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THERMAL CYCLING OF PLUTONIUM
PART I: OBSERVATIONS OF THE PHYSICAL DAMAGE
RESULTING FROM THERMAL CYCLING PLUTONIUM
THROUGH ITS LOW TEMPERATURE PHASE TRANSFORMATIONS

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ABSTRACT

Plutonium, thermally cycled through the low temperature allotropic transformations, exhibited extensive physical damage. The physical damage was greater than that reported for any other metal or alloy. The extent of physical damage was determined by measurements of fluid displacement and the dimensional changes. The internal porosity was examined metallographically. Physical damage varied considerably, depending on the cycling conditions and the characteristics of the metal (particularly inclusions and casting imperfections). Observations showed that increasing the specimen size produced a greater decrease in density as a function of the number of cycles. The degree of void formation was directly proportional to volume change associated with the phase transformation. Other variables such as the length-diameter ratio (constant diameter) had little or no influence on the amount of physical damage. The dimensional growth was both radial and longitudinal. The tensile strength and the yield strength of tensile specimens cycled ten times between the gamma and alpha phases were decreased to an average of 30,000 and 26,000 psi respectively.

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INTRODUCTION

The physical damage resulting from thermal cycling uranium and other metals through their phase transformations is well known and has been thoroughly investigated.⁽¹⁻⁴⁾ It has been shown that the nature of the physical damage induced by cycling is quite complex.

It was reported by Coffinberry and Waldron⁽⁵⁾ that thermal cycling of plutonium resulted in physical damage in the form of progressive decrease in density and surface distortion similar to that found for transformation cycling in uranium. They suggested the possibility of microvoids existing in dilatometer specimens after thermal cycling through the phase transformations. A study of the transformation kinetics of plutonium⁽⁶⁾ reported that macrovoids appeared after repeated cycling through the beta-to-alpha transformation.

The physical damage in plutonium when thermally cycled was very pronounced, greater than reported for any other metal. It has also been found that many variables affect the extent of void formation (e.g. -specimen size and inclusions). This paper is a presentation and discussion of observed physical damage in plutonium when it is thermally cycled between the alpha, beta, gamma, and delta phases.

SUMMARY

Thermal cycling plutonium through its allotropic transformations resulted in extensive physical damage. This was shown primarily by a density decrease as a function of the number of cycles. Thermally-cycled specimens contained voids and macrovoids which were observed metallographically.

The voids initially appeared as internal micro-cracks and upon repeated thermal cycling became somewhat symmetrical. After repeated cycling the distribution of the voids was generally quite uniform except at the specimen ends, where the voids were larger than those in the interior. Also, the voids were randomly oriented throughout the samples.

The experimental observations of cycling damage in plutonium were:

1. The physical damage resulting from thermal cycling plutonium through its allotropic transformations was greater than for any other metal.
2. The extent of physical damage varied considerably depending on the cycling conditions and the characteristics of the metal, particularly inclusions and casting imperfections. Specimens containing a larger number of inclusions and casting imperfections showed more extensive void formation.
3. The extent of physical damage, determined by density measurements, was directly proportional to the volume change associated with the allotropic transformations.
4. The presence of voids caused a decrease in mechanical properties. The tensile strength of specimens containing five to seven per cent voids decreased from 62,800 to 30,000 psi and the yield strength from 39,300 to 26,400 psi.
5. Increasing the specimen size resulted in a greater decrease in density as a function of the number of cycles.
6. Cycling rate may have affected the extent of void formation but probably not to the degree that specimen size and impurities did.
7. Varying the length-to-diameter ratio of rods having a constant diameter had little or no effect on the extent of void formation.
8. Bending of rods always occurred in thermally cycled rods.
9. Radial and longitudinal growth occurred in all of the cylindrical specimens that were thermally cycled. A depression also appeared in the ends of each rod.

10. Fracture frequently occurred when specimens were cycled.
11. Voids collapsed when thermally-cycled specimens were subsequently allowed to transform under 5000 psi.
12. The extent of void formation was directly proportional to the volume change associated with the phase transformation.

EXPERIMENTAL PROCEDURE

There are six allotropic forms of plutonium. The crystal lattice, transition temperature, and calculated density of each phase are shown in Table I. In the present work physical damage was investigated only for specimens thermally cycled between the alpha, beta, gamma, and delta phases.

TABLE I
CRYSTAL STRUCTURE DATA FOR THE PLUTONIUM PHASES

<u>Phase</u>	<u>Crystal Lattice</u>	<u>Transition Temp. to the Next Higher Phase</u>	<u>Calculated Density (g/cm³)</u>
Alpha	Monoclinic	120	19.82
Beta	Monoclinic	203	17.8 approx.
Gamma	Face-centered Orthorhombic	316	17.14
Delta	Face-centered Cubic	451	15.92
Delta Prime	Body-centered Tetragonal	480	16.00
Epsilon	Body-centered Cubic	640	16.51

The extent of physical damage to plutonium due to thermal cycling was determined primarily by metallography, density measurements, and by measuring dimensional changes. The density measurements were determined by a fluid displacement technique using either carbon tetrachloride or bromobenzene as the displacement medium. All densities reported were determined in this manner and do not refer to the theoretical density or the densities determined by X-ray diffraction.

It was found that the surface condition of the specimens, whether as-cast or machined, had no effect on the results. Therefore, all of the specimens were in the as-cast condition.

The four general conditions under which the specimens were cycled were: (1) an air atmosphere, (2) a peanut oil bath, (3) evacuated capsules, or (4) vacuum annealing furnaces. The cycling conditions for each specimen are indicated in the discussion of results. Also, the rate of heating and cooling are noted in the discussion.

DISCUSSION OF RESULTS

Effect of the Volume Change Associated with the Allotropic Transformation on the Extent of Void Formation

Four specimens were machined to identical sizes from the same as-cast ingot for the purpose of determining the effect of the volume change associated with the phase change upon the extent of physical damage resulting from thermal cycling. One specimen was cycled through the gamma-to-alpha transformation, one through the beta-to-alpha transformation, one through the gamma-to-beta transformation, and one through the delta-to-gamma transformation. All specimens were cycled ten times in vacuum at the same cycling rate. The specimen cycled between the beta and gamma phases also passed through the beta-to-alpha transformation during the final cooling cycle. The specimen cycled between the delta and gamma phases passed through both the gamma-to-beta and beta-to-alpha transformations during the final cooling cycle. The density of each specimen before and after cycling is shown in Table II.

The data clearly show that the extent of void formation is directly proportional to the volume change associated with the phase transformation.

TABLE II
DENSITY OF PLUTONIUM AFTER TEN CYCLES

<u>Phase Change</u>	<u>Allotropic Vol. Change (approx.)</u>	<u>Ratio*</u>	<u>Density Before Cycling</u>	<u>Density After Cycling</u>	<u>Volume of Voids</u>	<u>Ratio** (approx.)</u>
Gamma-to-Beta	3%	1	19.57	19.24	1.7%	1
Delta-to-Gamma	6%	2	19.54	18.90	3.4%	2
Beta-to-Alpha	9%	3	19.58	18.60	5.3%	3
Gamma-to-Alpha	12%	4	19.56	18.29	6.9%	4

* Ratio of the volume change associated with each phase transformation to that of the Gamma-to-Beta transformation.

** Ratio of the volume of voids formed with each phase transformation to that formed in the Gamma-to-Beta transformation.

Gamma-to-Alpha Thermal Cycling

Plutonium samples one-half inch in diameter and three-fourths inch long were cycled between the alpha and gamma phases at different rates of transformation. Peanut oil and vacuum were used as the heat treating media.

Regardless of the heating or cooling rate there was a rapid decrease in density, or increase in total volume of void space, as a function of the number of cycles (Figure 1). The transformations between the gamma phase and the alpha phase when cycled in peanut oil were quite rapid as the samples were allowed to transform from one phase to the other by direct immersion.

For the first few cycles the rapid heating and cooling rates resulted in the formation of more voids than by heating and cooling more slowly. It seemed unlikely that the formation of voids was appreciably affected by the transformation medium during the first few cycles. However, after the voids had grown to an appreciable size, and the density was less than 18.0 g/cm^3 , the heat-treating liquid penetrated the specimens and filled the voids near the surface.

The voids appeared initially as small micro-cracks (Figure 2a) and became somewhat symmetrical after repeated cycling (Figures 2b - 2d). Also it can be seen from the photomicrographs that few inclusions were present in the metal.

The extent of void formation after ten cycles between the gamma and alpha phases was very nearly the same regardless of the medium of transformation or the cycling rate. Eight samples were cycled ten times under different conditions. The average density of the eight samples was 18.3 g/cm^3 . The minimum density was 18.15 g/cm^3 and the maximum was 18.41 g/cm^3 .

A sample three-eighths inch in diameter and three-fourths inch long was sealed in an evacuated capsule and cycled thirty-five times between the gamma and alpha phases. The density decreased from 19.5 to 14.0 g/cm^3 . This was an average density decrease of 0.16 g/cm^3 per cycle. The distribution of voids was quite uniform throughout the sample. Figures 2b and 2c show that the distribution of voids was very nearly the same at the center as at the edge of the sample. The distortion of the sample was such that it grew both radially and longitudinally.

Beta-to-Alpha Thermal Cycling

Cycling conditions and the purity of the plutonium had a more pronounced effect on the formation of voids between the beta and alpha phases than when cycled between the gamma and alpha phases. This is shown in Figure 3 where the per cent decrease in density is plotted as a function of the number of cycles. The curve showing a greater rate of density decrease was plotted from data for a sample cycled in peanut oil and rapidly heated and cooled (i. e. - by direct immersion). The curve showing a lower rate of density decrease was of a sample cycled in vacuum and at slower heating and cooling rates (approximately 25 C/min). However, the difference between the curves was due primarily to purity. The purer sample was affected less by cycling.

The shape of the voids, when beta-to-alpha cycled, was similar to those occurring when the metal was gamma-to-alpha cycled. The voids, appearing first as micro-cracks and then becoming somewhat symmetrical, were similar to the photomicrographs shown in Figure 2. The distribution of voids was uniform from the center of the sample to the outside edge.

Delta-to-Alpha Thermal Cycling

The density decrease as a function of the number of cycles of a specimen cycled between the delta and alpha phases was approximately linear, except between four and eight cycles. The average density decrease was 0.14 g/cm^3 per cycle (Figure 4). The deviation of the curve from linearity between four and eight cycles could not be explained satisfactorily. The curve was similar to that obtained with a specimen cycled between the gamma and alpha phases (Figure 1). However, an exact correlation to the gamma-to-alpha cycling could not be made since the metal used for the delta-to-alpha cycling was unfortunately from a different heat than that used to study the gamma-to-alpha thermal cycling.

Influence of Specimen Size

Early in the investigation it was found that the density decrease upon thermal cycling plutonium between any of its allotropic transformations depended on the sample size. This led to a quantitative investigation of the influence of sample size on the physical damage to plutonium subjected to thermal cycling.

Unalloyed plutonium was cast into spheres having diameters of $1/4$, $1/2$, $3/4$, 1, and $1-1/2$ inches. A photograph of the as-cast spheres is shown in Figure 5. The density of each of the as-cast spheres was 19.45. Representative photomicrographs of the as-cast metal are shown in Figures 6 and 7. The metallographic samples were obtained from a portion of the casting joining the spheres.

The density decrease of each sphere after one, two, three, four, and eight cycles between the beta and alpha phases is shown in Table III and plotted in Figure 8. The spheres were cycled in vacuum at approximately 2 C/minute. The maximum cycling temperature was 180 C, at which the specimens were held for 30 minutes.

TABLE III
PER CENT DENSITY DECREASE OF PLUTONIUM
OF VARYING DIAMETER AFTER THERMALLY CYCLING
BETWEEN THE BETA AND ALPHA PHASES

<u>No. of Cycles</u>	<u>Diameter (inches)</u>				
	<u>0.25</u>	<u>0.50</u>	<u>0.75</u>	<u>1.00</u>	<u>1.50</u>
1	0.60	1.52	1.57	1.03	1.70
2	2.73	3.92	4.25	4.66	4.01
3	4.59	6.12	6.66	7.16	6.74
4	6.35	8.63	9.25	9.81	9.77
8	14.00	17.26	18.99	23.40	15.17

The data show that increasing the sample size produced a greater amount of physical damage. This damage reached a maximum at approximately one inch (Figure 9). Although specimen diameter noticeably influenced physical damage, its effect was not nearly as great as impurities and other casting variables.

The weight of the 1-1/2 inch sphere and ductility of the beta phase caused the voids to collapse. Hence, the density decrease of this sphere after eight cycles was significantly less than the 1/2, 3/4, and 1 inch spheres as reported in Table III. A flattening of the 1-1/2 inch sphere was also observed after eight cycles.

Plutonium rods, 0.375 inch in diameter with a varying length: diameter ratio, were cycled eight times between the beta and alpha phases. The densities after each cycle are shown in Table IV.

TABLE IV
PER CENT DENSITY DECREASE OF PLUTONIUM
OF A VARYING LENGTH:DIAMETER RATIO (0.375 INCH DIAMETER)
AFTER THERMALLY CYCLING BETWEEN THE BETA AND ALPHA PHASES

<u>No. of Cycles</u>	<u>Length:Diameter Ratio</u>				
	<u>1:1</u>	<u>3:2</u>	<u>2:1</u>	<u>3:1</u>	<u>5:1</u>
1	0.67	0.41	0.26	0.67	0.51
2	0.99	1.22	0.88	1.35	1.27
3	2.12	2.19	1.60	2.53	2.18
4	3.64	3.24	2.51	3.57	3.37
8	7.77	7.57	6.33	8.08	7.36

The data show that varying the length:diameter ratio had little or no effect on the resulting physical damage due to thermal cycling. The 2:1 ratio specimen showed a consistently lower density decrease and was believed to have contained a lower amount of inclusions. Likewise, the sample having a length:diameter ratio of 3:1 was believed to have contained a slightly greater amount of inclusions. This would have accounted for the density change being greater than for the other samples.

Three 0.75-inch long plutonium rods, with varying length:diameter ratios of 3:1, 1:1, and 3:4, were cycled eight times between the beta and alpha phases. The densities after each cycle are tabulated in Table V and shown graphically by Figure 10.

The data showed that decreasing the length:diameter ratio (increasing the specimen size) of a sample having a constant diameter resulted in a greater amount of physical damage due to beta-to-alpha thermal cycling.

TABLE V
PER CENT DENSITY DECREASE OF PLUTONIUM
OF A VARYING LENGTH:DIAMETER RATIO (0.75 INCH LENGTH)
AFTER THERMALLY CYCLING BETWEEN THE BETA AND ALPHA PHASES

<u>No. of Cycles</u>	<u>Length:Diameter Ratio</u>		
	<u>3:1</u>	<u>1:1</u>	<u>3:4</u>
1	0.22	0.23	1.08
2	0.26	0.98	2.10
3	1.29	--	--
4	1.83	2.26	4.26
6	2.98	4.26	6.52
8	4.23	5.87	8.73

Dimensional Changes Due to Thermal Cycling

The dimensional changes of plutonium rods were such that each specimen was slightly elongated and had a depression in each end. Also, there were both radial and longitudinal growth with the radius increasing appreciably more at the ends of the specimen than at the center. A slight roughening of the surface also occurred.

Specimens were also fractured as a result of thermal cycling. Figure 11 is a photograph of a sample that had fractured after ten cycles between the gamma and alpha phases. The fracture was quite smooth and uniform. The diameter of the sample at the fracture was the same as the initial diameter, whereas the diameter at each end of the sample had increased approximately ten per cent. Fracturing also occurred when plutonium was cycled between other phases. Figure 12 shows a specimen that had fractured when cycled between the delta and alpha phases. Both the radial and longitudinal dimension of the specimen increased with the largest radial growth being at the ends of the sample.

The dimensional changes of two specimens after 20 cycles between the beta and alpha phases were determined. A comparison of these specimens before and after cycling is shown in Figure 13. Prior to cycling the three cylindrical rods were 0.375 inch in diameter and 0.750-inch long.

Figure 13 and Table VI show that the specimens expanded both longitudinally and radially. The radial growth was four to five times greater at the ends than at the center.

TABLE VI
DIMENSIONS (INCHES) OF PLUTONIUM RODS
CYCLED TWENTY TIMES
BETWEEN THE BETA AND ALPHA PHASES
(approximately 2 C/hour)

	<u>Sample 1</u>		<u>Sample 2</u>	
	<u>Before</u> <u>Cycling</u>	<u>After</u> <u>Cycling</u>	<u>Before</u> <u>Cycling</u>	<u>After</u> <u>Cycling</u>
Diameter:				
Center of Specimen	0.375	0.406	0.375	0.393
End of Specimen	0.375	0.382	0.375	0.379
Opposite End	0.375	0.396	0.375	0.396
Length:				
Center of Specimen	0.750	0.830	0.750	0.816
Edge of Specimen	0.750	0.792	0.750	0.792
Opposite Edge	0.750	0.778	0.750	0.778

Influence of Thermal Gradients

A cylindrical plutonium rod 4.5 inches long and 0.375 inch in diameter was cycled 15 times between the beta and alpha phases with an average temperature gradient of 30 - 50 C per inch. One end of the rod was held at 50 C and the other end heated to 200 C. The bar was sectioned into 0.75-inch lengths and the density determined for each segment. The average density and the average maximum temperature of each piece are shown in Table VII and plotted in Figure 15.

TABLE VII
DENSITY VERSUS DISTANCE OF A PLUTONIUM ROD
SUBJECTED TO THERMAL CYCLING

<u>Distance (inches)</u> <u>From Cooler End</u> <u>of the Bar</u>	<u>Average</u> <u>Density</u> <u>g/cm³</u>	<u>Average</u> <u>Maximum</u> <u>Temperature</u>
3/8	19.45	70 C
1-1/8	19.48	95 C
1-7/8	18.95	130 C
2-5/8	16.50	170 C
3-1/8	15.13	190 C
3-7/8	15.53	200 C

Physical damage in the rod was in the form of voids which varied from micro-voids to large macro-cracks (Figure 16). There was also swelling with large pimpling on the surface of the specimen portion that had undergone repeated beta-to-alpha cycling. Metallography revealed that the zone between which there was an extensive amount of voids and no voids was quite small, approximately one millimeter.

Metallography showed that the distribution of voids across a transverse section of a cycled plutonium cylindrical rod, except at the ends, was uniform. Also, distribution of voids across a transverse section was very little different from that through a longitudinal section (Figure 2). At the extreme ends of the rods the distribution of voids was non-uniform. In addition, the shape of the voids at the ends of the rods was very irregular.

Influence of Imperfections

Many variables affected the amount of physical damage. The variable that most appreciably influenced the amount of void formation was the condition of the as-cast metal. This was perhaps best indicated by the densities of the as-cast metal. When the density of the metal was greater

than 19.60 g/cm^3 the density in some specimens decreased to only 18.8 g/cm^3 after 20 cycles. Conversely, the density decreased to as low as 14.0 g/cm^3 after only eight cycles when the density of the as-cast metal was 19.40 to 19.45 g/cm^3 . Plutonium metal having microstructures such as those shown in Figures 6 and 7 exhibited density decreases of approximately 0.6 g/cm^3 -cycle, whereas plutonium metal having very nearly no inclusions decreased approximately 0.03 g/cm^3 -cycle in density.

Effect of Void Formation on the Tensile Properties

Four one-fourth-inch tensile bars were cast slightly over-size, sealed in evacuated capsules and cycled ten times between the gamma and alpha phases. After cycling the tensile bars were machined to final size. The resulting tensile data are shown in Table VIII with a comparison to the average tensile properties of alpha metal that had not been cycled.

TABLE VIII

TENSILE PROPERTIES OF PLUTONIUM AFTER THERMAL CYCLING

No. of Cycles	Density (g/cm^3)		Ultimate Strength (psi)	Yield Strength 0.02% Offset (psi)	Diamond Pyramid Hardness	Impurity (ppm)
	Before Cycling	After Cycling				
0*	19.51	--	62,800	39,300	262	975
10	19.51	18.21	30,600	26,100	-	Approx 1000
10	19.45	18.27	33,900	27,100	-	Approx 1000
10	19.46	18.41	26,300	25,900	-	Approx 1000
10	19.45	18.16	29,800	26,300	180	Approx 1000

* Average tensile properties of as-cast unalloyed plutonium. ⁽⁷⁾

The data showed that the ultimate tensile strength, yield strength, and hardness decreased appreciably after thermal cycling. The decrease was due to the presence of voids as shown in Figure 14.

CONCLUSIONS

This investigation showed that the physical damage resulting from thermal cycling plutonium through its low temperature allotropic transformations was quite large. The density decreased as much as 23.40 per cent after only eight cycles through the beta-to-alpha phase transformation. Many variables affected the extent of physical damage, particularly inclusions and specimen size.

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FIGURE 1
Variation of the Density of
Plutonium During $\gamma \rightleftharpoons \alpha$ Cycling

(a)

Two cycles.
Magnification 100X.

(b)

Thirty-five cycles. Edge of
specimen - transverse section.
Magnification 50X.

(c)

Thirty-five cycles. Center of
specimen - transverse section.
Magnification 50X.

(d)

Thirty-five cycles. Center of
specimen - longitudinal section.
Magnification 50X.

FIGURE 2

Photomicrographs Showing Voids After Thermal Cycling
Between the Alpha Phase and the Gamma Phase

FIGURE 3
Variation of the Density
of Plutonium During $\alpha \rightleftharpoons \beta$ Cycling

FIGURE 4
Variation of the Density
of Plutonium During $\alpha \rightleftharpoons \delta$ Cycling

FIGURE 5

As-cast Plutonium Spheres
Used in the Thermal Cycling Studies
Diameters 1-1/2, 1, 3/4, 1/2, and 1/4 inch

FIGURE 6

Photomicrograph Showing Inclusions (predominantly carbides) of As-cast Spheres Shown in Figure 5
Magnification 100X

FIGURE 7

Same as Figure 6. Magnification 250X

FIGURE 8
Density Decrease of Plutonium Spheres
of Varying Diameter
Due to $\beta \rightleftharpoons \alpha$ Thermal Cycling

FIGURE 9

Effect of Specimen Diameter on the Density Decrease
Due to $\beta \rightleftharpoons \alpha$ Thermal Cycling

FIGURE 10

Effect of Specimen Size on the Density Decrease
of Plutonium When Thermally Cycled
Through the Beta-to-Alpha Phase Transformation

FIGURE 11

Fractured Plutonium Specimen after Thermal Cycling
Ten Times Between the Gamma and Alpha Phases

FIGURE 12

Fractured Plutonium Specimen after Thermal Cycling
Between the Delta and Alpha Phases

FIGURE 13

Plutonium Rods Thermally Cycled Between the Beta
and Alpha Phases (left and right)
Center Rod Was in the As-cast Condition

FIGURE 14

Photomicrograph of a Plutonium Tensile Specimen
Cycled Between the Beta and Alpha Phases
Magnification 50X

FIGURE 15

Density Distribution of a Plutonium Rod Subjected
to a Thermal Gradient of 30 - 50 C Per Inch

One-fourth Inch from the Transition Zone

Two Inches from the Transition Zone (see Figure 15)

FIGURE 16

Photomicrographs of a Plutonium Rod Subjected
to a Thermal Gradient of 30-50 C Per Inch
Magnification 50X

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