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A NEW TECHNIQUE FOR EXPERIMENTAL INVESTIGATION
OF HIGH-TEMPERATURE THERMAL DIFFUSIVITY
OF URANIUM DIOXIDE UNDER TRANSIENT CONDITIONS

by

Leslie D. Montgomery

Reactor Physics Division

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I. INTRODUCTION

Recent advancements in nuclear-reactor technology have placed increased emphasis upon the development of fuels that can be used at high temperatures. Since uranium and its lower alloys exhibit low melting points and structural instability at elevated temperatures due to the accumulation of fission gases, solid fuels in reactors of the future may consist of high alloys of uranium with nonfissionable materials or may consist of uranium compounds.

Work is now in progress to study various refractory uranium compounds as potential high-temperature fuels. The materials being studied include uranium oxides, carbides, silicides, nitride, and sulfide. Perhaps the most important trait of a desirable fuel compound would be good stability in irradiation at high temperatures. Other desirable characteristics are a high melting point, low neutron-absorption cross section, chemical and metallurgical inertness, high thermal conductivity, high uranium density, and good mechanical strength with resistance to thermal shock.

One compound will probably not possess all of the aforementioned characteristics. Uranium dioxide fulfills many of these requirements, but it lacks structural strength, has a low uranium atom density, and has poor thermal conductivity. Nevertheless, more experimental work has been performed on the investigation of the physical properties of uranium dioxide than upon any other uranium fuel compound. At present, it appears to be the most promising of the ceramic fuels.

Only limited data are available on the thermal conductivity of sintered uranium dioxide, at high temperatures and under irradiated conditions. The apparent reason for the lack of such data appears to be twofold: 1) The difficulty of conducting experiments at high temperatures by which to measure the thermal conductivity of uranium dioxide; 2) the difficulty of taking into account the effect of cracking produced by thermal stresses upon the thermal conductivity.

There are several transient and steady-state methods for measuring thermal conductivity; however, there is some dissatisfaction with the

length of time required to make reliable measurements. If the time required to measure the thermal conductivity is relatively long, errors due to the cooling of the sample may be introduced in the values obtained. A need exists for a rapid and reliable method for measuring the thermal diffusivity of uranium dioxide over wide ranges of temperature and under reactor operating conditions.

The object of this study is to test the feasibility of an entirely new method of measuring the thermal diffusivity of ceramic reactor fuel materials, with a specific application to uranium dioxide at temperatures in excess of 1000°C.

II. LITERATURE SEARCH

Some of the uranium compounds under investigation as potential, high-temperature, reactor fuel materials are uranium oxides, carbides, silicides, nitride, and sulfide. Although the bulk of the high-temperature fuel development is being carried on with UO_2 , it may be of interest to survey its competitors briefly.

Epremian¹ stated that under exceptional conditions the uranium borides might be utilized as a high-temperature fuel. Uranium forms the borides, UB_2 and UB_4 . Both compounds have melting points above 1500°C, but neither compound has good chemical stability with air or steam. Since boron has a high neutron-absorption cross section, the use of a boride would require the separated B^{11} isotope.

The carbides of uranium, UC and UC_2 , have high melting points and higher uranium density than UO_2 , and at elevated temperatures possess thermal conductivities five to six times as great as UO_2 . The thermal conductivity of uranium carbides remains fairly constant as temperatures increase; that of UO_2 decreases appreciably with increasing temperatures. Uranium carbides UC and UC_2 are chemically unstable in hot water or air but appear to be useful in gas-cooled or in liquid-metal-cooled reactors.

The nitride of uranium also has a very high melting point and uranium atom density. Its main disadvantages are chemical instability and a high neutron-absorption cross section.

Uranium sulfide is comparable to UO_2 in melting point and uranium atom density and is stable in boiling water but will readily oxidize in high-temperature air.

The uranium compounds of silicon possess favorable properties at high temperatures and have a better thermal conductivity than UO_2 . Under irradiation, the uranium-silicon compounds undergo severe distortion, swelling and, decomposition, which may limit their use as reactor fuels.

In addition to the compounds mentioned, several alkaline-earth uranites are being studied. These compounds are quite stable and have melting points in excess of 1500°C . Irradiation of such samples at 200°C caused fragmentation and complete release of fission gases. Decomposition occurred when these compounds were heated to 1800°C .

Although an extensive exploratory program is being conducted to develop a satisfactory high-temperature fuel material, more research has been conducted, and more information is available on UO_2 than on any other refractory fuel compound. For this reason, UO_2 at present is more widely utilized as a reactor fuel material than any other uranium compound.

Before a potential fuel material can be practically utilized in a reactor, its thermal conductivity must be measured accurately. Thermal conductivity may be measured by either static or dynamic methods. In dynamic methods, the temperature is varied suddenly or periodically for one portion of the sample, and the temperature change with time is measured to determine the thermal diffusivity, $k/\rho C_p$. In static methods, the sample is allowed to come to a steady state, and the temperature distribution is measured to determine the thermal conductivity, k .

A study of the literature by the author indicated that many methods have been used to measure the thermal diffusivity and thermal conductivity of materials. A survey of the methods that may be applied to refractory fuel compounds will illustrate some of the difficulties in an experiment such as the one proposed.

Two experimental groups^{2,3} utilized a xenon flash lamp to determine the thermal diffusivity of various nonfissionable materials over a wide range of temperatures. A high-intensity, short-duration, light pulse was absorbed in the front surface of a thermally-insulated specimen a few millimeters thick, coated with camphor black, and the resulting temperature history of the rear surface was measured by a thermocouple and recorded with an oscilloscope. The oscilloscope pattern was photographed, and the thermal diffusivity of the sample was calculated from the time-versus-temperature curve, as described by Parker *et al.*³

Loeb⁴ proposed an idealized experiment for measuring the thermal conductivity of ceramic materials at high temperatures. The experimental method consisted of surrounding a long, uniformly-heated cylindrical core by a hollow cylinder of the ceramic, and using the cylindrical form of the heat-flow equation in determining the conductivity from the average temperature gradient across a certain distance in the ceramic.

Upon further investigation, Loeb found that heat loss due to end effects of the sample produced appreciable errors in the obtained thermal-conductivity values. Experiments to alleviate this problem were in progress.

Preliminary experiments were performed by Firman et al.⁵ to develop a method for measuring thermal conductivity suitable for in-pile use. A transient method was employed involving the measurement of sinusoidally-varying temperature at two points along a specimen rod which was periodically heated at one end. The thermal diffusivity of the material was calculated from a simple relationship between the amplitude decrement and the velocity of the temperature wave within the sample.

Such an experiment was performed on nickel, Armco iron, and C.Y. 9 carbon, from which values were obtained that agree with existing data. Firman et al.⁵ stated that this method would yield accurate values with materials whose thermal diffusivity is above $0.02 \text{ cm}^2/\text{sec}$. This fact would prohibit the use of this method on UO_2 , but it may be applicable to other refractory fuels.

The thermal conductivity of UO_2 has been treated experimentally over a wide range of temperatures. Most of the experiments described below were conducted under out-of-pile conditions.

Kingery et al.⁶ reported data on UO_2 having 73% theoretical density. The measurement was made by means of a linear-flow-comparison method. Samples of known conductivity were placed in series with unknown samples. Kingery also used this method to measure the thermal conductivity of UO_2 having 95% theoretical density. This is the only conductivity information available on high-density uranium dioxide over a wide range of temperatures.

Hedge and Fieldhouse⁷ at Armour, using a stacked-disk method, measured the thermal conductivity of uranium dioxide that was 75% of theoretical density to 1670°C . The method consisted of measuring, during steady state, the radial heat flow and radial temperature drop in a vertical stack of disks composed of the material whose thermal conductivity was to be measured. The disks were in the form of annular rings, and the heat that flowed through the disks was supplied by an electric heater centered in the axial hole of the stacked disks. The temperature gradient was measured by means of thermocouples inserted in small vertical holes, one near the axial hole and the other near the outer edge of the disks. The thermal conductivity of the disk material was calculated from the power input and the distance of the inner and outer thermocouples from the axis of the disk, between the holes.

At approximately 1670°C , severe cracking of the sample occurred and prevented further experimentation. The values obtained by Hedge and Fieldhouse were appreciably lower than those measured by Kingery et al.

Scott⁸ at Harwell, England, conducted an experimental determination of the thermal conductivity of UO_2 of 96% theoretical density, over the range

of 800 to 1150°C. The experimental method was based on the measurement of the radial temperature difference between the center and surface of a cylindrical sample while heat was being generated throughout its volume by the passage of electric current.

Reiswig⁹ measured the thermal conductivity of 85% theoretical density UO_2 to 2100°C. A radial-heat-flow method, similar to that employed by Hedge and Fieldhouse, was used. Instead of thermocouples, as used by Hedge and Fieldhouse, Reiswig used a Pyro Micro-Optical pyrometer, focused down sight holes, to measure the radial temperature difference between the center and the outer edge of the sample. The values obtained by Reiswig agreed favorably with those of Kingery *et al.* and Hedge and Fieldhouse.

The chief source of error in Reiswig's experiment was probably an error in the measured power input. Cracks, due to thermal stresses, formed in all disks except one, allowing some of the measured power to escape from the heater rod to the radiation shield by direct radiation rather than by conduction through UO_2 .

Englander¹⁰ reported an average value of $0.01 \text{ cal-cm}^{-1}\text{-sec}^{-1}\text{-per } ^\circ\text{C}$ at approximately 70°C for material having a density of 9.2 g/cm^3 .

The first in-pile determination of the thermal conductivity of uranium dioxide appears to have been made by Flinta¹¹ in Sweden. The UO_2 samples had 91% of theoretical density and were subjected to a neutron flux of $1.8 \times 10^{12} \text{ n-cm}^{-2}\text{-sec}^{-1}$ in the research reactor, R1. The method was essentially a stacked-disk method. Five annular-shaped disks 15 mm in diameter and 2 mm long were stacked together, with a tungsten rod passing through the central hole and serving as a heat source in addition to the heat caused by the fissioning. The thermal conductivity was calculated by using the temperature gradient between two thermocouples, one near the center of the sample, and the second near the outside surface of the UO_2 pellets.

The thermal conductivity was erratic with neutron irradiation, and it dropped to $0.007 \text{ W-cm}^{-1}\text{-}^\circ\text{C}^{-1}$ after three heating cycles.

The latest in-pile measurement of the "effective" thermal conductivity of sintered uranium dioxide was made by Eichenberg¹² in the Materials Testing Reactor. PWR fuel-element pellets 0.3535 in. in diameter were stacked in a stainless-steel capsule and subjected to an estimated neutron flux of $0.7 \times 10^{20} \text{ nvt thermal}$. By measuring the temperature difference between the center on the sample pellets and the exterior, the thermal conductivity was determined up to 480°C. The values were about 50% of those obtained by Hedge and Fieldhouse.

The only determination of the thermal conductivity of UO_2 at temperatures in excess of 2100°C reported at the time of writing was carried out by Bates²² at Hanford Atomic Products Operation. Bates made laboratory investigations of unirradiated UO_2 at temperatures to the melting point and established the characteristic temperatures at which certain physical changes occur. By observing similar physical changes in post-irradiation specimens, Bates deduced the temperature profile of the sample during irradiation and, from this, estimates thermal conductivity. Such estimates lead to values larger than those now commonly assumed at temperatures greater than 1400°C .

Results and comparison of the values given by the various experimental methods are summarized in Fig. 1.

Fig. 1. Reported Thermal-conductivity Values of Uranium Dioxide

Due to the lack of "effective" thermal-conductivity data at high temperatures, since out-of-pile values cannot be extrapolated to meet conditions of irradiated material and to investigate the discrepancy of thermal-conductivity values above 1400°C , a direct in-pile method to measure the thermal conductivity of UO_2 is proposed.

III. FORMULATION

A. Derivation of Experimental Equations

An experiment has been prepared¹³ to determine the thermal diffusivity of UO_2 in the Transient Reactor Test Facility (TREAT). Two rods of different U^{235} enrichment are bonded end to end. The combined rod is then exposed to a uniform, intense burst of neutron radiation within TREAT, causing a temperature rise in each portion which is approximately proportional to its respective enrichment. The heat within the sample is generated internally by the fission process. By means of two thermocouples embedded within the rod at $\pm L$ from the interface, the temperature difference, ΔT , between the two thermocouple positions can be measured as a function of time. If the value of ΔT at a certain time is known, the thermal diffusivity of the sample may be determined by means of the formula (as derived by Golden¹³),

$$\Delta T = (T_{01} - T_{02}) \operatorname{erf} \frac{L}{2\sqrt{Dt}}. \quad (1)$$

Another method, from the same experiment, for determining the thermal diffusivity of the sample is by measuring the slope, $(\partial \Delta T / \partial t)_L$, of the photographic data trace, which is a record of Δt versus time, at a time, t , and using the formula (also derived by Golden¹³),

$$\left(\frac{\partial \Delta T}{\partial t} \right)_L = -\frac{1}{\sqrt{\pi}} \left(\frac{T_{01} - T_{02}}{t} \right) \frac{L}{2\sqrt{Dt}} e^{-\left(\frac{L}{2\sqrt{Dt}} \right)^2}. \quad (2)$$

In Eqs. (1) and (2),

$T_{01} - T_{02}$ = maximum temperature difference between the two portions of the sample,

ΔT = temperature difference between the two portions of the sample rod at $t > 0$,

t = time ($t = 0$ at time of maximum ΔT),

$\pm L$ = distance of each thermocouple from the interface,

and

D = thermal diffusivity of the sample material.

The following assumptions are made:

- 1) Thermal conductivity varies linearly with temperature over the range of temperatures from T_{01} to T_{02} .*
- 2) Temperature, as a function of time and position, can be described by the one-dimensional, rectangular form of the time-dependent heat equation.
- 3) The rod extends to $\pm\infty$.
- 4) The initial temperature (just following the neutron burst) in each of the two regions is uniform.
- 5) Radial heat loss by radiation is negligible for the anticipated time scale of the experiment (a few seconds).
- 6) The product of density and specific heat (the volumetric heat capacity) is constant.

The experiment in question is similar to that discussed by Stokes¹⁴ dealing with the diffusion of solutions of two concentrations across an initially-sharp boundary between two semi-infinite columns of liquid. Stokes gives the basic differential equation for one-dimensional diffusion with a diffusion coefficient, D , which is a function of concentration, as

$$\frac{\partial c}{\partial t} = \frac{\partial}{\partial x} \left(D \frac{\partial c}{\partial x} \right). \quad (3)$$

If temperature is substituted for concentration, Eq. (3) becomes

$$\frac{\partial T}{\partial t} = \frac{\partial}{\partial x} \left(D \frac{\partial T}{\partial x} \right), \quad (4)$$

which is applicable for the case in point. The solution of the above equation, given by Stokes,¹⁴ is presented in Appendix A.

To obtain an accurate determination of thermal diffusivity by application of the above model, the temperature distribution, within the sample, as a function of time, must be measured precisely. Since such measurements cannot readily be made in the TREAT reactor at high temperatures, this model is not considered further.

As a simplification of the above model, it was next assumed that the thermal conductivity is constant for the range of temperature in a

*Although the temperature variation of UO_2 thermal conductivity is clearly not linear (see Fig. 1), the actual curve may be approximated as a straight line over T_{01} to T_{02} for reasonable values of $T_{01} - T_{02}$, of the order of those used for the experiments.

given experiment. An analytical solution of the heat equation, under this condition, is given in Appendix B. Using this assumption and assumptions 2-6 above, it was found that, for a rod of infinite length,

$$T_1 = T_{01} - \frac{1}{2}(T_{01} - T_{02}) \operatorname{erfc} \frac{|x|}{2\sqrt{Dt}}, \quad (5)$$

and

$$T_2 = T_{02} - \frac{1}{2}(T_{01} - T_{02}) \operatorname{erfc} \frac{|x|}{2\sqrt{Dt}}. \quad (6)$$

Subtracting the above equations resulted in

$$\Delta T = T_1 - T_2 = (T_{01} - T_{02}) - (T_{01} - T_{02}) \operatorname{erfc} \frac{L}{2\sqrt{Dt}}$$

or

$$\Delta T = (T_{01} - T_{02}) \operatorname{erf} \frac{L}{2\sqrt{Dt}}, \quad (7)$$

yielding the basic equation for determining D by measuring ΔT at a given time, t .

Two thermocouples are placed at a distance $\pm L$ from the interface, connected in series, to measure the rate of change of temperature difference with respect to time. If Eq. (7) is differentiated with respect to time, the result is

$$\left(\frac{\partial \Delta T}{\partial t} \right)_L = (T_{01} - T_{02}) \frac{2}{\sqrt{\pi}} \frac{\alpha}{\alpha t} \int_0^{\frac{L}{2\sqrt{Dt}}} e^{-\lambda^2} d\lambda,$$

or

$$\left(\frac{\partial \Delta T}{\partial t} \right)_L = -\frac{1}{\sqrt{\pi}} \left(\frac{T_{01} - T_{02}}{t} \right) \left(\frac{L}{2\sqrt{Dt}} \right) e^{-\left(\frac{L}{2\sqrt{Dt}} \right)^2} \quad (8)$$

Define

$$\alpha = \frac{L}{2\sqrt{Dt}}.$$

Then

$$\left(\frac{\partial \Delta T}{\partial t}\right)_{L, \tau} = -\frac{1}{\sqrt{\pi}} \left(\frac{T_{01} - T_{02}}{\tau}\right) \alpha e^{-\alpha^2}, \quad (9)$$

which is the equation used to determine thermal diffusivity by measuring the slope of the photographic data trace at a given time, τ .

B. Feasibility of Experiment

The validity of Eqs. (7) and (9) will be established if the validity of the preceding assumptions can be proven.

Assumption 1 has been removed (see Appendix B).

Assumptions 2 and 4 are based upon the conclusion that the initial axial neutron distribution within each portion of the sample is constant. Since the internal heat is produced by the fission process that is initiated by neutron absorption, the temperature distribution will be uniform if the neutron distribution is uniform. Uniform heat generation will permit temperature as a function of time and (axial) position to be described by the one-dimensional rectangular form of the time-dependent heat equation.

Paine and Taraba¹⁵ derived the equation

$$\phi(Z, \tau) = A(\tau) e^{-[B(\tau)Z]} + C(\tau) \quad (10)$$

from experimental data which give the longitudinal distribution of thermal neutron flux in a cylindrical fuel specimen during irradiation, where

$\phi(Z, \tau)$ = the average cross-sectional flux at distance z
from the end of the cylinder,

$A(\tau), B(\tau), C(\tau)$ = constants varying with sample radius and
enrichment,¹⁵

τ is defined as $= a[0.91a(\Sigma_{ab} + \Sigma_s)]$,

and

a = radius of the sample.

Normalizing Eq. (10) yields

$$\frac{\phi}{C} = 1 + 0.278e^{-BZ}. \quad (11)$$

The calculated values of ϕ/C for various values of Z in a 10%-enriched UO_2 sample for $a = 0.64$ cm for which $B = 1.79 \text{ cm}^{-1}$ are tabulated in Table I.

TABLE I. Axial Flux Distribution

Z, cm	Normalized Z to End of Sample			ϕ/C , Normalized to End of Sample		
	BZ	e^{BZ}	$0.278e^{-BZ}$	ϕ/C		
0	0	1.00	0.278	1.278		1.00
0.2	0.1	1.43	1.194	1.194		0.935
0.4	0.2	2.05	0.136	1.136		0.891
0.8	0.8	4.18	0.067	1.067		0.836
1.0	0.5	5.98	0.046	1.046		0.820
1.2	0.6	8.58	0.032	1.032		0.813
1.4	0.7	12.30	0.023	1.023		0.804
1.6	0.8	17.46	0.016	1.016		0.796
1.8	0.9	25.03	0.011	1.011		0.793
2.0	1.0	35.87	0.008	1.008		0.789

The normalized values of ϕ/C in Table I indicate that the axial flux distribution within the UO_2 sample can be considered constant over the thermocouple region, thereby validating assumptions 2 and 4.

As a test of the validity of the third original assumption, the temperature difference, ΔT , is computed, both for a rod of infinite extent and for one whose length is 6 cm.

For the numerical calculations, the variables involved are given values that are considered to be typical for the experiment involved.

Let

$$T_{01} = 2300^\circ\text{K},$$

$$T_{02} = 2100^\circ\text{K},$$

$$\rho C_p = 0.86 \text{ cal/cm}^3\text{-}^\circ\text{K},$$

$$k = 0.015 \text{ cal/sec-cm-}^\circ\text{K},$$

and

$$D = 0.0174 \text{ cm}^2/\text{sec}.$$

Take

$$L = 1 \text{ cm},$$

and

$$\tau = 5 \text{ sec.}$$

Then

$$\alpha = \frac{L}{2\sqrt{Dt}} = \frac{1}{2\sqrt{0.0174(5)}} = 1.695.$$

The temperature difference, ΔT , for a rod of infinite length, is

$$\begin{aligned} \Delta T &= (T_{01} - T_{02}) \operatorname{erf} \frac{L}{2\sqrt{D\tau}} = 200^\circ\text{K}(0.98347) \\ &= 196.7^\circ\text{K}. \end{aligned}$$

For the rod whose length is 6 cm,

$$\begin{aligned} \Delta T &= (T_{01} - T_{02}) \left(\operatorname{erf} \frac{L}{2\sqrt{D\tau}} - \operatorname{erfc} \frac{2a - L}{2\sqrt{D\tau}} + \operatorname{erf} \frac{2a + L}{2\sqrt{D\tau}} + \operatorname{erfc} \frac{4a - L}{2\sqrt{D\tau}} \right) \\ &= 200^\circ\text{K}(0.98347 - \text{negligible}) = 196.7^\circ\text{K}. \end{aligned}$$

Thus, the validity of the third assumption is established.

To confirm the fifth assumption, the heat loss due to thermal radiation was calculated by means of the following model. The following conditions were used to determine the case of greatest heat loss:

- 1) All temperature gradients in the system were neglected.
- 2) The temperature of the sample was assumed to be T_{01} .
- 3) The emissivity of the surface of the sample was set equal to one.
- 4) The sample radiates to the interior of the TREAT transparent test capsule, which is composed of polished Zircaloy, having an emissivity of 0.08.
- 5) The ratio of the sample surface area to the effective enclosure surface area is 0.167.

Then if the sample is a solid cylinder of diameter, d , the instantaneous heat transfer rate per unit length at time zero is given by Jakob and Hawkins¹⁶ as

$$\frac{q}{\ell} \approx \sigma \pi d \frac{T_{01}^4}{1 + 0.167 \left(\frac{1}{0.08} \right) - 1} = \frac{\pi d^2}{4} \rho C_P \frac{dT_1}{dt},$$

from which

$$\frac{dT_1}{dt} \approx \frac{1.37 \sigma T_{01}^4}{\rho C_P d}$$

If $d = 1$ cm, and $T_{01} = 1900^\circ\text{K}$, which is a typical experimental temperature, then

$$\frac{dT_1}{dt} \approx 28^\circ\text{K/sec.}$$

Thus, at the end of 5 sec,

$$\Delta T_1 \approx 140^\circ\text{K},$$

which represents the maximum amount of heat that will be lost from the sample due to radiation under isothermal conditions.

In the actual experiment, several conditions exist that will appreciably lower the above radiation heat loss.

In the above calculations, the emissivity of uranium dioxide was given the maximum value of one. Under actual conditions, the emissivity will be less than one, which will tend to decrease the amount of heat lost from the sample because of radiation.

The calculated values of ΔT represent the amount of heat lost from the outside of the sample, neglecting all internal temperature gradients.

Calculations were made by means of the RE-147 computer code, as described by Heestand *et al.*,¹⁷ to obtain an accurate internal temperature distribution under nonisothermal conditions. From these calculations, it was concluded that radial radiant heat loss will have negligible effect upon the axial temperature of the sample because of the poor thermal conductivity of UO_2 , and that under nonisothermal conditions the axial temperature is not affected by radiant heat loss for as long as 6 sec.

Since the thermocouples are located on the axis of the sample and responsive to the axial flow of heat, any effect that radiant heat loss will have upon the experimental thermal diffusivity is negligible.

If experience proves the fifth original assumption invalid, it may be necessary to build a thermal radiation shield around the sample. Such a shield might be a thin shell of UO_2 , enriched to match closely the initial temperature distribution in the sample.

C. Description of Experimental Oscillograph Traces

The data to be analyzed from the given experiment will return from the National Reactor Testing Station at Idaho Falls, Idaho, in the form of a photographic oscillograph trace similar in shape to Fig. 2. The trace will be a measure of the temperature difference between the portions of different enrichments in the sample as a function of time. The given formula with which to compute the thermal diffusivity of a sample employs either a value of ΔT or of the slope at some point of time, t .

Fig. 2. Sample Oscillograph Trace

IV. DESCRIPTION OF EXPERIMENTAL APPARATUS

A uranium dioxide sample was fabricated by J. Handwerk, Argonne National Laboratory, with two enrichments of U^{235} . The sample consisted of two nominally 1/2-in.-dia cylinders of 11 and 10 w/o enrichments fused together into a single cylinder, having two separate enrichments with an interface. The fusing was accomplished by firing the green unfired extrusions of individual enrichments while they were held in close contact.

A 0.125-cm-dia hole was drilled in the fired sample from each end along its centerline, leaving 0.3125 cm of material between the bottom of

Fig. 3. Schematic Diagram of Sample

each hole and the interface. These holes permitted sheathed thermocouples to be inserted within the sample to measure the axial heat flow. Figure 3 is a schematic diagram of the sample showing the relative position of the holes to the interface.

The miniature, high-temperature thermocouples were tantalum-sheathed, thoria-insulated, tungsten 5% rhenium versus tungsten 26% rhenium. This type of thermocouple is capable of response times of the order of 0.1 sec and can withstand temperatures in excess of 2300°C.

The experimental sample was supported within a refractory metal framework (Fig. 4) by spring-loading the sheathed thermocouples into the axial holes. This mounting served the dual purpose of maintaining thermocouple contact during the transient and providing secondary shock mounting to the sample for shipment from Argonne National Laboratory to the TREAT facility at the National Reactor Testing Site in Idaho.

The sample, mounted in the refractory frame, was housed in a "transparent capsule" (Fig. 5),¹⁸ which provided a heat sink and particulate shield, and shipped to Idaho where it was sealed within the "transparent slot liner" (Fig. 6),¹⁸ which in turn was inserted in the TREAT reactor for transient experimentation (Fig. 7).^{19,20} The slot liner is a gas-tight encapsulation providing monitoring of released gaseous and airborne products while allowing motion-picture studies of the sample during a reactor transient.

Fig. 4. Experimental L-frame Support to Be Inserted in the Transparent Slot Liner within TREAT

Fig. 5. Transparent Meltdown Facility

Fig. 6. Inner and Outer Capsule Subassemblies

Fig. 7. Cutaway Isometric of TREAT

V. PREPARATION OF DATA FOR ANALYSIS

A. Graphing Experimental Data from Oscillograph Chart

The experimental data will return from the TREAT reactor in the form of a photographic oscillograph chart similar to the one in Fig. 2.

The course of the experimental transient is followed by recording power and temperature data on the direct-writing-type oscillograph with light-beam galvanometers. These recorded traces, in units of current versus time, must be manually calibrated and graphed in units of temperature or reactor power versus time. The resulting graphs of sample temperature and reactor power versus time will be similar to those shown in Figs. 8, 9, 10, and 11.

B. Determination of Thermal Diffusivity

The graphs of temperature and reactor power versus time are now used to determine the value of the thermal diffusivity of the sample by the following graphical approximation according to the formula,

$$\Delta T = (T_{01} - T_{02}) \operatorname{erf} \frac{L}{2\sqrt{Dt}}, \quad (12)$$

Fig. 8. Experimental Temperature and Power versus Time, Transient No. 539

Fig. 9. Experimental Temperature and Power versus Time, Transient No. 535

Fig. 10. Experimental Temperature and Power versus Time, Transient No. 536

Fig. 11. Experimental Temperature and Power versus Time, Transient No. 537

by the following procedure:

- 1) Using Eq. (12), plot

$$\frac{\Delta T}{T_{01} - T_{02}} = \operatorname{erf} \frac{L}{2\sqrt{Dt}}, \quad (13)$$

giving L the value of the L used in the given experiment, specifying several approximate values of D for the temperatures encountered in the given experiment, and varying time, t .

- 2) By inspecting the experimental graphs of ΔT versus t , for each transient, choose some time that is within the range of maximum, experimental, temperature difference. Call the theoretical value of $\Delta T / (T_{01} - T_{02})$ at this time $\Delta T_1 / (T_{01} - T_{02})$, and plot the ratios of

$$\left(\frac{\Delta T_1}{T_{01} - T_{02}} \right) / \left(\frac{\Delta T_n}{T_{01} - T_{02}} \right)$$

for successive times of the curves obtained in part 1 for each transient. Such curves represent $\Delta T_1 / \Delta T_n$ for various values of D for the given value of L . These curves will be similar to traces A, B, C, and D, shown later in Figs. 20, 21, and 22. By plotting the ratio, $\Delta T_1 / \Delta T_n$, the curves will be independent of the value of $T_{01} - T_{02}$.

3) Normalize the experimental curve of ΔT versus t for each transient to the same value of t chosen for that transient in part 2. Plot the experimental ratios of $\Delta T(t)/\Delta T(n)$; where

$\Delta T(t)$ = the value of ΔT at the time of normalization,

$\Delta T(n)$ = value of ΔT at successive times,

superimposed on the theoretical curves plotted in part 2.

4) By comparing the experimental curves obtained in part 3 to the theoretical curves obtained in part 2, approximate the value of D for a given transient, as is done in Figs. 20, 21, and 22.

VI. RESULTS AND CONCLUSIONS

The experimental transients that have been analyzed are listed in Table II.

TABLE II. Experimental Transients

Transient	L, cm	Enrichments	Diameter, in.	Maximum Temperature, °C
539	0.3125	10 versus 11%	1/2	1450
535	0.3125	10 versus 11%	1/2	1650
536	0.3125	10 versus 11%	1/2	1850
537	0.3125	10 versus 11%	1/2	2050

The values of the maximum temperatures attained by the various transients given in Table II were the average temperatures registered by the sample's thermocouples.

Temperature and power input data as taken from the transient oscillograph charts, are graphed in the following figures:

<u>Transient</u>	<u>Fig. No.</u>
539	8
535	9
536	10
537	11

Zero time for these graphs was taken as the starting time of the TREAT control-rod removal. The time for control-rod removal will vary for each reactor transient. To give a uniform time basis of comparison, the experimental zero time was taken as the time of peak power as indicated by the

oscillograph chart record (see Fig. 2). The time correction was accomplished by subtracting the period of time between the oscillograph zero time and the time of peak power from all experimental temperature data.

Graphs of the time-corrected transients are given in the following figures:

<u>Transient</u>	<u>Fig. No.</u>
539	12
535	13
536	14
537	15

For those transients in which the thermocouple traces cross, as in Figs. 12, 13, and 15, a temperature correction is necessary. This correction consists of adding the absolute measured temperature difference at $t \gg 0$, (approximately 35 sec) to the measured ΔT existing at smaller values of time, $0 < t \lesssim 20$ sec.

Fig. 12. Experimental Temperature versus Time, Transient No. 539, Time Zero = Time of Peak Reactor Power

Fig. 13. Experimental Temperature versus Time, Transient No. 535, Time Zero = Time of Peak Reactor Power

Fig. 14. Experimental Temperature versus Time, Transient No. 536, Time Zero = Time of Peak Reactor Power

Fig. 15. Experimental Temperature versus Time, Transient No. 537, Time Zero = Time of Peak Reactor Power

The values of corrected ΔT versus t for each transient are illustrated in the following figures:

<u>Transient</u>	<u>Fig. No.</u>
539	16
535	17
536	18
537	19

To facilitate comparison of experimental to theoretical data and to make the experimental data independent of $T_{01} - T_{02}$, the values of ΔT versus t for each transient are normalized. The time of normalization is arbitrary, but for ease of comparison, the time was chosen to be approximately the time of the maximum ΔT for each transient.

By comparing the experimental normalized curves to the theoretical curves of ΔT versus t , normalized to the same time, we can approximate the value of D for the conditions of each transient. A tabulation of the comparison of experimental to theoretical data plus the approximated values of D is given in Table III.

Fig. 16. Experimental Temperature Difference versus Time, Transient No. 539

Fig. 17. Experimental Temperature Difference versus Time, Transient No. 535

Fig. 18. Experimental Temperature Difference versus Time, Transient No. 536

Fig. 19. Experimental Temperature Difference versus Time, Transient No. 537

TABLE III. Approximate Values of Diffusivity, D

Transient	Comparison Shown in:	Approximate D
539	Fig. 20	0.007
535	Fig. 21	0.010
536	Fig. 22	0.025
537	Fig. 22	Indeterminate

Using the measured values of thermal diffusivity and the relationship $D = k/\rho C_p$, the thermal conductivity of UO_2 was calculated as a function of temperature. The volume of the sample was 5.0443 cm^3 and its weight was 47.141 g , which yielded a density of 9.345 g/cm^3 . This density is 85.6% of the theoretical density of UO_2 .

The specific heat, C_p , of UO_2 varies with temperature according to the relationship²¹

$$C_p(\text{cal/mole-}^\circ\text{C}) = 18.45 + 2.431 \times 10^{-3}T - 2.272 \times 10^{-5}T^2.$$

Fig. 20. Comparison of Normalized Experimental to Normalized Theoretical Temperature Difference versus Time for Various Values of Diffusivity, D, Transient No. 539

Fig. 21. Comparison of Normalized Experimental to Normalized Theoretical Temperature Difference versus Time for Various Values of Diffusivity, D , Transient No. 535

Fig. 22. Comparison of Normalized Experimental to Normalized Theoretical Temperature Difference versus Time for Various Values of Diffusivity, D , Transients No. 536 and 537.

At the average maximum temperature in the transients analyzed, the specific heat of UO_2 was found to be as follows:

Transient	Average Maximum Temperature, °C	C_p , cal/g-°C
539	1450	0.0815
535	1650	0.0830
536	1850	0.0845

The values of the thermal conductivity of UO_2 derived from the data of Table III are compared with existing measurements in Fig. 23. Also included in Fig. 23 are these values, corrected to theoretical density, assuming conductivity to be approximately proportional to density over the range of densities concerned.

Fig. 23. Reported and Experimental Thermal-conductivity Values of Uranium Dioxide

The thermal-diffusivity values obtained from transients 539 and 535 agree favorably with existing data, but the value obtained from transient 536 at 1850°C appears to be excessively high. The upward trend of thermal conductivity exhibited by the experimental data is in agreement with the prediction made by Bates²² that the thermal conductivity of UO_2 increases appreciably at high temperatures.

It can be shown that the optimum time range for determining D by means of ΔT at various times is $0.185L^2/D$ to $0.350L^2/D$, with the ideal time at $0.255L^2/D$. Within this range, the probable error in calculating the thermal diffusivity of the sample is 5% or less. At times other than those within the given range, the probable error in the determination of D promptly increases to ∞ .

Outside the given range, the value of $\Delta T_t/\Delta T(n)$ increases to infinity, indicating the influence of experimental errors. At small values of time, the thermocouple "lag" and the finite time of power input are the major sources of error.

Even though the transients have been "time-corrected" to the peak-power-input time, heat will continue to be generated for a short time. This upward "coasting" of temperature produces the rise to infinity at short periods of time.

At larger values of time, radial heat loss from the sample is the main source of error. Since radiant heat loss is proportional to the fourth power of the sample temperature, this error increases at elevated temperatures. This effect causes the normalized experimental trace of ΔT versus t to

increase to infinity at lower values of time as the transient maximum temperature is increased. Hence the optimum range for approximating thermal-diffusivity values decreases until the normalized trace of ΔT versus t becomes an acute parabola. This occurred in the case of transient 537, making it impossible to approximate the value of the sample's thermal diffusivity at 2050°C .

Fig. 24. Initial Cracking of Sample at the End of Transient No. 535

Motion pictures of the sample transients show that cracking occurred near the end of transient 535. Prints of the motion pictures were made at the time of initial cracking and are shown in Fig. 24. The cracks are visible as lines near the bottom of the sample. These cracks had little effect upon the thermocouple readings since they are well below the bottom thermocouple location.

Further cracking, as illustrated in Fig. 25, occurred near the end of transient 536. As can be seen, these cracks are quite near the bottom-thermocouple location.

Fig. 25. Extensive Cracking as Occurred during Transient No. 536

Even though these cracks do not become visible until the end of transient 536, they no doubt occurred internally early enough during the transient to be responsible for the high value of D at 1850°C . The cracking became more severe near the end of transient 536 and is the cause for the lack of "effective" data in transient 537.

VII. FURTHER EXPERIMENTATION

A. Suggestions

The feasibility of this experiment has been demonstrated by the work presented herein. However, this experiment should be performed on other samples of UO_2 to obtain an accurate measurement of the thermal diffusivity of UO_2 over a wider range of temperatures. For this reason, the following recommendations are made.

1. For values of D at lower temperatures, perform the experiment using a sample of the same dimensions with lower enrichments but with a larger difference of enrichments, i.e., 3% versus 5%.
2. To obtain values of D at higher temperatures, use the same experimental sample as in this report, with the addition of thermal shielding to decrease the effect of radial heat loss.

When the given experiment is applied to other ceramic materials, it may be necessary to introduce refinements that are dependent upon the properties of the material to be studied.

If the material to be studied possesses a large value of thermal diffusivity, it may be desirable to increase the sample's radius and value of L . Since volume is proportional to r^2 and area is proportional to r , an increase in the sample's radius will result in less heat loss per unit volume. Hence,

there will be less radiation from the sample, which will, in turn, decrease experimental errors. An increase in the sample's radius will also give greater leeway in the radial thermocouple position.

By increasing the value of L , better "time resolution" of temperature measurements may be attained. It will take longer for temperature changes to be felt by the thermocouples, which will, in turn, decrease the errors caused by the time "lag" of the thermocouples.

Multidimensional, nodal, heat-transfer calculations, performed on a high-speed computer, would make it possible to mock-up a real experiment much more closely than the analytical model, and would make it possible to obtain data from experiments for which the analytical analysis could not be used because of such phenomena as thermocouple lag.

B. Limitations

Limitations of the technique should be reviewed briefly. Experimental conditions can preclude extraction of valid values of D , because of such effects as thermocouple lag, heat flow to the center from sample regions of highest thermal-neutron flux, heat flow from the center due to heat loss from the surface, finite duration of heating pulse, temperature differences small compared with experimental error or uncertainty, and sample cracking. Although this technique does not appear to be one of high precision, it (or variations on it) may prove useful in obtaining data of engineering accuracy on materials that might be difficult to study otherwise, under conditions not readily achieved with more conventional techniques.

APPENDIX A

Heat Conduction When D Is a Linear Function of Temperature

Consider the solution of the heat equation,

$$\frac{\partial}{\partial X} \left[D \frac{\partial T}{\partial X} \right] = \frac{\partial T}{\partial t},$$

with the initial conditions,

$$T(X, t) = T_{01}, \quad X < 0, \quad t = 0;$$

$$T(X, t) = T_{02}, \quad X > 0, \quad t = 0;$$

and boundary conditions,

$$\lim_{X \rightarrow -\infty} T(X, t) = T_{01}, \quad \lim_{X \rightarrow \infty} T(X, t) = T_{02}.$$

This problem is treated by Stokes¹⁴ as follows: Let

$$Y = X(t)^{-1/2}.$$

With this substitution, the heat equation becomes

$$-2 \frac{\partial}{\partial Y} \left[D \frac{\partial T}{\partial Y} \right] = Y \frac{\partial T}{\partial Y}$$

with the transformed boundary conditions,

$$\lim_{Y \rightarrow -\infty} T(Y) = T_{01}, \quad \lim_{Y \rightarrow \infty} T(Y) = T_{02}.$$

Take the linear variation of D with T to be

$$D = \bar{D} \left[1 - \frac{a}{2}(T_{01} + T_{02}) + aT \right],$$

where for

$$T = \frac{T_{01} + T_{02}}{2}, \quad D = \bar{D}.$$

Let

$$\nu = \left[\frac{1 - \frac{a}{2}(T_{01} + T_{02}) + aT}{1 - \frac{a}{2}(T_{01} - T_{02})} \right]^2;$$

and

$$W = \left[1 - \frac{a}{2}(T_{01} - T_{02}) \right]^{-1/2} Y / 2\sqrt{D}.$$

Then the heat equation transforms to

$$\frac{d^2 \nu}{dW^2} = - \frac{2W}{\sqrt{\nu}} \frac{d\nu}{dW},$$

with the boundary conditions,

$$W \rightarrow +\infty, \quad \nu \rightarrow 1;$$

$$W \rightarrow -\infty, \quad \nu \rightarrow \left[1 + \frac{a}{2}(T_{01} - T_{02}) \right]^2 / \left[1 - \frac{a}{2}(T_{01} - T_{02}) \right]^2 \equiv 1/b^2.$$

The results of numerical integration of the above second-order differential equation appear in the literature; these are presented in terms of the independent variable,

$$Z = W \left(\frac{2}{1+b} \right)^{1/2} = \frac{X}{2\sqrt{Dt}}.$$

If the average value of the thermal diffusivity is kept constant, but the ratio of the diffusivities in the two regions at time zero is varied, the temperature does not vary greatly with $X/2\sqrt{Dt}$. Because of this relative insensitivity, a TREAT experiment based upon the linear thermal diffusivity model does not appear to be warranted.

APPENDIX B

Analytical Solution of One-dimensional Rectangular Constant-diffusivity Heat Equation for Rod of Finite Length

Consider the physical model of Fig. 26 and its associated equations:

$$\begin{array}{cc} \underline{-a < X < 0} & \underline{0 < X < a} \\ \frac{\partial T_1}{\partial t} = D \frac{\partial^2 T_1}{\partial X^2} & \frac{\partial T_2}{\partial t} = D \frac{\partial^2 T_2}{\partial X^2} \end{array}$$

$$T_1(X, 0) = T_{01} \quad T_2(X, 0) = T_{02}$$

$$\frac{\partial T_1(-a, t)}{\partial X} = 0 \quad \frac{\partial T_2(a, t)}{\partial X} = 0$$

$$T_1(0, t) = T_2(0, t)$$

$$\frac{\partial T_1(0, t)}{\partial X} = \frac{\partial T_2(0, t)}{\partial X}$$

Fig. 26. Physical Model

This is the classical-type problem that is readily solved by means of the Laplace transform. Define

$$L\{T_n(X, t)\} = \int_0^\infty e^{-Pt} T_n(X, t) dt = \theta_n(X, P), \quad n = 1, 2.$$

Then

$$P\theta_1 - T_{01} = D \frac{d^2 \theta_1}{dX^2};$$

$$\theta_1 = A_1 e^{-\sqrt{\frac{P}{D}} X} + B_1 e^{\sqrt{\frac{P}{D}} X} + \frac{T_{01}}{P}.$$

From the end-condition in this region,

$$A_1 = B_1 e^{-2\sqrt{\frac{P}{D}}a},$$

$$\theta_1 = B_1 \left[e^{\sqrt{\frac{P}{D}}X} + e^{-\sqrt{\frac{P}{D}}(2a+X)} \right] + \frac{T_{01}}{P},$$

and

$$\frac{\partial \theta_1}{\partial X} = B_1 \sqrt{\frac{P}{D}} \left[e^{\sqrt{\frac{P}{D}}X} - e^{-\sqrt{\frac{P}{D}}(2a+X)} \right].$$

Similarly,

$$\theta_2 = A_2 \left[e^{-\sqrt{\frac{P}{D}}X} + e^{-\sqrt{\frac{P}{D}}(2a-X)} \right] + \frac{T_{02}}{P},$$

and

$$\frac{\partial \theta_2}{\partial X} = A_2 \sqrt{\frac{P}{D}} \left[-e^{-\sqrt{\frac{P}{D}}X} + e^{-\sqrt{\frac{P}{D}}(2a-X)} \right].$$

Employing the coupling conditions, it is found that

$$A_2 = -B_1 = \frac{1}{2}(T_{01} - T_{02}) \frac{1}{P} \sum_{n=0}^{\infty} (-1)^n e^{-2na} \sqrt{\frac{P}{D}}.$$

Thus,

$$\theta_1 = \frac{T_{01}}{P} - \frac{1}{2}(T_{01} - T_{02}) \sum_{n=0}^{\infty} (-1)^n \left[\frac{1}{P} e^{-\sqrt{\frac{P}{D}}(2na-X)} + \frac{1}{P} e^{-\sqrt{\frac{P}{D}}[2a(n+1)+X]} \right],$$

and

$$T_1 = T_{01} - \frac{1}{2}(T_{01} - T_{02}) \sum_{n=0}^{\infty} (-1)^n \left[\operatorname{erfc} \frac{2na - X}{2\sqrt{Dt}} + \operatorname{erfc} \frac{2a(n+1) + X}{2\sqrt{Dt}} \right].$$

Similarly,

$$\theta_2 = \frac{T_{02}}{P} + \frac{1}{2}(T_{01} - T_{02}) \sum_{n=0}^{\infty} (-1)^n \left[\frac{1}{P} e^{-\sqrt{\frac{P}{D}}(2na+X)} + \frac{1}{P} e^{-\sqrt{\frac{P}{D}}[2a(n+1)-X]} \right],$$

and

$$T_2 = T_{02} + \frac{1}{2}(T_{01} - T_{02}) \sum_{n=0}^{\infty} (-1)^n \left[\operatorname{erfc} \frac{2na + X}{2\sqrt{Dt}} + \operatorname{erfc} \frac{2a(n+1) - X}{2\sqrt{Dt}} \right].$$

Consider the expansion of T_1 :

$$T_1 = T_{01} - \frac{1}{2}(T_{01} - T_{02}) \left\{ \operatorname{erfc} \frac{|X|}{2\sqrt{Dt}} + \operatorname{erfc} \frac{2a - |X|}{2\sqrt{Dt}} - \operatorname{erfc} \frac{2a + |X|}{2\sqrt{Dt}} - \operatorname{erfc} \frac{4a - |X|}{2\sqrt{Dt}} + \dots \right\}.$$

As a approaches infinity, all terms in the brackets but the first go to zero; i.e.,

$$T_1 = T_{01} - \frac{1}{2}(T_{01} - T_{02}) \operatorname{erfc} \frac{|X|}{2\sqrt{Dt}},$$

and similarly,

$$T_2 = T_{02} + \frac{1}{2}(T_{01} - T_{02}) \operatorname{erfc} \frac{|X|}{2\sqrt{Dt}}.$$

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