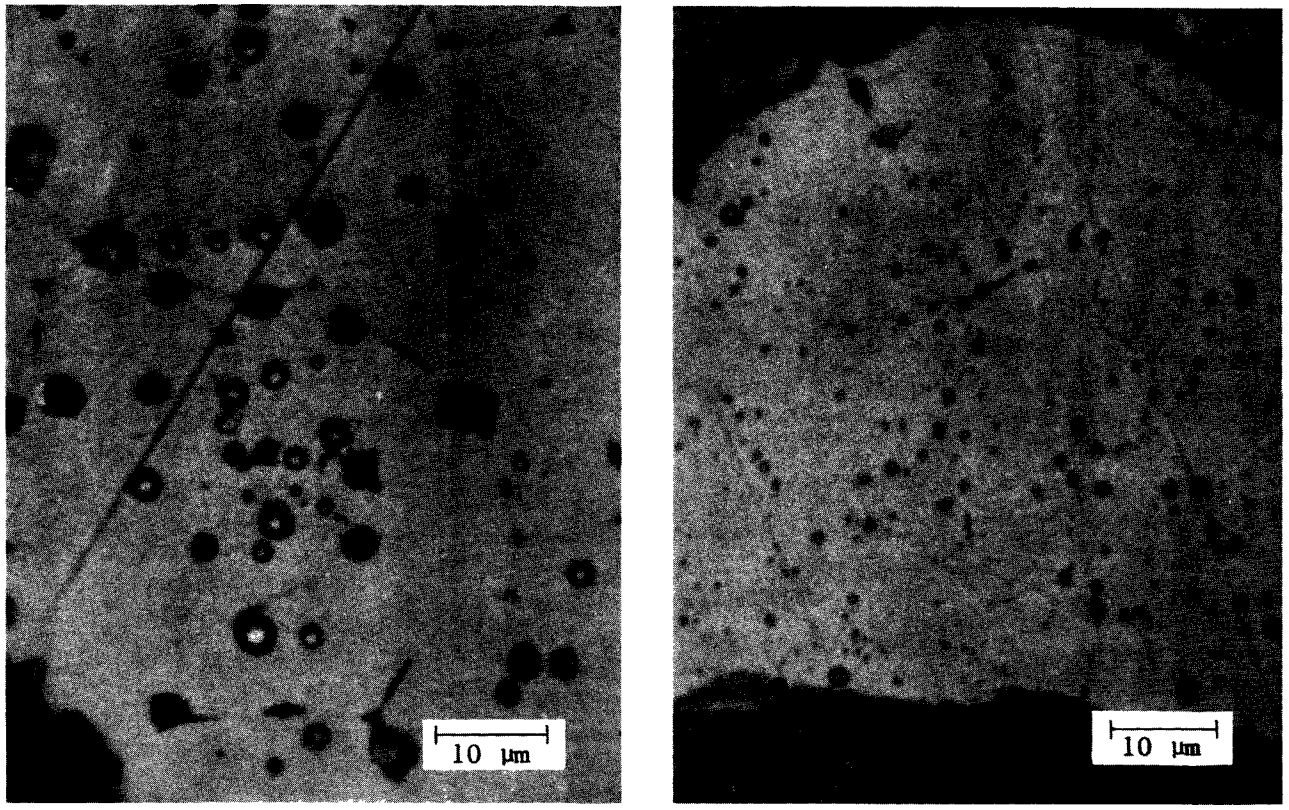


Sintered at SRL



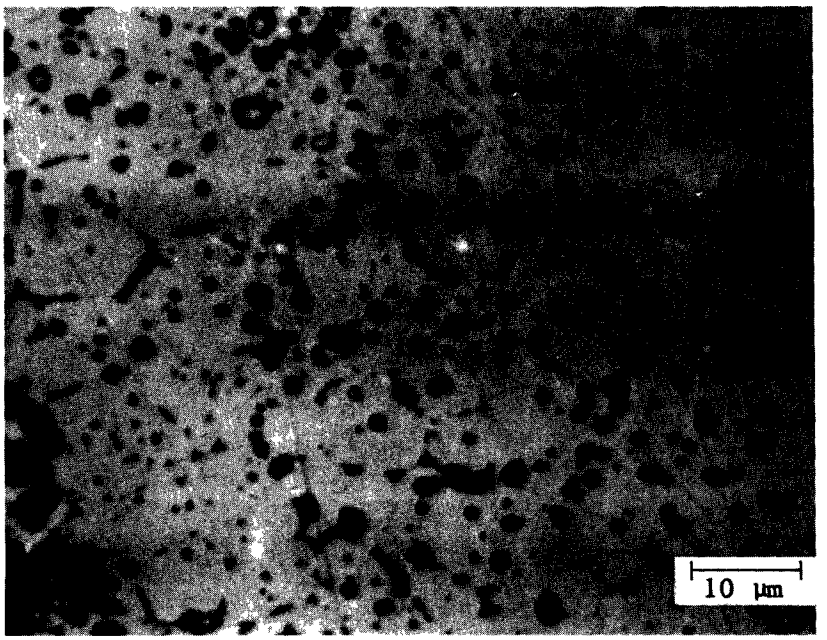
Sintered at LANL

FIGURE 3. $^{238}PuO_2$ Granules Fabricated at LANL, Sintered in Ar/H_2O. Note Lower Density of Material Sintered at LANL.

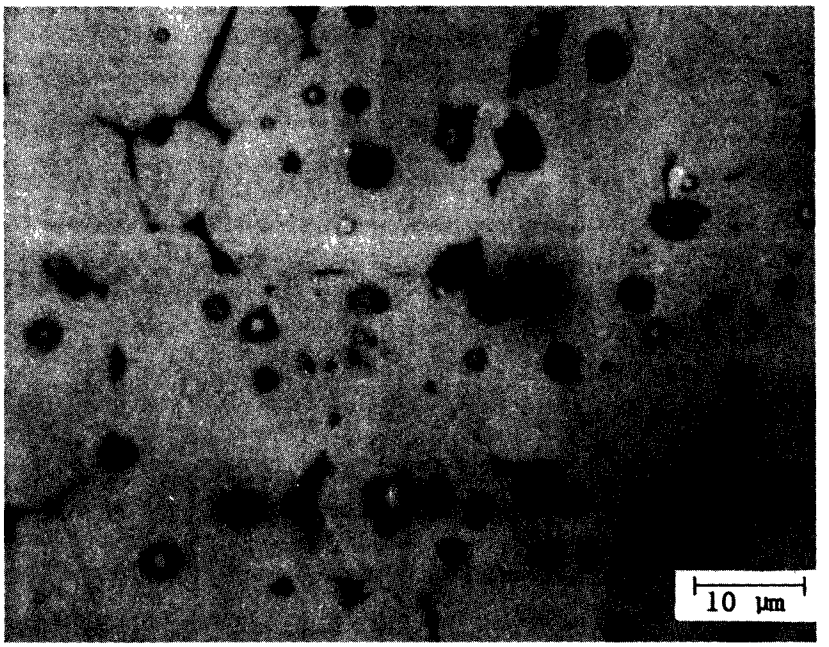


Sintered in Ar/H_2O Sintered in He/H_2O

FIGURE 4. Effect of Solubility of Cover Gas in $^{238}PuO_2$ on Density of SRL Oxide Granules. Note Higher Density of Granules Sintered in He/H_2O, a Gas Which Dissolves in and Reduces the Plutonia.



Sintered in Ar/5% O_2



Sintered in Ar/H_2O

FIGURE 5. Relationship Between Grain Size and Granule Sintering Atmosphere. Grain-Boundary-Etched Granules, Fabricated and Sintered at SRL. Note Larger Grain Size of Granules Sintered in Ar/H_2O.

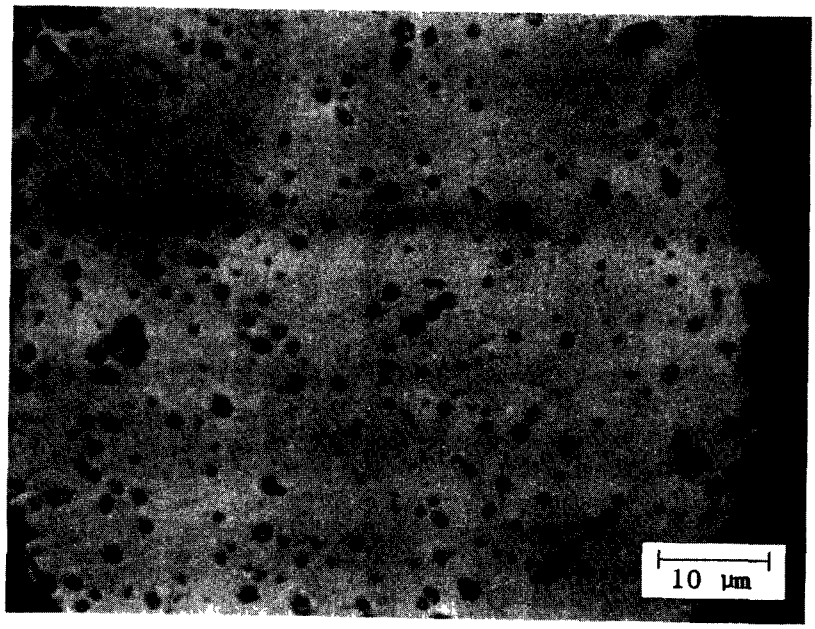
Interactions of Gas Solubility and Granule Stoichiometry

An important interaction effect between stoichiometry and sintering gas solubility was also found. As shown in Figure 5, if an insoluble cover gas is used for granule heat treatment, the use of a reducing atmosphere increases the rate of grain growth significantly but does not necessarily increase the rate of densification. In an atmosphere which is insoluble and reducing, rapid grain growth is accompanied by a significant increase in pore size by the pore coalescence mechanism discussed earlier and no significant reduction in the total pore volume in the granule. Likewise, the use of a soluble gas in an oxidizing atmosphere does not necessarily aid in the densification of $^{238}PuO_2$ granules (Figures 6 and 7).

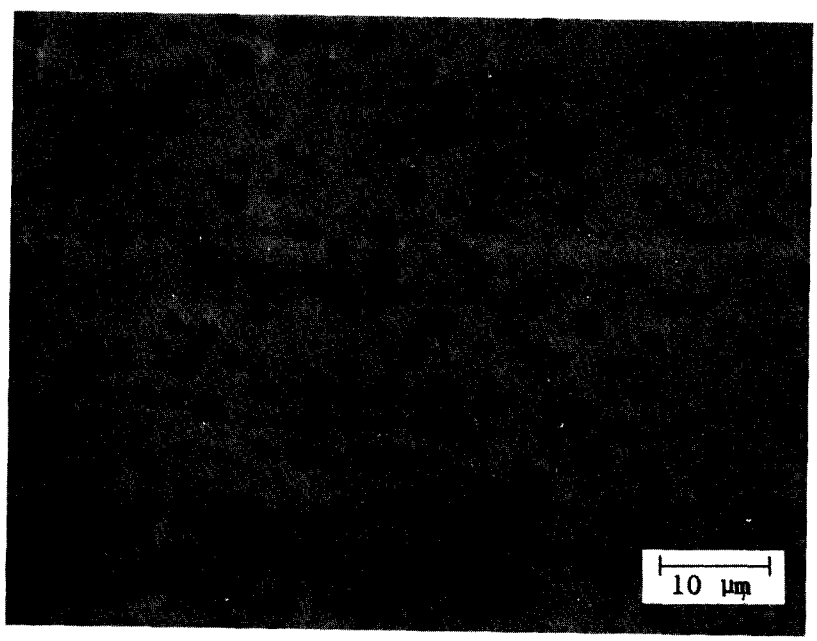
If the sintering rate is extremely slow, as is the case with $^{238}PuO_2$ in an oxidizing atmosphere at 1600°C, the effect of gas solubility is negligible, since the rates of grain growth and vacancy diffusion are very low. In summary, the ideal atmosphere to densify $^{238}PuO_2$ is a combination of a reducing atmosphere and a soluble gas viz., He/4% H_2 (see Figure 8).

Effects of Granule Sintering Atmosphere on Pellet Characteristics

Granule sintering atmosphere significantly affects GPHS fuel pellet characteristics. As discussed earlier, GPHS process development in the PEF was done with Ar used as the granule sintering atmosphere. As described in earlier reports,[1,10] GPHS pellets made with PEF centerline[1] conditions had a uniform density and very few cracks. The production process in the PuFF Facility uses Ar/$^{16}O_2$ as the cover gas. On the basis of this granule sintering study, since Ar/O_2 is an oxidizing atmosphere with an insoluble gas, the granule sintering rate is very slow. This slow sintering rate not only results in relatively low density, high-fired granules, but presumably may also result in low-density, low-fired granules with a lower crush strength relative to granules sintered in Ar. (Mounting and polishing techniques to characterize these low-fired granules were unsuccessful because they were too fragile to polish; consequently, confirmation of this presumption is still pending.)

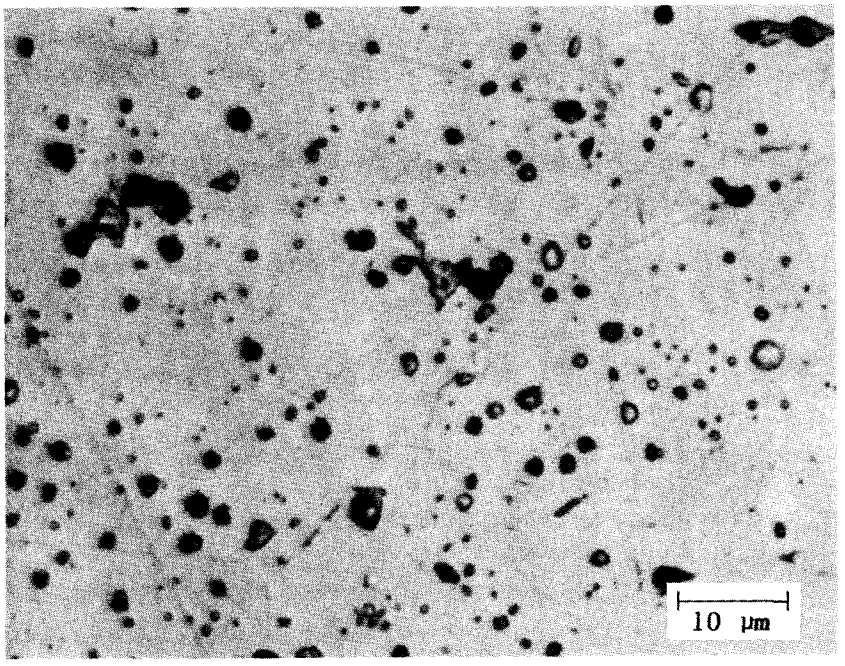


Sintered in He/5% O_2

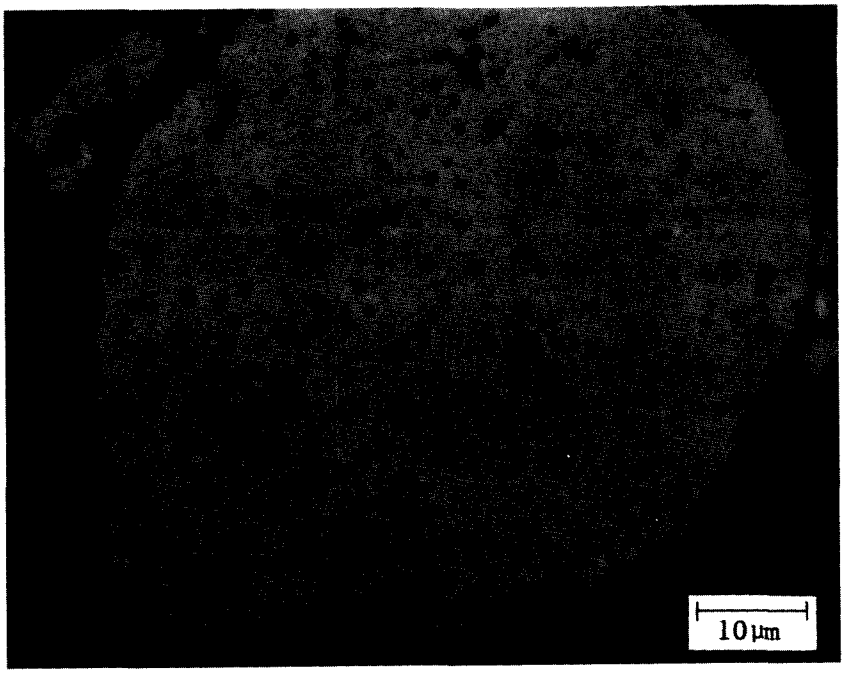


Sintered in Ar/5% O_2

FIGURE 6. Combined Effects of Oxide Stoichiometry and Solubility of Cover Gas in Oxide Granules



Sintered in O_2



Sintered in Air

FIGURE 7. Oxide Granules Sintered in Air (Left) and in O_2. Fabricated and Sintered at SRL

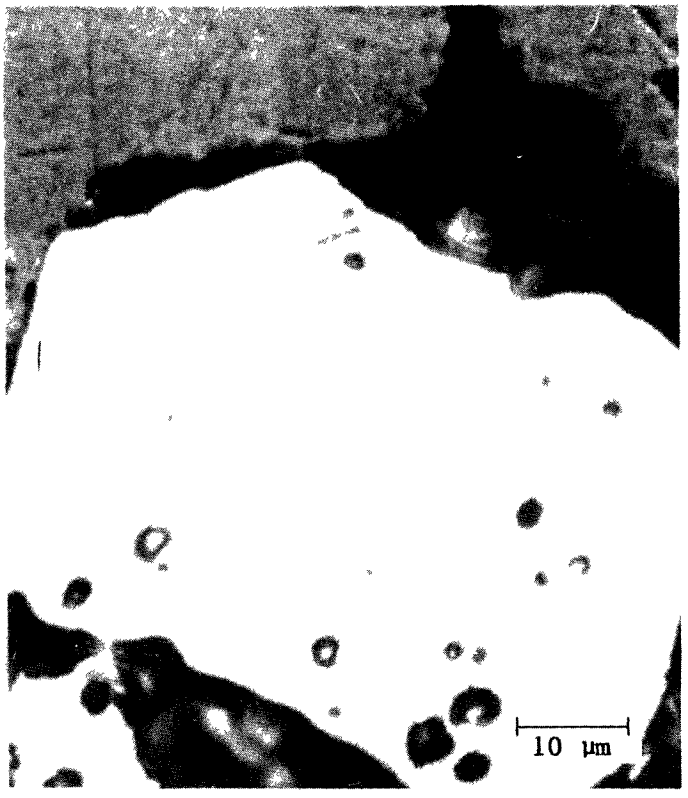


FIGURE 8. Granules Sintered in He/4% H_2 Fabricated and Sintered at SRL. Note Optimum Density that Results When Granules are Sintered in an Atmosphere that Reduces and Dissolves in Plutonia.

To test the effect of granule sintering atmosphere on the characteristics of a full-scale pellet, GPHS Pellet 39 was hot pressed in the PEF from granules sintered in Ar/5% O_2 (both the 1100°C and 1600°C shards). All other process parameters were "PEF centerline" conditions.[1] GPHS Pellet 39 had a density of 83.0% theoretical density (TD) as pressed and 85.0% theoretical density (TD)after heat treatment which is typical of other centerline pellets. The pellet was uncracked as pressed and only one hair-line crack was apparent on the bottom after heat treatment, also typical of centerline pellets.

Although density of GPHS Pellet 39 was typical of that of centerline pellets, metallographic analysis indicated that granules sintered in Ar/O_2 had a significant effect on the microstructure and internal cracking of this pellet. As shown in Figure 9, the cross section of the pellet revealed considerable cracking. Figure 10 compares the microstructure of a centerline PEF GPHS Pellet with GPHS Pellet 39. Apparently the integrity of the high-fired granules was retained but the low-fired granules were crushed during hot pressing in Pellet 39. This microstructure is apparently more susceptible to cracking.

The results of these granule sintering studies and pellet fabrication tests were useful in improving the quality of the early production pellets. The low initial yield of production pellets was improved by increasing the sintering temperature of the low-fired granules from 1100°C to 1150°C. This increase in temperature helped to compensate for the oxidizing atmosphere used in the production process.

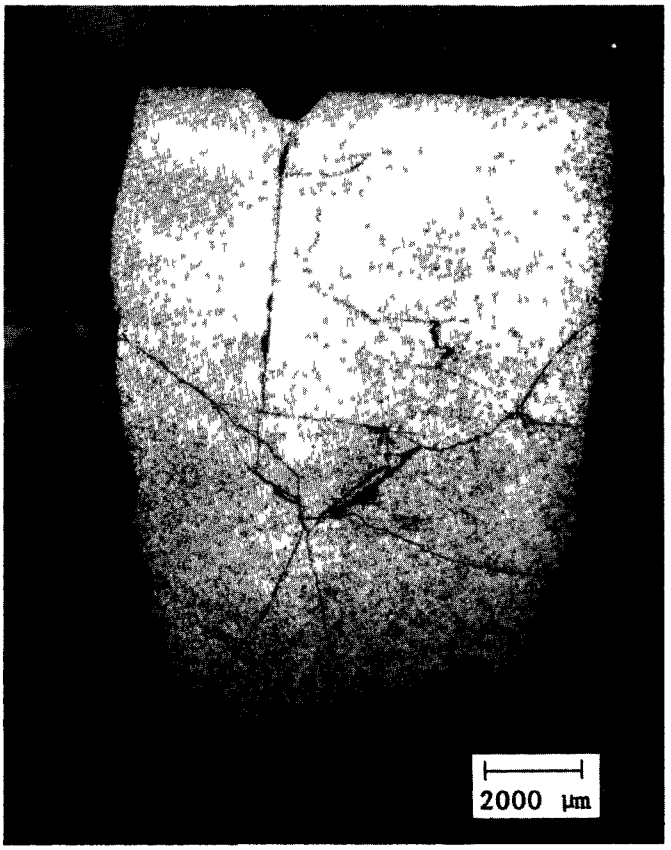
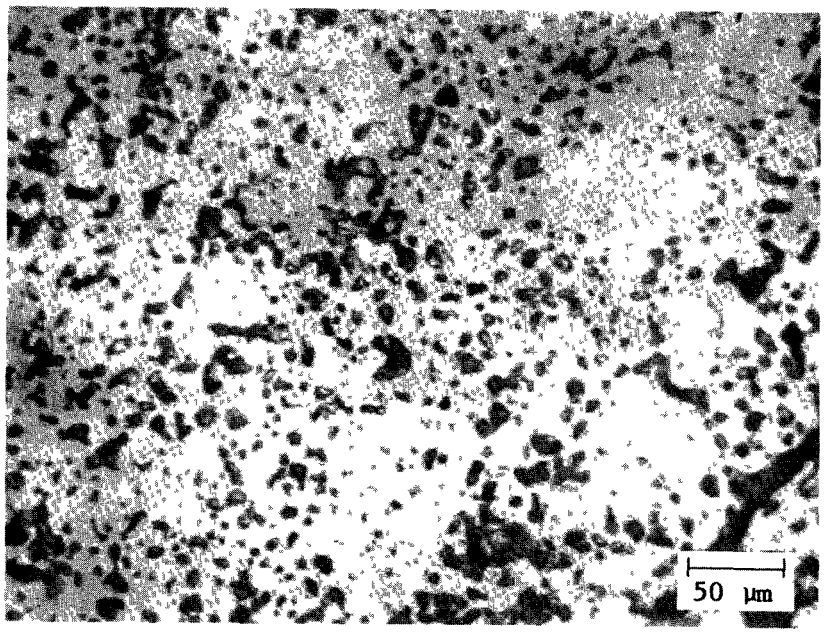
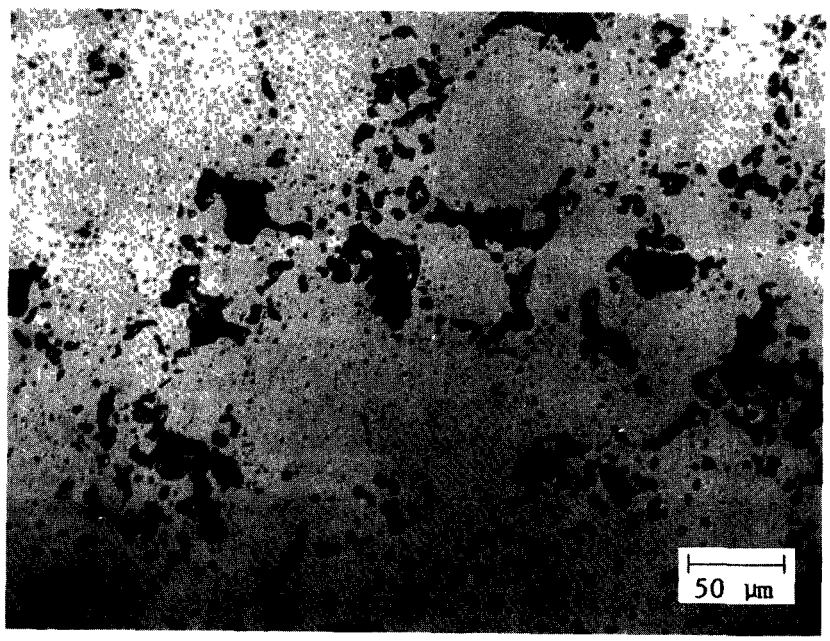


FIGURE 9. Cross Section of GPHS Pellet 39. Hot Pressed from Shards Sintered in Ar/5% O_2. Note Extensive Cracking.



GPHS Pellet 39
(Granules Sintered in Ar/5% O_2)



GPHS Pellet 32
(Granules Sintered in Ar)

FIGURE 10. Effect of Granule Sintering Atmosphere on Microstructure of Pellets

GPHS PROCESS IMPROVEMENTS

FULL-SCALE DEMONSTRATION OF A DIRECT FABRICATION PROCESS

Summary

An acceptable feed material for the direct fabrication of $^{238}PuO_2$ fuel was produced in the SRP HB-Line using a Pu(III)-oxalate direct-strike precipitation technique that was developed at SRL. This feed material was hot pressed to form an integral GPHS fuel pellet. This direct fabrication process will eliminate a number of powder conditioning steps and will therefore be a safer and simpler process for producing $^{238}PuO_2$ fuel forms. The entire direct fabrication process has been demonstrated successfully, but more development work will be necessary to establish centerline and process limit conditions, to perform impact verification tests, and to complete the required documentation before direct fabrication can be implemented as a production process.

Background

A two-step Pu(III) direct-strike technique was developed at SRL to precipitate rosette-shaped crystals of PuO_2 with a diameter of 40 to 50 μm.[3] This feed was precipitated in relatively small (1-g and 5-g) batches and characterized by scanning electron microscopy to establish centerline precipitation conditions and many of the process limits.[1] A small-scale PuO_2 pellet was hot pressed using feed from these precipitation tests to demonstrate that an integral pellet with an acceptable microstructure could be fabricated from Pu(III) direct-strike feed.[3] A $^{239}PuO_2$ feed material from JB-Line with a size and morphology similar to Pu(III) direct-strike feed was used as a simulant and hot pressed in the PEF to demonstrate that a full-scale GPHS pellet could be fabricated from a feed material having rosette morphology.[11]

Results

HB-Line Precipitations

The centerline precipitation conditions,[1] which called for a two-step approach (Table 1), established in the small-scale SRL tests were used for two 100-g Pu(III) direct-strike precipitations (Run 101 HA 217 and 101 HA 218) in the SRP HB-Line. These

precipitation tests were carried out during January 1981 with a process cooling water temperature of 21.5°C. (Results of the small-scale tests[3] indicated that a precipitation temperature of <25°C was required to produce the desired particle morphology.) SEM analysis showed that feed from these full-scale precipitations consisted of 30 to 50-μm-diameter rosettes which were very similar to the SRL feed as shown by Figure 11. (These particles were too large for quantitative particle size measurements using the Coulter Counter technique employed for standard $^{238}PuO_2$ feed.)

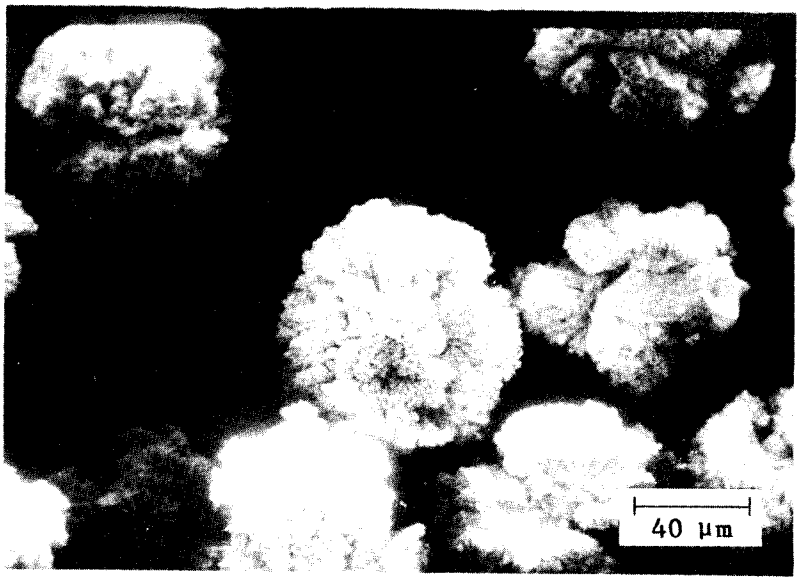
Two additional 100-g precipitations (Runs 101 HA 219 and 101 HA 220) performed in HB-Line using a one-step Pu(III)-oxalate direct-strike method (see Table 2) were not as successful as the two-step approach; however, laboratory-scale tests had demonstrated that this one-step precipitation technique also produced 40 to 50-μm rosettes (Figure 12). One of the Plant tests produced very fine particles (Run No. 101-HA-219) and the other test (Run No. 101-HA-220) produced some rosette-shaped crystals but also included a large volume of relatively small particles (Figure 13).

A second series of Plant precipitation tests was requested by SRL. This series of tests was carried out in HB-Line during March 1981 with a cooling water temperature of ~20°C. The two-step Pu(III) direct strike procedure was used to precipitate six 100-g batches (Runs No. 103-HA-248, 103-HA-249, and 103-HA-250) of feed for the development of a direct fabrication process. SEM analysis indicated that all three precipitation runs produced an acceptable feed (Figure 14).

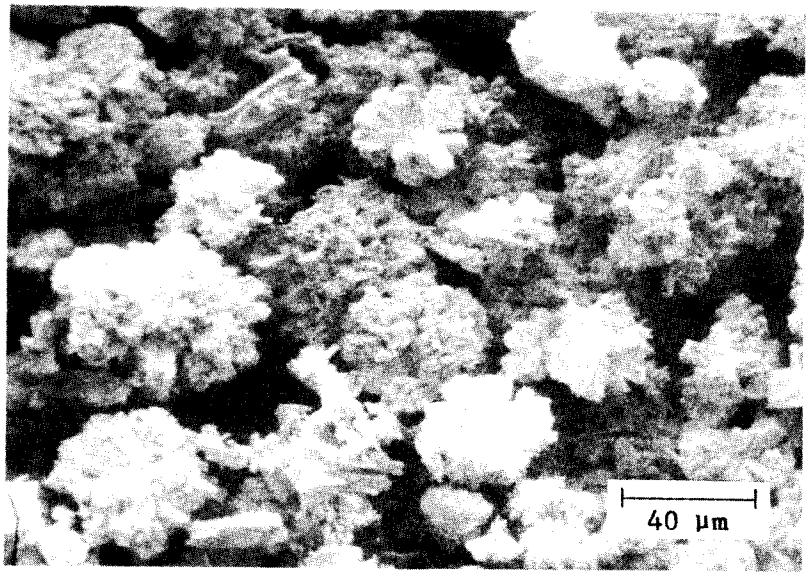
TABLE 1

Two-Step Pu(III) Process

1. Feed Adjustment
   - 1.2 ±0.2M HNO_3
   - 5 ±2 g Pu/L
   - 0.5M $N_2H_5NO_3$
   - 0.05M Ascorbic Acid
2. Transfer Adjusted Feed to Precipitator
3. Oxalic Acid Addition to Precipitator
   - 23° ±3°C
   - Oxalic Acid Adjustment No. 1 to $\frac{[H_2C_2O_4]}{[H^+]^3} = 0.011 \pm 0.002$ (Addition Time 10 Minutes)
   - Digest for 20 Minutes with Mixing
   - Oxalic Acid Adjustment No. 2 to 0.22 ±0.002M Oxalate in Supernate (Addition Time 20 Minutes)
   - Mix 5 Minutes (After Oxalic Acid Addition Complete)
4. Filter
5. Cake Wash (Technical Standard)
6. Calcine (Technical Standard)



SRL



SRP HB-Line

FIGURE 11. Pu(III) Direct-Strike Feed. Rosettes Produced by Two-Step Technique

TABLE 2

Flowsheet for Direct-Strike Pu(III) Oxalate Precipitation (One-Step Procedure)

1. Feed Adjustment

   1.2 ±0.2 M HNO_3

   5 ±2 g Pu/L

   0.05M $N_2H_5NO_3$

   0.05M Ascorbic Acid

2. Transfer Adjusted Feed to Precipitator

3. Add Oxalic Acid Continuously to Precipitator to 0.22 ±0.02M Oxalate in Supernate

   Temperature <25°C

   Addition Time 50-60 Minutes

   Mix 5 Minutes (After Oxalic Acid Addition Complete)

4. Filter

5. Cake Wash (Technical Standard)

6. Calcine (Technical Standard)

Pellet Fabrication Tests

Feed from the first two two-step, 100-g plant precipitations (Runs 101-HA-217 and 101-HA-218) was used to demonstrate the direct fabrication process in the PEF. A blend of 40 wt % rosettes sintered for 6 hr at 1600°C in He/H_2O and 60% as-calcined rosettes was hot pressed in a graphite die to form DF Pellet 3. A load of 7000 lb (compared to 2600 lb for the GPHS process)[1] was applied using a slow preload followed by a six-minute parabolic load ramp. Die closure was completed two minutes after the maximum load was applied and at a temperature of 1540°C.

DF Pellet 3 was easily ejected from the die and was uncracked as pressed. After heat treatment in oxygen the pellet had minor hairline cracks in both ends (Figure 15a) similar to the cracks observed in most GPHS pellets fabricated in the PEF. DF Pellet 3 had a final density of 85.7% TD and a linear shrinkage of 0.6% during heat treatment. This shrinkage is only slightly greater than that typically observed in GPHS pellets and may be reduced if the percent fines in the feed can be reduced by controlling the precipitation parameters.

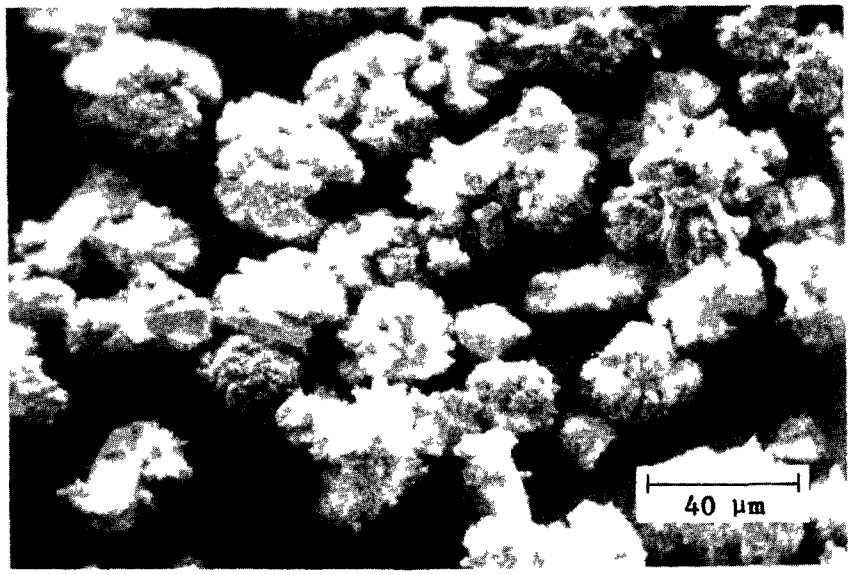
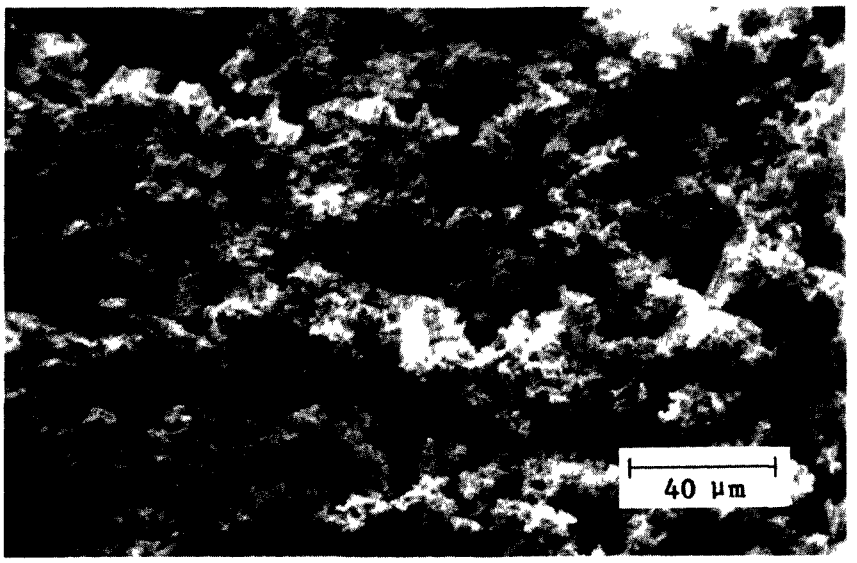
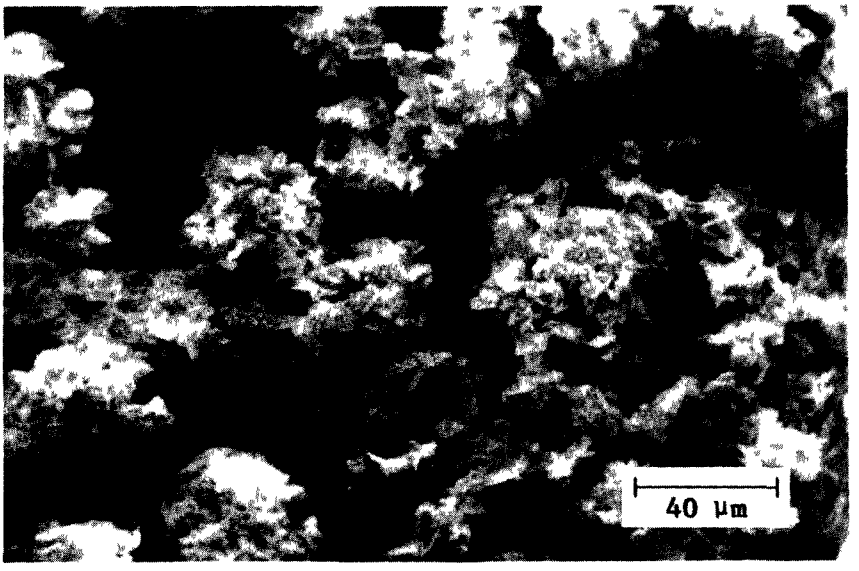


FIGURE 12. Pu(III) Direct-Strike Feed. Rosettes Produced by One-Step Technique at SRL

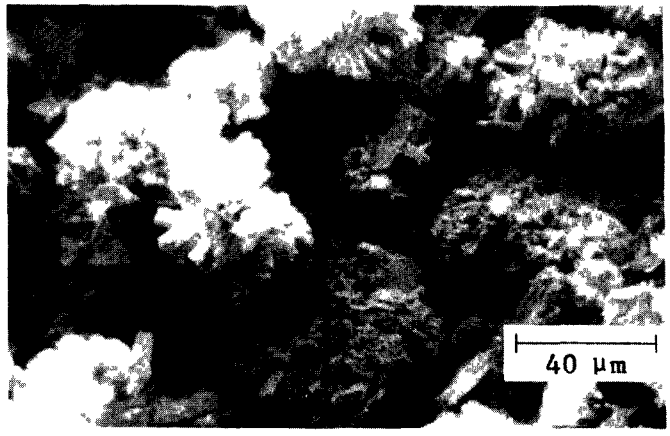


Run No. 101-HA-219

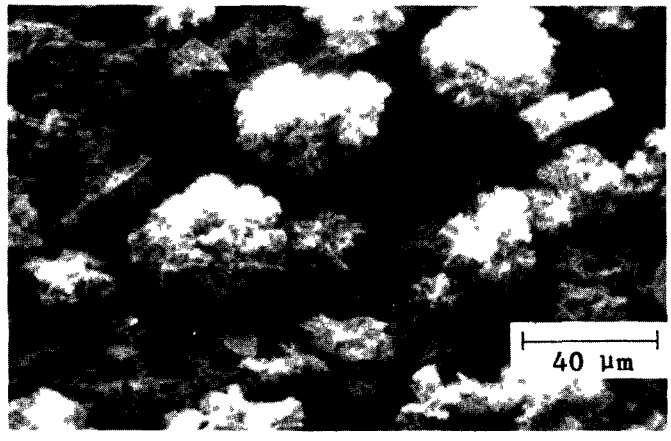


Run No. 101-HA-220

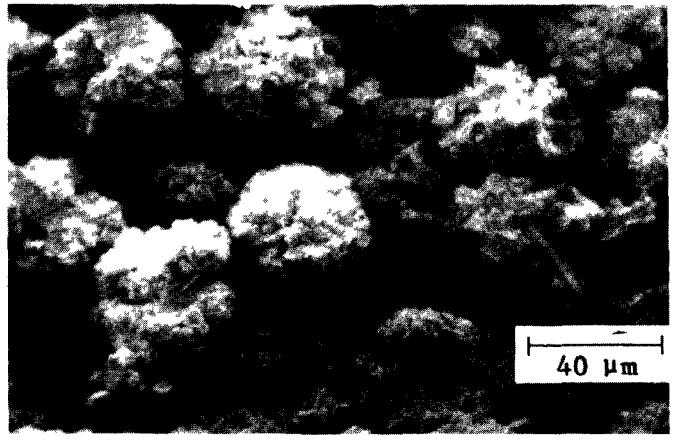
FIGURE 13. Pu(III) Direct-Strike Feed Produced in HB-Line by One-Step Technique



Run No. 103-HA-248

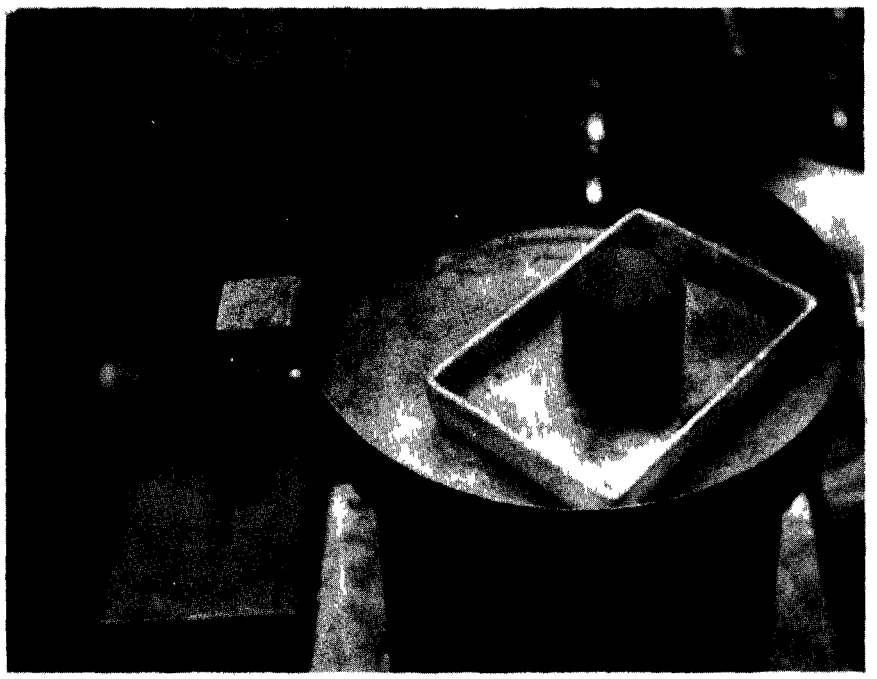


Run No. 103-HA-249

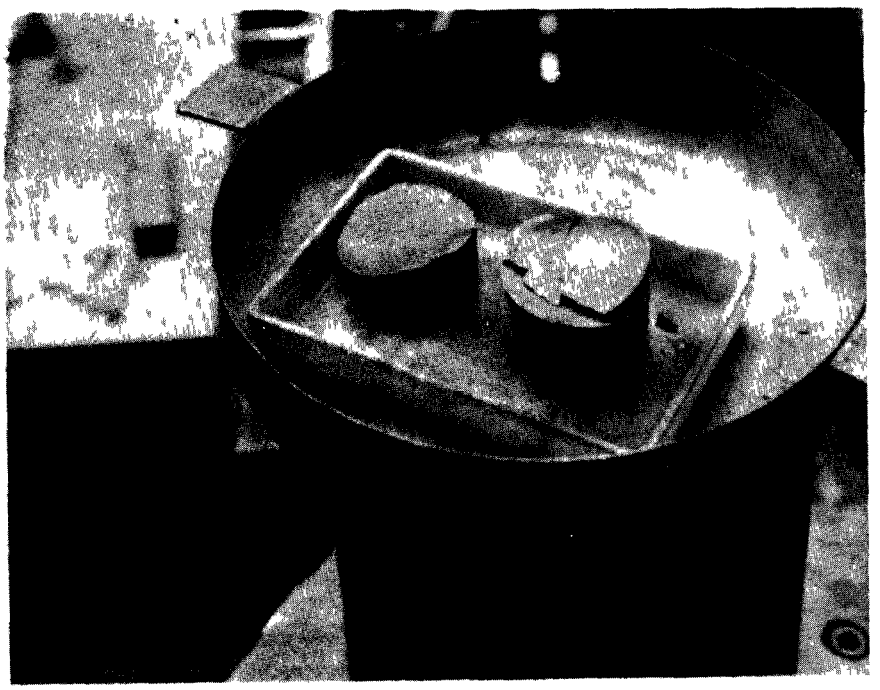


Run No. 203-HA-250

FIGURE 14. Pu(III) Direct-Strike Feed Produced in HB-Line by Two-Step Technique



a. Heat-Treated Pellet



b. Sectioned Pellet Showing Cleavage Surfaces

FIGURE 15. DF Pellet 3

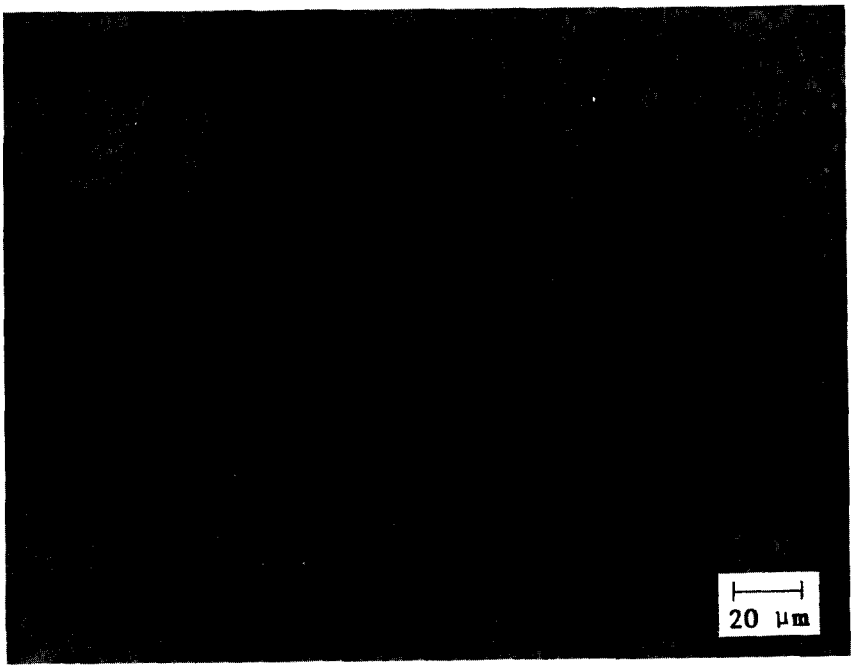
The pellet was sectioned with an Isomet* diamond for metallographic examination. After sectioning, the piece of pellet held by the clamp sheared along a plane perpendicular to the pressing direction, (Figure 15b) probably along a pressing lamination. Future development work may be able to reduce the load required for closure, thereby reducing the likelihood of pressing laminations.

Microstructural analysis of three sections of DF Pellet 3 indicated that the pellet had the desired granular-type microstructure with many pores in the 10 to 15-μm range (Figure 16). These relatively large pores in a matrix of grains with an average size of <10 μm give to pellet some microstructural and dimensional stability at high temperatures. Two of the sections were uncracked and the third section was cracked during sample preparation. At a magnification of about 3x greater, the direct fabrication microstructure is similar to the GPHS microstructure (Figure 17).

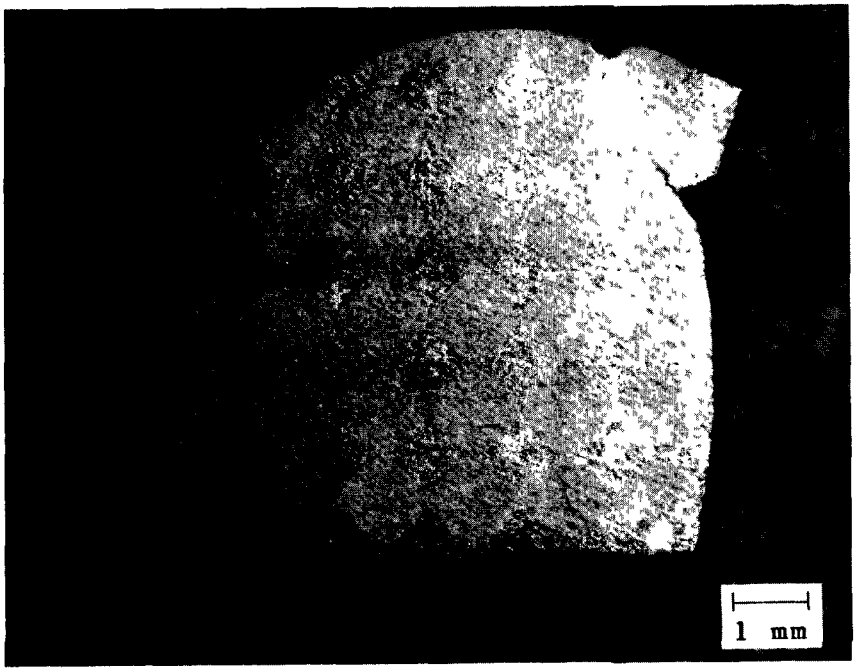
Program

Test results obtained so far are very encouraging but, a major amount of work remains before a direct-fabrication production process can be implemented at SRP. A number of process parameters must be optimized to determine the centerline precipitation and pellet fabrication conditions. New instrumentation must be selected, purchased, and installed in a containment facility to measure particles in the size range 0.1 to 100 μm. Additional small-scale precipitation tests will be carried out to determine the process limits at 16°C. This is the minimum cooling water temperature which can be obtained in HB-Line if the chilled water lines are rerouted to provide cooling for low-temperature precipitation. Small-scale tests have indicated that lower precipitation temperatures improve the morphology (more rosettes, fewer fines) of Pu(III) direct-strike feed. After these small-scale tests are complete and the chilled water lines are rerouted, continuity tests in HB-Line to produce at least 2 kg of $^{238}PuO_2$ direct fabrication feed will be required to complete the demonstration of the precipitation process and provide feed for pellet fabrication tests. Pellet fabrication tests and microstructural analysis will be required to define centerline and process limit conditions. Documentation, including technical standards and a technical manual including technical standards and a technical manual will be required before the direct fabrication process can be used for production at SRP. Design verification impact tests and other evaluations must be carried out to verify the acceptability of direct-fabrication pellets for flight use. Finally, DOE approval must be obtained prior to implementation of this process at SRP.

* Beuhler Ltd., 2120 Greenwood St., Evanston, IL 60204

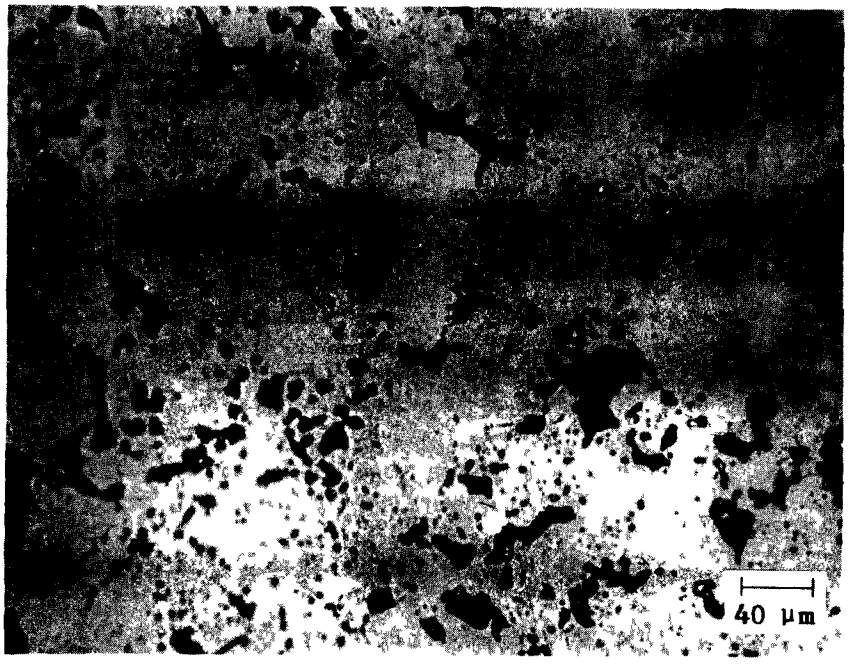


Microstructure (Grain-Boundary Etch)

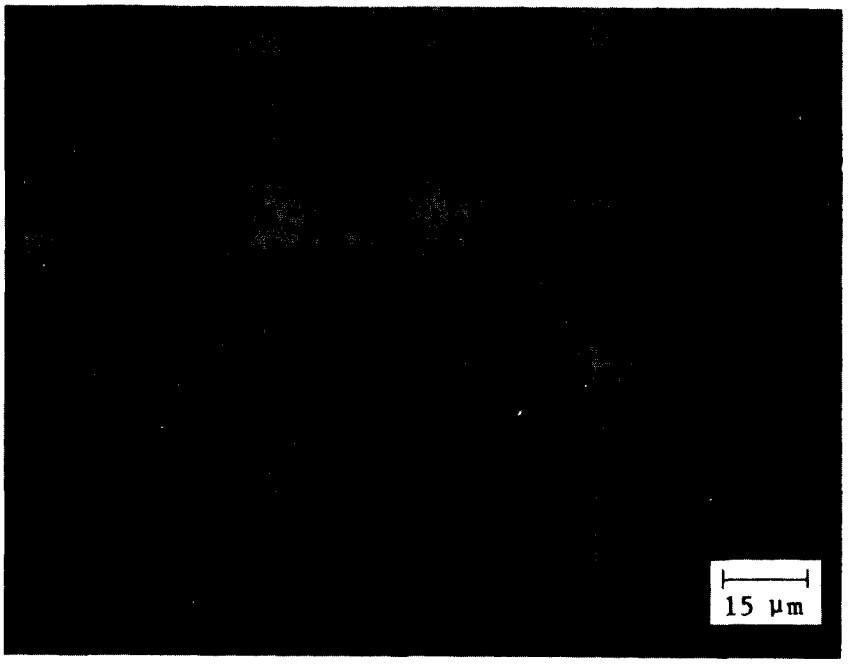


Cross Section

FIGURE 16. Microstructure of DF Pellet 3



GPHS



DF

FIGURE 17. Comparison of Microstructures of DF and GPHS Pellets. Note Different Magnifications.

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