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COMPRESSIVE CREEP OF URANIA-PLUTONIA FUELS

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COMPRESSIVE CREEP OF URANIA-PLUTONIA FUELS

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ABSTRACT

Measurements of steady-state creep have been carried out on the urania-plutonia system using a compression technique. The results support (but do not establish) hypotheses regarding mechanisms of creep to the effect that:

1. *At low stress (less than 3000 psi), creep is controlled by grain boundary diffusion (an inverse cube dependence on grain size is observed).*
2. *At higher stress (greater than 6000 psi), creep is controlled by dislocation motion.*

The most sensitive parameter for fast reactor fuel is stoichiometry; increasing O/M from 1.95 to 2.00 increases the strain rate by a factor of 8 (for UO_2 -20% PuO_2). Strain rate also increases with plutonium content, porosity, and temperature. An empirical equation has been developed which relates strain rate with all of these parameters.

Activation energies for the creep of UO_2 -20% PuO_2 of -100 and -140 Kcal/mole observed here are similar to those reported for UO_2 .

1. INTRODUCTION

The creep properties of ceramic oxide nuclear fuels are an important parameter in determining the performance of a fast reactor fuel element. During reactor operations, stresses and strains develop within the fuel as a result of interactions with the cladding, differential thermal expansion, and volume changes induced by fission products. Expansion of fuel with burnup can result in high hoop stresses in the cladding. The resultant fuel-cladding interaction may lead to significant deformation of the cladding and limit the life of the fuel pin in the reactor. In a fast ceramic reactor, fuel burnups in excess of 100,000 MWd/Te are desired. It is therefore important to have quantitative data on the thermal creep response of oxide fuels to establish fuel design criteria that minimize cladding strain during the life of the core. The object of this work was to obtain this type of information on mixed plutonium-uranium oxide fuels. The effects of stoichiometry, density, temperature, stress, grain size and plutonium content on the creep characteristics of mixed oxides were studied.

A test apparatus was constructed to permit definitive measurement of the creep rate of mixed plutonium-uranium oxide fuels under conditions of interest in reactor operation. The trial operation of this equipment was performed on uranium dioxide (natural and depleted) and the results were presented by Bohaboy, et al.¹

The inherent radial temperature gradient in an operating fuel element results in gross microstructural changes across the fuel radius. These changes include grain growth and fuel migration during the formation of columnar grains, resulting in an increase in the fuel density. A small ring of fuel immediately adjacent to the cladding which does not experience microstructure change maintains its as-fabricated density. Since creep is a function of stress, temperature, and density, the deformation behavior of the oxide fuel will vary within the different regions of the fuel matrix as a function of temperature and density gradient.

Present fuel design incorporate porosity within the fuel to accommodate fuel volume increases and minimize the subsequent strain in the cladding. The success of this concept is partially determined by the ability of the oxide fuel to creep into the available pore volume.

The purpose of this investigation was to provide useful information on the out-of-reactor creep behavior of mixed oxide fuels under various controlled conditions of stress, temperature, grain size, stoichiometry, plutonium content, and density. By knowing how a fuel behaves as a function of these variables, it should be possible to make more reliable predictions of the fuel-cladding mechanical interaction and to optimize fuel element design.

A compression test was chosen for this investigation. Under reactor operating conditions, the fission product swelling and differential thermal expansion result in radial force components that are opposed by cladding and fuel restraint. These conditions are best duplicated in a compression test. The results obtained in this investigation are reported herein.

2. CONCLUSIONS

1. The results of this investigation indicate that the steady-state creep behavior of coprecipitated UO_2 - PuO_2 of O/M = 1.95 - 1.96 under compressive loads can be described by the equation.

$$\dot{\epsilon} = A \sigma \exp(-100,000/RT) + B \sigma^{4.5} \exp(-140,000/RT)$$

where:

- $\dot{\epsilon}$ = steady state creep rate, hour^{-1}
- $A = 3.1 \times 10^7 \exp[33.3(1-D)] \exp(3.56X)/G^3$
- $B = 4.37 \times 10^{-4} \exp[10.3(1-D)] \exp(3.56X)$
- σ = compressive stress, psi (1000 - 10,000)
- $R = 1.987 \text{ cal/mole } ^\circ\text{K}$
- T = temperature, $^\circ\text{K}$ (1473-1898) or $^\circ\text{C}$ (1200-1625)
- D = fractional density (0.88-0.95)
- G = grain size, microns (4-35)
- $X = \text{Pu}/(\text{Pu}+\text{U})$ (0.2 to 0.3)

2. The experimental results are in agreement with existing theoretical creep mechanisms. The cubic dependence on grain size indicates that grain boundary diffusion may be controlling deformation behavior in the linear stress dependence region. Creep at high stresses can be attributed to dislocation climb.
3. Addition of plutonium to the UO_2 lattice enhances the strain rate in both stress regions investigated. The exact mechanisms for this enhancement are not clear.
4. A decrease in density results in an enhancement in the strain rate in agreement with creep behavior in UO_2 .
5. Change in oxygen to metal ratio from 1.95 to 2.00 in UO_2 -20% PuO_2 results in an eightfold increase in strain rate.
6. The empirical equation is valid to within $\pm 42\%$ (of strain rate) based upon 90% confidence limits.

3. BACKGROUND AND LITERATURE REVIEW

Creep is best defined as the strain rate induced under constant stress as a function of time. The phenomenon of creep has been observed in all types of solids, but theories of creep behavior have been developed mainly to explain metallic deformation. The application of these theories to ceramic materials has been a significant step in the ultimate understanding of mechanical behavior in ceramic materials.

Experimental studies of creep behavior have established that there are four types of deformation under the influence of a constant stress. As the stress is initially applied, a certain amount of elastic deformation occurs which is recoverable and reproducible (this is not creep). The first stage of plastic deformation takes place following the elastic strain and is known as primary creep. The major indication of primary creep is decreasing strain rate with time under constant stress. After an initial period of primary creep, the strain rate becomes constant with time, resulting in steady-state or secondary creep. It is this segment of creep behavior which is of the most importance because it is likely to be the major creep mechanism in nuclear fuels. When a sample is subjected to considerable strain, the strain rate usually begins to accelerate with time, resulting in tertiary creep which ends in failure of the sample.

Two basic creep processes may be identified; those occurring through diffusion in the absence of slip, and those occurring as a result of driving forces which influence the flow of dislocations.

3.1 CREEP MECHANISMS IN CERAMIC MATERIALS

The creep deformation of single phase polycrystalline ceramics is a complex process generally attributed to stress enhanced diffusion, dislocation movement, and grain boundary sliding. These thermally activated processes which become operative at temperatures of approximately half the melting point can contribute simultaneously to the overall deformation of the material. The mechanism which dominates the behavior, however, is directly related to the temperature, stress, density, grain size and impurity content. Changes in any of these variables can alter the controlling mechanism and significantly change the mechanical deformation behavior.

Most experimental data on the creep behavior of ceramic materials can be described by the following form of the Arrhenius equation:

$$\dot{\epsilon} = A \sigma^n \exp(-Q/RT)$$

where:

$\dot{\epsilon}$ = creep rate

A = material parameter determined by microstructure

σ = stress

n = stress exponent

Q = activation energy

R = gas constant

T = temperature

Attempts have been made to correlate the parameters A, n, and Q with existing creep theories. In general, the experimentally determined values of Q have been consistent with the activation energy for cation self-diffusion (usually the slowest moving ion in the material). However, because of uncertainties in the experimental data, only rarely has it been possible to establish unequivocally a relation between a particular creep mechanism and the observed behavior.

3.2 STRESS ENHANCED DIFFUSION

Two types of creep behavior based on stress enhanced diffusion have been identified. Nabarro² and Herring³ have quantitatively discussed deformation which results from flow of vacancies from grain boundaries in tension to those in compression. The vacancy flow is through the grains and can be expressed in terms of volume diffusion mechanisms. This mechanism leads to strain rates which are linearly dependent upon the stress and inversely proportional to the square of the grain size.

Coble⁴ has proposed a similar mechanism which is based upon flow of vacancies along grain boundaries; hence, using grain boundary diffusion coefficients. Strain rates in Coble's creep proposal are linearly dependent upon stress and inversely proportional to the cube of the grain size.

3.3 DISLOCATION MOVEMENT

The movement of dislocations due to applied shear stresses within the crystal structure results in a macroscopic movement of material and permanent deformation. At high temperatures, dislocation climb* can occur and results in an increase in deformation rate by allowing dislocations to surmount barriers (impurity precipitates, porosity, grain boundaries, etc.) which normally would restrict dislocation movement. Weertman⁵ has proposed that high temperature dislocation movement should result in the creep-rate being proportional to stress raised to the 4.5 power ($\dot{\epsilon} \propto \sigma^{4.5}$).

3.4 GRAIN BOUNDARY SLIDING

The grain boundaries, which are areas of poor lattice registry and high dislocation density, give rise to a complex deformation process which is not well understood. Without the occurrence of either diffusional or dislocation movement mechanisms to change the grain's shape during deformation, grain boundary sliding cannot occur on a continuous basis while maintaining grain boundary continuity. However, grain boundary sliding in cooperation with other deformation mechanisms can have the effect of increasing the overall creep rate and reducing the geometric restrictions imposed on the other mechanisms.

3.5 CREEP OF $\text{UO}_2 - \text{PuO}_2$

Seltzer, Perrin, Clauer, and Wilcox⁶ have produced a review of creep behavior in nuclear fuel materials which describes all experiments which had been published prior to March 1971, including in-pile tests of UO_2 and UC.

4. EXPERIMENT DESIGN

4.1 TEST FURNACE

The test furnace has been previously described (reference 1). For these experiments the furnace was placed in an alpha enclosure for use with plutonium bearing materials. A photograph of this installation is shown in Figure 1. The only modification for this study was the substitution of a molybdenum muffle for boron nitride around the test specimen. This was necessary since some work was done in N_2 -6% H_2 saturated with H_2O , which precludes the use of boron nitride.

* Dislocation climb is thought to be important in explaining creep behavior and can be best described as the addition of vacancies or interstitials to the end of an edge dislocation which can permit the dislocation to move out of its slip plane and slip onto an adjoining plane, thus providing a mechanism for surmounting barriers.

Figure 1. Furnace for Measurement of Compressive Creep in $\text{UO}_2\text{-PuO}_2$

4.2 INSTRUMENTATION

The instrumentation was identical to that used in the UO_2 study discussed in reference 1. Creep of $\text{UO}_2\text{-PuO}_2$ was measured by the continuous LVDT readout technique described in that study.

4.3 TEST SPECIMENS

All but a few of the specimens used in this investigation were prepared from coprecipitated $\text{UO}_2\text{-PuO}_2$ powder with 10, 20, and 30 wt% PuO_2 . The precipitations were carried out by the reverse strike process, adding the solution feed to ammonium hydroxide. The filter cakes were dried in air and then calcined to the oxide form in $\text{He-6\% H}_2$ at 750°C . The powders were ballmilled for one hour and then screened < 65 mesh.

The powders were cold pressed in cylindrical steel dies and the resulting right circular cylindrical pellets were sintered at 1650°C for 5 to 7-1/2 hours in $\text{He-6\% H}_2$ in which the water content was varied to control stoichiometry. After sintering, the pellets were centerless ground to achieve uniform diameters.

Plutonia samples were prepared from oxalate derived PuO_2 powder. Normal cold press and sinter techniques as described above were used to fabricate pellets.

Physically mixed samples of $\text{UO}_2\text{-20\% PuO}_2$ were prepared from UO_2 and PuO_2 powders blended in a twin-shell blender and ballmilled to improve homogeneity. Pellets were fabricated by normal cold press and sinter techniques described above.

Small grain size material was prepared from coprecipitated $\text{UO}_2\text{-20\% PuO}_2$ by sintering at 1400°C for 3 hours. The physical properties of the samples used in this investigation are shown in Table 1.

The stoichiometry of the finished pellets was determined by the thermogravimetric method.⁷ Polished metallographic sections of the as-fabricated specimens are shown in Figures 2, 3 and 4.

Table 1
PROPERTIES OF SINTERED PELLETS

Composition w/o Pu	Density Range (%)	Approximate Grain Size (Microns)	Sintered Stoichiometry O/M $\pm$.01	Test Stoichiometry O/M $\pm$.01
10	93.5–94.5	20	1.98	1.98
20	94.5–95.5	20	1.98	1.95
	92.5–94.0	22	1.98 and 2.00	1.95 and 2.00
	88.0–91.0	22	1.98 and 2.00	1.95 and 2.00
	93.8	4	1.98	1.95
30	93.0–94.0	18	1.97	1.95
100	93.0	31	1.78	1.78
20 Physically Mixed	95.0	12	2.00	2.00

Figure 2. As-Fabricated Microstructure of $(U_{0.9}Pu_{0.1})O_{1.98}$ with 20 μ Grain Size and 93% T.D. 100X

Figure 3. As-Fabricated Microstructure of $(U_{0.8}Pu_{0.2})O_{1.95}$ with 32 μ Grain Size and 93% T.D. 100X

Figure 4. As-Fabricated Microstructure of $(U_{0.7}Pu_{0.3})O_{1.94}$ with 19 μ Grain Size and 93% T.D.

100X

4.4 EXPERIMENTAL PROCEDURE

The experimental procedure was described by Bohaboy (reference 1) and only deviations from that procedure are reported here.

The furnace temperature was monitored throughout the tests with a micro-optical pyrometer which was calibrated to a tungsten-5% rhenium vs tungsten-26% rhenium thermocouple touching the side of the test pellet. After the initial elastic compression, the deformation rate decreased until a minimum or constant rate was obtained. Depending upon the test conditions, from three to ten hours were required to reach a constant creep rate. The deformation of the sample was then recorded continuously as a function of time. A single long-term creep test was also conducted.

At the completion of each test, if further tests with that pellet were to be performed, a small load was applied to the sample during cooldown and subsequent reheating to retain the matched pellet-to-tungsten ram surface and prevent "bedding in" problems due to sample shifting. This was necessary due to slight deformation at the ram contact which was observed during long term testing.

5. RESULTS AND DISCUSSION

5.1 EMPIRICAL EQUATION

Experiments were carried out on specimens of $\text{UO}_2\text{-PuO}_2$ to determine the effects of density, stoichiometry, composition, grain size, and homogeneity on the compressive creep behavior. Measurements were carried out under conditions of constant temperature and load until steady state was reached. Measurements at a series of loads were made while keeping the temperature constant. The data were plotted as logarithm of strain rate versus logarithm of stress to provide a convenient format for identification of plastic deformation mechanisms. The data presented in the plots has been normalized (see Appendix B) to specific test conditions to make the graphical comparison of data from different tests clearer and more meaningful. The data were interpreted using an empirical equation of the form:

$$\dot{\epsilon} = A \sigma \exp(-Q_1/RT) + B \sigma^{4.5} \exp(-Q_2/RT)$$

which is a two term form of an Arrhenius type equation as discussed in Section 3.1. Q_1 and Q_2 are activation energies, and the empirical constants A and B were determined as a function of the various experimental parameters such as density, grain size and composition. The data of Figure 5 (see Appendix B) were used to determine an empirical equation based upon this model. Digital computer codes were used to facilitate the handling of the calculations.

The exact form of this equation has already been described above. This equation is applicable for data in the composition range of 20 to 30 wt % Pu. The stoichiometry dependence was not included since the nature of the variation between low stoichiometry and near stoichiometry was not measured. Figure 5 gives a comparison of experimental data for $\text{UO}_2\text{-20% PuO}_2$ of 1.95 stoichiometry, 23 micron grain size, and 93% theoretical density versus the empirical equation for three temperatures. In this and all succeeding figures, the creep rates indicated by the various plots were often normalized to certain test conditions so that many creep rates could be compared. For example, two creep rates at 1540°C and 1560°C respectively, could both be normalized to 1550°C by application of the exponential temperature dependence defined by the empirical equation. These two data points could then be compared with other data at 1550°C. Normalization of other test parameters such as stress follow in a similar manner as outlined in reference 1.

The stress exponents used in the empirical equation were indicative of diffusion mechanisms for low stresses (1020 to 3000 psi) as discussed by Nabarro,² Herring,³ and Coble⁴ and a dislocation climb mechanism at high stresses (4000 to 8000 psi) as proposed by Weertman.⁵

The data used in this study are reported in Appendix B. Comparison of the data in Figure 5 with the empirical equation developed shows that agreement is within $\pm 42\%$ (of strain rate) based upon 90% confidence limits.

5.2 EFFECT OF DENSITY

The effect of density on creep of $\text{UO}_2\text{-20% PuO}_2$ of 1.95 stoichiometry and 23 micron grain size at 1550°C is shown in Figure 6 for 1000 psi and 6000 psi. The variation in strain rate due to density change is much less at high stresses than for low stresses. This observation is in qualitative agreement with Armstrong, Irvine, and Martinson⁸ who attribute their observed creep rate variation with density in UO_2 to presence of grain boundary sliding which would be enhanced by grain boundary porosity.

Figure 5. Steady-State Compressive Creep of $(U_{0.8}Pu_{0.2})O_{1.95}$

Figure 6. Effect of Density on Creep of $(U_{0.8}Pu_{0.2})O_{1.95}$

5.3 EFFECT OF STOICHIOMETRY

The effect of stoichiometry on creep of UO_2 -20% PuO_2 of 23 micron grain size and 93% of theoretical density at 1550°C is illustrated in Figure 7. An eight-fold increase in strain rate occurs when the stoichiometry is changed from 1.95 to 2.00. The increase is similar for both stress regions. Since both the dislocation climb and Nabarro-Herring^{2,3} or Coble⁴ mechanisms are ultimately dependent upon diffusion of the slowest moving species, the relative creep rates can be linked qualitatively with explanations of the effect of stoichiometry on diffusion behavior. Diffusion of Pu in $(\text{U,Pu})\text{O}_2$ is not well established, but diffusion of U in $(\text{U,Pu})\text{O}_2$ has been shown by Lindner, Reimann, and Schmitz⁹ to be comparable to that in UO_2 . If U+4 ions as proposed by Lidiard¹⁰ are presumed to be the diffusing species via vacancies controlling creep behavior, then an increase in an ion vacancies would tend to decrease the uranium vacancies resulting in an overall decrease in strain rate. Presumably this decrease would be evident in both high and low stress regions.

The exact form of the dependence of stoichiometry on creep in mixed oxides was not established since it was possible to measure the strain rate only at two stoichiometries, 1.95 and 2.00. Therefore, the stoichiometry dependence was not included in the empirical equation for creep.

5.4 EFFECT OF GRAIN SIZE

The results of compressive creep measurements on UO_2 -20% PuO_2 of 1.95 O/M 94% T.D., and 4 micron grain size are shown in Figure 8. The lines through the data are empirical equations plotted for the appropriate temperatures, grain size (GS), and density. The data indicate a best fit to a $(\text{GS})^{-3}$ dependence in the low stress region in comparison with a $(\text{GS})^{-2}$ dependence exhibited for UO_2 .¹ Coble⁴ has formulated a theory for diffusion creep based on grain boundary diffusion which gives a $(\text{GS})^{-3}$ dependence as opposed to lattice diffusion as proposed by Nabarro² and Herring³ with its $(\text{GS})^{-2}$ dependence. Both models predict a linear stress dependence.

Figure 9 shows the effect of grain size on the creep of UO_2 -20% PuO_2 of 1.95 stoichiometry and 93% of theoretical density for two different grain sizes. These observations were based only on three grain sizes and the results shown in Figure 9 indicate an inverse cubic dependence of creep on grain size. This was determined by measuring the slope of the parallel lines on the log-log plot of creep versus grain size.

This slope was found to be approximately equal to -3 indicating the inverse-cubic relationship.

A long term test was conducted with 4 micron grain size, 93% dense UO_2 -20% PuO_2 1200°C and 1200 psi to test the extrapolation of the data to lower temperatures. A creep rate of 2.1×10^{-5} in./in./h was obtained which is in good agreement with the calculated creep rate of 1.77×10^{-5} in./in./h for these experimental conditions.

Seltzer, Clauer, and Wilcox¹¹ have pointed out that for creep phenomena exhibiting two different mechanisms, such as Nabarro-Herring diffusion creep plus dislocation climb, a transition stress should exist which is dependent upon grain size. If the two mechanisms exhibit different temperature dependences then the transition stress will be temperature dependent as well. The transition stress for 4 micron grain size material at 1390°C as calculated from the empirical equation is 11,000 psi, so that in the range of stresses used in this study, the small-grain material should exhibit only creep with a linear stress dependence. No dependence of creep on grain size was observed in the higher stress regions (i.e., those stress regions where the second term of the empirical equation proportional to $\sigma^{4.5}$ dominated creep) and hence there was no grain size dependence in the high stress dominant term of the empirical equation. In all cases, the transition stress was higher than the maximum stress used for any of the tests. From Figure 8, it is evident that this is the case.

5.5 EFFECT OF COMPOSITION

The effects of variation in plutonium content on creep properties of UO_2 - PuO_2 of a constant O/Pu = 1.78 are exhibited in Figure 10. The two points shown for UO_2 (X=0) were obtained from the study by Bohaboy, et al.¹

As the plutonium content is increased, a definite softening of the material takes place. The degree of softening appears to be similar in both the low and high stress regions. The strain rates for PuO_2 as a function of stress are plotted in Figure 11. The activation energies for creep deformation in PuO_2 are notably smaller than for UO_2 -20% PuO_2 of the same oxygen activity, being -70 Kcal/mole and -100 Kcal/mole for the low and high stress regions respectively. The same stress exponents are observed as in the mixed oxide case indicating that the same creep mechanisms may be operating in PuO_2 as in mixed oxide. The lack of data on self-diffusion of Pu in PuO_2 renders a comprehensive analysis of these data difficult.

Results of a creep measurement on UO_2 -10% PuO_2 at 1550°C (18 micron grain size, 94% density, and 1.98 O/M) are shown in Figure 12 in comparison with UO_2 -20% PuO_2 (23 micron grain size, 94% density, and 1.95 and

Figure 7. Variation in Steady-State Creep of $(U_{0.8}Pu_{0.2})O_{2-x}$ with Stoichiometry

Figure 8. Steady-State Creep in $(U_{0.8}Pu_{0.2})_{0.95}$ of 94% T.D. and 4μ Grain Size

Figure 9. Effect of Grain Size on the Creep of $(U_{0.8}Pu_{0.2})O_{1.95}$

Figure 10. Effect of Plutonium Content on Creep of $(U_{1-X}Pu_X)O_{2-Y}$

Figure 11. Steady-State Creep of $\text{PuO}_{1.78}$

Figure 12. Steady-State Creep in $(U_{0.9}Pu_{0.1})O_{1.98}$ Compared with Stoichiometric and Hypostoichiometric $(U_{0.8}Pu_{0.2})O_2$ at $1550^\circ C$

2.00 stoichiometric compositions). The 10% PuO_2 data agreed quite well with the 20% PuO_2 data when corrections were made for grain size variations as shown in Figure 12. The limited data, however, did not lead to specific conclusions. The combined effects of stoichiometry (2.00 versus 1.98) and plutonium (10 to 20%) differences were not discernable within the accuracy of the experimental apparatus.

5.6 EFFECT OF INHOMOGENEITY

Creep experiments were conducted on physically blended mixed oxides to establish any difference between this fuel type and the coprecipitated fuel. The physically blended UO_2 -20% PuO_2 was prepared by blending UO_2 and PuO_2 powders in the proper ratio in a twin shell blender. The results of creep measurements on this fuel in comparison with the plot of the empirical equation for 5 micron grain size is shown in Figure 13. In addition to containing potentially inhomogeneous Pu distribution, this specimen contained regions of widely different grain size. The majority of the specimens exhibited 12 micron grains, but a substantial minority of the sample exhibited 5 micron grains. Since the specimen was fabricated by a physical blending process, either the 12 micron or 5 micron grain size regions were probably enriched in plutonium. It was not possible to determine which was the case. It would be expected that the creep properties would be more closely related to the 12 micron grain size since it was the majority region; however, the data was in good agreement with the empirical equation for the 5 micron grain size. This indicated either that the 5 micron grain size region was controlling the creep or that the 12 micron grain size region was sufficiently enriched in plutonium that it behaved as if it had a smaller grain size. Therefore, it was not possible to make any firm conclusion concerning plutonium inhomogeneity from the current data.

5.7 COMPARISON OF RESULTS WITH PREVIOUSLY REPORTED DATA

Creep results on UO_2 - PuO_2 materials have been reported by Houston, Kruger, and Pardue,^{1,2} Marples and Hough,^{1,3} and Krummerer and Vollath.¹⁴ Slagle¹⁵ has reported creep tests in four-point bending. All these workers have reported similar activation energies with stress exponents varying from 1 to 4.5. A summary of these results along with the results of this study are given in Table 2. The creep rates determined by Houston, et al.,^{1,2} for physically mixed UO_2 -22% PuO_2 , with 4 micron grain size, and 94% density are in good agreement with the data reported here for coprecipitated UO_2 -20% PuO_2 of similar parameters. Their O/M ratio was higher (1.99) compared to 1.95 used here. They report an activation energy of 77 Kcal/mole. The data reported by Marples and Hough^{1,3} exhibits creep rates 10 to 15 times greater than the data in this study with similar parameters for both coprecipitated and physically blended fuel; however, their activation energies and stress dependences are in good agreement with those reported here.

Table 2
STEADY-STATE CREEP OF $\text{UO}_2\text{--PuO}_2$

Investigators	This Study	Houston, et al. (12)	Marples & Hough (13)	Kummerer & Vollath (14)	Slagle (15)
Material Preparation	Coprecipitation	Physical Blending	Coprecipitation & Physical Blending	Physical Blending	Coprecipitation
Grain Size (μ)	4--31	3--15	3--80	5--30	20
Density (% T.D.)	89--95	90--94	94--99	92--95	96
O/M	1.95--2.00	1.99	1.995--2.00	—	1.98
PU Content (Pu/U+Pu)	10,20,30, 100	22	2,5,10,15, 20,30,40	19.5	20
Type of Measurement	Compression	Compression	Compression	Compression	Bend
Stress Range (psi)	900--8000	1100--3200	1200--2000	425--3560	2500--9000
Temperature Range ($^{\circ}\text{C}$)	1250--1625	1100--1330	1150--1500	1200--1700	1475--1560
Stress Exponent	1 & 4.5	1.4	1 & 4.5	—	1.08--1.75
Activation Energy (Kcal/mole)	100 & 140	77	103 & 133	98	110

Figure 13. Steady-State Creep of Physically Mixed $(U_{0.8}Pu_{0.2})O_{1.95}$ at $1390^{\circ}C$

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APPENDIX A. PRECISION OF EXPERIMENTAL TECHNIQUE

The precision of the experimental technique has been outlined in the UO_2 portion of this study.¹ The treatment here concerns only deviations from that report.

A.1 TEMPERATURE

A calibrated tungsten-5% rhenium versus tungsten-26% rhenium thermocouple was used to calibrate a micro-optical pyrometer to the optical arrangement of the furnace. The pyrometer was used for all temperature measurements. The pyrometer measurements could be made with a reproducibility of $\pm 3^\circ\text{C}$. Since the thermocouple measurements were accurate within $\pm 1.0\%$ of the measured temperature, the overall precision of the temperature measurement was less than $\pm 20^\circ\text{C}$.

A.2 DENSITY

Sample densities were calculated from dimensional and gravimetric measurements. The overall precision of this technique is $\pm 0.6\%$.

A.3 CONFIDENCE LIMITS

The data of Figure 5 was used to calculate 90% confidence limits for the equation given in Section 5. The confidence limits gave an indication of the degree of fit of the equation to the experimental data, and they indicate that 90% of the time the experimental creep rate was contained within these limits of the calculated creep rate. These confidence limits were calculated as follows:

- (1) From Appendix B, Figure 5 data:

$M(+)$ = positive arithmetic mean percent difference in creep rates = 39.1%

$M(-)$ = negative arithmetic mean percent difference in creep rates = 29.0%

M = combined arithmetic mean percent difference in creep rates = 33.7%

$S(+)$ = standard deviation for a sample of 15 positive percent differences in creep rates = 29.8%

$S(-)$ = standard deviation for a sample of 17 negative percent differences in creep rates = 19.1%

S = combined sample standard deviation for a sample of 32 percent differences in creep rates = 25.6%

- (2) Standard error

SE = standard error

N = sample number

$$SE(+) = \frac{S(+)}{\sqrt{N-1}} = \frac{29.8\%}{\sqrt{14}} = 7.97\%$$

$$SE(-) = \frac{S(-)}{\sqrt{N-1}} = \frac{19.1\%}{\sqrt{16}} = 4.78\%$$

$$SE = \frac{S}{\sqrt{N-1}} = \frac{25.6\%}{\sqrt{31}} = 4.60\%$$

- (3) Upper and lower confidence limits:

$CI(+)$ = upper confidence limit

= $M(+) + (\Delta) SE(+)$

= $+39.1 + (1.65) (7.97) = 52.3\%$

$CI(-)$ = lower confidence limit

= $M(-) - (\Delta) SE(-)$

= $-29.0 - (1.65) (4.78) = 36.9\%$

CI = combined (symmetric + or -) confidence limit

= $M \pm (\Delta) SE = 41.3$

= $33.7 \pm (1.65) (4.60) = \approx 42\%$

$\Delta = 1.65 = 90\%$ confidence factor from tables of the normal distribution function

- (4) Since $CI(+)$ and $CI(-)$ were not equal, the confidence limits appeared to be skewed slightly. This indicated that the equation would be slightly more likely to under-predict the true creep rate than to over-predict it. Since the creep rate varied as a logarithmic function of temperature, slight variations in temperature would effect much larger changes in creep rate. Hence the combined $\pm 42\%$ confidence limits were reasonable for these types of measurements.

APPENDIX B. DATA

The experimental data used in this study are listed in this Appendix in order of the figure in the report to which it applies. The experimental creep rates (in./in./h) are listed along with the experimental parameters for each test number: stress (psi), temperature ($^{\circ}$ K), density (% of theoretical), grain size (microns), and fractional plutonium content. The creep rate (in./in./h) calculated using the empirical equation given in Section 5 for each specific set of test conditions is also given along with the percent differences (based upon the calculated creep rate) between the measured and calculated creep rates. Corrections were made for stoichiometry variations in the experimental creep rates so that they could be compared to the calculated values. Significance should only be attached to the statistical data (sample standard deviation, etc.) for Figure 5, given in Table B-1.

The experimental data were for the most part grouped about specific values of temperature, density, and grain size. Small variations from these nominal values, however, were present in most of the data. For example, a number of tests were intended to be made at a nominal temperature of 1650° C. The actual temperatures of these individual tests were anywhere from 1640 to 1660° C. Similar variations existed in the density and grain size numbers. A graphical presentation of the data in this form made interpretation difficult and had little value. Consequently, experimental data that were grouped about the same nominal values were "normalized" to the same temperature, grain size and density. The technique used to normalize the data was analogous to that used in the previous UO_2 study.¹

Table B-1
FIGURE 5 DATA

Test Number	Stress (psi)	Temperature (°K)	Theoretical Density (%)	Grain Size (Microns)	Normalized Experimental Creep Rate	Calculated Creep Rate	Difference in Creep Rates (%)	Pu
47	1000	1898	93.0	23	3.08E-04	1.67E-04	84.08	0.20
47	4760	1898	93.0	23	7.30E-03	5.67E-03	28.65	0.20
48	4700	1898	93.0	23	1.09E-02	5.39E-03	102.12	0.20
49	2780	1898	93.0	23	8.13E-04	8.89E-04	-8.50	0.20
50	2860	1898	93.0	23	5.48E-04	9.61E-04	-42.97	0.20
51	4760	1098	93.0	23	7.16E-03	5.67E-03	26.18	0.20
51	1727	1898	93.0	23	2.22E-04	3.33E-04	-33.24	0.20
51	1690	1898	93.0	23	2.63E-04	3.22E-04	-18.26	0.20
52	1245	1898	93.0	23	7.30E-05	2.15E-04	-65.98	0.20
52	1200	1898	93.0	23	8.38E-05	2.05E-04	-59.22	0.20
35	7864	1823	93.0	23	9.30E-03	1.06E-02	-12.39	0.20
35	6207	1823	93.0	23	1.97E-03	3.85E-03	-48.85	0.20
35	4004	1823	93.0	23	7.67E-04	7.08E-04	8.40	0.20
35	1973	1823	93.0	23	1.17E-04	1.28E-04	-8.74	0.20
37	5760	1823	93.0	23	2.46E-03	2.82E-03	-12.89	0.20
37	2250	1823	93.0	23	1.77E-04	1.60E-04	10.86	0.20
37	3970	1823	93.0	23	5.48E-04	6.87E-04	-20.27	0.20
39	4160	1823	93.0	23	6.67E-04	8.08E-04	-17.43	0.20
39	967	1023	93.0	23	6.90E-05	5.32E-05	29.79	0.20
39	947	1823	93.0	23	6.10E-05	5.26E-05	16.02	0.20
42	5950	1823	93.0	23	3.08E-03	3.23E-03	-4.62	0.20
45	6630	1823	93.0	23	2.15E-03	5.09E-03	-57.74	0.20
46	6020	1823	93.0	23	3.43E-03	3.39E-03	1.10	0.20
46	4710	1823	93.0	23	1.39E-03	1.27E-03	9.27	0.20
47	4790	1823	93.0	23	1.89E-03	1.36E-03	39.34	0.20
38	7030	1748	93.0	23	1.85E-03	1.29E-03	43.52	0.20
38	5310	1748	93.0	23	2.69E-04	4.20E-04	-35.99	0.20
40	5790	1748	93.0	23	9.62E-04	5.86E-04	64.14	0.20
42	3020	1748	93.0	23	5.44E-05	7.67E-05	-29.09	0.20
42	3050	1748	93.0	23	6.48E-05	7.84E-05	-17.35	0.20
44	3140	1748	93.0	23	1.51E-04	8.37E-05	80.34	0.20
44	6180	1748	93.0	23	1.08E-03	7.59E-04	42.22	0.20

Arithmetic Mean (Absolute Differences) = 33.74

Mean (Positive Differences) = 39.07

Mean (Negative Differences) = -29.03

Sample Standard Deviation = S = 25.5917

S(+) = 29.8

S(-) = 19.1

Table B-2
FIGURE 6 DATA

Test Number	Stress (psi)	Temperature (°K)	Theoretical Density (%)	Grain Size (Microns)	Normalized Experimental Creep Rate	Calculated Creep Rate	Difference in Creep Rates (%)	Pu
57	1000	1823	94.0	23	3.40E-05	4.01E-05	-15.19	0.20
39	1000	1823	93.0	23	6.60E-05	5.57E-05	18.52	0.20
124	1000	1823	88.0	23	2.90E-04	2.91E-04	-0.31	0.20
122	6000	1823	88.0	23	4.30E-03	6.78E-03	-36.59	0.20
94	6000	1823	94.0	23	2.00E-03	2.96E-03	-32.32	0.20
46	6000	1823	93.0	23	3.10E-03	3.34E-03	-7.27	0.20

Arithmetic Mean (Absolute Differences) = 18.37
Mean (Positive Differences) = 18.52
Mean (Negative Differences) = -18.34
Sample Standard Deviation = 14.0432

Table B-3
FIGURE 7 DATA

Test Number	Stress (psi)	Temperature (°K)	Theoretical Density (%)	Grain Size (Microns)	Normalized Experimental Creep Rate	Calculated Creep Rate	Pu	O/M
103	1018	1823	94.0	18	2.92E-04	8.43E-05	0.20	2.00
95	990	1823	94.0	18	2.79E-04	8.19E-05	0.20	2.00
95	1585	1823	94.0	18	5.86E-04	1.37E-04	0.20	2.00
94	2793	1823	94.0	18	2.57E-03	3.16E-04	0.20	2.00
94	2831	1823	94.0	18	2.88E-03	3.24E-04	0.20	2.00
96	1060	1748	94.0	18	1.52E-04	2.68E-05	0.20	2.00
99	4630	1748	94.0	18	1.42E-04	2.77E-04	0.20	2.00
105	2860	1748	94.0	18	3.51E-04	9.01E-05	0.20	2.00
99	4540	1748	94.0	18	2.76E-04	2.61E-04	0.20	1.95
38	7030	1748	94.0	18	2.03E-03	1.42E-03	0.20	1.95
38	5310	1748	94.0	18	3.31E-04	5.17E-04	0.20	1.95
40	5790	1748	94.0	18	1.13E-03	0.91E-04	0.20	1.95
42	3020	1748	94.0	18	9.36E-05	1.32E-04	0.20	1.95
42	3050	1748	94.0	18	1.11E-04	1.34E-04	0.20	1.95
44	3140	1748	94.0	18	2.54E-04	1.41E-04	0.20	1.95
44	6180	1748	94.0	18	1.24E-03	8.72E-04	0.20	1.95

Table B-4
FIGURE 8 DATA

Test Number	Stress (psi)	Temperature (°K)	Theoretical Density (%)	Grain Size (Microns)	Normalized Experimental Creep Rate	Calculated Creep Rate	Difference in Creep Rates (%)	Pu
154	2840	1663	94.0	4	1.79E-03	1.49E-03	20.15	0.20
155	1680	1663	94.0	4	1.32E-03	8.80E-04	49.98	0.20
156	1030	1663	94.0	4	8.06E-04	5.39E-04	49.40	0.20
177	970	1663	94.0	4	1.61E-03	5.08E-04	216.89	0.20
177	985	1663	94.0	4	1.64E-03	5.16E-04	217.88	0.20
178	1360	1663	94.0	4	1.20E-03	7.12E-04	68.45	0.20
179	2360	1663	94.0	4	1.73E-03	1.24E-03	39.85	0.20
151	2540	1598	94.0	4	3.36E-04	3.89E-04	-13.56	0.20
151	3780	1598	94.0	4	1.43E-03	5.80E-04	146.72	0.20
151	8040	1598	94.0	4	2.43E-03	1.27E-03	90.79	0.20
167	7150	1598	94.0	4	4.14E-03	1.12E-03	269.82	0.20
168	4900	1598	94.0	4	2.06E-03	7.54E-04	173.15	0.20
168	2650	1598	94.0	4	5.55E-04	4.06E-04	36.84	0.20
169	1630	1598	94.0	4	5.37E-04	2.49E-04	115.38	0.20
169	954	1598	94.0	4	1.81E-04	1.46E-04	24.05	0.20
175	2870	1598	94.0	4	7.52E-04	4.39E-04	71.15	0.20
175	2780	1598	94.0	4	7.84E-04	4.26E-04	84.23	0.20
175	2750	1598	94.0	4	8.66E-04	4.21E-04	105.73	0.20
176	4650	1598	94.0	4	1.67E-03	7.15E-04	133.59	0.20
176	978	1598	94.0	4	3.18E-04	1.50E-04	112.59	0.20
172	4850	1523	94.0	4	1.69E-04	1.58E-04	7.09	0.20
173	7280	1523	94.0	4	2.82E-04	2.39E-04	17.84	0.20
174	2850	1523	94.0	4	9.68E-05	9.25E-05	4.67	0.20
174	2830	1523	94.0	4	1.45E-04	9.18E-05	57.90	0.20
174	2830	1523	94.0	4	1.40E-04	9.18E-05	52.46	0.20

Arithmetic Mean (Absolute Differences) = 87.21
Mean (Positive Differences) = 90.28
Mean (Negative Differences) = -13.56
Sample Standard Deviation = 71.9798

Table B-5
FIGURE 9 DATA

Test Number	Stress (psi)	Temperature (°K)	Theoretical Density (%)	Grain Size (Microns)	Normalized Experimental Creep Rate	Calculated Creep Rate	Difference in Creep Rates (%)	Pu
156	1000	1823	94.0	4	1.12E-02	7.46E-03	50.15	0.20
103	1000	1823	94.0	18	4.50E-05	8.27E-05	-45.59	0.20
47	1000	1823	94.0	23	7.40E-05	4.01E-05	84.59	0.20
176	4000	1823	94.0	4	7.09E-02	3.03E-02	134.20	0.20
102	4000	1823	94.0	18	8.20E-04	7.66E-04	7.04	0.20
35	4000	1823	94.0	23	6.46E-04	5.96E-04	8.47	0.20

Arithmetic Mean (Absolute Differences) = 55.01
 Mean (Positive Differences) = 56.89
 Mean (Negative Differences) = -45.59
 Sample Standard Deviation = 48.4266

Table B-6
FIGURE 10 DATA

Test Number	Stress (psi)	Temperature (°K)	Theoretical Density (%)	Grain Size (Microns)	Normalized Experimental Creep Rate	Calculated Creep Rate	Difference in Creep Rates (%)	Pu
39	1000	1823	93.0	23	6.60E-05	5.57E-05	18.52	0.20
130	1000	1823	93.0	18	9.90E-05	1.64E-04	-39.77	0.30
132	1000	1823	93.0	31	1.20E-03			1.00
39	4000	1823	93.0	23	5.50E-04	7.05E-04	-22.01	0.20
128	4000	1023	93.0	18	7.00E-04	1.35E-03	-48.00	0.30
132	4000	1823	93.0	31	7.90E-03			1.00

Arithmetic Mean (Absolute Differences) = 32.08
 Mean (Positive Differences) = 18.52
 Mean (Negative Differences) = -36.59
 Sample Standard Deviation = 14.12

Table B-7
FIGURE 11 DATA

Test Number	Stress (psi)	Temperature (°K)	Theoretical Density (%)	Grain Size (Microns)	Normalized Experimental Creep Rate	Pu
131	734	1823	90.0	31	1.29E-03	1.00
131	1550	1823	90.0	31	3.00E-03	1.00
132	1290	1823	90.0	31	1.57E-03	1.00
132	2460	1823	90.0	31	1.27E-02	1.00
132	2320	1748	90.0	31	1.68E-03	1.00
132	3070	1748	90.0	31	2.06E-02	1.00
132	1940	1748	90.0	31	1.32E-03	1.00
133	3019	1748	90.0	31	2.09E-02	1.00
134	1700	1748	90.0	31	1.34E-03	1.00
134	1040	1748	90.0	31	4.37E-04	1.00
136	2970	1748	90.0	31	1.88E-02	1.00
134	1100	1663	90.0	31	1.02E-04	1.00
137	1510	1663	90.0	31	5.33E-04	1.00
137	2530	1663	90.0	31	1.65E-03	1.00
138	3840	1663	90.0	31	2.48E-02	1.00
138	3630	1663	90.0	31	1.47E-02	1.00
138	3550	1663	90.0	31	1.03E-02	1.00
143	915	1663	90.0	31	1.32E-04	1.00
144	940	1663	90.0	31	1.99E-04	1.00

Table B-8
FIGURE 12 DATA

Test Number	Stress (psi)	Temperature (°K)	Theoretical Density (%)	Grain Size (Microns)	Normalized Experimental Creep Rate	Calculated Creep Rate	Difference in Creep Rates (%)	Pu
191	5030	1823	94.0	18	3.33E-02			0.10
191	3070	1823	94.0	18	3.07E-03			0.10
192	1148	1823	94.0	18	6.45E-04			0.10
35	7864	1823	93.0	23	9.30E-03	1.06E-02	-12.39	0.20
35	6207	1823	93.0	23	1.97E-03	3.85E-03	-48.85	0.20
35	4004	1823	93.0	23	7.67E-04	7.08E-04	8.40	0.20
35	1973	1823	93.0	23	1.17E-04	1.28E-04	-8.74	0.20
37	5760	1823	93.0	23	2.46E-03	2.82E-03	-12.89	0.20
37	2250	1823	93.0	23	1.77E-04	1.60E-04	10.86	0.20
37	3970	1823	93.0	23	5.48E-04	6.87E-04	-20.27	0.20
39	4160	1823	93.0	23	6.67E-04	8.08E-04	-17.43	0.20
42	5950	1823	93.0	23	3.08E-03	3.23E-03	-4.62	0.20
45	6630	1823	93.0	23	2.15E-03	5.09E-03	-57.74	0.20
46	6020	1823	93.0	23	3.43E-03	3.39E-03	1.19	0.20
46	4710	1823	93.0	23	1.39E-03	1.27E-03	9.27	0.20
47	4790	1823	93.0	23	1.89E-03	1.36E-03	39.34	0.20
94	2831	1823	94.0	23	3.30E-04	2.04E-04	62.03	0.20
94	2793	1823	94.0	23	2.90E-04	1.97E-04	47.43	0.20
94	2745	1823	94.0	23	2.80E-04	1.88E-04	48.71	0.20
95	1585	1823	94.0	23	6.30E-05	6.90E-05	-8.68	0.20
95	990	1823	94.0	23	2.90E-05	3.97E-05	-26.88	0.20
96	932	1823	94.0	23	2.80E-05	3.72E-05	-24.71	0.20
96	922	1823	94.0	23	2.50E-05	3.68E-05	-32.00	0.20
102	2890	1823	94.0	23	2.50E-04	2.15E-04	16.29	0.20
103	1960	1823	94.0	23	8.50E-05	9.46E-05	-10.15	0.20
103	1018	1823	94.0	23	3.20E-05	4.09E-05	-21.70	0.20

Arithmetic Mean (Absolute Differences) = 23.94
Mean (Positive Differences) = 27.06
Mean (Negative Differences) = -21.93
Sample Standard Deviation = 18.1397

Table B-9
FIGURE 13 DATA

Test Number	Stress (psi)	Temperature (°K)	Theoretical Density (%)	Grain Size (Microns)	Normalized Experimental Creep Rate	Calculated Creep Rate	Difference in Creep Rates (%)	Pu
180	8160	1663	95.0	5	3.15E-03	1.81E-03	74.42	0.20
182	4640	1663	95.0	5	7.72E-04	9.11E-04	-15.22	0.20
182	4860	1663	95.0	5	9.50E-04	9.57E-04	- 0.75	0.20
183	2840	1663	95.0	5	4.79E-04	5.48E-04	-12.58	0.20
184	1110	1663	95.0	5	1.91E-04	2.13E-04	-10.49	0.20

Arithmetic Mean (Absolute Differences) = 22.69
 Mean (Positive Differences) = 74.42
 Mean (Negative Differences) = - 9.76
 Sample Standard Deviation = 29.4269

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