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THE STEADY-STATE CREEP
OF HIGH-PURITY PLUTONIUM

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THE STEADY-STATE CREEP
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R. D. Nelson, T. K. Bierlein,
and F. E. Bowman

Metallurgy Research Section
Reactor and Materials Technology Department

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PACIFIC NORTHWEST LABORATORY
RICHLAND, WASHINGTON

THE STEADY-STATE CREEP OF HIGH-PURITY PLUTONIUM

INTRODUCTION

This report presents results of steady-state, compressive creep experiments on the six allotropes of plutonium. These results are discussed and correlated with similar information about more common metals. Steady-state creep data were needed for the analysis of transformation creep data which provided information on the type of mechanism that might be responsible for these transformations.

SUMMARY AND CONCLUSIONS

Compressive creep experiments were performed on the six allotropes of plutonium at constant stress and isothermal conditions. The following conclusions were made:

- The creep rates of the allotropes formed by heating a low temperature-stable allotrope decrease in the order of

$$\epsilon > \delta' > \delta > \beta > \gamma > \alpha \quad .$$
- The creep rates of the allotropes formed by cooling a high temperature-stable allotrope decrease in the order of

$$\epsilon > \delta' > \delta > \gamma > \beta > \alpha \quad .$$
- For strains between 10^{-4} and 10^{-1} hr^{-1} the steady state creep rates of the beta and gamma phases vary as the fifth power of stress; the steady-state creep rate of the delta phase varies as the sixth power of stress.
- The average activation energies for steady-state creep based on an Arrhenius relationship are 26, 30, 35, 31, 28, and 25 kcal/g-atom for the α , β , γ , δ , δ' , and ϵ phases, respectively.
- Activation energies for self-diffusion were calculated from an empirical expression that utilizes modulus compensated creep rates. These activation energies are 23, 27, 31, 30, 28, and 25 kcal/g-atom for the α , β , γ , δ , δ' , and ϵ phases, respectively.

EXPERIMENTAL PARAMETERS

The metal used in this investigation was high-purity as-reduced plutonium that had been vacuum melted and vacuum cast. The americium content was estimated to be 100 ppm. Typical impurity contents were: 50 ppm Fe, 60 ppm C, 50 ppm U, 20 ppm Ni, 10 ppm Al, 5 ppm Mg, and 10 ppm Si; the concentration of all other impurities was considered to be less than 5 ppm. The high value for the density of the metal, greater than 19.60 g/cm^3 , was considered to be a necessary confirmation of metal quality and purity. ⁽¹⁾

The compressive creep experiments were performed on solid cylindrical rod specimens. The metal was melted in a tantalum crucible by heating to 1000 C in a vacuum of 10^{-4} torr and then cast into preheated graphite molds coated with a fused alumina and silica paint. The preheat temperature of the molds was 550 C. After the ingots had cooled to approximately 150 C, they were transferred to an oil bath at 110 C and held at temperature for 2 hr so the metal could transform completely to beta. Transformation of beta to alpha was accomplished by a procedure that minimizes microcracking introduced during the $\beta \rightarrow \alpha$ transformation. This procedure calls for quenching to a temperature of -80 C or lower followed by cycling between -80 and +100 C ⁽²⁾ to transform any retained phases.

The rods were machined to a final diameter of either 0.200, 0.250, 0.375, or 0.500 in. and cut to a length equal to one and one-half times the diameter. The 0.200 and 0.250 in. diameter specimens were used for alpha, beta, and gamma creep studies. Because the delta, delta-prime, and epsilon phases have creep rates which are high compared to those of the low temperature allotropes, larger, 0.375 and 0.500 in. diameter specimens were used for these phases. Barrelling and total strain for a given load is less in specimens with a large cross-section. Consequently errors in measurement are also reduced. The crystal structures of the allotropes of plutonium, temperature ranges of stability, and densities are given in Table I.

TABLE I

CRYSTAL STRUCTURE, RANGES OF STABILITY,
AND DENSITIES FOR PLUTONIUM ALLOTROPES

<u>Phase</u>	<u>Crystal Lattice</u>	<u>Number of Atoms per Structure Cell</u>	<u>Stability Range, C</u>	<u>Calculated Density, g/cm³</u>
Alpha	Simple Monoclinic	16	Below 112	19.82 (25 C)
Beta	Body-Centered Monoclinic	34	112-185	17.8 (150 C)
Gamma	Face-Centered Orthorhombic	8	185-310	17.14 (235 C)
Delta	Face-Centered Cubic	4	310-451	15.92 (320 C)
Delta Prime	Body-Centered Tetragonal	2	451-480	16.00 (480 C)
Epsilon	Body-Centered Cubic	2	480-640	16.51 (510 C)

Creep testing was performed at a pressure of 10^{-3} torr in the apparatus shown in Figure 1. Tantalum sheet, 0.07 mm thick, was placed between the sample and the steel platens to prevent alloying of the plutonium and steel. Alloying occurs quite readily above 415 C due to the formation of a low melting Pu-Pu₆Fe eutectic.

FIGURE 1

Apparatus in Which the Creep
Experiments were Performed
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The experiments were performed in the glovebox shown in Figure 2. The flange of the furnace vacuum tube located below the glovebox, was welded directly to the floor of the glovebox. Power to the tube furnace heater equipped with a control thermocouple was supplied by a controller-variatic combination. A thermocouple attached to the specimen showed that the temperature during an experiment was maintained within $\pm 1\text{ C}^\circ$. Relative displacement of the two platens was used as a measure of the creep strain of the specimen. Two externally positioned differential transformers sensitive to 0.0001 in. changes and two dial indicators graduated in units of 0.0001 in. were used. The engineering strain, (e) , was determined as a function of time and then converted to true creep strain, ϵ , by the relationship $\epsilon = \ln (1 + e)$.

FIGURE 2

Glovebox and Instrumentation for Performing
the Compression Creep Experiments

The experiments were performed at constant stress under isothermal as well as under variable temperature conditions. The stress was maintained constant by increasing the load on the platen in direct proportion to the increase in the cross-sectional area of the specimens. The actual stress deviated from the prescribed stress by an amount less than 0.5%. The determination of the cross-sectional area was based on the assumptions that the volume of the specimen remained constant, and that barrelling of the specimen did not occur during the test. Experimentally, the latter assumption was true for strains up to 0.20. Thus, the experiments were generally terminated before this strain was reached.

Before each test, the specimens were heated to a temperature high in the range of stability of the phase that was to be tested and held at this temperature for at least 2 hr. Specimens were then brought to the desired test temperature.

The data presented in this report apply only to specimens that were obtained by transformation from the phase stable at a low temperature to the phase stable at the higher temperature. For example, beta phase specimens were obtained by heating the specimens from the alpha phase region to 180 C, holding for 2 hr, and then cooling to the desired test temperature in the range of beta stability. Transforming specimens from a low temperature phase to the phase stable at the higher temperature was adopted as a standard procedure because the creep rate of beta formed from gamma was observed to be different than the creep rate of beta formed from alpha.⁽³⁾

EXPERIMENTAL RESULTS

Representative compression creep curves obtained at different stresses and temperatures for the different allotropes of plutonium are shown in Figures 3 and 4. The creep curves are typical in that they consist initially of primary creep followed by secondary or steady-state creep.

FIGURE 3

Compression Creep Curves of Plutonium

FIGURE 4

Compressive Creep Curves of Plutonium

Sherby has derived an empirical expression relating strain rate to temperature and stress.⁽⁴⁾ A more recent version of this equation has the form^(5, 6)

$$\dot{\epsilon} = K \gamma^{3.5} D \left(\frac{\sigma}{E} \right)^n \quad (1)$$

where

K = constant for all metals

γ = stacking fault energy

σ = applied stress

n = constant equal to about 5

E = modulus of elasticity

$D = D_o e^{-Q_{sd}/RT}$

In the expression for D,

D_o = universal constant

Q_{sd} = activation energy for self diffusion

T = test temperature in degrees Kelvin

R = universal gas constant.

The original equation⁽⁴⁾ contained a grain size term; the more recent equation is grain size independent. Because the grain size of the allotropes, excluding alpha, can be neither determined nor maintained constant, the new form of the equation has obvious advantages in analyzing creep data of several allotropes of the same metal. The stacking fault energies of the six allotropes of plutonium are not known, but they were assumed to be constant during the creep tests. The values for the moduli of elasticity of the different allotropes over a range of temperatures are given in Table II.

TABLE II
VALUES FOR THE MODULUS OF ELASTICITY⁽⁷⁾

Phase	Temperature, C	Modulus of Elasticity, psi
Alpha	115	12.3×10^6
Beta	115	6.2
Beta	185	5.5
Gamma	185	5.5
Gamma	305	4.6
Delta	305	2.5
Delta	450	2.4
Delta Prime	450	(a)
Epsilon	475	(a)
Epsilon	475	(a)

(a) No modulus of elasticity values available.

Sufficient data were obtained for some of the allotropes to conclude that there was a range of strain rate values where n is a constant and is approximately equal to 5. For low values of $\dot{\epsilon}$, n is less than 5, and for high strain rates n is greater than 5. Most of the experimental work was performed at stresses where $\dot{\epsilon} \propto \left(\frac{\sigma}{E}\right)^5$. The applied stress was compensated for the modulus of elasticity according to Equation (1) so that the ratio $\frac{\sigma}{E}$ would remain constant at different temperatures. Representative data showing the dependency of the creep rates of the beta, gamma, and delta phases on the modulus compensated stress, i. e., $\frac{\sigma}{E}$, are shown in Figures 5 through 7. It is apparent from Figure 5 that n has a value of 6 for delta; n has a value of 5 for most metals.

FIGURE 5

Dependency of the Creep Rate of Beta Plutonium on $\frac{\sigma}{E}$

FIGURE 6
Dependency of the
Creep Rate of Gamma
Plutonium on $\frac{\sigma}{E}$

FIGURE 7
Dependency of the
Creep Rate of Delta
Plutonium $\frac{\sigma}{E}$

Because the creep rate of the alpha phase was low at stresses below 15,000 psi, and because the stable temperature ranges of the delta-prime and epsilon phases are relatively high, only a limited amount of data was obtained on the creep of the alpha, delta-prime, and epsilon phases. Only enough data on these three phases were obtained to compare all six of the allotropes.

Figure 8 permits a comparison of the logarithm of the steady-state creep rate versus reciprocal temperature for all plutonium allotropes. There was a very marked difference between the creep rates at the same stress of two, single phase allotropes, one stable and the other unstable, that can each exist at temperatures where transformation is extremely sluggish. If the creep rate and temperature are related by the Arrhenius equation, $\dot{\epsilon} = Ae^{-Q_c/RT}$, then the creep activation energy can be determined from the slope of the plots in Figure 8, i. e.

$$Q_c = -\frac{2.3 R d \log \dot{\epsilon}}{d (1/T)} \quad (2)$$

FIGURE 8
Temperature Dependency
of the Creep of Plutonium
(The creep stresses are
in psi.)

The average values for Q_c are 26, 30, 35, 31, 28, and 25 kcal/g-atom, respectively, for the α , β , γ , δ , δ' , and ϵ phases. The activation energy, Q_c , is related to the self-diffusion activation energy by the relationship:⁽⁸⁾

$$Q_c = Q_{sd} - \frac{nRT^2}{E} \frac{dE}{dT} \quad (3)$$

At a given temperature the creep rate of the phase normally stable at higher temperatures is greater than the creep rate of the phase normally stable at the lower temperatures with but one exception--the creep rate of gamma is less than the creep rate of the beta phase near the $\beta \rightleftharpoons \gamma$ equilibrium transformation temperature. It is to be noted that this exception applies only to beta that is formed from alpha. The creep rate of beta material formed from alpha is 200 to 500 times greater than the creep rate of beta formed from gamma tested at the same temperature and stress conditions.⁽⁹⁾ Consequently, the creep rate of beta that is formed from gamma is less than the creep rate of the gamma phase under the same temperature and stress conditions. The effect of prior transformation on the mechanical properties of beta plutonium will be discussed in a subsequent report.

The activation energy for self-diffusion can be calculated from the strain rates at two temperatures according to the relationship:

$$\frac{\dot{\epsilon}_1}{\dot{\epsilon}_2} = \frac{K\gamma^{3.5} \left(\frac{\sigma}{E_1} \right)^n D_o e^{-Q_{sd}/RT_1}}{K\gamma^{3.5} \left(\frac{\sigma}{E_1} \right)^n D_o e^{-Q_{sd}/RT_2}} \quad (4)$$

If the ratio $\frac{\sigma}{E}$ is held constant by experimentally changing σ to compensate for the temperature dependency of E , then the relationship becomes:

$$\frac{\dot{\epsilon}_1}{\dot{\epsilon}_2} = \frac{e^{-Q_{sd}/RT_1}}{e^{-Q_{sd}/RT_2}} \quad \left(\text{Constant } \frac{\sigma}{E} \right) \quad (5)$$

The activation energy, Q_{sd} , can also be calculated at constant stress using known values of E and n from the relationship:

$$\frac{\dot{\epsilon}_1 E_1^n}{\dot{\epsilon}_2 E_2^n} = \frac{e^{-Q_{sd}/RT_1}}{e^{-Q_{sd}/RT_2}} \quad (6)$$

In the present investigation the technique of holding the stress constant and compensating for the modulus was used rather than holding $\frac{\sigma}{E}$ constant. Representative data of the temperature dependency of the modulus compensated strain rate are given in Figure 9. The moduli used for compensating the strains of the delta-prime and epsilon phases were extrapolated values. The difference between the modulus compensated strain rates of two phases at their mutual transformation temperature, apparent in Figure 9, is generally less than the difference between the noncompensated strain rates, Figure 8. This is because the modulus of elasticity of the stable phase above the transformation temperature is generally less than that of the stable phase below the transformation temperature; an exception is the beta and gamma phases at the $\beta \rightleftharpoons \gamma$ transformation temperature, where $E_\gamma = E_\beta$. It is also apparent from Figure 9 that the strain rate of the higher temperature phase for a given stress is generally greater than the strain rate of the lower temperature phase; an exception to this generalization is that the strain rate of the gamma phase is less than the strain rate of the beta phase, if the beta phase is formed from alpha.

The activation energy for self-diffusion can be obtained from the slope of the curves shown in Figure 9. The average activation energies that have been calculated for the six allotropes are given in Table III. These activation energies are for stresses where $n = 5$ in the proportionality $\dot{\epsilon} \propto \left(\frac{\sigma}{E}\right)^n$. Additional data, not included, tend to show that the activation energies are higher at stresses when $n > 5$, and they are lower when $n < 5$.

With the calculated self-diffusion activation energies substituted into Equation (1), creep data obtained for the six allotropes can be presented in the form shown in Figure 10, where $\log \dot{\epsilon} e^{Q/RT}$ is plotted as a function of $\log \left(\frac{\sigma}{E}\right)$. The slopes of the curves correspond to the exponent n . The

FIGURE 9
Modulus Compensated
Creep Rate Versus
Reciprocal Temperature
for the Six Allotropes
of Plutonium

TABLE III
CHANGES, TEMPERATURES, ENERGIES, AND STRUCTURE FACTORS
FOR THE SIX ALLOTROPES OF PLUTONIUM

Phase	Enthalpy and Entropy Changes for Normal Transformation cal/g-atom ⁽¹⁾		T _m Effective Melting Temperature,		Activation Energy, kcal/g-atom		Structure Factor (K _o + V) ^(c)
	$\Delta H^{i \rightarrow (i+1)}$	$\Delta S^{i \rightarrow (i+1)}$	K	C	Q _c ^(a)	Q _{sd} ^(b)	
α	900	2.28	557	284	26	23	21
β	160	0.33	748	475	30	27	18
γ	148	0.25	804	531	35	31	19.5
δ	10	0.01	843	570	31	30	18
δ	444	0.59	842	569	28		17 ^(d)
ε	676	0.74	913	640	25		14 ^(d)

(a) Derived from Figure 8

(b) Derived from Figure 9

(c) $(K_o + V) = \frac{Q_{sd}}{RT_m}$

(d) Q_c was assumed equal to Q_{sd}

FIGURE 10

Creep Data Correlated According to the
Proportionality $\dot{\epsilon} e^{Q_{sd}/RT} \propto \left(\frac{\sigma}{E}\right)^n$

difference in the values of $\dot{\epsilon} e^{Q_{sd}/RT}$ at constant $\left(\frac{\sigma}{E}\right)$ for the allotropes other than alpha can be attributed to differences in stacking fault energies. These data imply that there is little difference in stacking fault energy of the beta, gamma, delta, delta-prime, and epsilon phases.

For the data of the alpha phase to correspond to this model, the stacking fault energy would have to be one-tenth the stacking fault energy of the higher temperature phase. More detailed experiments on creep of alpha plutonium may reveal that the activation energy, Q_{sd} , of alpha is greater than 23 kcal/g-atom. It should be remembered that creep of the alpha phase may not be described by Equation (1).

Using tracer techniques, Tate and Cramer⁽¹⁰⁾ obtained an activation energy of 23.8 kcal/g-atom for self-diffusion of the delta phase of plutonium between 350 and 440 C. Their value is somewhat lower than the value of 30 kcal/g-atom derived from the creep data. No diffusion data are available to make similar comparisons between activation energies for creep and self-diffusion for the other allotropes.

Sherby and Simnad⁽¹²⁾ derived an expression for the self-diffusion activation energy, Q_{sd} , in terms of the effect of absolute melting temperature, T_m ; the valence, V ; and a crystal structure parameter, K_o . Their relationship is:

$$Q_{sd} = RT_m (K_o + V) \quad . \quad (7)$$

The effective melting temperature is essentially the theoretical melting temperature of an allotrope. If the liquid, epsilon, delta-prime, delta, gamma, beta, and alpha phases are designated 0, 1, 2, 3, 4, 5, and 6, respectively, then the effective melting temperature of each phase can be calculated according to the method derived by Ardell⁽¹³⁾, i. e.,

$$T_m^i = \frac{\sum_{i=1}^n \Delta H^{i \rightleftharpoons (i+1)}}{\sum_{i=1}^n \Delta S^{i \rightleftharpoons (i+1)}} \quad (8)$$

In this expression $\Delta H^{i \rightleftharpoons (i+1)}$ and $\Delta S^{i \rightleftharpoons (i+1)}$ are the enthalpy and entropy changes associated with the transformation of a particular phase to the next higher temperature phase. The effective melting temperatures of all six allotropes calculated in this manner are tabulated in Table III. The enthalpy and entropy changes associated with each transformation used in these calculations are also listed in Table III. Ianniello⁽¹⁴⁾ calculated the effective melting temperature to be 622 K, which is slightly higher than the value calculated by Ardell's method. The fact that every transformation temperature is greater than $0.5 T_m$ is noteworthy.

Plutonium offers a unique opportunity to determine the values of six crystal structure factors for a single metal. From the activation energies and the effective melting temperatures in Table III, values for $(K_o + V)$ can readily be calculated from the expression:

$$K_o + V = \frac{Q_{sd}}{RT_m} . \quad (9)$$

The values calculated for $(K_o + V)$ of all six allotropes are given in Table III.

Unfortunately, values for V in the above relationship have not been established. Sherby and Simnad⁽¹²⁾ assigned zero to V for uranium. By analogy, zero for V may also be applicable to plutonium. However, the significance of such a value for the valency term would be very doubtful. If V does equal zero, then values for K_o will be the same as the values given for $(K_o + V)$ in Table III.

Sherby and Simnad⁽¹²⁾ determined K_o to be 14, 17, and 21 for the body-centered cubic, face-centered cubic, and diamond structures, respectively. If $V = 0$, the K_o for the body-centered cubic and face-centered cubic allotropes of plutonium are 14 and 18 which are essentially in agreement with Sherby's and Simnad's values derived from other metals.

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