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Metallurgy and Ceramics

ARGONNE NATIONAL LABORATORY

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Lemont, Illinois

DIMENSIONAL STABILITY OF URANIUM POWDER COMPACTS UPON THERMAL CYCLING

by

R. M. Mayfield, S. T. Zegler and H. H. Chiswik

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For The Atomic Energy Commission

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Chief, Declassification Branch

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DIMENSIONAL STABILITY OF URANIUM POWDER COMPACTS UPON THERMAL CYCLING

by

R. M. Mayfield, S. T. Zegler and H. H. Chiswik

I. INTRODUCTION

Thermal cycling tests^{(1)*} on uranium have shown that the dimensional changes that occur on cycling in the alpha range are directly related to both the texture of the material and its grain size: cold rolled rods generally elongate in the direction of rolling, while the same rods, after a beta-treatment,** grow at rates several orders of magnitude lower. This considerable improvement by beta-treatment has been attributed to the texture randomization accompanying the heat-treatment. In the course of this heat-treatment, however, considerable grain growth occurs, which has the effect of causing surface roughening on cycling (also referred to as "bumping"); fine grained material generally retains a smooth surface. These observations led to the speculation that the most desirable structure in uranium, from standpoint of dimensional stability, is one that combines both a random texture and a fine grain size. Heat treatment of rolled rod offered no easy method to obtain such a product; powder metallurgical techniques, however, appeared ideally suited for this purpose.

To this end, early in 1949, the Sylvania Electric Products Company initiated a program to develop suitable techniques for producing uranium powder compacts having the above-mentioned desired characteristics. Because of the availability of thermal cycling equipment at Argonne, the Metallurgy Division of the Laboratory has undertaken to evaluate the stability of the various experimental compacts produced in the developmental phases of the program. This report contains the results of these evaluations.

*Numbers in parenthesis refer to bibliography at end of report.

**Water quenching from beta phase; about 725°C.

II. MATERIALS AND METHODS OF EVALUATION

The compacts made available for stability evaluation were prepared by one of the following methods:

1. Cold compacting and sintering at temperatures in the high gamma range - 1095° to 1120°C.
2. Cold compacting, sintering, repressing, and final annealing either in the alpha range or the gamma range.
3. Hot pressing in the high alpha temperature range.

These methods have been described in detail by Sylvania,^(2,3) and will not be described here. Tables I and II contain summaries of the pertinent fabrication details as supplied by Sylvania, such as the raw materials and powder sizes used, and the pressing, sintering and annealing variables. The densities of the compacts ranged from 16.2 to 18.92 gms/cc, with the higher values attained in the hot pressed compacts. The grain sizes of the sintered specimens were very large and irregular; the hot pressed compact grains were uniformly fine with average diameters less than 0.025 mm.

The dimensional stability of the materials were evaluated from the results obtained upon thermal cycling in either the vertical tube (Woosher) or induction cycling units.⁽⁴⁾ The cycling temperature limits in the Woosher were 50°C and 600°C, using a 5 second transfer time on heating and cooling and a 5 minute holding time at each temperature. In the induction cycling unit the temperature limits were 100°C and 550°C, the heating time 10 to 12 minutes, and the cooling time 25 to 30 minutes, depending on the size of the compacts.

The thermal cycling results will be discussed with reference to the methods of fabrication outlined above. Tables III and IV contain summaries of the experimentally measured dimensional changes of all the cycled compacts, along with qualitative observations regarding surface quality and characteristics such as roughening, seams, cracks, etc.

III. RESULTS

1. Cold Compacted and Sintered Specimens (Nos. U-34A, B, C and E)*

These specimens were cut from a rectangular piece that had been cold compacted and sintered at 1125°C. The "as-sintered" dimensions were: 0.365 in. wide by 0.450 in. thick by 10.5 in. long, with the thickness dimension representing the direction of pressing on compacting. The cycling specimens were cut so that their length, width, and thickness directions were parallel to the corresponding directions in the "as-sintered" piece. The cycling specimens were finished to the dimension shown in Table III by grinding. Upon Woosher cycling for 200 times, all except U-34E grew in all directions, with the largest growth occurring generally in the thickness dimension (pressing direction). Specimen U-34E shrank in length and grew in width and thickness. After 150 cycles, the maximum values of growth and shrinkage in any direction were 12.4% and -0.26% respectively. The recorded dimensional changes are subject to considerable error because of surface roughening which, as shown in Figure 1, was quite pronounced after only 100 cycles. After 200 cycles, the specimens were roughened and distorted to a degree that precluded even an approximate measurement of the dimensional changes. From past observations the pronounced roughening may be attributed to the coarse grain size established at the high sintering temperature, as illustrated in Figure 2.

After 200 cycles the specimens were reground and their bulk densities determined from their weights and volumes (the latter measured by micrometer). The densities decreased slightly from an initial value of 17.2 gms/cc to values ranging from 17.1 to 16.7 gms/cc. This decrease may be attributed to the initial porous nature of the compacts (see Figure 3), and the severe cracking that occurred during cycling (see Figure 1).

2. Cold Compacted, Sintered, Repressed and Annealed Specimens

Cycling specimens No. 76B-1 and 2 were cut from a cold compacted and sintered specimen that had been repressed at room temperature and annealed for 1 hour at 500°C. The cycling specimens were finished to size by surface grinding. The thickness dimension represented the repressing direction. Upon Woosher cycling both specimens grew more or less uniformly in all directions. The maximum growth observed in any direction after 500 cycles was 5.88%. Roughening was slight but the specimens developed deep surface cracks (see Figure 4). A qualitative indication of the extent of the cracking is provided by the fact that the specimens exuded NaK for some time after testing. The porosity in this material, illustrated in Figure 5, was somewhat more prominent than in the U-34 specimens. The microstructure could not be satisfactorily resolved but the grain structure

*Identification numbers of specimens are those supplied by Sylvania.

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appeared large and irregular. Sub-graining has been noted by Sylvania on similarly treated material.⁽²⁾ The difficulty of revealing the microstructure metallographically arose from the fact that the voids enlarged during electropolishing to the point of scattering the incident light beam so much that the image was blotted out. Best results, although not very satisfactory, were obtained by electropolishing directly after lead lapping on 900 grit abrasive. This procedure tended to fill the voids, thus avoiding excessive preferential dissolution around them. This technique revealed grain outlines in large-grained specimens, but was inadequate for fine-grained specimens.

Densities were obtained after cycling in a manner similar to that used for the U-34 specimens. The densities decreased from initial values of 16.4 and 16.2 gms/cc to 13.6 and 14.1 gms/cc. Porosity and cracking are probably the cause of this decrease, with the tendency of the NaK to expand the pores during cycling, a possible contributing factor.

Specimens No. 168-1 and 2 were cut in the form of small cubes from a rectangular sintered compact about 6 in. long by 0.8 in. thick by 0.5 in. wide. The cubes were first repressed in the same direction as the original compact (thickness direction) and then repressed again on the other faces (rotations of 90°). The specimens were then annealed for one hour at 1090°C and machined to approximately 0.5 in. on each side. In 200 Woosher cycles, growth varied from 3.71% to 8.38% in width, 3.04% to 4.30% in thickness, and 1.54% to 2.38% in length.* It is apparent that in some directions growth occurred at rates notably higher than in the 76B series above. The above growth rates are to be considered as approximate because of errors of measurement due to surface roughening (see Figure 6). Densities, initially 18.03 and 18.1 gms/cc, remained essentially unchanged, as determined by the carbon tetrachloride immersion method. The specimens did not show any indications of cracking.

3. Hot Pressed Specimens Made from Uranium Powders

Cycling specimens No. 509-1 and 510-1 were cut and ground from discs, 1.44 in. in diameter by approximately 0.3 in. thick, that had been made by hot pressing at 600°C, with the pressure applied in the thickness direction. The thickness direction in the cycling specimens corresponded to the direction of pressing; the length and width directions were arbitrary radial directions with reference to the "as pressed" discs. The specimens were of uniform fine grain size (average diameter 0.025 mm or less) and the densities were considerably higher (18.77 and 18.78 gms/cc) than those of any of the sintered specimens described above.

*These are arbitrarily assigned directions and may not be related to corresponding directions in "as pressed" specimens.

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Upon Woosher cycling both specimens shrank in thickness (pressing direction) and grew in width and length (radial directions of disc). Maximum shrinkage after 500 cycles was 2.6%; maximum growth 1.6%. Both pieces were very slightly roughened and warped as shown in Figure 7. All edges were slightly raised producing a "wire edge" effect. No evidence of cracking or porosity was seen in either specimen after cycling. Density measurements after cycling (by carbon tetrachloride immersion method) showed no significant decrease.

In an attempt to remove orientation effects, the specimens were re-ground, beta treated (4 minutes at 725°C - water quenched) and recycled another 500 times. Dimensional changes were inconsistent between the two specimens. No. 509-1a grew in all directions with the greatest growth (1.5%) occurring in the thickness direction, while No. 510-1b shrank in width and thickness (1.1% and 0.6% respectively) and grew in length (0.9%). In consequence of the coarsened grain structure resulting from the beta treatment, (compare Figures 8 and 9) the surface roughening was considerably more pronounced (compare Figure 10 with Figure 7).

For comparison with these compacts, two rolled and beta treated uranium rods, 1 in. long x 3/8 in. in diameter, were similarly cycled. After 500 cycles the growths ranged from 1.27% to 1.40% in length and from 0.2% to 0.47% in diameter, the latter values being subject to considerable experimental error due to surface roughening (see Figure 11). From the results it may be inferred that the order of magnitude of the dimensional changes was the same for both the powder compacts and the rolled and beta treated specimens. Roughening, as might be expected, was more pronounced after beta treatment.

Cycling specimens No. 611-1 through 7 were cut from a 1.4 in. diameter x 4 in. long rod which had been hot pressed from uranium powder in a single pressing operation, with the pressing direction parallel to the cylindrical axis of the rod. The rectangular cycling specimens were sectioned so that the pressing direction was represented by the thickness dimension in specimens 1, 4, and 7, and by the length dimension in specimens 2, 3, 5 and 6. Their grain structures were uniformly fine (about 20 microns) and their densities ranged from 18.56 to 18.72 gms/cc. Their thermal cycling stability was similar in all respects to that of the hot pressed compacts described previously: shrinkage occurred in the pressing direction and growth in directions normal to the pressing direction. Maximum shrinkage in 500 cycles was 1.5%; maximum growth 3.4%; roughening and warp were very slight (see Figure 12).

Specimen Nos. 671, 673 and those of the 1300 series (see Tables I and III) were hot pressed in a single operation at 600°C, into slugs 1.3 in. in diameter by 2 and 4 in. long and were cycled in full size. The average grain diameters of Nos. 671 and 673, ascertained from a hardness relationship,⁽²⁾ were of the order of 0.02 mm or less. The six slugs in the 1300

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series were of slightly finer grain size with average diameters of the order of 0.01 mm. The densities of these specimens varied from 18.74 to 18.92 gms/cc. Upon induction cycling, all slugs shrank in length (pressing direction) and grew in diameter. Shrinkage in 250 cycles ranged from 0.52% to 0.81% in Nos. 671 and 673 and from 0.17% to 0.52% in all compacts of the 1300 series. Representative compacts of the 1300 series shrank upon further cycling (700 times) from 0.88% to 1.19%. The centerless ground compacts of this series shrank slightly less than those cycled "as pressed and electropolished."

Compact No. 673 was the only one in this group that showed any notable evidence of surface roughening (see Figure 13). Compacts 671 and 673 showed slight to pronounced "wire edge" effects. This "wire edge" effect was observed on only one end of the compacts, usually the upright end on vertical cycling. Compacts of the 1300 series had only slight "wire edge" effects (Figure 14), except No. 1392 which showed a pronounced "wire edge" effect as well as definite circumferential cracks or crevices. The only other significant physical defect was a vee-shaped crack, about 3/8 in. deep, observed on the circumference of slug No. 1361. In all the 4 in. slugs (1300 series) the die parting line was evident after 250 cycles; after 700 cycles a slight ridge at the parting line was noted.

Specimens No. 645 and 651 were hot pressed and repressed to a final size of 1.2 in. in diameter by 4.0 in. long. Similarly cycled, both compacts shrank in length (pressing direction) and grew in diameter. After 250 cycles, shrinkages in length ranged from 0.39% to 1.32%, which is of the same order of magnitude as that obtained on the 2 in. compacts discussed above (Nos. 671 and 673). Roughening of No. 651 was slight to moderate; in No. 645 it was quite pronounced, as shown in Figure 15. Circumferential crevices were noted at regular intervals along the length of both compacts.

4. Hot Pressed Specimens Made from Uranium Hydride Powders

Specimens No. 625-1 and 2 and 634-1 and 2 were sectioned from hot pressed discs approximately 1-3/8 in. in diameter x 0.3 in. thick. The pressing direction in the sectioned cycling specimens was represented by either the width or thickness dimensions. Growth after 500 cycles on 625-1 and 2 was greatest in the thickness direction (maximum 2.4%), due in most part to surface roughening, shrinkage occurred in the width of both specimens (0.29%) and in the length of specimen No. 625-1 (0.58%). Surface roughening on the thickness surfaces was quite pronounced; the other surfaces remained relatively smooth, as shown in Figure 16a. Sharp edges were present on both samples after 200 cycles.

Compacts No. 634-1 and 2 were cut from a similarly hot pressed disc, using sufficient coolant in cutting to prevent overheating, since it was thought that the roughening on the thickness surfaces of the previous specimens

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might have arisen from such circumstances. Upon similar cycling, very little surface roughening was evident after 500 cycles, as shown in Figure 16b. Both specimens shrank in length (maximum 1.8%) and grew in width (maximum 1.2%) and thickness (maximum 3.4%). These dimensional changes are probably more nearly representative of this material, since improper sectioning was avoided.

Specimens No. 674, 25, 26, 31, 35, 36 and 48 were cylindrical slugs, 1.3 in. in diameter by 2 or 4 in. long, hot pressed in a single operation at 600°C at a pressure of 11 tsi. Except for starting material (UH₃ powder) fabrication details were the same as for the compacts in the 671-673 and 1300 series discussed above. Average grain diameters were 0.01 mm or less and densities ranged from 18.84 to 18.92 gms/cc. With the exception of No. 674, all compacts shrank in length (pressing direction) and grew in diameter upon induction cycling. After 250 cycles the maximum shrinkage was 0.85% and maximum growth 0.63%. After 700 cycles, representative compacts had shrunk 1.67% and grown 1.24% (maximum values in both cases). The range of values for all compacts (with exception of No. 674) were of about the same order of magnitude regardless of whether surfaces had been electropolished or centerless ground prior to cycling. No. 674 grew in length (0.88%) and became elliptical in cross section after 250 induction cycles. No evidences of roughening or "wire edge" effects were noted in any of the above compacts after cycling (see Figures 13 and 14); however, die parting lines were evident on all the 4 in. compacts.

Specimens No. 680 and 696 were made by sinterwelding initially hot pressed components, approximately 2 in. long by 1.3 in. in diameter, into 4 and 8 in. slugs respectively. A charge of approximately 25 grams of UH₃ powder was placed between the components and hot pressed at 600°C in the usual manner. Slug No. 696 was machined and centerless ground to 3/4 in. in diameter for cycling. Average grain diameters in both compacts were 0.025 mm or less, with densities ranging from 18.90 to 18.92 gms/cc. Upon induction cycling, No. 680 grew only 0.5% in 250 cycles. Changes in diameter were erratic in that one of the original 2 in. components shrank a maximum of 0.54%, while the other grew a maximum of 0.41%. Pronounced wire edges were evident on the upright end as shown in Figure 17, but only very slight surface roughening was noted. The sinterweld area and the die parting line were visible after cycling. Scattered longitudinal hairline cracks were noted.

The 8 in. compact, No 696, was induction cycled 700 times. For comparison, a beta treated uranium rod (No. 108)* of equivalent length and diameter was cycled along with the compact. The sinterwelded compact grew 0.40% in length and 0.66% in diameter, while the beta treated rod grew

*Rod No. 108 was rolled 38% at 300°C, beta treated for 8 minutes at 725°C, water quenched and centerless ground.

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3.28% in length and shrank 0.67% in diameter. The surface appearance of both rods after cycling is shown in Figure 18. The compact had no "wire edges" and the surface was considerably less roughened than that of the beta treated rod; however, there was considerable out-of-roundness, giving the compact a wavy, irregular surface along its length. The diameters of the compact at the weld areas were about 0.004 in. smaller than at other locations.

5. Effects of Cycling Variables

To evaluate the effects of cycling variables, two specimens, 3/8 in. in diameter by 1 in. long, were machined from the centers of compact Nos. 25 and 1354 and Woosher cycled under two different conditions. A transfer time of 5 seconds heating and cooling was used on two specimens, while a 15 minute heating - 5 second cooling transfer time was used on the other two specimens. Cycling occurred between 50°C and 550°C with holding times of 2 minutes in the cold tank and 5 minutes in the hot tank. The results after 200 cycles are presented below.

<u>Cycling Conditions</u>	<u>Number of Cycles</u>	<u>Percent Change in Length</u>	
		<u>A - 25</u> <u>(UH₃ Powder)</u>	<u>A-1354</u> <u>(U Powder)</u>
5 seconds heating and cooling transfer times	200	-0.57	-1.16
15 minute heating - 5 second cooling transfer times. ("Slow heating-fast cooling" cycle)	200	+0.15	+0.91

Using the "equal heating and cooling rate" cycle, specimen No. 1354 had a higher shrinkage rate than No. 25. The "slow heating-fast cooling" cycle caused both specimens to grow, with No. 1354 having the greater rate. This influence of cycling variables upon the direction of dimensional changes is consistent with that observed in alpha rolled as well as beta treated uranium.⁽⁵⁾ A "slow heating-fast cooling" cycle increases the growth in either case.

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IV. DISCUSSION OF RESULTS

Uranium powder compacts fabricated by cold compacting and sintering in the gamma range were of low density, approximately 17 gms/cc, and of extremely large and irregular grain size. Thermal cycling produced large, and non-uniform dimensional changes, excessive cracking, and pronounced surface roughening.

Cold compacted, sintered, repressed and gamma annealed compacts were also characterized by a large and irregular grain size. The final density varied widely depending upon pressures, temperatures and repressing operations; however, at best it was considerably below theoretical values. Dimensional changes and surface roughening upon thermal cycling were moderate to severe, depending upon the grain size and density. Cold repressing followed by alpha annealing, as contrasted to gamma annealing, produced a somewhat more stable compact. No correlation between pressing directions and the directions of the dimensional changes upon thermal cycling could be found for the materials that had been heated into the gamma temperature range prior to cycling. The pronounced surface roughening made accurate measurements of the dimensional changes difficult, and the reported values should be interpreted as approximations only.

Hot pressing of uranium or uranium hydride powders in the high alpha temperature range produced compacts of higher densities and uniformly smaller grain sizes. Hot pressed specimens made from uranium powders invariably shrank in the direction of pressure application. No such correlation could be found in the hot pressed specimens made from uranium hydride powders. Upon Woosher cycling, the dimensional changes of hot pressed compacts, made from either uranium or uranium hydride powders, were of about the same order of magnitude as those obtained in beta-treated rolled uranium, except for considerably diminished surface roughening effects. In this connection, it should be emphasized that a beta treatment of the hot pressed compacts reduced the dimensional changes only slightly but increased the surface roughening noticeably, presumably because of increased grain size.

The magnitude of dimensional changes of hot pressed compacts upon induction cycling was lower in all cases than upon Woosher cycling. This is a characteristic of the two cycling units that is directly related to the cycling variables, chiefly heating and cooling rates.^(4, 5) The surface roughening of full-sized rods pressed in a single operation was negligible, but other defects were occasionally noted, such as metal overlapping, circumferential cracks or crevices, wire edges, and die parting marks. "Wire edge" effects were less evident in the compacts made from uranium hydride and were further minimized by finishing to size by surface grinding.

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The two compacts that were produced by pressing and repressing in a graphite sleeve were less stable in all respects. Shrinkage was high (1.32% in only 250 cycles), roughening was pronounced, and surface defects were present. Based on surface appearance alone, one would hesitate to recommend this fabrication technique of pressing and repressing as a means of producing suitable 4 in. compacts, although it should be borne in mind that only two such compacts were examined.

The two "sinterwelded" compacts grew only 0.66% maximum after 700 induction cycles. Surface roughening was very slight, but slight "wire edges," out-of-roundness, and surface cracks were present. It should be noted that even though the individual components, from which the 8 in. long rod was made, shrank in length and grew in diameter, the composite tended to grow in all directions. The weld areas were visible after cycling in all cases.

The dimensional changes of the 8 in. long sinterwelded compact were lower in magnitude than those of a beta-treated rolled uranium rod of similar dimensions and cycled under identical conditions (0.40% vs. 3.28% growth). From past observations on numerous other beta-treated rolled rods, however, the noted growth of this particular beta-treated rod appears abnormally high, and a comparison of the relative growth stability of the two types of products based on only a single set of measurements cannot be made without considerable reservations. No such qualifications exist, however, in regard to surface roughening; the problem is practically eliminated in suitably fabricated compacts.

In general, the cycling results indicate that even in the hot pressed compacts there is retained a minor orientation effect, arising from the directionality of the pressing operation, which makes itself felt in a preferential directional growth or shrinkage on thermal cycling. The influence of this orientation effect upon the dimensional changes may be altered by cycling variables in a manner consistent with that observed on alpha rolled as well as beta treated uranium.

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V. CONCLUSIONS

The data in this report indicate that compacts of nearly theoretical density and fine grain size can be obtained by hot pressing uranium or uranium hydride powders in the high alpha temperature range. Large diameter compacts (1.4 in. in diameter) of varying length are best made by hot pressing in a single operation or by sinterwelding of individual hot pressed components. The magnitudes of growth or shrinkage of such compacts upon thermal cycling approximate those of beta-treated rolled uranium; the surface wrinkling problem, however, associated with beta treatment is practically eliminated in the alpha pressed powder compacts.

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Figure 1.

17

Surface appearance of Compacts U-34A, B, C and E after "Woosher" Cycling between 50°C and 600°C.

(Cold Compacted and Sintered)

Figure 2.

Microstructure of Specimen U-34C before cycling.

Micro #6987 H_3PO_4 Electrolytic Polish 50x
Polarized Light

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Figure 3.

Porosity in Specimen U-34C before cycling.

Micro #6986 Mechanical Polish 100x
Bright Field.

Figure 4.

Surface Appearance of Compacts 76B-1 and 2
after 500 Woosher Cycles.
(Cold Compacted, Sintered, Repressed and Annealed)

76B-1 76B-2
Macro #4640 approx. 1x

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Figure 5.

Porosity of Specimen 76B-2 before cycling.
(Cold Compacted, Sintered, Repressed and Alpha Annealed)

Micro #6982 Mechanical Polish 100x
Bright Field.

Figure 6.

Surface Appearance of Compacts 168-1
and 2 after 200 Woosher Cycles.
(Cold Compacted, Sintered, Repressed and Gamma Annealed)

Macro #7904

approx. 2x

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Figure 7.

Surface Appearance of Hot Pressed
Compacts 509-1 and 510-1 after
500 Woosher Cycles.

509-1
Macro #9046

510-1
approx. 2x

Figure 8.

Microstructure of Hot Pressed Compact 510-1
after 500 Woosher Cycles.

Micro #10,197 H_2PO_4 Electrolytic
Polish, Polarized Light. 250x

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Figure 9.

Microstructure of Beta Treated Hot Pressed
Compact 509-1 after 500 Woosher Cycles.
(Compact previously cycled 500 times in as-
received condition)

Micro #10,199 H_3PO_4 Electrolytic 250x
Polish, Polarized
Light.

Figure 10.

Surface Appearance of Beta Treated Hot Pressed
Compacts after 500 Woosher Cycles.
(Compacts previously cycled 500 times in as-received condition)

509-1
Macro #10,153

510-1
approx. 2x

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Figure 13.

Surface and End Appearances of Compacts Nos. 671, 673 and 674
after 250 Induction Cycles Between 100°C and 550°C.
(Pressed in single operation)

a. End appearances.

No. 671, U-Powder
Macro #11,764

No. 673, U-Powder

No. 674, UH_3 Powder
1x

b. Surface appearances.

No. 671, U-Powder
Macro #11,763

No. 673, U-Powder

No. 674, UH_3 Powder
1x

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Figure 14.

Surface Appearance of Compacts Nos. 1359, 1392, 26 and 48 After 700 Induction Cycles.

(Pressed in Single Operation)

Macro #13,596
1359

1392

26

approx. 1x
48

Raw Material: U
Surface: As Pressed
and Electropolished

U
Centerless
Ground

UH₃
As Pressed
and Electropolished

UH₃
Centerless
Ground

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Figure 15.

Surface Appearance of Compacts Nos. 645 and
651 After 250 Induction Cycles.

(Fabricated by Pressing and Repressing Tech-
niques)

Macro #11,766
No. 645

approx. 1x
No. 651

Macro #11,765
Raw Material: U-Powder

approx. 1x

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Figure 16.

Surface Appearance of Hot Pressed
Compacts Nos. 625 and 634 After
500 Woosher Cycles.

625-1
Macro #11,309

625-2
approx. 2x

634-1
Macro #13,431

634-2
approx. 2x

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Figure 17.

Surface Appearance of 4" Long Sinter-
welded Compact No. 680 after 250 In-
duction Cycles.

Macro #11,766 approx. 1x

Macro #11,765 approx. 1x

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Figure 18.

Surface Appearance of Beta Treated Rolled
Uranium Rod and Sinterwelded Compact No.
696 After 700 Induction Cycles.

Macro #11,977
Beta Treated
Rod #108

3/4 x
Compact #696

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Table I

FABRICATION DATA OF SPECIMENS MADE FROM URANIUM POWDERS⁽⁴⁾

Specimen No.	Raw Materials and Fabrication Methods	Compacting, Sintering, Pressing & Annealing Data			Density (gms/cc)	Grain Size (Avg Dia. in mm)	Approx. Size " as fabricated, " (in) (3)				Remarks
		Compacting & Sintering	Pressing	Annealing			W	T	D	L	
U-34 A,B,C & E	Uranium powder < 325 mesh Cold compacted and sintered.	65 tsi, 240 min. at 1125°C.	---	---	17.2	Very large & irregular (> 10 mm)	0.4	0.45		10.5	Pressure applied in thickness direction.
76B-1 76B-2	Uranium powder < 325 mesh Cold compacted, sintered, repressed, and annealed.	65 tsi, 120 min at 1095°C.	Repressed at R.T., 139 tsi	60 min. at 500°C	16.4 16.2	Very large & irregular	0.39	0.24		7.0	Pressure applied in thickness direction.
168-1 168-2	Uranium powder < 325 mesh. Cold compacted, sintered, repressed, and annealed.	50 tsi, 180 min. at 1090°C.	Repressed at R.T. in 3 directions 90° apart; 100-175 tsi.	60 min at 1090°C	18.03 18.1	Very large & irregular	0.5	0.5		0.5 ⁽¹⁾	Cubes for repressing cut from rectangular sintered piece 6" long. Repressed at room temperature three times on different faces. Directions of final pressing not identified.
509-1 510-1	Uranium powder < 325 mesh. Hot pressed.	---	25-30 tsi, 15 min. at 600°C.	---	18.77 18.78	Small & uniform, 0.025 or less	---	0.3	1.44	---	Pressure applied in thickness direction
611-1 thru 611-7	Uranium powder < 325 mesh. Hot pressed. Single pressing.	---	25 tsi, 15 min. at 570°C.	---	18.56 to 18.72	Small & uniform, About 0.020.	---	---	1.4	4	Pressure applied in length direction of 4" slug.
671 673	Uranium powder < 4 mesh Hot pressed.	---	11 tsi, 15 min. at 600°C	---	18.74 18.78	Approx 0.02 ⁽²⁾	---	---	1.4	2	Die lubricated with aquadag Pressure applied in length direction of slug.
1354 1359 1361	Uranium powder < 80 mesh. Hot pressed	---	11 tsi 15 min. at 600°C	---	18.88 18.86 18.90	< 0.01 0.012 to < 0.01 0.017 to < 0.01	---	---	1.2	4	Die lubricant used, no sleeve. Pressure applied in length direction of slug.
1358 1380 1392	Uranium powder < 80 mesh. Hot pressed.	---	11 tsi 15 min at 600°C	---	18.89 18.91 18.92	< 0.01	---	---	1.2	4	Die lubricant used, no sleeve. Pressure applied in length direction of slug
645 651	Uranium powder < 4 mesh. Hot pressed and repressed.	---	10 tsi, 15 min at 600°C, 2 pressings.	---	18.75 18.81	---	---	---	1.3	4	Graphite sleeve used in die. Pressure applied in length direction of slug.

(1) Dimensions of cubes cut from sintered plate which was 0.5" wide x 0.8" thick x 6" long "as sintered"

(2) From hardness vs grain size relationship (SEP 72 - Reference 2).

(3) W - width, T - thickness, D - diameter, L - length

(4) Fabrication data supplied by Sylvania

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Table II

FABRICATION DATA OF SPECIMENS MADE DIRECTLY FROM URANIUM HYDRIDE POWDERS⁽⁴⁾

Specimen No.	Raw Materials and Fabrication Methods	Compacting, Sintering, Pressing & Annealing Data			Density (gms/cc)	Grain Size (Avg. Dia. in mm)	Approx. Size "as fabricated" (in.) (3)				Remarks
		Compacting & Sintering	Pressing	Annealing			W	T	D	L	
625-1 625-2 634-1 634-2	Uranium hydride powder < 325 mesh. Hot pressed.	---	10 tsi; 15 min. at 600°C. 5 tsi; 15 min. at 600°C.	---	18.81 18.84	Small & uniform; < 0.01 mm.	---	0.3	1-3/8	---	Pressure applied in thickness direction.
674	Uranium hydride powder < 10 microns. Hot pressed.	---	11 tsi; 15 min. at 600°C.	---	18.84	Approx. 0.01 ⁽²⁾	---	---	1.4	2	Die lubricant with aquadag. Pressure applied in length direction of slug.
25 26 31	Uranium hydride powder; 10 microns and less. Hot pressed.	---	11 tsi; 10 min. at 600°C.	---	18.89 18.86 18.85	< 0.01	---	---	1.3	4	Die lubricant used; no sleeve. Pressure applied in length direction of slug.
35 36 48	Uranium hydride powder; 10 microns and less. Hot pressed.	---	11 tsi; 10 min. at 600°C.	---	18.89 18.91 18.92	< 0.01	---	---	1.3	4	Die lubricant used; no sleeve. Pressing in length direction.
680	Uranium hydride powder; 10 microns or less. Sinterwelded from 2 prior hot pressed 2-inch slugs ⁽²⁾	---	10 tsi; 15 min. at 619-642°C; for sinterwelding: 20 tsi & 15 min. at 645°C.	---	18.90	0.025 to < 0.01	---	---	1.3	4	Die lubricant used; no sleeve. Pressing in length direction.
696	Uranium hydride powder; 10 microns or less. Sinterwelded from 4 prior hot pressed 2-inch slugs.	---	11 tsi; 15 min. at 624-636°C; same for sinterwelding.	---	18.92	Approx. 0.02 ⁽¹⁾	---	---	1.3	8	Die lubricant used; no sleeve. Pressing in length direction.

⁽¹⁾From hardness vs. grain size relationship (SEP 72 - Reference 2).⁽²⁾Sinterwelding consisted of placing approximately 25 gms of UH₃ between the components and hot pressing at 600 - 650°C.⁽³⁾W - width; T - thickness; D - diameter; L - length.⁽⁴⁾Fabrication data supplied by Sylvania.

Table III

THERMAL CYCLING DATA OF SPECIMENS MADE FROM URANIUM POWDERS

Specimen No.	Initial Dimensions ⁽¹⁾ (inches)				Type Cycle	No. of Cycles	Dimensional Changes Upon Cycling (%)				Remarks
	Width	Thickness	Diameter	Length			Width	Thickness	Diameter	Length	
U-34A	0.4325	0.3400		1.7371	Woosher	50 100 150	+0.60 +1.24 +4.21	+7.70 +5.88 +12.4		Nil Nil +0.41	Cycling specimens cut out & ground so that their length, width, & thickness directions were parallel to the corresponding directions "as sintered." All surfaces rough and irregular after 50 cycles. After 150 cycles surfaces were extremely irregular and cracks easily discernible.
U-34B	0.4348	0.3343		1.6979	Woosher	50 100 150	+0.23 +0.34 +0.85	+1.08 +2.24 +3.83		+0.44 +0.71 +0.61	
U-34C	0.4328	0.3324		1.6854	Woosher	50 100 150	+0.53 +1.25 +0.69	+0.21 +0.53 +0.63		Nil +0.10 ---	
U-34E	0.4298	0.3322		1.6665	Woosher	50 100 150	+0.56 +1.60 +2.58	+0.69 +1.69 Nil		-0.23 -0.17 -0.26	
76B-1	0.3670	0.2300		1.6654	Woosher	100 200 400 500	+0.87 +1.66 +3.27 +5.20	+1.26 +2.09 +3.56 +4.96		+0.95 +1.98 +3.64 +5.27	
76B-2	0.3460	0.2057		1.6771	Woosher	100 200 400 500	+1.10 +1.88 +3.21 +4.77	+1.51 +2.53 +3.45 +5.88		+1.02 +2.41 +4.0 +5.60	
168-1	0.4850	0.4478		0.4993	Woosher	100 200	+2.06 +3.71	+2.72 +4.30		+1.34 +1.54	
168-2					Woosher	100 200	+1.95 +8.38	+1.69 +3.04		+1.58 +2.38	
509-1	0.3103	0.1717		1.0349	Woosher	100 200 300 500	+1.2 +1.6 +1.5 +1.2	Nil -0.7 -1.4 -2.6		+0.5 +0.7 +0.9 +1.2	
510-1	0.2500	0.1748		1.1098	Woosher	100 200 300 500	+0.3 +0.8 +0.9 +1.6	-0.4 -1.4 -1.0 -1.4		+0.3 +0.4 +0.7 +1.5	
509-1a ⁽²⁾	0.2952	0.1478		0.7882	Woosher	100 200 300 500	+0.3 +0.3 +0.3 Nil	+0.7 +1.4 +1.7 +1.5		+0.3 +0.4 +0.5 +0.5	
510-1b ⁽²⁾	0.2441	0.1631		0.8536	Woosher	100 200 300 500	Nil -0.4 -0.5 -1.1	--- -0.4 -0.3 -0.4		+0.4 +0.6 +0.7 +0.9	
611-1	0.1242	0.1247		1.1242	Woosher	500	+2.6	-1.1		+2.8	Slight warp in all specimens. Roughening very slight. Cycling specimens cut so that the pressing direction was represented by the thickness in 1, 4, & 7 and by the length in 2, 3, 5 & 6.
611-4	0.1236	0.1250		1.1248	Woosher	500	+2.4	-1.2		+3.4	
611-7	0.1236	0.1253		1.1251	Woosher	500	+2.8	-0.9		+2.6	
611-2	0.1233	0.1251		1.1239	Woosher	500	+1.8	+2.2		-1.0	
611-3	0.1247	0.1247		1.1228	Woosher	500	+2.2	+2.0		-1.5	
611-5	0.1258	0.1251		1.1251	Woosher	500	+2.4	+1.9		-1.2	
611-6	0.1226	0.1249		1.1250	Woosher	500	+2.7	+2.4		-1.2	
671			1.4385	2.0778	Induct.	250			+0.24 to 0.52	-0.81	Slight "wire edge" effect; Roughening slight. Pronounced "wire edge" effect; roughening pronounced. Cycled as pressed & electropolished.
673			1.4472	2.0760	Induct.	250			Nil to +0.47	-0.52	
1354			1.365	4.089	Induct.	250			+0.22	-0.29	Slight wire edges. Roughening - none.
1359			1.364	4.082	Induct.	250			+0.38	-0.37	
1361			1.365	4.085	Induct.	750			+0.73 +0.07	-0.88 -0.17	
1358			1.352	4.010	Induct.	250			+0.22	-0.30	No wire edge. Slight wire edge. Pronounced wire edge at one point on circumference. Shallow crack. Roughening - none. Diameters furnished by surface grinding.
1380			1.352	4.013	Induct.	250			+0.37	-0.30	
1392			1.352	4.015	Induct.	250			+0.37	-0.52	
1392			1.352	4.015	Induct.	700			+1.33	-1.19	
645			1.2292	3.8950	Induct.	250			+0.14 to 0.91	-0.39	Shallow circumferential crevices. Roughening pronounced. Two deep circumferential crevices. Slight roughening.
651			1.2878	4.000	Induct.	250			+0.25 to 1.65	-1.32	

(1) Dimensions of cycling specimens. Surfaces finished ground except as indicated.

(2) Specimens 509-1 and 510-1 beta treated (4 min. at 725°C - water quenched).

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Table IV

THERMAL CYCLING DATA OF SPECIMENS MADE FROM UH_3 POWDERS

Specimen No.	Initial Dimensions (1) (inches)				Cycle Type	No. of Cycles	Dimensional Changes Upon Cycling (%)				Remarks
	Width	Thickness	Diameter	Length			Width	Thickness	Diameter	Length	
625-1	0.2505	0.2518		1.2236	Woosher	100	+0.92	-0.32		-0.38	Slight wire edges; roughness on thickness faces; other faces relatively smooth. Pressing direction represented by either thickness or width dimension.
						200	+0.92	+0.36		-0.35	
						400	-0.20	+1.5		-0.52	
						500	-0.20	+2.4		-0.58	
625-2	0.2427	0.1516		1.2991	Woosher	100	+0.74	-0.02		Nil	
						200	-0.54	+0.01		+0.13	
						400	-0.29	+0.03		+0.70	
						500	-0.29	+0.52		+0.84	
634-1	0.3014	0.2862		1.1965	Woosher	250	+0.26	+2.3		-0.71	
						500	+1.2	+3.4		-1.8	
634-2	0.2662	0.2438		1.1952	Woosher	250	+1.8	+0.49		-0.44	
						500	+0.7	+0.9		-0.52	
674			1.449	2.0065	Induct.	250			-0.27 to +0.96	+0.88	No wire edges. Roughening - none. Cycles "as pressed and electropolished."
25			1.365	4.135	Induct.	250			+0.15	-0.36	No wire edges. No roughening. Cycled "as pressed and electropolished."
26			1.366	4.139	Induct.	250			+0.15	-0.65	
						700			+1.24	-1.67	
31			1.367	4.082	Induct.	250			+0.07	-0.42	
35			1.352	4.012	Induct.	250			+0.33	-0.47	No wire edges. No roughening.
36			1.352	4.013	Induct.	250			+0.33	-0.46	
48			1.352	4.012	Induct.	250			+0.63	-0.85	
						700			+1.18	-1.62	
680			1.4292	4.0852	Induct.	250			-0.54 to +0.41	+0.15	Roughening slight; pronounced wire edge marks at weld; scattered small hair-line surface cracks.
696			0.7488	7.955	Induct.	700			+0.66	+0.40	Roughening-none; marks at weld visible; diameter out of round and wavy.

(1) Dimensions of cycling specimens. Surfaces finished ground except as indicated.