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Mechanical Properties of Carbide and Nitride Reactor Fuels

by

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ABSTRACT

Mechanical-property evaluations of plutonium-bearing carbides and nitrides conducted at the Los Alamos Scientific Laboratory in study of fast reactor fuels are presented, along with a review of the literature on the mechanical properties of uranium and plutonium carbides and nitrides.

I. Introduction

The physical and thermodynamic properties of the carbides of uranium and plutonium are fairly well known. Their specific heat, vapor pressure, heat of vaporization, heat of formation, free energy of formation, electrical resistivity, thermal conductivity, thermal expansion, melting point, crystal structure, and lattice parameter as a function of carbon/metal (C/M) ratio have been studied. Many of these properties have been measured with great accuracy and, where appropriate, over a wide temperature range. Some information of this type is also available on the nitrides of uranium and plutonium and on mixed uranium and plutonium nitrides and carbides, but much remains to be learned.

In contrast, little information on the mechanical properties of these materials is available. The nature and scope of the available mecahnical-property data are shown in Table I. It is evident that most pertain to the uranium carbides and mononitride. Except for the creep[1-8] and hot-hardness[9-13] data, most were obtained at room temperature. The quantity of data is small; e.g., room-temperature hardness is the only mechanical-property measurement reported for U_2C_3,[14] UC_2,[15] Pu_3C_2,[16] PuC,[16-18] Pu_2C_3,[19] and $(U,Pu)_2C_3$.[20] No mechanical-property measurements of any kind have been reported on PuC_2. Poisson's ratio has been reported for UN[21-25] only.

This paper reviews available mechanical-property measurements of the carbide and nitride fuel materials. A review of the literature and some new information obtained in the Los Alamos Scientific Laboratory (LASL) research and development program on fast reactor fuels are included.

II. Hot-Hardness Testing

Equipment. The hardness of a material at elevated temperatures is easily measured, and the values can be used in qualitative comparisons or predictions of other properties such as creep resistance or compressive strength. Figure 1 is a schematic of a hot-hardness tester intended for use at the LASL plutonium facility. This equipment is a modification of a design with a maximum temperature capability of about 1000°C which is now in use The new unit is designed for temperatures up to at least 1500°C, which capability will be achieved largely through substitution of more refractory materials for those now used. For example, the radiation shields will be tantalum instead of stainless steel and the indentor will be B_4C rather than diamond or sapphire.

In the apparatus now in use, the specimen rests on an anvil that can be raised or lowered by a push rod. When a micro-indentation is made, the specimen is raised until it contacts the indentor and lifts it off its support. At this instant the electrical contacts at the indentor support are broken. This provides a means of determining the indentation time.

With this type of hardness testing apparatus, the indentations cannot be measured *in situ*. After the indentations have been made, the specimen is cooled and transferred from the glove box to a standard Leitz hardness tester where the indentations are measured. Because of alpha contamination special efforts are made to clean the specimen before it is taken out of the glove box. This is accomplished by washing the specimen in Vythene in an ultrasonic cleaner, then transferring it immediately out of the glove box to a clean disposable glass plate. No contamination problems have been encountered using this technique.

TABLE I

MEASURED MECHANICAL PROPERTIES OF PLUTONIUM AND URANIUM CARBIDES AND NITRIDES

UC	U_2C_3	UC_2	UN
H–H[a](25 to 1420°C) Refs. 9-13	Hardness (25°C) Ref. 14	Hardness (25°C) Ref. 15	H–H(25 to 1200°C) Ref. 13
Creep (Compressive) Refs. 1-6 (Tensile) Ref. 7			Creep (Compressive) Refs. 2,8
E (25 to 1300°C) Ref. 31			E(25°C) Refs. 21-25 E(25 to 1300°C) Ref. 31
Modulus of Rupture (25°C) Refs. 21-23,30			M. O. R. (25°C) Refs. 23,24
Compressive Strength (25°C) Refs. 8-10, 25			ν (25°C) Refs. 21-25

PuC	Pu_3C_2	Pu_2C_3	PuC_2	PuN
Hardness (25°C) Refs. 16-18	Hardness (25°C) Ref. 16	Hardness (25°C) Ref. 17	---	H–H(25 to 1000°C)[b]
H–H(25 to 1000°C)[b]				

(U,Pu)C	$(U,Pu)_2C_3$	(U,Pu)N
H–H(0 to 1000°C) Ref. 9	Hardness (25°C) Ref. 20	H–H(10 to 1000°C) Ref. 9
E^b (25°C)		E(25 to 1300°C) Ref. 31
G^b (25°C)		
ν^b (25°C)		
B^b (25°C)		
$\theta_D{}^b$ (25°C)		

[a]Hot-Hardness

[b]Reported here for the first time.

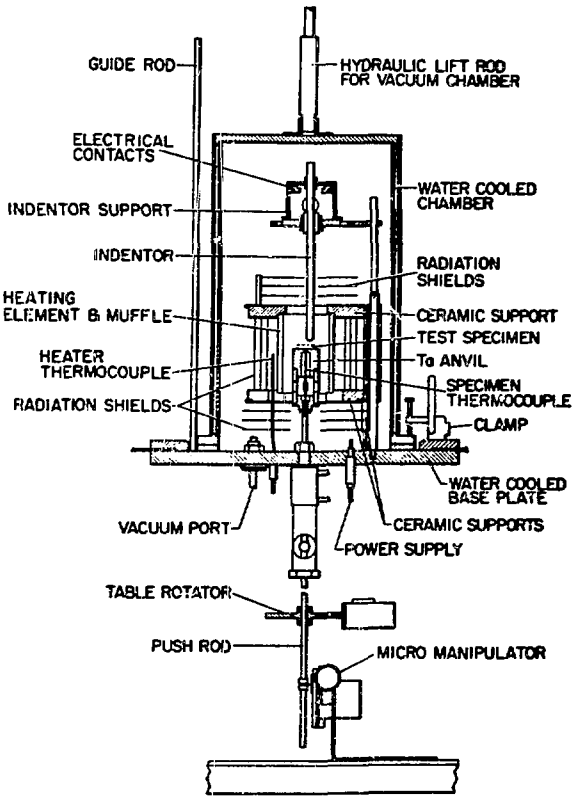

Fig. 1.
Hot-hardness tester.

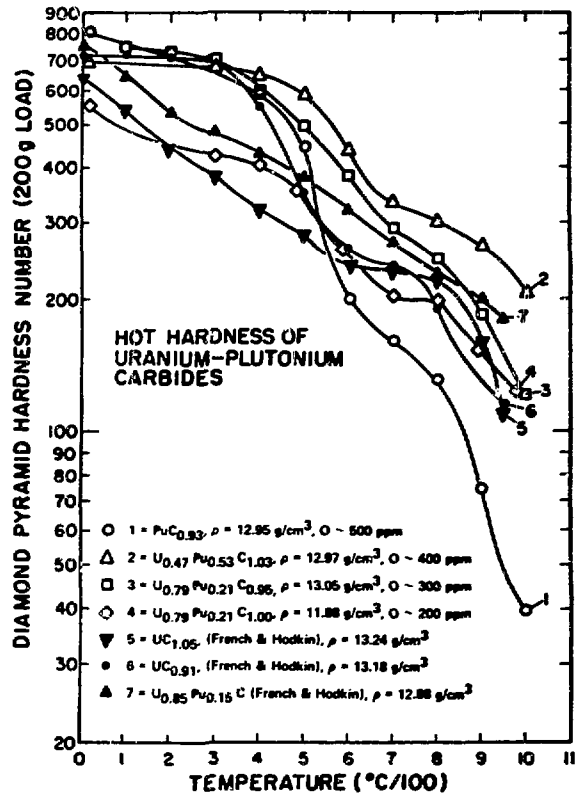

Fig. 2.
Hot hardness of uranium-plutonium carbides.

Results. Hot-hardness curves for sintered specimens of various uranium-plutonium carbide compositions are shown in Fig. 2.* Curves 5 and 6 for UC and curve 7 for $U_{0.85}Pu_{0.15}C$ were taken from the literature[9] and are shown for comparison. Each data point for curves 1 through 4 represents the mean value of at least five readings.

Curve 1 shows the hot-hardness behavior of a PuC specimen containing about 10 vol% Pu_2C_3. Up to about 500°C, this material's hardness is comparable to that of the mixed carbides, and is, in fact, greater than the literature values for UC or $U_{0.85}Pu_{0.15}C$, but above 500°C it drops rapidly.

The hot-hardness behavior of a hypostoichiometric specimen of PuC containing some Pu_3C_2 has also been determined but is not shown in this figure. This material had a diamond pyramid hardness of about 270 at room temperature, dropping to 36 at 550°C.

That some solid solution strengthening is obtained from the addition of PuC to UC is evident from comparison of the UC curves with those for the mixed carbides. The highest hardness values at high temperatures were for specimen No. 2 which contained about a 1:1 mole ratio of plutonium to uranium.

The hot-hardness behavior of specimen No. 4 (which contained some M_2C_3) was somewhat surprising in that the single-phase $U_{0.8}Pu_{0.2}C$ material (specimen No. 3) was considerably harder, at least up to 900°C. The sesquicarbide in specimen No. 4 had been expected to have a hardening effect. Part of the low hardness might be attributed to this specimen's relatively low density. However, French and Hodkin[9] obtained an analogous result with UC. This may be seen by comparing curve 5 (for slightly hyperstoichiometric UC) with curve 6 (for hypostoichiometric UC) and noting that below 700°C, the hypostoichiometric material is considerably harder. Evidently the sesquicarbide is an effective hardener only at high temperatures. Electron microprobe examination of hyperstoichiometric uranium-plutonium carbides shows that the sesquicarbide is enriched in plutonium relative to the matrix monocarbide. Whether the plutonium-uranium distribution changes with temperature and how this could affect the hardness of the matrix is not known.

*Details of specimen preparation are given in "Synthesis and Fabrication of Pure, Single-Phase Uranium-Plutonium Monocarbide Pellets," Los Alamos Scientific Laboratory report LA-4283 (1969), by M. W. Shupe, A. E. Ogard, and J. A. Leary.

The hot-hardness behavior of PuN, UN,[13] and $U_{0.7}Pu_{0.3}N$[9] is shown in Fig. 3. As might be expected, the nitrides are considerably softer than the carbides at low temperatures, but above 800°C PuN is harder than PuC. For example, at 1000°C the diamond pyramid hardness of PuC is ~40. Figure 3 shows that strengthening or hardening is obtained by alloying the UN with PuN, because the PuN-UN solid solution is harder than either component compound alone. Work at LASL will include further investigation of the effects of stoichiometry and uranium-plutonium ratio on the hot hardness of the monocarbides as well as measurements of the hot hardness of compounds, such as the sesquicarbides, which have not been tested before. As mentioned earlier, these measurements will be made at higher temperatures than heretofore possible.

III. Creep

Creep of Uranium Carbide. An important high-temperature mechanical-property measurement is that of creep. Most creep studies reported have been on UC.[1-7] A comparison of the results of some of these studies is given in Table II.

It is interesting to compare the values obtained for the stress exponent, n(in the creep equation). Theoretically, if the rate-controlling creep mechanism is stress-directed vacancy migration (called Nabarro-Herring creep), n equals 1. However, a value of n ~ 5 indicates that the creep mechanism is related to dislocation motion;

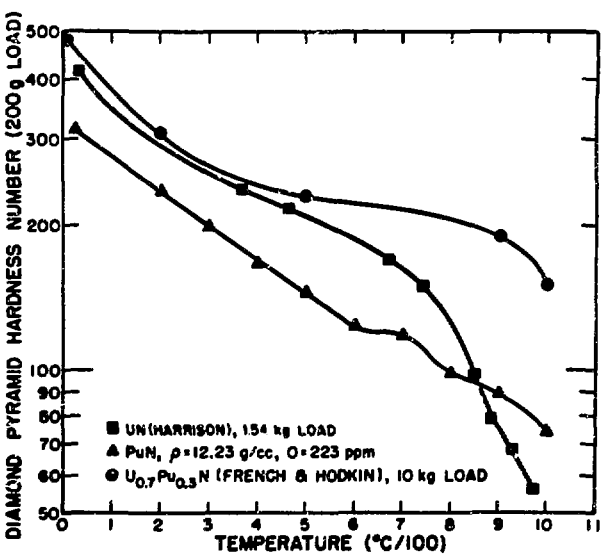

Fig. 3.
Hot hardness of uranium-plutonium nitrides.

for example, nonconservative motion of jogs on screw dislocations or dislocation climb. Values of n in Table II range from 1.8 reported by Norreys[1] to 5 reported by Chang.[4] Some investigators have postulated that Nabarro-Herring creep should become rate-controlling as the temperature is increased, but as the table shows, correlation of the temperature ranges used to measure creep in UC with the corresponding n values reported reveals that n increases with test temperature. Furthermore, although Stellrecht et al.[3] reported an average n value of 3, they found n to be 2.4 at 1300°C, 3.3 at 1400°C, and 4 at 1500°C. These results seem to indicate that as the temperature increases, the creep mechanism changes from vacancy migration to dislocation climb.

It is also interesting to compare the reported activation energies for creep of UC. Generally, the activation energies for high-temperature creep (at temperatures $\geq 1/2\ T_m$) in materials whose elastic modulus does not change much with temperature are close to that for self-diffusion. In an intermetallic compound such as UC, the slower diffusing species are rate-controlling. The activation energy for self-diffusion of carbon in UC was reported to be 50 kcal/mole by Chubb et al.[11] and 54 kcal/mole by Lee and Barrett.[26] Hence, the activation energies for creep reported by Norreys[1] and Fassler[2] (see Table II) are close to that for self-diffusion of carbon in UC. On the other hand, the activation energy for self-diffusion of uranium in UC was determined as 104 kcal/mole by Lee an Barrett and 64 kcal/mole by Chubb et al. Moreover, Lindner et al.[27] reported that for both hypo- and hyperstoichiometric UC the activation energy for self-diffusion of uranium in UC was ~ 30 kcal/mole at low temperatures, whereas at high temperatures they observed different activation energies: 70 ± 5 kcal/mole for the hypostoichiometric and 90 ± 6 kcal/mole for the hyperstoichiometric UC. This does not explain the relatively low activation energy for creep of UC reported by Chang, who worked with hypostoichiometric UC over a relatively high temperature range. Owing to the conflicting and confusing results of the creep and self-diffusion studies, no interpretation of the creep mechanism of UC can be made. Until both creep and self-diffusion measurements are conducted at the same laboratory on the same material, the rate-controlling creep mechanism will remain in doubt.

Effect of Microstructure. The effect of microstructure on creep in UC has not been systematically studied. Thus the effects of grain size, porosity, and amount and distribution of secondary phases have not been studied. For example, most UC creep studies have involved arc-cast material of about 200- to 400-μm grain size. Whereas grain size would be expected to have little effect if the creep mechanism is dislocation climb, it is important in Nabarro-Herring creep and in dislocation glide. Furthermore, in several materials, the proportion of creep deformation caused by grain-boundary sliding has been found to change with grain size, at least over a limited grain size range.

TABLE II

COMPARATUVE LITERATURE DATA ON CREEP OF URANIUM CARBIDE

Subject	Norreys[1]	Fassler[2]	Stellrecht[3]	Chang[4]
wt. % C	4.9 to 5.2	4.9	5.2	hypostoichiometric
Stress (psi)	2000 to 6000	6000 to 10,000	3000 to 10,000	-------
Temp. Range (°C)	1000 to 1500	1100 to 1300	1200 to 1600	1500 to 1900
Grain Size (μm)	~ 300	~ 30	~ 200	300 to 400
Specimen Preparation	Arc-Melted and Cast	Cold-Pressed and Sintered	Arc-Melted and Cast	Arc-Cast
Q(kcal/mole)	49 ±8	44	90	37.5
Stress Exponent, n	1.8	2.3	3	5

$$\dot{\epsilon} = A\,\sigma^{n} e^{-Q/RT} ,$$

where A = constant, Q = activation energy,
σ = stress, R = gas constant,
n = stress exponent, T = temperature.

Most studies of creep in UC are in general agreement on the effect of carbon content. The creep rate is highest for hypostoichiometric material with free metal in the microstructure and lower for hyperstoichimoetric material. Yet what is meant, for example, by the term "slightly hyperstoichiometric"? Figure 4 shows the microstructure of a specimen described[3] as "slightly hyperstoichiometric" UC. It is arc-cast material with a grain size of ~ 200μm. The large white areas are U_2C_3. Compare this to the "slightly hyperstoichiometric" UC shown in Fig. 5. This is hot-pressed material with a grain size of ~ 40μm. Although UC_2 platelets are present, there is no U_2C_3. Is it reasonable to expect the creep rates of these two "slightly hyperstoichiometric" specimens to be the same? Only opinions, not data, can be offered in answer to this question, because creep studies on UC have not emphasized microstructural effects.

Porosity might also be expected to significantly affect creep rate in specimens fabricated from sintered powders. Vasilos and Spriggs[28] and others[29] have developed and proposed several expressions to quantitatively correlate creep rate with porosity, usually by modifying the creep stress by some factor. No relationship has been found to hold for more than a very few cases, all of them oxides, not carbides. The problem is further complicated because some account must be made for the distribution or location of the pores; e.g., grain-boundary pores can affect creep by grain-boundary sliding. Much remains to be done in this area.

Although most creep studies on UC (and UN) have been of compressive creep, tensile creep in UC is now being studied by Battelle Memorial Institute. The creep resistance of UC is reported to be greater in tension than in compression.[7]

Creep of Uranium Nitride. Fassler et al.[2] and Vandervoort et al.[8] have studied creep in UN. Their data

Fig. 4.
"Slightly hyperstoichiometric" UC, arc-cast (Ref.3.).

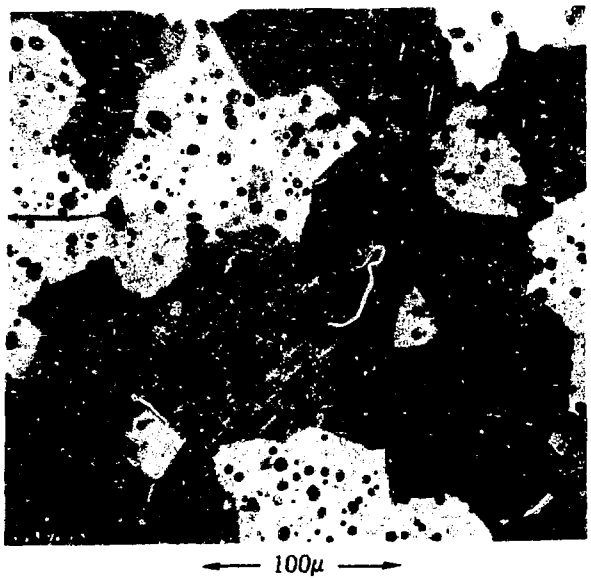

Fig. 5.
"Slightly hyperstoichiometric" UC, hot-pressed.

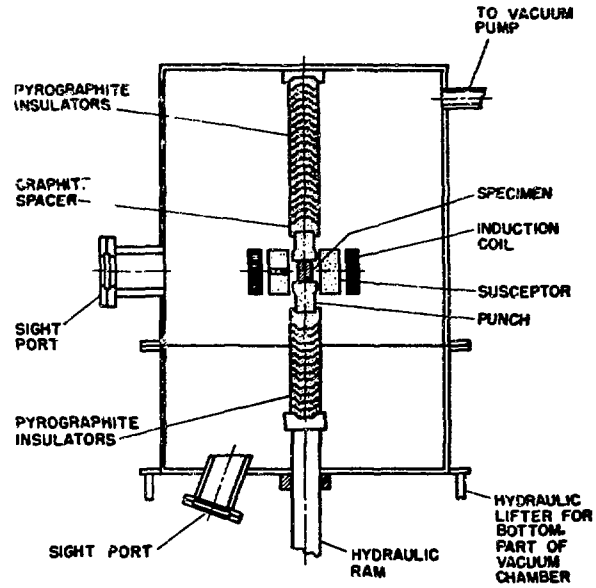

Fig. 6.
Deformation testing equipment.

indicate that the creep resistance of UN increases with oxygen content. Fassler et al. reported a value of 4.15 for the stress exponent (n) (compared to Vandervoort's 6.0), indicating that the creep mechanism in UN is related to dislocation climb or nonconservative motion of jogs on screw dislocations. According to Vandervoort, diffusion of the uranium ion is rate-controlling for creep.

Creep Measurement Apparatus at LASL. At Los Alamos, the compressive creep of mixed UC-PuC fuels is measured using equipment adapted from a design developed for hot pressing UC-graphite composites. Figure 6 is a schematic of this equipment. The specimens are solid right cylinders about 0.5 in. long and 0.4 in. in diameter. The specimen is heated by induction in a graphite susceptor. The compressive load is applied by a hydraulic ram. No graphite parts actually touch the specimen; instead, carburized tantalum disks are placed at either end of the specimen and act as diffusion barriers for graphite. Temperature readings are taken by an optical pyrometer through a blackbody hole in the susceptor. Specimen deformation is measured using an optical extensometer sighted onto the flat faces of the graphite pieces immediately above and below the susceptor.

Studies at LASL have tried to elucidate the creep mechanism or mechanisms in well-characterized uranium-plutonium carbides. Preliminary results show that most creep in specimens strained ~ 20% or less can be attributed to grain-boundary sliding. We base this evaluation on measurements of the average grain shape, defined in terms of the parameter (L/B) where L is the average grain length and B is the width, relative to the compressive stress direction. Sample areas in the sectioned, polished, and etched specimens are photographed, and individual grains are measured. The change in interior grain shape is compared with the change in specimen shape (total specimen deformation), and the amount of grain-boundary sliding is deduced from the difference. For example, the L/B ratio for a $U_{0.8}Pu_{0.2}C$ specimen deformed 23.6% was determined to be 0.99. From this it appears that most of the creep was from grain-boundary sliding. This result, plus similar findings on other specimens, indicates that sliding probably causes all the initial deformation. Of course grain-boundary sliding cannot contribute to deformation indefinitely without auxiliary processes, because some intragranular deformation is necessary to maintain geometrical continuity between the grains. When such deformation within the grains is absent, pores, and eventually cracks, form at triple points.

IV. Isotropic Elastic Constants

Literature. Some data on the isotropic elastic constants of monocarbides and mononitrides of uranium have been taken,[21-23,25,30,31] and there is one set of published data[31] for the isotropic elastic constants of $U_{0.82}Pu_{0.18}N$ as a function of temperature, but there are no published data on the elastic constants of carbides containing plutonium. Padel and DeNovion[31] present the most complete data on the elastic constants of UC and UN as a function of porosity and temperature.

Elastic Constants of $U_{0.8}Pu_{0.2}C_{1-x}$. Room-temperature longitudinal and transverse sound velocities of polycrystalline single-phase $U_{0.80}Pu_{0.20}C$ have been measured at LASL using a conventional ultrasonic pulse-echo technique. The isotropic elastic moduli and Poisson's ratio

were calculated from the measured sound velocities (Table III) and were evaluated as functions of material density. The Debye temperature of the single-phase carbide solid solution was computed from the extrapolated sound velocities for fully dense carbides.

The results of this investigation are presented in Table IV. Because there are no published elastic data for this compound, 100% dense elastic moduli of single-phase $U_{0.80}Pu_{0.20}C$ cannot be directly compared. Table V is a comparison of the data obtained in our investigation with the UC elastic data obtained by French workers.[31] The extrapolated elastic moduli of $U_{0.80}Pu_{0.20}C$ are slightly smaller than those for UC. The Poisson's ratio of theoretically dense $U_{0.80}Pu_{0.20}C$ was calculated to be 0.29, which agrees well with the 0.295 for UC reported by Battelle Memorial Institute[21] and the 0.286 reported by Padel and DeNovion.[31] Poisson's ratio decreased with increasing porosity, which corroborates data found in other elastic-constant investigations.[31-33] Data reported here apparently agree with the UC data, but are preliminary and have an estimated accuracy of ± 5%.

The Debye temperature of this material, calculated from the theoretically dense sound velocities, is approximately 323°K with a standard deviation of 5°K. From data on the elastic and thermodynamic properties of UC and PuC, Kempter[34] calculated the Debye temperature of PuC to be 271°K. This estimate, combined with the 330°K Debye temperature for UC,[34] allows one to estimate the Debye temperature of $U_{0.80}Pu_{0.20}C$ compositions to be 318°K, which is in good agreement with the data obtained in this investigation.

TABLE III

EQUATIONS USED TO CALCULATE THE ROOM-TEMPERATURE ISOTROPIC ELASTIC CONSTANTS AND THE DEBYE TEMPERATURE OF SINGLE-PHASE $U_{0.80}Pu_{0.20}C$

Technique:	Pulse-Echo
Frequency:	10 MHz
Specimen Size:	~ 3/8-in. Diam by 1/2-in. Length

$$V_\ell = \frac{2L}{t} \qquad \text{(Logitudinal Velocity)}$$

$$V_s = \left[\left(\frac{\Delta t}{D}\right)^2 + \left(\frac{t}{2L}\right)^2\right]^{-1/2} \qquad \text{(Shear Velocity)}$$

$$\overline{V}_m = \left[1/3\left(\frac{1}{V_\ell^3} + \frac{2}{V_s^3}\right)\right]^{-1/3} \qquad \text{(Average Velocity)}$$

$$\nu = \frac{1-2\left(\frac{V_s}{V_\ell}\right)^2}{2-2\left(\frac{V_s}{V_\ell}\right)^2} \qquad \text{(Poisson's Ratio)}$$

$$G = V_s^2\,\rho \qquad \text{(Shear Modulus)}$$

$$E = 2G(1+\nu) \qquad \text{(Young's Modulus)}$$

$$B = V_\ell^2\rho - (4/3)\,G \qquad \text{(Bulk Modulus)}$$

$$\theta = \frac{h}{k}\left[\frac{3N_o\rho}{4\pi M}\right]^{1/3}\overline{V}_m \qquad \text{(Debye Temperature)}$$

where

- t = time of longitudinal flight
- Δt = time between logitudinal and transverse flight
- ρ = sample density
- D = sample diameter
- L = sample length
- h = Planck's constant
- k = Boltzmann constant
- N_o = Avogadro's number
- M = molecular weight of sample

V. Summary

Data on the mechanical properties of the uranium and plutonium carbides and nitrides are sparse, but attempts are being made at LASL and elsewhere to rectify this. Because mechanical-property measurements are generally much more dependent on structure than are measurements of thermodynamic or physical properties, at LASL special emphasis is being placed on determining the effects of microstructure, particularly the effects of grain size, porosity, and secondary phases on properties such as creep. This sort of information is necessary and desirable not only from an engineering or design standpoint, but also because it can be used in correlation with diffusion data to better our understanding of basic high-temperature mechanisms in the actinide carbides and nitrides. Mechanical-property measurement of reactor fuels has not kept pace with research on the thermodynamic and physical properties, but surely knowledge of these properties will soon be further advanced than it is today.

Acknowledgments

The authors are indebted to Ronald D. Strong for his suggestions on ultrasonic measuring technique and to Richard E. Honnell, Robert W. Walker, and H. G. Moore for sample preparation.

TABLE IV

ROOM-TEMPERATURE ISOTROPIC ELASTIC CONSTANTS FOR SINGLE-PHASE $U_{0.8}Pu_{0.2}C_{1-x}$

wt % U = 76.04	a_o = 4.9651 ± 0.0003 Å[a]	Oxygen Content:	260 ppm
wt % Pu = 19.25	ρ_o = 13.576 g/cm³	Nitrogen Content:	265 ppm
wt % C = 4.71	Grain Size = 20 μm	Metallic Impurities:	< 400 ppm

Elastic Constant	Porosity Dependence (P = volume fraction porosity)	Standard Deviation
Young's Modulus	E = 2023 (1 − 1.54 P) (kbar)	54.4 (kbar)
Shear Modulus	G = 785 (1 − 1.52 P) (kbar)	26.1 (kbar)
Bulk Modulus	B = 1603 (1 − 1.73 P) (kbar)	110.7 (kbar)
Poisson's Ratio	ν = 0.290 (1 − 0.21 P)	0.018

[a]Lattice parameter at room temperature; 95% confidence interval with respect to internal consistency.

TABLE V

COMPARISON OF $U_{0.8}Pu_{0.2}C$ AND UC ELASTIC CONSTANTS

	$U_{0.80}Pu_{0.20}C_{1-x}$	UC[b] Ref. 31
Young's Modulus (kbar)	E = 2023 (1 − 1.54P[a])	E = 2249 (1 − 2.30 P)
Shear Modulus (kbar)	G = 785 (1 − 1.52P)	G_o = 873 (1 − 1.92 P)
Bulk Modulus (kbar)	B = 1603 (1 − 1.73 P)	B_o = 1768 (1 − 2.49 P)
Poisson's Ratio	ν = 0.290 (1 − 0.21P)	ν = 0.288 (1 − 0.59 P)

[a]P = volume fraction porosity.
[b]Calculated from first-order-P Mackenzie equation.

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