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TECHNOLOGY OF CERAMIC COMPONENTS

PART 3 OF THE TORY II-C PROGRAM

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UCRL-7036 Pt. 3

Nuclear Reactors for
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M-3679 (26th Ed.)

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UNIVERSITY OF CALIFORNIA
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Livermore, California

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TECHNOLOGY OF CERAMIC COMPONENTS — PART 3 OF THE TORY II-C PROGRAM

(Title: Unclassified)

A. J. Rothman

August 28, 1962

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FOREWORD

This is Part 3, "Technology of Ceramic Components," of the 10-part report UCRL-7036. A list of all 10 parts, including titles and major subheadings, appears on the following two pages.

LIST OF PARTS ISSUED UNDER UCRL-7036
THE TORY II-C PROGRAM:

Part 1 Introduction and General Description

Part 2 Structure of Reactor

(Description of Mechanical System, Fuel Loading, Structural Analysis)

Part 3 Technology of Ceramic Components (This Report) Page No.

a. Fabrication Procedures 1
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Part 4 Design Aspects

(Neutronic — Design criteria, Fuel map, Control system; Radiation Effects — Nuclear heating, External radiation levels, Activation of components; Heat Transfer and Fluid Flow — Design criteria, Pressure profiles, Component temperatures, Performance.)

Part 5 Control System

(System Requirements, Mechanical Description, System Analysis)

Part 6 Instrumentation

(Nuclear, Nonnuclear)

Part 7 Test Vehicle

(Description, Safety Features)

Part 8 Test Facility

(Site Description, Safety Features)

Part 9 Operations

(Staff Organization, Test Program, Emergency Plan)

Part 10 Hazards of Operation

(Normal Operation, Credible Accidents, Incredible Accident, Site Radiation Levels)

TECHNOLOGY OF CERAMIC COMPONENTS
PART 3 OF THE TORY II-C PROGRAM

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Livermore, California

August 28, 1962

A core of ceramic fueled tubes constitutes the heart of the Tory II-C reactor. The scope of this report is the description of these fueled tubes, an outline of the technique used in their fabrication, and their operating requirements and ability to meet these requirements.

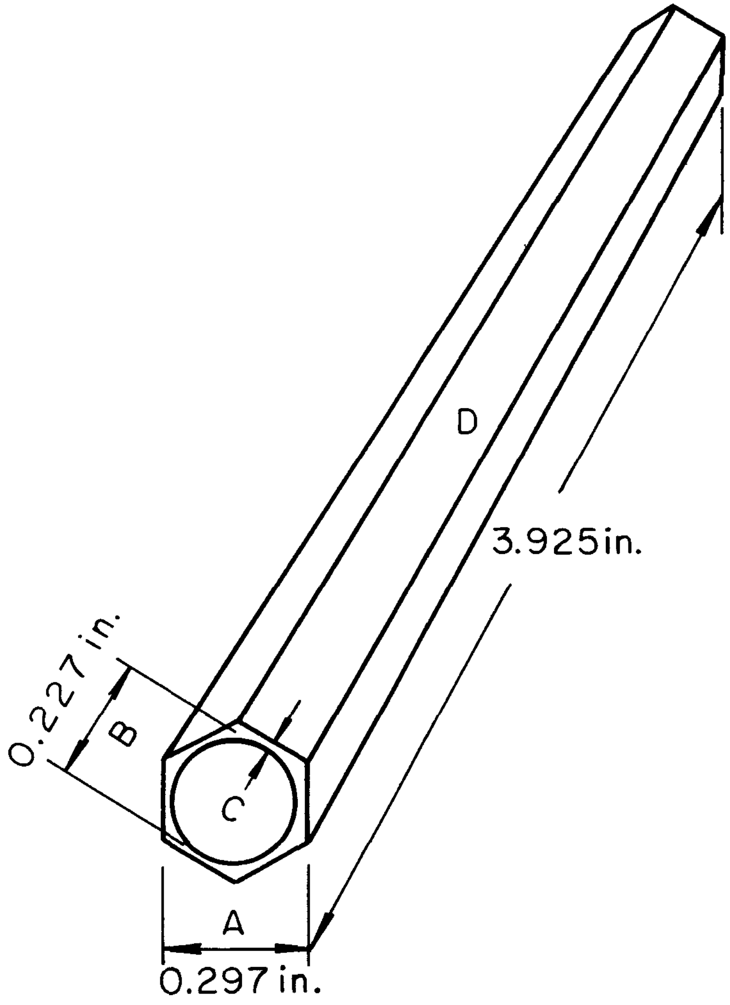
a. Fabrication Procedures

1. Description of Tubes

The tubes are hexagonal prisms 4 inches long and 0.297 in. across flats, having a central hole 0.227 in. in diameter. (See Fig. 1.). The tubes consist of a continuous BeO matrix containing in solid solution a fine dispersion of urania (UO_2), zirconia (ZrO_2), and yttria (Y_2O_3). The Y_2O_3 and ZrO_2 are used to help stabilize the tubes against phase transformation to U_3O_8 near 1200°C and to reduce fuel loss in the high oxygen pressures encountered in Tory II-C.

The mean BeO grain size in the matrix varies from about 5 to 20 microns, depending on the fuel loading and firing temperature. The fuel particles ZrO_2-UO_2-Y_2O_3 (hereafter referred to as "Horseradish") are typically 0.5 to 1 micron in diameter, although they are distributed in size from well under 0.5 to occasionally 10 microns. They are distributed within grains as well as on grain boundaries.

The fuel loadings are described as weight percent urania in the total BeO-UO_2-ZrO_2-Y_2O_3 matrix. They vary from about 1 to 7%, excluding the unfueled elements. (Details of fuel loading classes are given elsewhere in other reports.) The weight ratio of $UO_2/ZrO_2/Y_2O_3$ is fixed at 45.00% to 19.38% to 35.62% for all loading classes. Alternatively expressed, this is approximately 1 mole Y_2O_3 to 1 mole ZrO_2 to 1.057 moles UO_2.



MUL-18304

Fig. 1. Pluto fuel element.

DIMENSIONS

A. All must be 0.297 ± 0.001, average of all must lie within 0.0005 of 0.297.

B. All must be 0.227 ± 0.002, average of all must lie within 0.0005 of 0.227.

C. Must be 0.035 ± 0.003 for fueled tubes.

D. All surfaces "D" must lie entirely within the space between two concentric hexagonal boundary surfaces whose dimensions "A" are 0.2985 and 0.2955, respectively.

The phase observed in these fueled tubes by x-ray diffraction is face centered cubic, with lattice parameter (room temperature) $a_0 = 5.31 - 5.32$ Å as prepared (after hydrogen firing). Samples of the hydrogen-fired fuel mixture, both in the pure form and mixed intimately with BeO, were annealed at various temperatures up to 1400°C at 75 psia. Times of annealing were 10 to 16 hours. After the high-pressure oxygen anneals, the lattice constant decreased to 5.27_4 Å. No difference in lattice parameter was observed when BeO was present during the anneal.

Typical densities of the ceramic elements are as follows:

Wt % UO_2	g/cm^3	
0	2.95	(98%)
2	3.06	
5	3.15	
8	3.33	

Other than for pure BeO, the densities are 99-100% of "theoretical" density, calculated on the basis:

$$D_{\text{theoret.}} = \frac{3.01}{W_1 + 0.277W_2 + 0.570W_3 + 0.479W_4},$$

where W_1 through W_4 are the weight fractions, respectively, of BeO, UO_2, Y_2O_3, and ZrO_2.

2. Fabrication Techniques

High-purity raw materials are used throughout: sinterable BeO powder, uranyl nitrate (U-235), yttrium nitrate, and zirconyl nitrate. The nitrate solutions are slurried with the BeO powder, and the fuel is coprecipitated by the addition of ammonium hydroxide (Fig. 2). This mixing technique is referred to in this report as slurry-precipitation or "slurpitation." The term "pre-coprecipitation" refers to coprecipitation of the fuel components in the absence of BeO, followed by mixing the precipitate and fuel together in a liquid suspension. Slurpitation is now the standard technique used in production.

The mixture is filtered, dried, and calcined at 1000°F to render it uniform. It is then blended with half its weight of a binder mixture containing polyvinyl alcohol, methyl cellulose, and water, and finally extruded under about 8,000-10,000 psi pressure through a die. The tubes are dried, the binders are burned out in air at 1500°F, and finally the fuel elements are

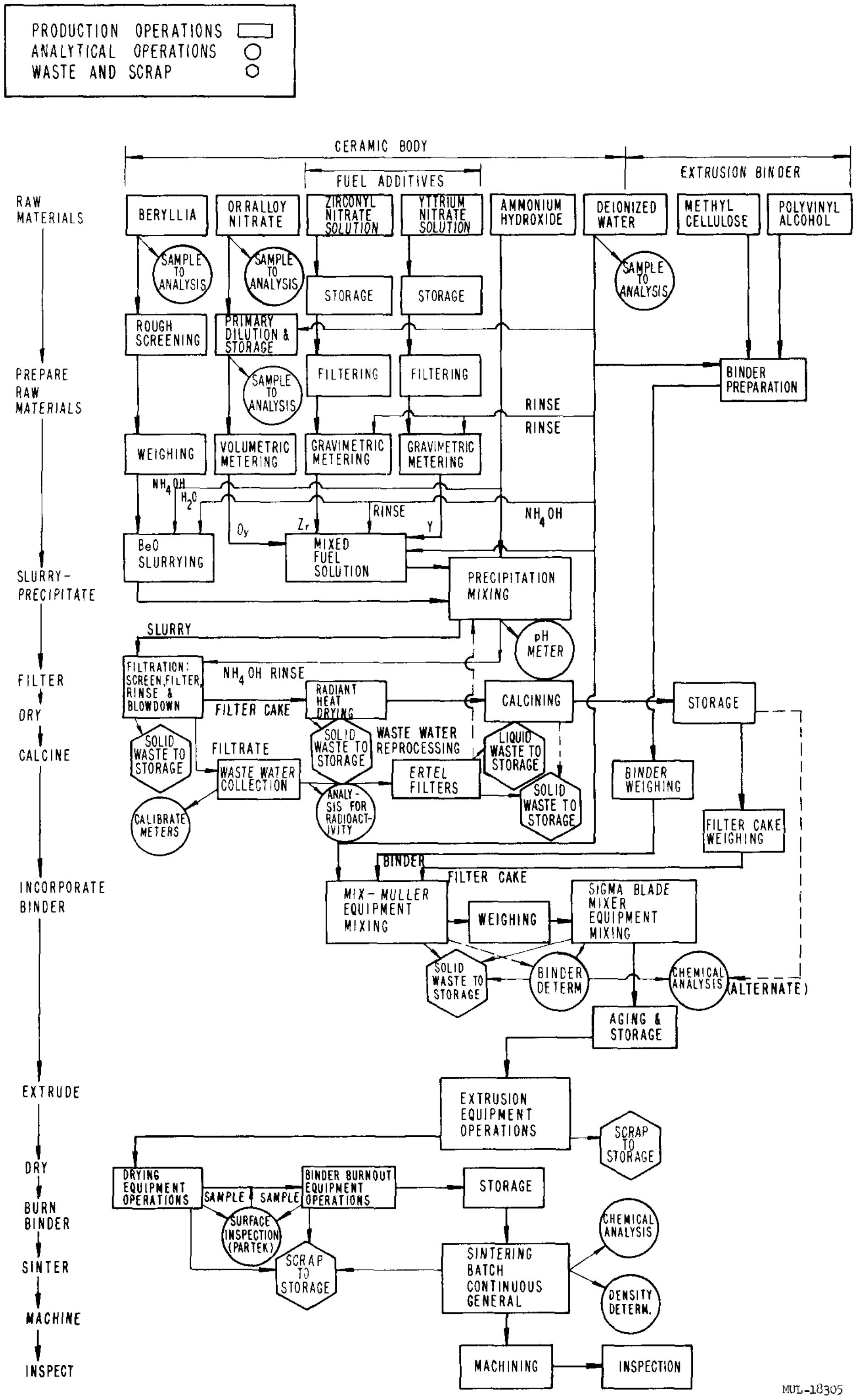


Fig. 2. Schematic flow sheet for fuel element fabrication.

fired in hydrogen at 3090°F (1700°C) to densify them. The firing temperature may be reduced 100 or 200°F based on subsequent evaluation.

b. Characteristics of Ceramic Elements

Fuel elements for Tory II-C must meet the following special requirements:

1) Withstand mechanical stresses, including those originating from thermal gradients.

2) Withstand thermal cycling stresses.

3) Retain fuel while in a high-pressure air stream.

4) Withstand radiation fields.

Fuel elements must also withstand attack of moist air, although this factor is unimportant in a typical 5-minute Tory II-C run. But for Pluto engines like Tory II-C the water attack problem is marginal for 10-hour runs at 2500°F wall temperature, and hence coatings designed to prevent water attack are being studied. Such coatings will not be a part of Tory II-C, however.

1. Mechanical Stresses

As in Tory II-A, mechanical stresses per se will be small. However, those originating from temperature gradients (thermal stresses) will be appreciable.

Thermal stress. In practice, a thermal stress resistance parameter $\sigma k/(1 - \nu)E\alpha$ is determined by measuring how much power must pass through the sample wall to cause cracking in the sample. The results may be plotted as $\sigma k/E\alpha$ (Fig. 3) for any assumed ν (Poisson's ratio, here 0.33) and the results compared with the Tory II-C demand. Details of the apparatus and assumptions made in the calculations are given by Wells and Cline.[1]

Earlier data on BeO and BeO-UO_2 were conflicting. Blowpipe data indicated that only slight cracking would occur,[2] while steady-state thermal stress tests using thick-wall specimens in static gas gave more pessimistic results. These earlier samples had BeO grain sizes of about 20 to 50 microns.

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[1] W. M. Wells and C. F. Cline, "Thermal Stress Tests of BeO and BeO-UO_2," UCRL-6852 (1962).

[2] J. W. Hadley, "Tory II-A — A Nuclear Ramjet Test Reactor," UCRL-5484 (1959).

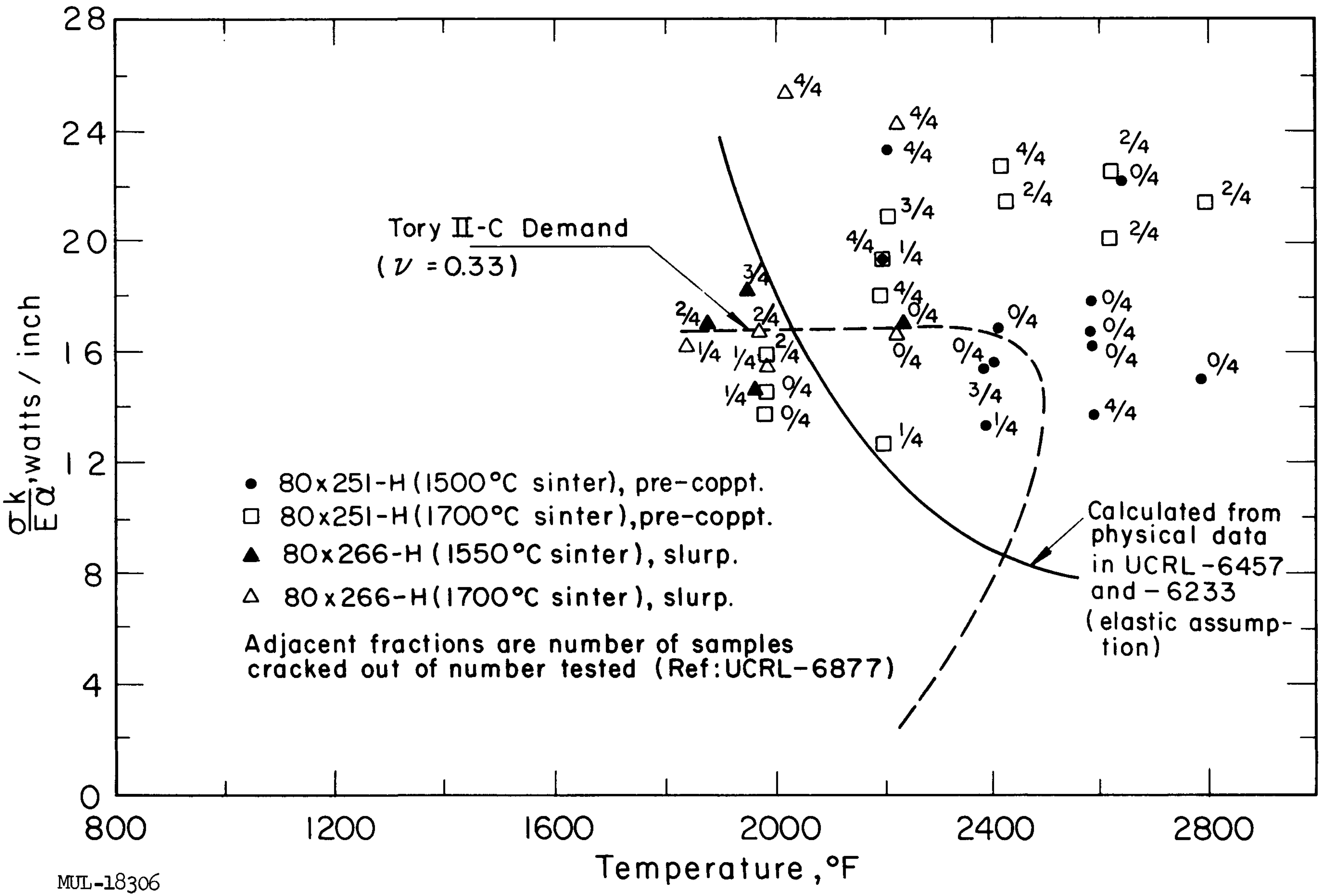

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Fig. 3. Thermal stress resistance of Horseradish (8% UO_2) samples.

Recent process improvements utilizing Horseradish fuel added by slurry-precipitation have resulted in finer grained fueled BeO with improved elastic strength at low temperatures and probably more creep relaxation near the higher temperatures of Tory II-C. As a result the material exhibits better thermal stress resistance in the temperature range studied (see Fig. 3). The grain size may be kept down to 8 microns at and above 5% UO_2, although it is larger for very low fuel concentrations (near 1%). Thermal stress tests of the thick-wall specimens in static gas are not yet complete, but they indicate that strengths of tubes with high fuel loading are adequate to meet Tory II-C demands without excessive cracking (Fig. 3).

Actually, some failures plotted were not typical in that cracks apparently occurred on cooling. If the sample undergoes creep during the application of thermal stress, then subsequent cooling will cause a stress reversal whose magnitude is about double* the original thermal stress for sample geometry like ours.[3] In our experimental arrangement the heat flows from the inside of the sample outward during application of the stress. Fracture of the 1700°C sintered material at test temperatures above 2400°F originated in the bore, thus indicating that failure occurred on cooling.

Yet to be assessed are the low fuel loadings, which may show somewhat lower thermal stress resistance because of larger grain size.

It should be noted parenthetically that evaluation of the Tory II-A fuel elements to date shows that cracking of tubes in central areas of the core has not been excessive. The large number of tubes which cracked in cooled peripheral areas of high temperature gradient in the reactor, did so because of high temperature gradients across their diameters rather than in their walls. Even these did not shatter, but maintained integrity. Thus, cracking of some tubes would not appear to be disasterous to the reactor.

Modulus of rupture. Typical values of modulus of rupture of 4-inch tubes measures on a 3-inch, three-point span are 30,000-40,000 psi. Modulus of rupture is a function of density and grain size, and therefore is influenced by sintering temperature. Figure 4 shows some recent measurements of strength vs sintering temperature for slurpitated tubes. More data are being obtained to augment these. Other data obtained show somewhat higher strengths for all sintering temperatures, especially those below 1600°C.

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*This doubling of stress is true for the configuration used in the test referred to. It is not intended that it be applied to the reactor situation.

[3] S. Timoshenko and J. N. Goodier, Theory of Elasticity (McGraw-Hill Book Co., Inc., New York, 1951), p. 413.

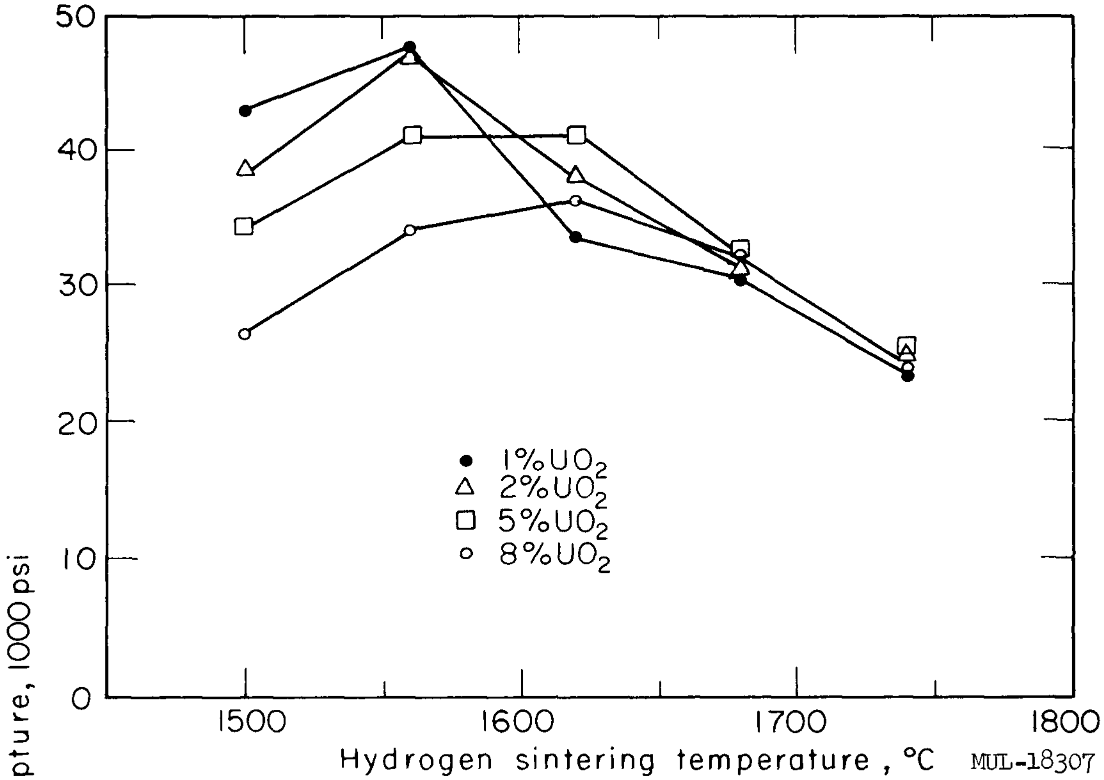

Fig. 4. Modulus of rupture: three-point loading, 3-in. span, 30-lb/min load.

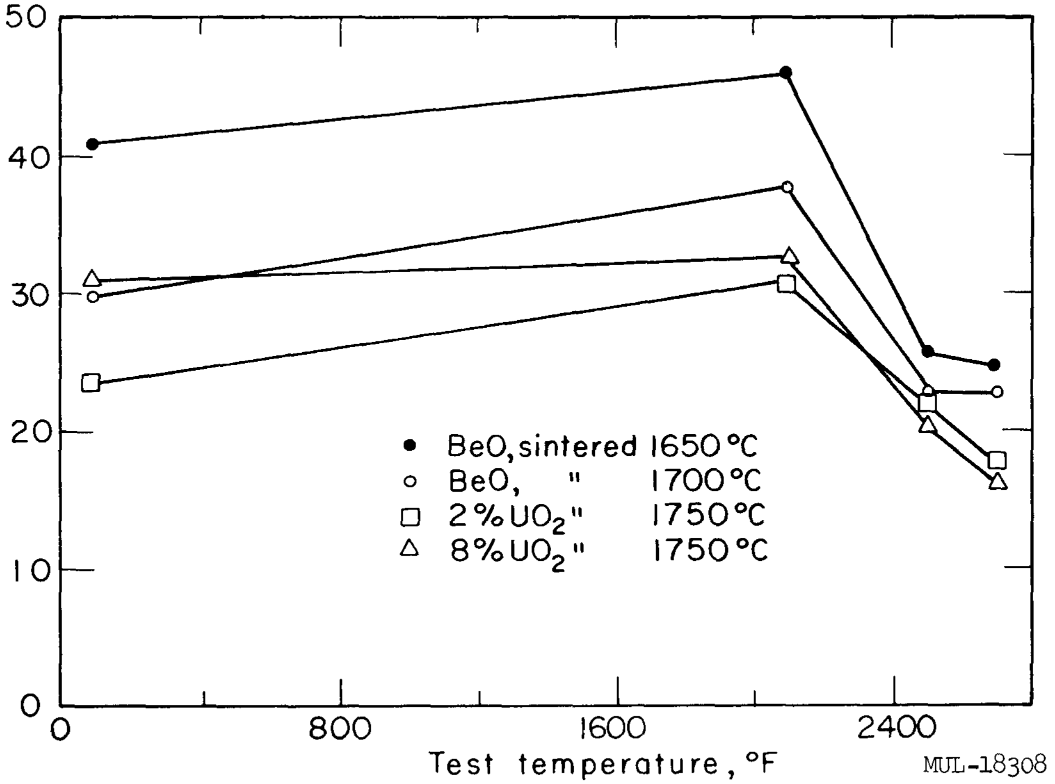

Fig. 5. Modulus of rupture vs test temperature.

Figure 5 illustrates modulus of rupture as a function of test temperature for several fuel loadings. These results are intended to show the relative variation of strength with temperature for tubes sintered at some arbitrary temperatures, not necessarily the optimum. In fact, the sintering temperatures for the fueled tubes are definitely higher than optimum. Note that the strengths at first increase with test temperature, then fall. The values recorded at 2500 and 2700°F are certainly too high since creep was observed to occur in the test.

Compressive creep. Compressive creep of BeO fueled with Horseradish has been measured in air and is listed below. As indicated in UCRL-6826, creep rate is inversely proportional to the square of the grain size. Consequently, statements about creep must include characterization of the grain size. Typical creep values for fueled BeO tubes are given in Table I.

Table I. Typical compressive creep measurements on fueled BeO tubes.

Specimen number	Stress (psi)	Temp (°F)	Time (hours)	Strain (%)	Predicted strain (%)	Density (g/cc)	Grain size[a] (microns)
11-1	3000	2700	5.00	0.10	0.11	3.37	~30
11-3	500	3100	3.52	0.25	0.26	3.35	~30
41-1	3000	2700	10.2	6.82	7.45	3.34	~5
41-2	500	3000	12.0	10.78	14.10	3.33	~5

[a] These values listed are found by the conventional ASTM intercept method.

To summarize the effects of compressive creep:

1) The effect of compressive creep in producing distortion of the fuel elements in a 10-hour Tory II-C mission may be neglected. Extrapolation would indicate 0.2% for the finest grained material (assumed to be 5 μ) at 2550°F under a stress of 300 psi for 10 hours.

2) Creep under other conditions may be estimated using the Nabarro-Herring creep relationship with ΔH = 96 kilocalories as follows:

$$\dot{\epsilon} = K \frac{s}{d^2} e^{-\Delta H/RT} \approx 5 \times 10^7 \frac{s}{d^2} e^{-48,314/T}$$

where

$\dot{\epsilon}$ = creep rate, in./in.-hr,

s = applied stress, psi,

d = mean grain diameter as determined by the intercept method, microns,

T = absolute temperature, °K.

It must be cautioned that the above constant K is satisfactory only for the particular material tested. Extrapolation even to the same material, but with a different thermal history, must be done with caution. Ideally one should determine the constant from at least one creep test on a like material. Furthermore, the density effect is not known. The above equation applies to high-density materials (>98.5%).

3) As is evident from a previous part of this report, grain size may be changed to vary creep. Thus one might reduce or increase creep by varying the sintering temperature of the fuel elements.

2. Fuel Loss and Structural Stability

Fuel loss and cycling. Of interest in a consideration of reactor performance are the questions of how much fuel is volatilized due to oxidation, and whether the fueled tubes retain integrity after oxidation exposure. These questions are dealt with below.

Fuel loss was measured in slowly flowing oxygen (75 psia) at 1650°C (3000°F) for 10 hours, using gamma counting of U^{235}. The average loss was 2.5% of the uranium for both 2 wt % UO_2 and 8 wt % UO_2 Horseradish-fueled BeO. The range of values for 33 samples was 1 to 4%. A few samples were tested at 1500°C in oxygen at 75 psia, but at this temperature the fuel loss is about the same (~0.5%) as the probable error in our counting. A plot of fuel loss vs time is given in Fig. 6 for a temperature well in excess of Tory II-C's and for longer times.

After fuel loss testing, the elements were measured for modulus of rupture. Only minor losses in strength were noted. See Table II.

Table II. Modulus of rupture[a] of fueled tubes before and after testing, given in thousands of psi. (Pre-coprecipitated tubes sintered 1700°C/4 hr in H_2.)

	Before test		After thermal cycling		After fuel loss	
Tube	Mean	Range	Mean	Range	Mean	Range
8% UO_2	28	27-30	24	23-26	24	17-29
2% UO_2	26	24-29	23	19-24	25	23-28

[a] Three-point loading, $1\frac{1}{2}$-in. span.

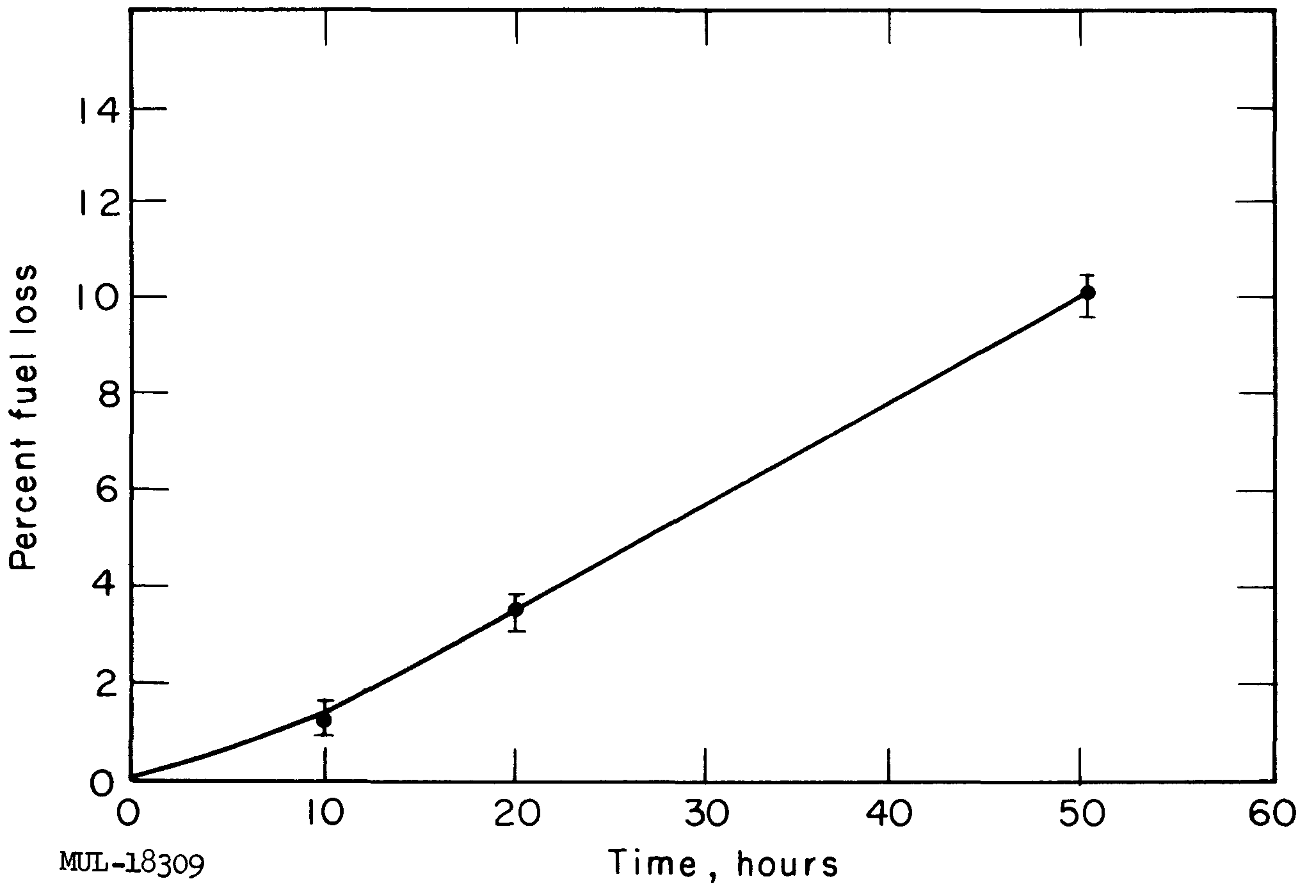

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Fig. 6. Fuel loss vs time at 1650°C (3000°F), 75 psia oxygen.

Thermal cycling to 1500°C consisted of heating to temperature in 10 minutes, holding for 10 minutes, and cooling to approximately 200°C in 10 minutes for a total of 10 cycles, to simulate reactor tests. All experiments were done in 75 psia oxygen. There was no evidence of structural degradation in any of the samples tested. Fuel losses were less than 0.5% uranium. As in fuel loss testing, only minor losses in strength were noted (see Table II).

X-ray diffraction studies made in 75 psia oxygen up to 1400°C show that the UO_2-Y_2O_3-ZrO_2 solid solution is stable under these conditions. Further, a negligible volume change results upon converting the compound from the oxygen-poor state (as sintered in hydrogen) to the oxidized form.

Similarly, an x-ray diffraction analysis of a tube which had been tested for fuel loss at 1650°C for 10 hours in 75 psia oxygen revealed the same face-centered structure with the same lattice constant. Microscopic examination of a thin section of the tube before and after testing revealed no significant change in the microstructure. There was no indication that reaction had occurred between the fuel phase and the BeO.

Thermal expansion results indicate that the Horseradish material has a higher coefficient of thermal expansion than BeO, although this does not seem to have any effect on the mechanical properties. The expansion coefficients are:

UO_2-Y_2O_3-ZrO_2 (in air or 1 atm O_2): $a_{21-1000°C} = 12.6 \times 10^{-6}$ in./in.-°C
BeO: $a_{21-1000°C} = 8.6 \times 10^{-6}$ in./in.-°C

In conclusion, fuel losses are negligible and fuel stability is good in the temperature region of interest for Tory II-C. Tubes of high fuel loading may swell slightly during oxidation, but not excessively in this temperature region. (See "Swelling" below.)

Swelling. Although no cracking or gross loss in strength is observed when the fuel elements are oxidized, "swelling" or volume expansion of tubes is observed in some cases. This is determined by measuring tubes at room temperature both before and after tests in oxygen at high temperature. In extreme cases, even blistering has been observed. The evidence is strong that the combination of fuel components and residual sulfur in the BeO is responsible for the swelling.

The swelling phenomenon is related to hydrogen sintering temperature (and perhaps sintering cycle), fuel loading (% UO_2), and oxidation test temperature. It is apparently also related to initial sulfur in the BeO. It may also be related to the method of adding fuel to BeO and to freedom of circulation of hydrogen in the sintering operation. There is appreciable variability in both the amount of swelling and in sulfur analyses. Consequently, we do not have complete definition of the problem for all conditions, but have determined the effect of each variable in a limited series of tests.

In summary, these are the pertinent observations after oxidation testing for 10 hours at 1500°C:

1) At low fuel concentration (2% UO_2 in tests), negligible swelling is observed for materials sintered at or above 1650°C.

2) At intermediate (5% UO_2) and high (8% UO_2) fuel concentrations, tests indicated appreciable swelling for low sintering temperatures (2-3% volume at 1600°C sintering temperature), and very small swelling (< 1% volume and < 1 mil across flats) for a sintering temperature of 1650 to 1700°C.

3) Swelling of the 5 and 8% UO_2 was found to be larger for several high-sulfur (1100 ppm S) BeO batches and less for low-sulfur (500 ppm S) batches. There is still a question as to whether sulfur content alone is sufficient to cause the swelling problem, since duplicate batches containing the same nominal sulfur content showed grossly different results.

4) Low sintering temperatures (near 1600°C) are probably desirable on other grounds to maximize strength (see "Thermal Stress"). Reasonable compromises may be made between strength and swelling, and final sintering temperatures will be chosen to keep swelling acceptably low (< 1% volume and < 1 mil across flats).

5) Note that at Tory II-C conditions, the swelling problem exists, but is not severe since temperatures are low compared to swelling test temperatures, and since by far the majority of the fuel elements contain < 3% fuel (see Table III), while only a small portion (10%) of the reactor consist of fuel loadings of 4% or above.

Table III. Distribution of tubes according to fuel concentration.

Approximate fuel concentration, a (% UO_2)	Percent of tubes having concentration $\leq$ a
2	40
3	74
4	91
5	96

3. Irradiation Damage Effects

Ample evidence exists that neutron exposures equivalent to those required for a 10-hour Pluto mission have no significant effect on unfueled BeO at temperatures of interest. There is a question, however, about the behavior of fueled BeO.

A program of irradiations is in progress to assess the effect of reactor irradiation on the properties of fuel elements. Tests are made in static oxygen or in an inert gas, depending on the conditions desired. The sample is fueled with oralloy and heated by its own fission energy. The irradiations to date have been on pre-coprecipitated tubes, containing 8 wt % $U^{235}O_2$, and having densities greater than 3.3 grams/cm^3. The irradiations were made in the LPTR, at a dose rate about 30% that of the maximum Tory II-C power level. The total dose ranged from 20 to 200% of the dose a Tory II-C fueled tube would receive in 10 hours at full power.

The only property which seems to be affected by radiation at exposures of Pluto level is the thermal conductivity. The room-temperature thermal diffusivity is measured before and after irradiation. The data obtained to date are given in Table IV. From these data, it appears that a real, but small, effect on the thermal diffusivity is produced by radiation at these temperatures. It also appears that a saturation value is reached in about 2 hours of equivalent full-power Tory II-C operation. The experiments will continue, with the intent of examining this effect at lower temperatures, shorter times, and at flux densities more nearly comparable to those expected for the Tory II-C reactor. It is expected from preliminary evidence that the effect will be significantly greater at lower temperature.

In addition to the thermal diffusivity measurements, the tubes are examined for dimensional changes and changes in modulus of rupture. The effect of irradiation on these properties is negligible.

Table IV. Relative room-temperature thermal resistivity before and after irradiation.

Sample	Irradiation temperature (°C ± 50)	Integrated power density[a] (MW-hr/ft^3)	Time, t (hr)	Atmosphere	R/R$_0$[b]
1	1450	536	63.1	Oxygen	1.35 ± 0.1
2	1450	493	58.0	Oxygen	1.51 ± 0.1
3	1400	0 - control	60.0	Oxygen	0.90 ± 0.1
4	1400	0 - control	60.0	Argon	1.1 ± 0.1
5	1400	320	37.7	Neon	1.4 ± 0.2
6	1400	51	6.0	Neon	1.37 ± 0.1

[a] The peak integrated power density in Tory II-C for 10 hours in the temperature range 1800 to 2500°F is 266 MW-hr/ft^3.

[b] $R/R_0 = \frac{\text{Thermal resistivity after irradiation}}{\text{Thermal resistivity before irradiation}}$.

In conclusion, irradiation decreases thermal conductivity measurably in the regions of interest, but not markedly at the maximum Tory II-C temperature. At minimum Tory II-C temperatures, the conductivity loss is expected to be worse.

/mr