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PHYSICAL DAMAGE IN PLUTONIUM

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PHYSICAL DAMAGE IN PLUTONIUM

INTRODUCTION

When plutonium is transformed from beta to alpha, it sustains extensive physical damage as indicated by cracks. These cracks can be either microcracks or macrocracks depending upon the previous mechanical and thermal history of the metal. The most significant variables that influence the amount, size, and distribution of these cracks are the temperature of the $\beta \rightarrow \alpha$ transformation, the metal quality, the specimen size and shape, and the number of previous $\beta \rightarrow \alpha$ transformations.

The physical damage in plutonium after repeated $\beta \rightarrow \alpha$ transformations may be quite pronounced.^(1, 2, 3) Large macrocracks, or even fracture of the metal, may occur, however, after only one $\beta \rightarrow \alpha$ transformation. Coffinberry and Waldron⁽⁴⁾ reported that in early work at Los Alamos thermal cycling of plutonium between phases resulted in a progressive decrease in density and increase in surface distortion similar to that found in uranium subjected to transformation cycling. It has been suggested that the microcracks formed primarily during the $\gamma \rightarrow \beta$ transformation because this was the first transformation between two complex crystal structures on cooling from elevated temperatures.^(4, 5)

This report presents some additional experimental work and observations on physical damage in plutonium. The occurrence and extent of this damage has been evaluated largely on the basis of optical metallographic observations and precision density determinations.

SUMMARY AND CONCLUSIONS

High quality as-cast plutonium may contain a large volume of microcracks that have formed during the $\beta \rightarrow \alpha$ transformation. The volume of microcracks increases with increasing transformation temperature, higher metal purity, increased specimen size, and after $\alpha \rightleftharpoons \beta$ thermal cycling.

Thermal cycling produces growth in all directions, general distortion, and extensive surface rumpling. Plutonium containing no microcracks can be produced by quenching from the beta phase to -75 C during casting. No physical damage has been observed in metal that initially contains no microcracks after either $\gamma \rightleftharpoons \delta$, $\delta \rightleftharpoons \gamma$, or $\delta \rightleftharpoons \delta$ thermal cycling.

MICROCRACKING IN AS-CAST PLUTONIUM

Metallography and ultrasonic testing reveal that as-cast plutonium may contain a large volume of microcracks. The volume of these microcracks varies from less than 0.1% to as high as 2% of the total specimen volume depending upon the impurity content and the rate of $\delta \rightarrow \alpha$ transformation. Metal containing less than 200 ppm total impurities of which less than 100 ppm are delta stabilizers, such as aluminum, gallium, and americium, that is cooled through the $\delta \rightarrow \alpha$ transformation at 1 to 20 C/min will contain a distribution of microcracks similar to that revealed by the microstructure in Figure 1. These microcracks form predominantly at the final alpha grain boundaries as indicated in Figure 2.

The size, distribution, and concentration of microcracks generally vary considerably within a given piece of metal. The dependency of microcracks on the $\delta \rightarrow \alpha$ transformation rate and impurity level is discussed in greater detail in the section on thermal cycling damage. In general, a higher $\delta \rightarrow \alpha$ transformation temperature and higher purity will result in more extensive microcracking.

The microcracking appears to occur after the $\delta \rightarrow \alpha$ transformation is 50 to 60% complete. Loasby and Lowe⁽⁵⁾ have plotted the percent of void formation against the percent of beta that has transformed into alpha and obtained a linear relationship in the range 50 and 100% beta transformed. Nelson⁽³⁾ found this relationship to be as shown in Figure 3. The deviation depends upon such factors as specimen size, isothermal transformation temperature, and impurity content.

The volume associated with microcracks in as-cast plutonium is decreased only slightly by hydrostatic compression at 6000 atmospheres at a temperature of 90 C, which is within the alpha phase stability range. Accordingly, the metal density increases only slightly, 0 to 0.05 g/cm³. The major portion of the observed density change is the volume decrease due to transformation of retained phases. Hydrostatic pressing at the same pressure but at 180 C followed by slow cooling to the alpha phase underpressure results in larger density increases, 0.10 g/cm³ or more. The amount of increase depends upon the temperature of pressing and the extent of microcracks, inclusions, and other imperfections in the metal. If microcracks comprise 1% of the volume of metal and they are eliminated during compression, the density will increase 0.2 g/cm³. Densities up to 19.80 g/cm³ at 25 C have been obtained. Microcracks are not discernible at 250X after this treatment.

The slight density increase of high quality plutonium after pressing at 90 C is attributed primarily to transformation of a small amount of retained beta phase and not to decreasing the volume of microcracks. The density increase after pressing at 180 C is due primarily to decreasing the volume of microcracks and only a small percentage results from transformation of retained phases. The volume decrease associated with the transformation of retained phases is the same in either pressing operation; however, in beta pressing the volume decrease associated with compressing the microcracks is much greater than in alpha pressing. The amount of density increase brought about by pressing depends upon the previous history of the metal.

PHYSICAL DAMAGE AFTER REPEATED $\alpha \rightleftharpoons \beta$ TRANSFORMATIONS

Repeated $\alpha \rightleftharpoons \beta$ transformations result in an increase in the extent of internal micro- and macrocracking. The size of the cracks increases progressively with continued thermal cycling (Figure 4). The extent of thermal cycling damage depends upon the metal quality, sample size, and $\beta \rightleftharpoons \alpha$ transformation temperature during thermal cycling.

Plutonium that is thermally cycled through the $\beta \rightarrow \alpha$ transformation by heating and cooling at 2 C/min may form voids up to 3% per cycle (0.6 g/cm^3 density decrease per cycle), resulting in growth, general distortion, and extensive surface rumpling (Figure 5). Growth is always observed in both the radial and longitudinal directions. Occasionally one to four very slow $\beta \rightarrow \alpha$ transformations are sufficient to cause fracture of a specimen. Densities of certain specimens have decreased from 19.7 to 6 g/cm^3 after 100 thermal cycles.

Heating plutonium into one of the high temperature phases and cooling very slowly into the alpha phase region will cause macrocracks and even fracture after only one transformation cycle. Heating into the epsilon phase region and slow cooling produces the most damage. For example, a high purity plutonium specimen, 1 cm in diameter and 1 cm long that had been epsilon heat-treated at 600 C, cooled to 95 C at 10 C/hr and then cooled to 60 C at 1 C/hr contained a macrocrack 1/2 cm long and 1/16 cm wide. The cooling rate was changed at 95 C to permit a very slow $\beta \rightarrow \alpha$ transformation. The $\beta \rightarrow \alpha$ transformation was presumed complete at 60 C.

The physical damage is greater for metal transformed from the beta phase at high alpha temperatures, e. g., 70 C, than at subzero temperatures. Samples from the same melt were allowed to transform isothermally to the alpha phase at -80 C, -23 C, 25C, 60 C, and 73 C after beta heat treating 30 min at 180 C. Each specimen was allowed to transform at the appropriate alpha temperature until apparent completion of the transformation was observed. The densities after each transformation were then determined at room temperature (Figure 6).

After the first cycle, the decreases in density of the specimens transformed isothermally at -23 and -80 C were essentially identical, 0.12 g/cm^3 . This density decrease was due to both the retention of the lower density beta and the development of microcracks in the specimens. Further cycling to six cycles in the subzero range resulted in a very small density change and hence very little physical damage. Increasing the transformation

temperature above -23 C resulted in more extensive physical damage and less retained beta. The amount of damage was greatest in the metal transformed at 73 C with the density decrease being 0.40 g/cm^3 per cycle. The amount of damage was slightly less, 0.30 g/cm^3 per cycle in the specimen transformed at 60 C.

Plutonium rods 1 cm diameter or less that do not initially contain microcracks will not form microcracks upon repeated $\alpha \rightleftharpoons \beta$ transformations by direct quenching from the beta phase to subzero temperatures. Microcracks initially present within the metal, however, will be enlarged by such thermal cycling treatment. For many studies, such as elevated temperature creep, it is particularly advantageous to have sound metal. Plutonium is therefore quenched from the beta phase to a subzero temperature to provide a crack-free specimen. Plutonium metal quenched in this manner has a very fine-grained microstructure and is extremely brittle.

The following experiment and results verify the previous generalizations. Microcracks were formed in plutonium rods during the casting process by permitting the beta to transform to alpha slowly (high alpha temperatures). Such rods containing microcracks were thermally cycled 17 times by direct quenching into the beta phase at 180 C and into the alpha phase at a subzero temperature of -80 C. Such $\alpha \rightarrow \beta \rightarrow \alpha$ cycling was followed by cycling within the alpha phase (100 C to -80 C) to transform any retained phase present to alpha. The rods were 1 cm long and 1 cm in diameter. That the $\alpha \rightarrow \beta \rightarrow \alpha$ thermal cycling enlarged microcracks was confirmed by metallography and density measurements. Plutonium that was quenched from the beta phase during casting to avoid formation of microcracks and then thermally cycled in the same manner sustained no microcracking and therefore less physical damage. The microstructure and the density of the rods after the thermal cycling treatments were essentially the same as before the cycling treatments. The pertinent data from these experiments are shown in Table I.

TABLE I
EFFECT OF $\alpha \rightleftharpoons \beta$ TRANSFORMATION CYCLING
(- 80 $\rightleftharpoons$ + 180 C) ON THE DENSITY OF PLUTONIUM

	<u>Metal A</u>	<u>Metal B</u>	<u>Metal A</u>	<u>Metal B</u>
Density Before Cycling	19.60 g/cm ³ *	19.55 g/cm ³ *	19.72**	19.60**
Density After 17 $\alpha \rightleftharpoons \beta$ Cycles	18.98	19.24	19.68	19.56
Density After 5 Subsequent Cycles Within the Alpha Phase	19.04	19.35	19.72	19.61

* Contained Microcracks

** Contained no Microcracks

The temperature dependence of physical damage in cylindrical 1 cm rod specimens is presented in Figure 7. The data were obtained by two methods. First, the percent microcracking per cycle was determined from the average slope of the curves between Cycles 2 and 6 in Figure 6. Second, the density change was observed in metal that had been beta-pressed after casting, transformed to alpha under pressure, beta heat-treated 30 min at 160 C, quenched and isothermally transformed to alpha at different temperatures, and then finally thermally cycled between -75 C and 100 C to transform retained beta to alpha. A schematic of this procedure is shown in Figure 8.

Although microcracking in high purity plutonium is pronounced at high alpha transformation temperatures, the amount of retained phases in metal cycled between the beta phase and the high alpha temperatures is quite small. The retention of beta, however, is quite significant below room temperature, particularly during the first cycle.

The voids and microcracks, after repeated $\alpha \rightleftharpoons \beta$ transformations, are somewhat uniformly distributed throughout specimens, but large macrocracks have been observed at or near the center of specimens. For example, spheres approximately 1 1/2 in. in diameter, thermally cycled at 2 C/min between 175 and 25 C contained voids 1 to 1 1/2 cm in diameter at the center. Large macrocracks have also been observed at the center of thermally-cycled rods.

Increasing the specimen size generally increases the amount of void formation (Figure 9). However, 1/4 in. diameter rods have been shown to have nearly as large a density decrease as 1 in. diameter rods when thermally cycled at a rate of 2 C/min. Similar data have been obtained for spheres of varying diameter. This indicates specimen size is not as important as the metal quality or the $\beta \rightarrow \alpha$ transformation conditions at this slow transformation rate.

The effect of specimen size on microcracking increases with increasing transformation rate. The amount of microcracking is not great at subzero temperatures in small specimens, but rods or spheres greater than 1 1/2 in. diameter will readily fracture when quenched from the beta phase to subzero temperatures where the $\beta \rightarrow \alpha$ transformation rate is a maximum. This is due to large temperature differences within the metal which lead to the formation of the more dense and less ductile alpha phase at the exterior of the specimens, while the interior consists of the lower density beta phase that subsequently transforms and contracts away from the alpha. This leads to fracture.

The physical damage associated with thermal cycling between the beta and alpha phases depends upon the amount and type of impurities present in the metal. Elements such as iron and nickel, which have little solubility in solid plutonium, and elements such as aluminum, which have extensive solubility in the delta phase mitigate the cyclic thermal damage. Some

elements such as carbon may possibly enhance microcracking; however, no detailed investigation of this has been performed. Nickel or iron in the amount of 1000 ppm decrease the amount of microcracks to 1/4 that of unalloyed plutonium containing less than 250 ppm total impurities. Addition of 600 ppm aluminum decreases the amount of physical damage to 1/10 that of the same unalloyed plutonium. However, this amount of aluminum significantly increases the amount of the retained delta and beta phases.

PHYSICAL DAMAGE AFTER $\alpha \rightleftharpoons \gamma$ CYCLING

The physical damage associated with thermal cycling between the gamma and alpha phases increases with decreasing $\gamma \rightarrow \alpha$ transformation temperature as revealed by density measurements, Figure 10. This temperature dependence is in the reverse direction but the absolute magnitude is not as great as after repeated $\beta \rightarrow \alpha$ thermal transformation. However, the change in transformation rate between -75 C and +73 C of the $\gamma \rightarrow \alpha$ transformation is not as great as that for the $\beta \rightarrow \alpha$ transformation. This temperature interval is where the gamma phase transforms directly to the alpha phase. Fracture generally occurs after the first few cycles during either $\delta \rightleftharpoons \alpha$ or $\gamma \rightleftharpoons \alpha$ cycling in which the quenching medium is at -75 C. The fracture results from the large volume changes between phases. These volume changes are approximately 25 and 15% respectively.

The physical damage at temperatures where the gamma phase first transforms to the beta phase and then to alpha has not been measured, but is quite large. The least amount of damage occurs when the gamma phase transforms to the beta phase and the metal is then quenched to subzero temperature.

PHYSICAL DAMAGE AFTER $\gamma \rightleftharpoons \beta$, $\delta \rightleftharpoons \gamma$, AND $\delta \rightleftharpoons \beta$ CYCLING

No physical damage has been observed during the $\gamma \rightleftharpoons \beta$, $\delta \rightleftharpoons \gamma$, or $\delta \rightleftharpoons \beta$ thermal cycling in metal that initially contains no microcracks. Any microcracking that is initially present in the metal will be enhanced by these transformations. However, a 0.08 g/cm³ density decrease has been

observed after the first $\delta \rightleftharpoons \gamma$ cycle. This can be attributed to a retention of a small percentage of the delta phase at 190 C. A comparison of the physical damage associated with $\delta \rightleftharpoons \gamma \rightleftharpoons \beta \rightleftharpoons \alpha$ thermal cycling as revealed by density measurements is given in Figure 11. The data were obtained from metal that had been cooled only to the beta phase during casting. This metal supposedly contained no microcracks initially. Similar data were obtained from plutonium which had initially been hydrostatically pressed at 180 C and cooled under pressure.

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FIGURE 1

Microcracks in Electrorefined Plutonium
Small Inclusions are PuO_2
As-Polished, 100X

FIGURE 2

Microstructure of Electrorefined Plutonium
Showing Microcracks at the Grain Boundaries
Polarized Light Illumination, 200X

FIGURE 3

Void Formation as a Function of % $\beta \rightarrow \alpha$ Transformation at 73 C

One $\alpha \rightleftharpoons \beta$ Transformation
Cycle

Two $\alpha \rightleftharpoons \beta$ Transformation
Cycles

Four $\alpha \rightleftharpoons \beta$ Transformation
Cycles

FIGURE 4

Representative Photomicrographs
Showing Microcracking in Plutonium
Cycled Between the Beta and Alpha Phases 50X

(a) (b) (c)

FIGURE 5

Plutonium Rods Thermally Cycled Between 180 C (beta phase) and Room Temperature (alpha phase) at an Approximate Rate of 2 C/min.

a) No Cycling

b) 50 Cycles

c) 100 Cycles

FIGURE 6

Alpha $\rightleftharpoons$ Beta Transformation Damage

FIGURE 7
Microcracking in Plutonium
as a Function of the Transformation Temperature

FIGURE 8

Schematic Diagram
Showing the Procedure for Determining the vol% of Microcracks
and Retained Phases in a Specimen by Density Measurements

FIGURE 9

Effect of Specimen Size on Density Decrease of Plutonium
when Thermally Cycled Through $\alpha \rightarrow \beta$ Transformation
at a Cooling Rate of 2 C°/min

Alpha $\rightleftharpoons$ Gamma Transformation Damage

FIGURE 10
Alpha $\rightleftharpoons$ Gamma Cyclic Damage

FIGURE 11

Physical Damage in Plutonium Due to Phase Transformation Cycling

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