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PREPARATION OF HIGH-DENSITY OXIDES AND VIBRATORY COMPACTION IN FUEL TUBES

S. D. Clinton
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CHEMICAL TECHNOLOGY DIVISION
and
METALLURGY DIVISION

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ABSTRACT

Initial experimental studies of simplified technologies for recycle of fuel and fertile materials to heterogeneous reactors have demonstrated chemical preparation of high-density oxides and vibratory compaction to give high densities in cladding tubes. Preparation of thorium or thorium-uranium sols followed by careful drying and calcination at 1200°C gave oxides with densities 98 to 99.4% of theoretical. Vibratory compaction by either pneumatic or electronic vibrators of mixed sized fractions of powders gave ThO_2 or ThO_2-UO_2 densities in stainless steel tubes up to 89% of theoretical. These procedures should decrease costs for direct fabrication and should permit remote preparation and compaction of oxide powders into fuel elements that have been assembled with empty pins.

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1.0 INTRODUCTION

Initial experimental studies directed at simplified processes for preparing high-density thorium oxide, uranium oxide, or mixed oxides and simplified procedures for fabricating these oxides into high-density metal-clad fuel element shapes are reported. This is part of an over-all study being made of the technology of recycling fuel and fertile materials to heterogeneous reactors. Development of fuel preparation and fabrication procedures adaptable to operation behind shielding, in addition to complete containment, is desirable for the thorium—U-233 fuel cycle because of the presence of U-232 and Th-228 decay chains in recycled materials. Shielding thicknesses of up to 2 ft of concrete may be necessary for parts of a U-233 fuel-fabrication plant.

Procedures now applied to the production of heterogeneous reactor fuel elements are complicated and costly. For this study, vibratory compaction of oxide particles into cladding tubes was selected as a promising fabrication procedure; this has been only briefly reported for UO_2 and has not previously been tested for ThO_2. With this method fuel elements could be assembled with empty pins by ordinary direct fabrication and inspection procedures and then be remotely filled by oxide powders vibratorily compacted to the required densities. This procedure will require development of techniques for remotely making and inspecting the final closures.

The authors acknowledge assistance from a number of sources, particularly from Gerald Doughty and other members of the Redstone Arsenal Environmental Test Section; H. G. Marsh and G. R. Cole of the Reactor Materials Division at the Savannah River Project; and G. R. Wilson's group in the ORNL Analytical Chemistry Division.

2.0 PREPARATION OF HIGH-DENSITY FUEL MATERIALS

Fabrication of satisfactory fuel elements by vibratory compaction can be effective only with fuel materials of near theoretical density, of proper particle size distribution, and with sufficient particle strength to maintain their size until compacted into tubes. Such a material can be made by arc melting of oxide. However, this is a difficult process to contain and operate remotely. Chemical methods are being developed which are promising for production of thoria, thoria-urania, and urania particles of near theoretical density and of controlled particle size and which are adaptable to remote procedures.

2.1 Chemical Preparation of Dense Thoria Particles

A procedure for preparing dense thorium or uranium-thorium oxide particles, which can be vibratory-compacted to a high density, is outlined in Fig. 2.1. The thorium oxide feed may be simply prepared by the thermal

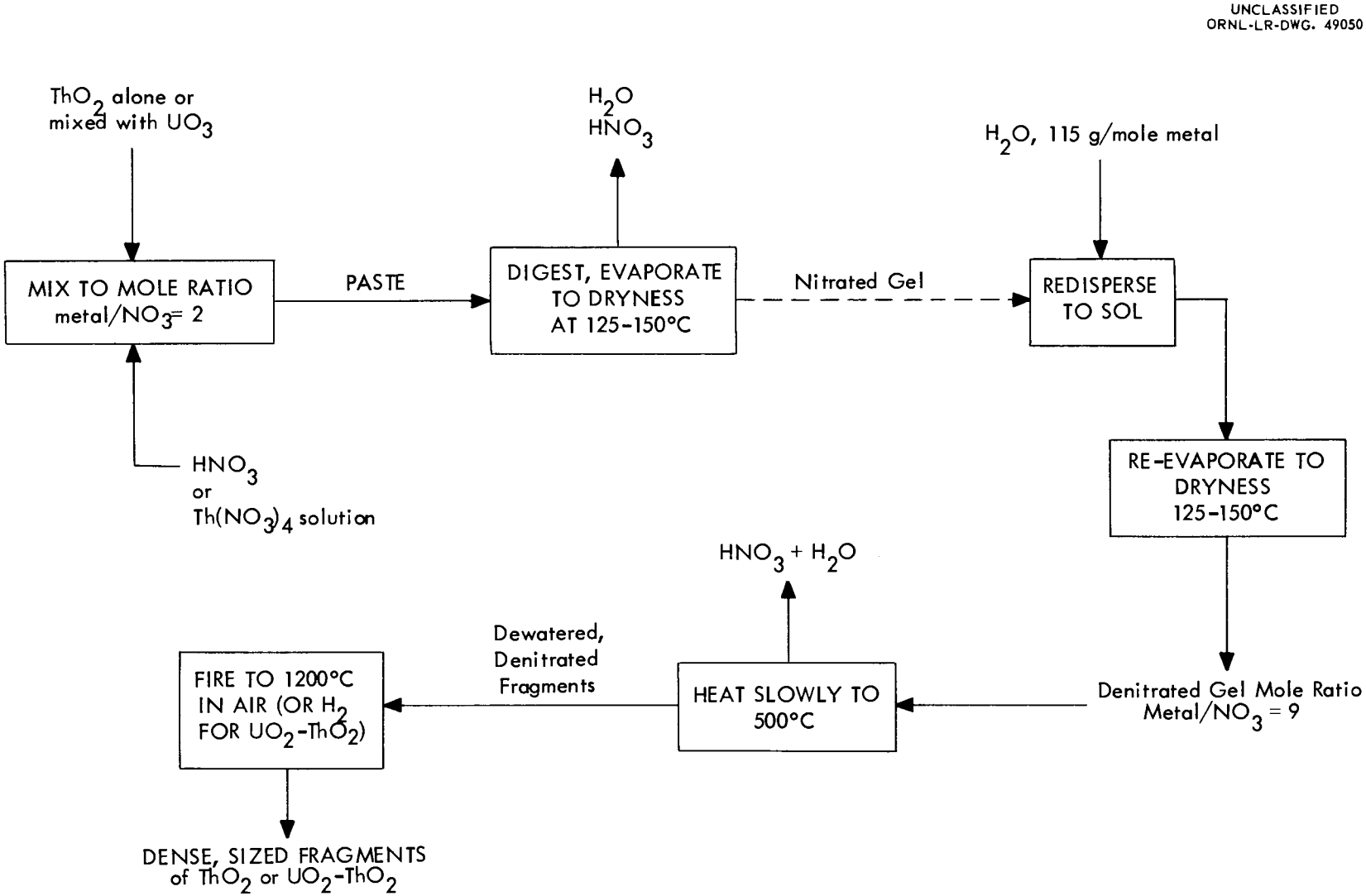

Fig. 2.1. Preparation of dense, sized fragments of ThO_2 or UO_2-ThO_2 by the sol-gel route.

denitration of thorium nitrate or mixed nitrates with superheated steam. Thorium oxide is dispersed by treatment with nitric acid or thorium nitrate followed by evaporation to dryness at 125-150°C. The resulting glassy cake is then easily dispersed by water to a bluish-white sol. The nitrate content of the cake is further reduced by repeated reslurryings and reevaporations to yield a material containing ~9 moles ThO_2/mole nitrate. The dense vitreous-appearing cake can then be heated slowly to 500°C to give dehydrated, denitrated fragments of thorium oxide with a particle size of a few millimeters. This material can then be densified by a final calcination at 1200°C to produce particles with a density approaching 99% of theoretical.

The initial thorium oxides used in successful experiments were prepared by precipitating the oxalate from thorium nitrate solutions and calcining the washed oxalates at 650 or 800°C. Two oxides calcined at 650°C had well-ordered relic structures of 1.0 and 2.3 μ average particle size while a third, fired at 800°C, had poorly ordered relics of 7 μ average size. When these oxides were treated with nitric acid or thorium nitrate the relic particles were broken down to the ultimate crystallites, or small multiples thereof (Fig. 2.2). The crystallite size was unchanged by treatment with nitrate and remained the same through the 500°C firing (Table 2.1). The 1200°C calcination increased the crystallite size to 500-1300 Å.

Table 2.1. Dispersion and Growth of ThO_2 Crystallites in Sol-Gel Procedure

Thorium Oxide Used in Gel Preparation	Crystallite Size, A			
	Initial ThO_2	After Evaporation with $Th(NO_3)_4$ at 125-150°C	Intermediate Calcination at 650-850°C	After Calcination at 1200°C
Well crystallized, 650°C-fired, 4 hr	120	120	150	500
Disordered, 800°C-fired, 2 hr	190	190	-	1300
Steam denitrated at 400°C	60	-	80	-

After the nitrate has been reduced to about 9 moles of thorium per mole of nitrate, the material may be calcined.

Slow approach (100°C per hour) to 500°C removed enough of the remaining water and nitrogen to prevent decrepitation to small sizes in the final firing step. Too rapid temperature increase caused decrepitation or crazing

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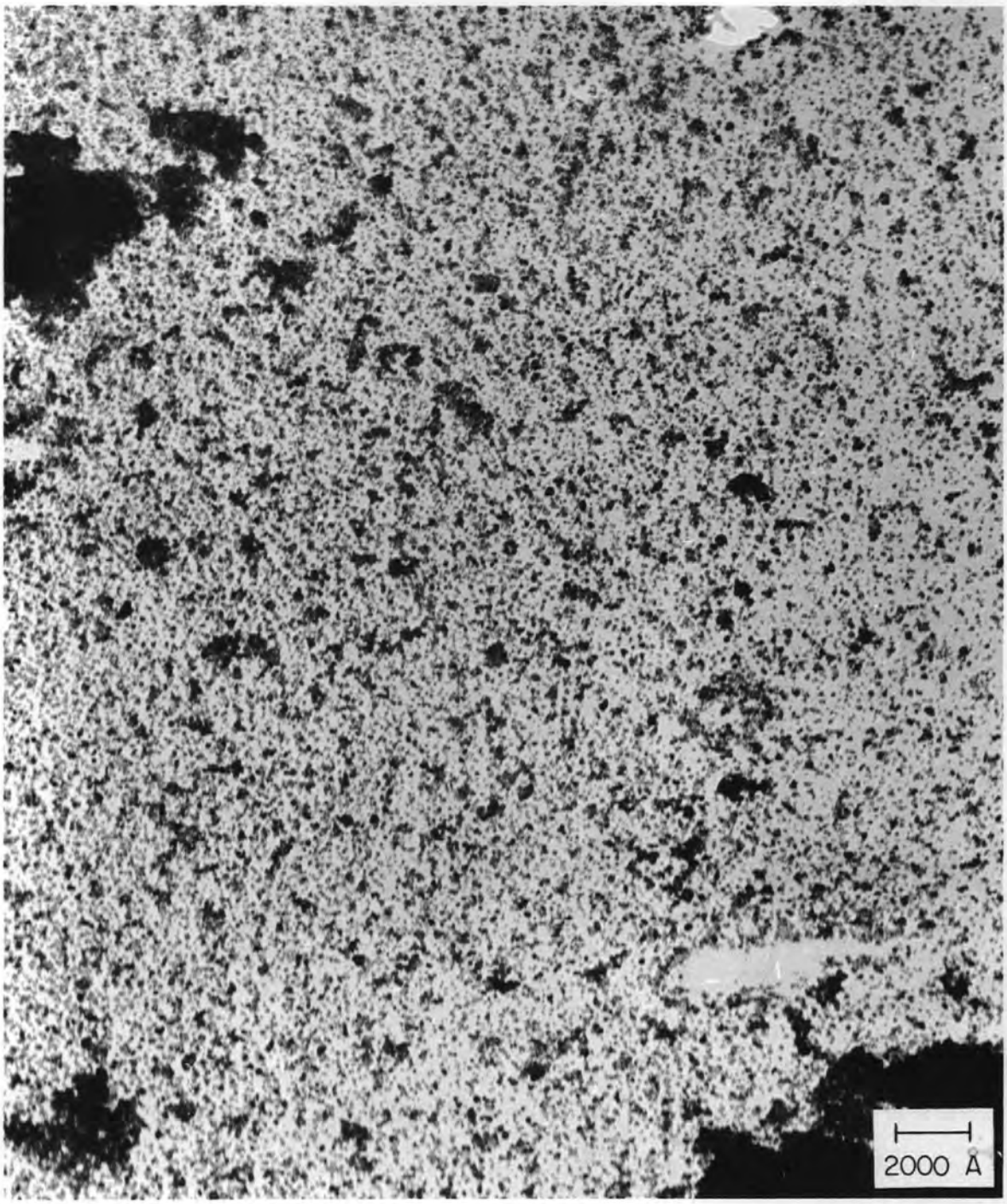

Fig. 2.2. Dispersion of ThO_2 relics in 2 M $Th(NO_3)_4$ solution. The smallest particles shown are about 100 A.

with consequent weakness of particles. After the slow firing to 500°C, the material may be densified by raising the temperature to 1200°C. Figure 2.3 shows thoria fragments >99% of theoretical density and ~1.4 mm size.

With the procedure described above, 300-g batches of thoria were prepared of particle density varying from 9.91 to 9.97 g/cc. One batch had the following particle size distribution:

Percent	Size Range, US Standard Mesh
60	10-16
25	80-100
15	-325

The tap density of this preparation was 7.95 g/cc, and the packed density after vibration of a selected size distribution in a 2-ft-long 3/8-in.-dia stainless steel tube with an air vibrator was 8.66 g/cc.

2.2 Chemical Preparation of Dense Thoria-Urania Particles

The procedures discussed above were used with only minor modifications to prepare high-density urania-thoria fuel particles of controlled size. Solid uranium trioxide dihydrate sufficient to give the desired U/Th ratio (e.g., ~0.05) was added to the ThO_2 suspension in 2 M $Th(NO_3)_4$ solutions prior to evaporation, or to any reslurrying step prior to calcination to 500°C. A deep red sol formed when water was added to the glassy cake, and evaporation of the sol gave a uniformly deep red glassy solid. Three preparations were made, one of which involved more than 200 g of oxide.

A small sample of cake that had been evaporated to ~150°C was slowly heated in air to 850°C. The red, glassy transparent appearance was retained, and the product had a particle density of 9.8 g/cc. However, when it was heated to 1200°C for 4 hr in air, the density decreased to 9.7 g/cc, and the particles changed to gray, opaque masses but did not decrepitate. Heating the red, glassy cake to 1230°C for 4 hr in a hydrogen atmosphere gave green, vitreous particles with a density of 9.76 g/cc, 97.0% of theoretical for a 5 mole % UO_2-ThO_2 solid solution. Mixed oxides prepared as above, air-fired to 1300°C for 4 hr, had a bulk density, after vibratory compaction of 8.23 g/cc, 82% of theoretical. Fragments of this type of mixed oxides are shown in Fig. 2.4.

In a modified procedure, hydrated uranium trioxide was dissolved in 2 M $Th(NO_3)_4$ to give a mixed solution with a U/Th atom ratio of 5/95. The solution was denitrated by direct evaporation until the yellow residue was syrupy and at a temperature of ~150°C. Six successive resuspensions in water and evaporations to increasingly high residue temperatures up to 280°C produced sols of increasingly deep red color and solids of increasingly glassy character. From the seventh evaporation, a dilute (31 g in

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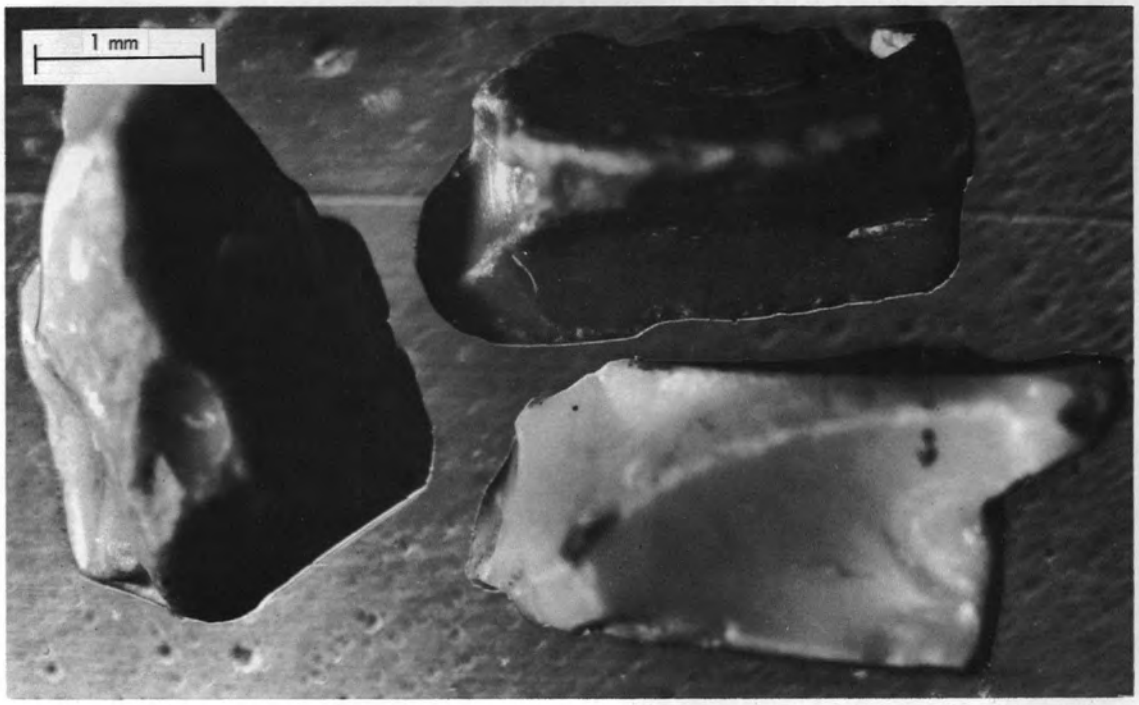

Fig. 2.3 Millimeter-size thoria gel fragments calcined at 1200°C.

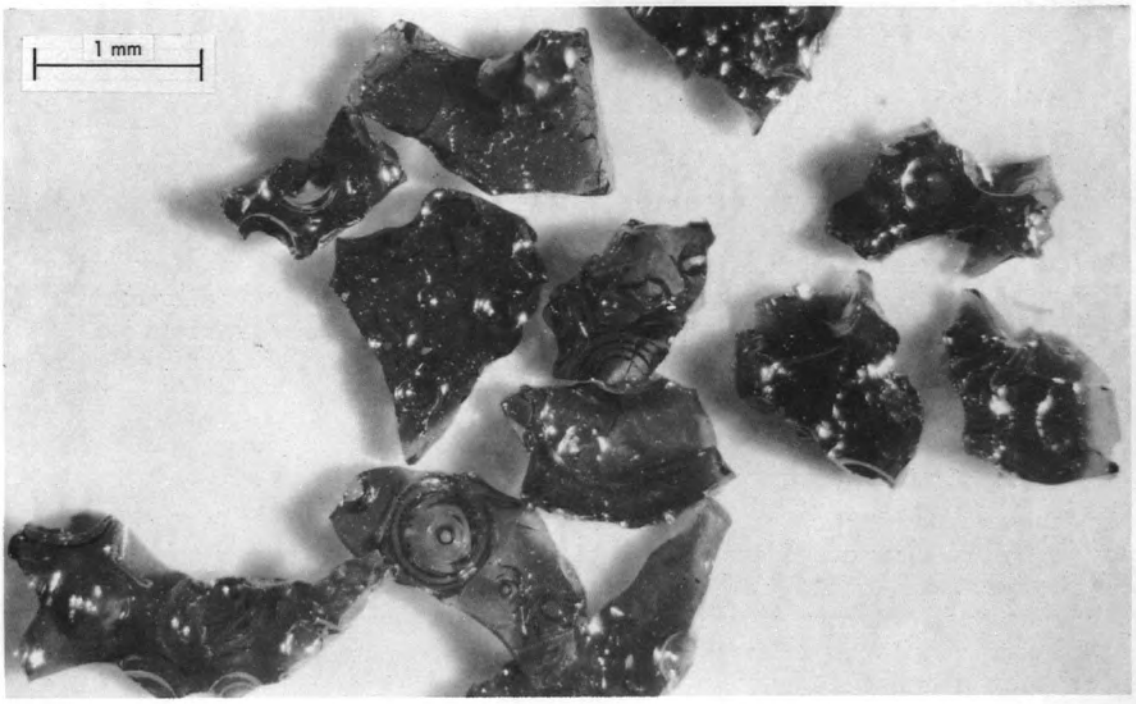

Fig. 2.4 Millimeter-size thorium uranium oxide gel fragments calcined at 1300°C.

4 liters of water) sol was prepared and reevaporated. The residue after heating to 500°C was hydrogen-fired at 1260°C for 4 hr to a particle density of 9.9 g/cc. This material was vibratory compacted to a bulk density of 8.3 g/cc.

2.3 Chemical Preparation of Dense Urania Particles

The problem of preparation of dense particles of UO_2 is complicated by the variety of valence states present in the uranium-oxygen system. Efforts to prepare dense glassy oxides from UO_3 and uranyl nitrate solution or nitric acid have, to date, resulted in failure.

One preparation which showed promise for behavior similar to that of the thoria-nitrate system was one in which efforts were made to retain the U(IV) oxidation state. Uranous oxalate was thermally decomposed under an unpurified argon blanket. The residue, containing some free carbon, was peptized with HCl solution (Cl/U mole ratio = 1) and evaporated under argon to a black glassy solid. The solid was reslurried with deionized water and reevaporated under argon three times to decrease the chloride content. The final evaporation was carried to 500°C under argon. After cooling to room temperature, the black glassy solid was transferred to a hydrogen furnace at 1200°C. An immediate evolution of white fumes indicated that the HCl had not been completely expelled at 500°C. After firing at 1200°C for 4 hr and cooling under hydrogen, the density of the nonglassy solid was measured pycnometrically, using toluene, and found to be 10.2 g/cc, 93% of the theoretical 10.97 g/cc. It is thought that the residual carbon or the initial evolution of gases at 1200°C introduced some porosity, which accounted for the comparatively low density of the final product.

3.0 VIBRATION TO GIVE HIGH COMPACTED DENSITIES

Arc fused thorium-uranium oxide was crushed, sized, and vibrated to bulk densities of 85 to 89% of the theoretical crystallite density (Table 3.1). These bulk densities were achieved in stainless steel tubes, 2 ft long, which were vibrated 10 min with a NAVCO pneumatic vibrator (1.25-in.-dia piston, single impact type). Many vibration variables must be explored in greater detail before an optimum pneumatic vibrating procedure can be selected for loading reactor tube bundles.

3.1 Effects of Equipment, Materials, and Procedures

Of the several types of pneumatic vibrators tested, the NAVCO "Bin Hopper" 1.25-in.-dia model with a single impacting piston produced the highest compacted densities. The success of this vibrator may be attributed directly to the one-directional impact of the piston. A BH 2-in. vibrator, air cushioned at both ends of the piston housing, gave ~10% lower compacted densities.

Table 3.1. Effects of Equipment and Materials on the Vibrated Bulk Density

Standard Conditions

Vibrator: BH - 1 1/4-in. single-impacting piston with impact on the upward stroke

Tube holder: Swagelok fitting (Fig. 3.1)

Tube: 2-ft-long 3/8-in. S.S. tubing, 35-mil wall

Oxide powder: fused ThO_2-UO_2 (3.4 wt % UO_2); 60% -10+16 mesh, 15% -70+100 mesh, and 25% -200 mesh

Parameter Varied from Standard Conditions	Vibrated Bulk Density, % of theoretical
None (Standard Conditions)	89
Vibrator: BH - 1 1/4-in. (piston impact on the downward stroke)	86
Vibrator: BH - 2-in. (piston air-cushioned at both ends of housing)	79
Tube holder: tube rigidly supported its entire length	84
Tube: 8 ft long, 3/8-in. S.S. tubing	87
Tube: 8 ft long, 3/8-in.-dia aluminum tubing	86
Tube: 6 ft long, 3/4-in.-dia Zircaloy-2 tubing	84
Tube: 2 ft long, 3/8-in. lucite tubing	81
Oxide powder: fused ThO_2	87
Oxide powder: hydrothermally denitrated ThO_2	87
Oxide powder: hydrothermally denitrated ThO_2-UO_2 (5.0 wt % UO_2)	85

The pneumatic vibrators were mounted beneath a spring-suspended base plate which also held the tube to be vibrated (Fig. 3.1). By inverting the piston in the vibrator, the impact force was imparted upward or downward with respect to the vibrating tube. Allowing the piston to strike the vibrator housing on the upward stroke produced a bulk density increase of 2-3% over that obtained when the piston delivered a downward impact.

The tube to be vibrated was mounted on the base plate by a Swagelok tubing-to-pipe connector. Originally the tubes were secured to the base plate by a holder which rigidly supported the entire tube length (Fig. 3.1). By using a Swagelok connection which fastened only the bottom end of the tube, acceleration measurements, taken from an M-B velocity pickup mounted on the top (free end) of a 2-ft tube, were 1.5 to 3 times those obtained from a similar tube clamped along its entire length. These increased accelerations were probably due to resonance effects in the tube. With the effective higher G level in the tube, vibratory compacted oxide densities were increased 3-5%.

The majority of the vibratory compaction runs were made in 3/8-in.-dia stainless steel tubes, 2 ft long, with 35 mils wall thickness. In this tube geometry, with 200-g batches of mixed-oxide powder, bulk densities up to 9.0 g/cm^3 were achieved with pneumatic vibration. In one experiment mixed oxide was compacted with pneumatic vibration into aluminum tubes 8 ft long x 3/8 in. dia to 8.7 g/cm^3, stainless steel tubes 8 ft x 3/8 in. dia to 8.8 g/cm^3, and Zircaloy-2 tubes 6 ft x 3/4 in. dia to 8.5 g/cm^3.

With an electronic vibrator with sine wave input, Hanford has attained bulk densities of 90 to 91% of theoretical in Zircaloy-2 tubes using UO_2 Using electronic vibrators located at Huntsville, Alabama,[2] and Savannah River, South Carolina,[3] attempts to achieve Hanford's results with mixed urania-thoria oxide in stainless steel tubes were generally unsuccessful. Bulk densities obtained at Savannah River with sine wave input were 5 to 6% less than those reported by Hanford.

Four types of oxide powders were pneumatically vibrated to bulk densities greater than 85% of the theoretical or particle density (Table 3.2). The oxide powders for compaction were crushed and blended from three screened size fractions as follow: 58 wt % -10+16 mesh, 15 wt % -70+100 mesh, and 27 wt % -200 mesh. Optimum powder packing depends on many variables, but the greatest single effect is probably the coarse/fine size ratio, which should be at least of the order of 30-50. An upper limit for this ratio must exist due to tube wall effects at the coarse end of the oxide size spectrum and unexplained surface interactions when the oxide particles become too finely divided (less than 5 μ).

The most satisfactory procedure for filling the tubes was to pour the blended oxide powders through a funnel with the pneumatic vibrator running at a reduced speed. At the end of the loading operation, the oxide compacted to approximately 82% of theoretical density. Bulk densities were usually

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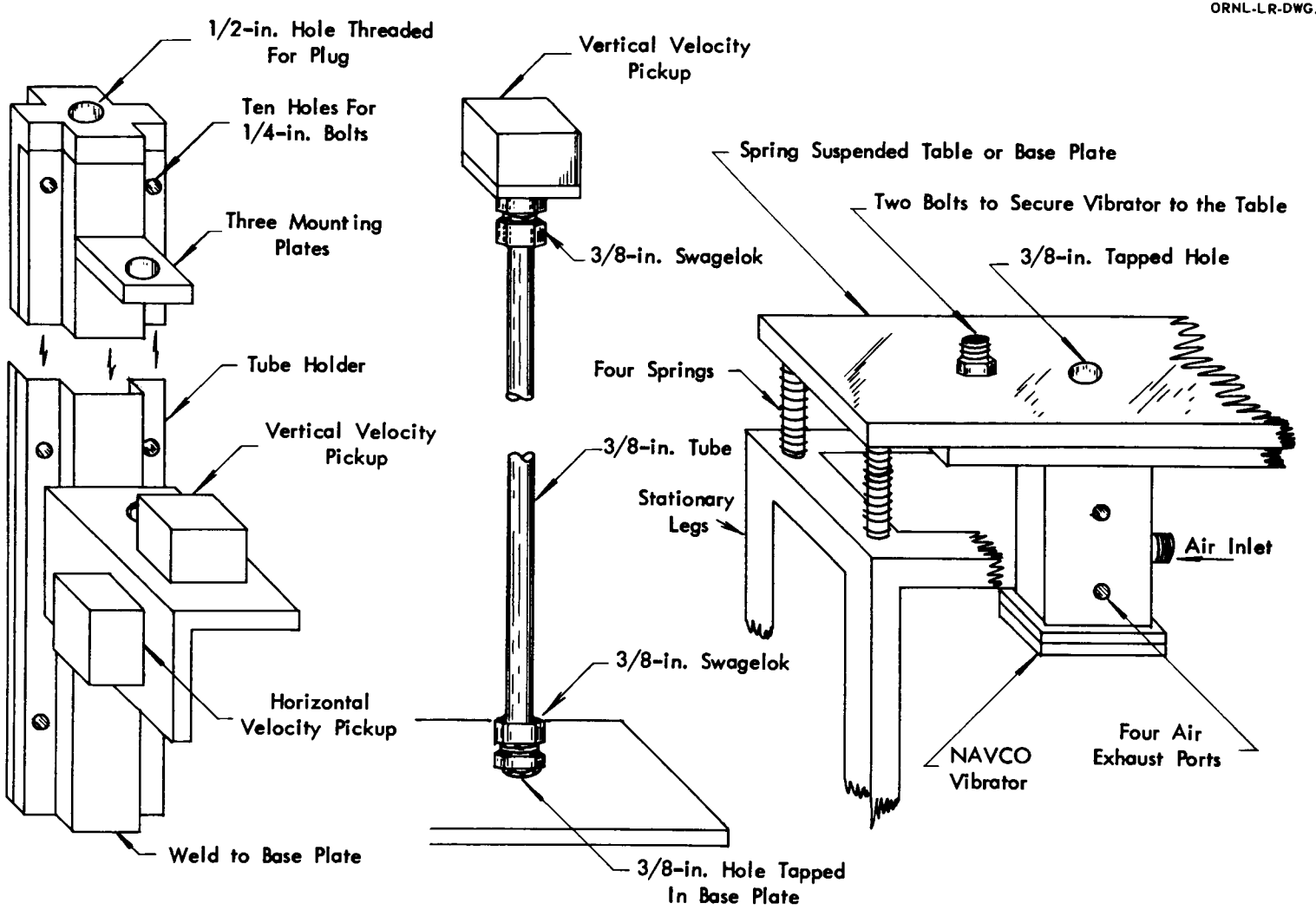

1a. Full Length Clamp

1b. Swagelok Connectors

1c. Vibrator and Table

Fig. 3.1. Vibration test geometry.

Table 3.2 Density of Oxide Powders Which Were Compacted by Pneumatic Vibration

Oxide Powders	Density, g/cc	
	Particle	Maximum Vibrated Bulk
Arc-fused ThO_2	10.0	8.7
Arc-fused ThO_2-UO_2 (3.4 wt % UO_2)	10.1	8.9-9.0
Hydrothermally denitrated ThO_2[a]	10.0	8.7
Hydrothermally denitrated ThO_2-UO_2 (5.0 wt % UO_2)	9.8	8.3

[a]One run only.

greater than 85% of theoretical after 3 to 5 min of continuous pneumatic vibration at the maximum service air pressure (about 85 psig). Blending the different powder size fractions prior to filling the tube would be expected to be a necessary step in the loading procedure; however, a bulk density of 8.5 g/cc was obtained by vibrating fused ThO_2-UO_2 with no premixing of the three different size fractions. The coarse, medium, and fine size fractions were added separately in the order listed, and apparently sufficient mixing took place in the 3/8-in.-dia tube to give a reasonably high compacted density.

3.2 Effects of Vibration Characteristics

During compaction the tube acceleration was the vibration parameter of major interest. Acceleration measurements were taken from the free end of the vibrating tube by a velocity pickup. As previously stated, the highest densities were produced with the BH 1.25-in. vibrator, and the corresponding tube accelerations were indicated to be between 80 and 150 G. The tube (3/8 in. o.d., 2 ft long) and oxide powder weighed approximately 0.8 lb, and the vibrator housing plus mounting plate weighed 12 lb.

For compacting multiple-tube bundles, the tubes plus oxide would become a significant part of the vibrating load and may therefore require a larger vibrator to deliver the same acceleration. To obtain an idea concerning the importance of acceleration, two bundles of different design were vibratorily compacted with the BH 1.25-in. vibrator. A small, rigid bundle of seven tubes (each 1/4 in. i.d., 20 in. long), weighing about 1-1/4 lb, was loaded with 2 lb of oxide. The densities obtained ranged from 8.5 to 8.7 g/cm^3 and the average of the seven tubes was 8.60 $\pm$ 0.07 g/cm^3. An EGCR bundle consisting of seven tubes (each 3/4 in. o.d., 27 in. long) and weighing 3-1/2 lb was loaded with 15-1/2 lb of oxide. Four tubes had

dummy loads. In the three tubes containing the same oxide as in the previous work, the densities obtained were 8.46, 8.42, and 8.23 g/cm^3. It appears that as the load increases a decrease in density can be expected for a given vibrator. However, larger pneumatic vibrators are available and cost less than $100 each.

The maximum frequency of available piston pneumatic vibrators is of the order of 200 cps. Since the variation of vibration frequency is limited, a major portion of the pneumatic compaction studies was at a single frequency, between 75 and 80 cps, with the BH 1.25-in. vibrator. Pulsing the vibrating frequency between 0 and 80 cps did not increase the bulk oxide density; however, visual observation in a vibrating lucite tube indicated that the bulk oxide powder tends to increase in volume at one or two frequencies between 0 and 80 cps. With an electronic vibrator Hanford achieved bulk urania densities between 90 and 91% of theoretical by pulsing the vibration frequency between 50 and 2000 cps; with these higher frequencies, Hanford was able to attain the results at an armature acceleration of approximately 10 G.[1] From these data it appears that high pulsing frequencies and low accelerations may have the same effect on densifying powders as high accelerations at a specified lower frequency.

The vertical tube displacement produced by the BH 1.25-in. vibrator was between 15 and 20 mils. A schematic trace of the displacement wave form produced by the single impacting piston-type vibrator is shown in Fig. 3.2. A sine wave displacement curve can be generated by a pneumatic

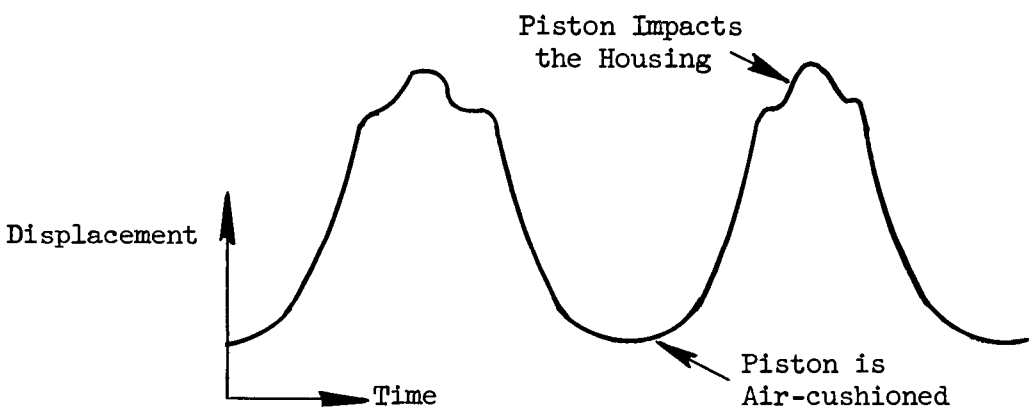

Fig. 3.2. Displacement wave form produced by BH 1.25-in. single-impact pneumatic vibrator.

vibrator in which the piston is air-cushioned at both ends of the housing; however, as previously stated, this type of displacement wave form produced bulk powder densities ~10% lower than those obtained with the single-impacting vibrator. Hanford has done all its compaction work with a sine-

wave input signal to their electronic vibrator.[1] With the electronic vibrator at Savannah River,[3] four vibratory compaction runs were made with a sine wave input signal on fused ThO_2-UO_2. By pulsing the frequency of vibration between 50 and 2000 cps in these four runs, bulk powder densities between 8.5 and 8.6 g/cc were obtained. In a fifth run at Savannah River with a saw tooth wave input signal the bulk density was 8.8 g/cc.

3.3 Other Observed Effects

A static load was applied to the surface of the fused oxide powder during several pneumatic vibratory compaction runs, resulting in bulk density increases of 1 to 2%. A 25-lb lead brick served as the load source, and the downward force was transmitted to the powder surface through a 0.25-in. steel rod. Since any applied load should be transmitted to a powder depth of only 1 to 2 in., any significant increase in bulk densification would require continuous or at least semicontinuous application of the downward force while filling the tube.

The variation of surface temperature with tube length was determined for an empty and oxide-filled stainless steel tube vibrating under the influence of the BH 1.25-in. vibrator. The top and bottom regions of an oxide-filled tube were 30 to 50°C hotter than the same regions of an empty tube. In a center region of approximately one-third the tube length, the temperature increase was only 5 to 10°C greater than in the middle third of an empty tube. This particular heating effect indicates a high degree of particle mixing in the lower and upper regions of the tube.

Visual observation of oxide powder in a vibrating lucite tube did not indicate any regions of vigorous agitation; the coarse oxide particles appeared relatively stationary, and only the smaller particles showed any degree of continuous motion. At certain frequencies the fine particles apparently had a net downward movement through the "fixed" coarse oxide structure until all the lower voids were filled.

4.0 APPLICATION TO FUEL RECYCLE

Both chemical densification to produce high-density fuel oxides and vibratory compaction to give high-density loadings of fuel into cladding appear simple processes. Either might be substituted for other more complex procedures. The two procedures together appear the most promising possibility for practical remote handling for recycle of fuel materials.

Figure 4.1 shows one of a number of possible combinations of these procedures into fuel recycle schemes. If the recycled fuel material must be handled remotely because of its radioactivity, this flowsheet shown completes the maximum amount possible of the fuel element assembly by direct fabrication methods before the empty assembly and the radioactive

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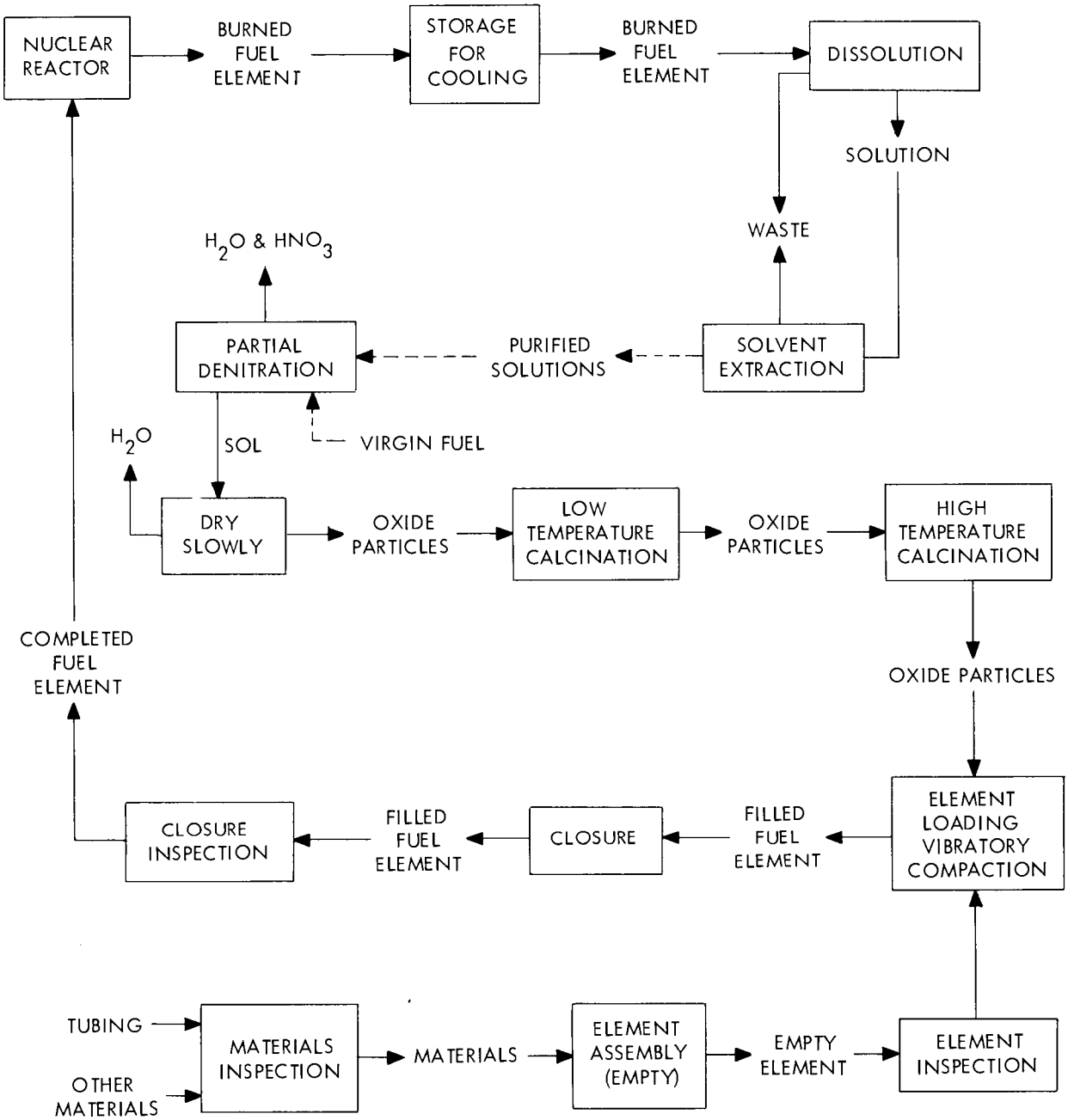

Fig. 4.1. Flowsheet for application of chemical densification and vibratory compaction to fuel recycle.

fuel material are brought together. The procedures for loading to a high density, making the closure, and inspecting the closure appear less difficult to carry out remotely than those required for other procedures such as oxide pellet fabrication or swaging. The chemical operations required to convert a solution to high-density oxide particles by the sol route appear well-suited to remote operation. The precipitation—washing—sol formation operations would probably be carried out in a single piece of equipment without transfer of solids or slurries. The high-temperature calcination step at 1200°C is at lower temperatures and easier to carry out than the calcination usually used for densification of ThO_2 or UO_2 pellets or the arc fusion of ThO_2 or UO_2 to be used for swaging.

Other processing steps might be substituted for those shown in Fig. 4.1. Processing methods that did not give complete decontamination might be substituted for solvent extraction if the chemical densification could be done remotely. The product from the chemical densification procedure would probably be well-suited to swaging operations. High-density oxides precipitated from fused fluorides might be excellent particles for vibratory compaction. Vibratory compaction as a fuel element loading procedure could be applied to other high-density fuel particles of a proper size distribution with minimum/maximum diameter ratios of about 50.

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