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THE HEAT TREATMENT OF PLUTONIUM

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THE HEAT TREATMENT OF PLUTONIUM

INTRODUCTION

The element plutonium is unique among metals in that it has six allotropic forms in the solid state. These have been designated as the alpha, beta, gamma, delta, delta-prime, and epsilon phases. The respective phase transformations occur at approximate temperatures of 122, 310, 319, 450, and 480 C with a melting point at 640 C.

With this number of phase transformations it becomes pertinent to consider the effect of heat treatment and cooling rate in the various phases on the microstructure and hardness of plutonium. Initially to determine the effect of a wide range of cooling rates it was decided to employ the Jominy end-quenching technique which is commonly used to study the hardenability of steel.

In addition, since the presence of iron in amounts greater than 500 ppm is common in plutonium and results in a network of the Pu-Pu₆Fe eutectic it was decided to study heat treatment effects on two bomb-reduced plutonium buttons with different iron contents. A low-iron button containing 165 ppm Fe and a high-iron button containing 678 ppm Fe were selected for this comparison.

SUMMARY AND CONCLUSIONS

Two groups of plutonium bars, one group containing 165 ppm iron and the other 678 ppm iron were end-quenched from the beta, gamma, delta, delta-prime and epsilon phases.

In general, cooling rate was found to have three striking effects on microstructure. The alpha grain size was found to decrease with increasing cooling rate during cooling from all phases. Upon cooling high-iron plutonium from the epsilon phase, the size of the Pu-Pu₆Fe eutectic network decreases with increasing cooling rate. Cooling high-iron plutonium from temperatures between 413 and 460 C produces a wide variety of Pu-Pu₆Fe configurations in the microstructure. Finally, the size of an unidentified spheroidal inclusion,

soluble in the delta and higher temperature phases, decreases as cooling rate increases across the delta to gamma transformation. The above effects of cooling rate can be used to indicate the cooling rate or thermal history that a particular plutonium specimen has encountered.

A linear relationship between the alpha grain size and the Pu-Pu₆Fe eutectic network size was observed. The network size is roughly two times larger than the alpha grain size.

Hardness traverses on each of the bars indicated no apparent effect of cooling rate during quenching on hardness.

A density decrease was noted near the quenched end of both low and high-iron bars which indicates that faster cooling rates promote microcracking. However, a greater decrease in density was obtained for the low-iron bars which was attributed to their greater tendency to microcrack. The presence of the Pu-Pu₆Fe eutectic network in the high-iron bars inhibits microcracking.

EXPERIMENTAL PROCEDURE

Experimental Material

The cylindrical bars for Jominy quenching were cast from button stock in vacuo in MgO coated graphite molds. Metal pouring temperature was approximately 950 C and the molds were preheated to 300 C prior to pouring. After removal from the mold the specimens were cold treated at -34 C for 24 hours and then machined to 0.5 inch diameter by 2.5 inch long cylinders.

Chemical analysis and density data for the two groups of Jominy specimens containing different iron contents are presented in Table I. Except for iron the two groups are comparable within the limits of analytical accuracy.

TABLE I
CHEMICAL ANALYSIS⁽¹⁾ AND DENSITY DATA

<u>Casting Heat</u>	<u>Al</u>	<u>Ca</u>	<u>Cr</u>	<u>Cu</u>	<u>Fe</u>	<u>Mg</u>	<u>Mn</u>	<u>Ni</u>	<u>Si</u>	<u>Zn</u>	<u>C</u>	<u>Total</u>	<u>Total Excluding Iron</u>	<u>Density (g/cc)</u>
19-12-1	32	2	100	50	678	5	50	100	<1	50	60	1128	450	19.55
20-2-1	3	3	10	<5	165	20	50	20	5	<50	50	381	216	19.66

(1) Reported in parts per million.

Representative specimens from the low-iron and high-iron bars were taken for metallographic examination. The low-iron specimen was found to have extensive microcracking (Figure 1). In addition, numerous unidentified spheroidal inclusions were present (Figure 18). The average grain size of the low-iron plutonium is 0.068 mm (Figure 5). In the high-iron plutonium, a Pu-Pu₆Fe eutectic network is prominent (Figure 8). The average size of the network is 0.100 mm. Unidentified spheroidal inclusions were also common in the high-iron plutonium. The average grain size of the high-iron plutonium is 0.036 mm.

Experimental Technique

A Jominy end-quenching fixture was fabricated for glove box use (Figure 2). A 2.5-inch water height was used with an orifice of 0.250 inch ID. The orifice to specimen distance was maintained at 0.5 inch. To obtain cooling curves during end-quenching, radial holes 0.040 inch in diameter by 1/8 to 1/4 inch deep for thermocouples were drilled at distances from the quenched end of 1/16, 1/8, 3/8, 3/4, 1-1/4, and 2 inches. Chromel-alumel thermocouples were peened into each hole prior to heat treatment. Cooling curves were recorded on three strip chart recorders with 0.25 second full scale response times and chart speeds of 6, 12, and 20 inches/minute. The temperature scale on each recorder was adjusted to obtain maximum sensitivity over the temperature range covered by each quench.

Annealing temperatures of 160, 265, 400, 465, and 535 C were chosen for the study of the effect of quenching from the beta, gamma, delta, delta-prime, and epsilon phases on microstructure and hardness. At the 160 and 265 C temperatures an agitated peanut oil bath was used for annealing. For the higher temperatures a 50:50 NaNO_2 - KNO_3 salt bath was used. Annealing times ranged from 25 to 65 minutes in the oil bath while in the salt bath a 5 minute time was used. The quench water temperature ranged from 5-10 C.

From the cooling curves obtained, cooling rate calculations were made for temperature intervals of 100-110, 160-170, 330-340, 420-430, and 490-500 C. These temperature intervals were chosen for their proximity to transformation temperatures because it was felt that they would be critical for determining the effect of cooling rate on structure. The cooling rate versus distance from the quenched end curves for each annealing temperature are presented in the Appendix.

After end-quenching, parallel flats for hardness testing were milled 0.015 deep on each specimen. Hardness traverses were made at 1/16 inch intervals for the first 1/2 to 3/4 inch and at 1/8 inch intervals for the rest of the bar. Hardness testing was done on a glove boxed Tukon Hardness Tester using a 136 degree diamond indenter and a 10 kg load.

After hardness testing each specimen was cut into three equal sections. A density determination was made on the section including the quenched end of the bar. All sections were mounted in cold setting resin for metallographic study. The metallographic specimen preparation procedure is described elsewhere.⁽¹⁾

Results and Discussion

During the metallographic studies on the end-quenched Jominy specimens three striking effects on microstructure were observed. The alpha grain size, the configuration of the $\text{Pu-Pu}_6\text{Fe}$ eutectic network and the size of an unidentified spheroidal inclusion were all markedly affected by cooling rate.

The effect of cooling rate on alpha grain size as indicated by distance from the quenched end of the Jominy specimens is illustrated in Table II. If distance from the quenched end is translated into cooling rate by means of the cooling rate curves in the Appendix, it is possible to graph grain size as a function of cooling rate. This relationship is illustrated in Figure 3 for cooling rates determined in temperature intervals of 100-110 C, 160-170 C, 330-340 C and 490-500 C while quenching from the beta, gamma, delta and epsilon phases respectively. From Table II and the above figure it is evident that for all phases grain size increases as cooling rate decreases, i.e., as distance from the quenched end increases. In addition, the effect of increasing the annealing temperature prior to end-quenching is to decrease the grain size at a particular position in the Jominy specimens. For example, at the quenched end of the Jominy specimens, the marked decrease in grain size as annealing temperature increases is illustrated in Figure 4.

Photomicrographs illustrating the effect of cooling rate on grain size for specimens end-quenched from the beta, gamma, delta, and epsilon phases are presented in Figures 5 and 6. Note that the grains present in the beta quenched specimen are very nonuniform in size and shape while those in the specimens quenched from higher temperatures are relatively uniform and equiaxed.

The second effect of cooling rate on microstructure concerns the configuration of the Pu-Pu₆Fe eutectic network in the high-iron Jominy specimens. Since the eutectic between Pu and Pu₆Fe melts at 413 C it was expected that this would be a critical temperature for heat treatment studies. In addition, the British work on the plutonium-iron phase diagram by Mardon, Haines and Pearce⁽²⁾ (Figure 7), indicated that a variety of substantially different structural configurations of the Pu-Pu₆Fe eutectic could be obtained by heat treating at various temperatures above 413 C.

In the high-iron Jominy specimen quenched from 527 C in the epsilon phase it was found that the eutectic network size was 0.003 mm in diameter at the quenched end and increased by a factor of 10 to 0.035 mm as cooling

TABLE II

ALPHA GRAIN SIZE VERSUS DISTANCE FROM QUENCHED END OF JOMINY SPECIMENS

Specimen Number	19-12-1A	20-2-1L	19-12-1D	20-2-1N	19-12-1E	20-2-1P	19-12-1G	20-2-1Q	19-12-1H	20-2-1O
Annealing Temperature ($^{\circ}\text{C}$)	156	166	270	266	396	420	473	465	533	537
As-Cast Grain Size (mm)	0.036	0.068	0.036	0.068	0.036	0.068	0.036	0.068	0.036	0.068
Quenched End	0.010	(1)	0.008	0.010	0.003	0.003	0.002	0.002	0.001	0.001
0.1	0.012	(1)	0.015	--	0.007	--	0.006	--	0.003	0.002
0.2	--	(1)	--	0.014	--	0.006	0.010	0.006	0.006	0.008
0.3	0.013	(1)	0.020	--	0.009	--	0.012	--	--	0.012
0.4	--	(1)	0.020	0.027	--	0.009	--	0.010	0.009	--
0.5	--	(1)	--	--	0.013	--	0.014	--	--	0.020
0.6	0.020	(1)	0.020	0.035	--	0.012	--	0.012	0.009	--
0.9	--	(1)	--	0.040	0.014	0.014	0.014	0.014	0.010	0.024
1.6	(1)	(1)	0.024	0.040	0.020	0.014	0.014	0.014	0.010	0.024
2.5	(1)	(1)	0.024	0.040	0.020	0.014	0.014	0.014	0.010	0.024

(1) Wide range of grain sized present, not possible to get good estimate.

rate decreased along the length of the specimen (Figure 8). The original as-cast network size was 0.100 mm. Graphically the relation between cooling rate and eutectic network size is illustrated in Figure 9. The process by which the network forms is a eutectoidal decomposition of the epsilon phase at 430 C to delta plutonium plus a liquid which is distributed around the islands of delta plutonium. The liquid network further decomposes in a eutectic reaction at 413 C to delta plutonium plus Pu_6Fe .

To further explore the effect of heat treatment temperature on eutectic configuration, the temperature ranges of 430 to 460 C and 413 to 430 C were chosen for study from the plutonium-iron phase diagram. Plutonium analyzing 1142 ppm Fe with an as-cast network size of 0.070 mm was used for this phase of the work (Figure 10).

Between 430 and 460 C in the delta plus epsilon phase region, quenching results in a mixture of islands of plutonium plus islands of a fine network of the $\text{Pu-Pu}_6\text{Fe}$ eutectic (Figures 11 and 12). The islands of fine eutectic network are obtained during the eutectoidal decomposition of the epsilon phase. The variation in network size between the structures illustrated in Figures 11 and 12 is the result of a variation in cooling rate during quenching. The somewhat coarser network in Figure 12 was caused by a slower cooling rate during quenching.

Between 413 and 430 C, the stable phases are delta plutonium plus liquid. The structure obtained on quenching from this temperature is illustrated in Figure 13. The large islands are agglomerated $\text{Pu-Pu}_6\text{Fe}$, the matrix is transformed delta, i.e., alpha plutonium.

The effect of a slow cool at 2.7 C/hr from temperatures above 460 C on the $\text{Pu - Pu}_6\text{Fe}$ eutectic network is illustrated in Figure 14. The network is approximately 0.3 mm in average diameter. This result is graphed on Figure 9. Note the islands of alpha plutonium apparent in the Pu_6Fe matrix of the network.

During the course of this work it was determined that the $\text{Pu-Pu}_6\text{Fe}$ eutectic network will spheroidize in either the gamma phase or delta phase

below 413 C (Figures 15 and 16). Note the boundaries of equiaxed grains apparent in Figure 16 where the spheroidized Pu-Pu₆Fe inclusions appear as dark circular areas as the result of a longer etching time than used for the specimen in Figure 15. The equiaxed grains are the grains present in the delta phase at the 400 C annealing temperature used for this specimen which was quenched after annealing. They average 0.050 mm in diameter compared to an alpha grain size of 0.010 mm as viewed under polarized illumination. Thus the grain size has been refined by a factor of five during quenching through the subsequent phase transformations to room temperature.

In the Jominy and other experimental specimens containing the Pu-Pu₆Fe eutectic network which were continuously cooled from the epsilon phase, it is possible to determine the relation between the grain size outlined by the network and the alpha grain size of plutonium. A linear relation is obtained (Figure 17). Algebraically the relation is expressed by the equation $y = 2.25x + 0.0032$ where y is the network size and x is the alpha grain size. Thus the alpha grain size is always finer than the network size by roughly a factor of 2. Since the network size is actually the grain size present in the epsilon phase, it is evident that the additional alpha grain refinement takes place during subsequent lower temperature phase transformation.

It is pertinent to note that in high-iron plutonium, the existing network size or Pu-Pu₆Fe configuration can be used as an indication of the thermal history, i.e., cooling rate, or control thereof during casting or heat treatment.

Considering the variation in configuration of the Pu-Pu₆Fe eutectic network which can be obtained by heat treatment, it would not be surprising if significant effects on properties could be produced by heat treatment of plutonium containing 500-1500 ppm Fe.

A third significant metallographic feature was observed in the Jominy bars quenched from the delta and higher temperature phases. The size of an unidentified spheroidal inclusion was observed to vary with cooling rate

(Figure 18). It was subsequently determined that this inclusion is soluble in the delta and higher temperature phases of plutonium. During quenching to room temperature the inclusion precipitates and its size is influenced by cooling rate. Inclusions of this type have been grown as large as 0.001 inches by cooling at 2.7 C/hr across the delta to gamma transformation. It has also been determined that this type of inclusion will preferentially precipitate at carbide and nitride inclusions (Figure 19). Considering the spheroidal shape of this inclusion, it is not probable that it will produce any great effect on the properties of plutonium.

Hardness traverses made along the length of specimens end-quenched from all phases did not indicate a significant effect of either annealing temperature, or cooling rate on hardness (Figure 20). This was true of both low-iron and high-iron plutonium. There was more scatter in the low-iron hardness data and this was attributed to the much greater frequency of microcracking in the low-iron plutonium (Figure 1). The high-iron plutonium was practically microcrack free, because of the microcrack inhibiting action of the Pu-Pu₆Fe eutectic.⁽¹⁾

Since it is believed that most of the microcracking in plutonium occurs during the beta to alpha transformation, the effect of cooling rate on microcracking as indicated by a decrease in density near the quenched end was studied by using the cooling rate at 0.1 inches from the quenched end over a temperature interval of 100-110 C. In Figure 21, the data are presented for both high and low-iron plutonium. As expected, based on the inhibiting effect of the Pu-Pu₆Fe eutectic on microcracking, the low-iron specimens had the greatest decrease in density indicating a greater number and/or size of microcracks.

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50 X

FIGURE 1

Microcracking Present in Low-Iron Plutonium, 165 ppm Fe

1/2 X

FIGURE 2

Apparatus for End-Quenching Plutonium

As-Cast 0.068 mm

Quenched End 123 C/min 27 C/min

End-Quenched from 160 C in Beta Phase, Cooling Rate for 100-110 C

Quenched End 130 C/min 48 C/min
0.010 mm 0.037 mm 0.040 mm

End-Quenched from 265 C in Gamma Phase, Cooling Rate for 160-170 C

FIGURE 5

Effect of Cooling Rate on Grain Size for Beta and Gamma Quenches
150 X

Quenched End
0.003 mm

520 C/min
0.009 mm

143 C/min
0.014 mm

End-Quenched from 420 C in Delta Phase, Cooling Rate for 330-340 C

Quenched End
0.001 mm

2300 C/min
0.008 mm

510 C/min
0.024 mm

End-Quenched from 535 C in Epsilon Phase, Cooling Rate for 490-500 C

FIGURE 6

Effect of Cooling Rate on Grain Size for Delta and Epsilon Quenches
150 X

FIGURE 7

The Plutonium-Rich Portion of the Plutonium-Iron Equilibrium Diagram

As Cast
0.100 mm

Quenched End
0.003 mm

5700 C/min
0.006 mm

1080 C/min
0.019 mm

600 C/min
0.023 mm

190 C/min
0.035 mm

FIGURE 8

Effect of Cooling Rate on Size
of Pu-Pu₆Fe Eutectic Network in Plutonium Containing 678 ppm Fe
End Quenched from 533 C, Cooling Rate for 420-430 C
150 X

FIGURE 10

As Cast, 1143 ppm Fe. Pu-Pu₆Fe network, 0.070 mm
150 X

FIGURE 11

Annealed at 460 C and Water Quenched.
Note Islands of Fine Grained Pu-Pu₆Fe Eutectic Network
150 X

150 X

FIGURE 12

Annealed at 460 C and Air Cooled Briefly Prior to Water Quenching.
Islands of Pu-Pu₆Fe Eutectic Network Coarser Grained
than Preceding Figure

150 X

FIGURE 13

Annealed at 430 C and Water Quenched.
Note Islands of Pu-Pu₆Fe Eutectic

FIGURE 14

Annealed at 500 C for Several Hours and Cooled to 350 C at 2.7 C/hr. Pu-Pu₆Fe Network Coarse, 0.3 mm with Substructure Apparent.

150 X

FIGURE 15

Spheroidization of Pu-Pu₆Fe Eutectic Obtained During Annealing in Gamma Phase or Delta Phase Below 413 C

150 X

FIGURE 16

Spheroidized Pu-Pu₆Fe (dark circles)
Note Boundaries of Equiaxed Grains with Average Grain Size of 0.050 mm

150 X

As-Cast

Quenched End

1600 C/min

820 C/min

375 C/min

143 C/min

FIGURE 18

Effect of Cooling Rate on Size
of Unidentified Spheroidal Inclusions in Plutonium
End-Quenched from 420 C, Cooling Rate for 330-340 C
250 X

500 X

250 X

FIGURE 19

Spheroidal Precipitate Nucleated at Carbide Inclusions (needles) in
Upper Photo, and at Pu-Pu₆Fe Eutectic Network in Lower Photo
6187, 5843

APPENDIX

The graphs on the following pages indicate the relationship between cooling rate and distance from the quenched end of the plutonium Jominy specimens. Curves are presented for end-quenching from the beta, gamma, delta and epsilon phases.

Cooling rates were determined at temperature intervals considered to be critical from the standpoint of effect on metallurgical characteristics.

FIGURE 3

Relation Between Cooling Rate and Alpha Grain Size
for End-Quenching from Beta, Gamma, Delta and Epsilon Phases

FIGURE 4

Relation Between Annealing Temperature and Grain Size
of Plutonium at Quenched End of Jominy Specimen

FIGURE 9

Relation Between Cooling Rate and Pu-Pu₆Fe Eutectic Network Size

FIGURE 17

Relation Between Alpha Plutonium Grain Size and Grain Size
of Pu-Pu₆Fe Eutectic Network

FIGURE 20

Hardness Versus Distance from Quenched End of Jominy Bar

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