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THERMAL CONDUCTIVITIES OF ^{90}Sr HEAT SOURCE MATERIALS

E. E. Ketchen

R. E. McHenry

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Isotopes Division

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E. E. Ketchen and R. E. McHenry

ABSTRACT

The thermal conductivities of SrO , Sr_2TiO_4 , and SrTiO_3 (of a composition which simulates these compounds prepared from nuclear fission fuels waste) are reported. The thermal conductivities were measured using a comparator apparatus which was altered to evaluate several factors necessary to establish a procedure for the measurement of the thermal conductivities of radioactive heat source fuel. The errors in the reported values are assessed and a procedure for high power density radioactive samples is described.

INTRODUCTION

The thermal conductivities of SrO , Sr_2TiO_4 , and SrTiO_3 have been measured using a modified version of a commercially available apparatus. The apparatus was modified in order to evaluate the effects of several factors which are not subject to rigorous control in thermal conductivity measurements of high power density radioactive materials. Thermal conductivity data of radioactive heat source materials are necessary for the calculation of thermal gradients in heat sources. Although the conductivities of radioactive materials which are suitable as heat sources were not measured in this work and are not reported in the literature, sufficient data and experience have been accumulated to establish a procedure for such materials.

Several ^{90}Sr isotopic power sources have been used in a series of power generators (SNAP-7A, B, C, D, and E) which were used in remote installations.¹ Second generation ^{90}Sr power generators such as SNAP-23 will require an isotopic heat source of 4 in. dia, which delivers 1200 watts of thermal power. An accurate knowledge of the thermal conductivity of these large sources will be required in calculation of thermal gradients and the consequent change in physical properties.

Conventional methods for the thermal conductivity measurements of ceramic materials require large well-instrumented samples. Unavailability

of sufficient materials (e.g., ^{244}Cm) and the difficulty of fabricating instrumented samples render conventional methods infeasible. Also, large thermal gradients in high power density isotopic fuels limit the size of the sample which can be used; for instance, a temperature difference of 50°C exists across a slab of 0.40-in.-thick ^{90}SrO at 800°C if all the heat flows from one side of the slab.

The thermal conductivities reported for SrO , Sr_2TiO_4 , and SrTiO_3 were measured in a thermal comparator apparatus which was modified to measure 1/2-in.-high by 1-in.-dia disk-shaped samples without thermocouples. The data obtained using several materials demonstrate that systematic error can be corrected by using empirical calibration constants and that random error is small. Additional modifications are required to adapt the apparatus for the thermal conductivity measurement of high power density radioisotopic samples; the proposed procedure is discussed.

EXPERIMENTAL

Apparatus and Materials

A modified 3M TC-200 thermal conductivity apparatus² was used to measure the thermal conductivity. A schematic diagram of the modified apparatus is shown in Fig. 1. The apparatus is a thermal comparator in which a heat flow (Q) supplied by a heater (a) flows in sequence through a specimen of known thermal conductivity (the standard), a specimen of unknown thermal conductivity (the sample), and another standard to a heat sink (b). The first standard, the sample, and the lower standard are referred to as S_1 , S_2 , and S_3 , respectively. The assembly is referred to as the thermal conductivity stack. The temperature at which the thermal conductivity is measured is controlled by a heater in the heat sink. The heat (Q) flowing through the standards (S_1 and S_3) can be calculated from the equation:

$$Q = \frac{kA\Delta T}{\Delta h}$$

Fig. 1. Cross Section of Thermal Conductivity Apparatus.

where k = thermal conductivity of the standard,

A = cross sectional area of the standard,

ΔT = temperature drop in the standard over a distance Δh .

The temperatures of the lower end of standard S_1 and the upper end of standard S_3 are obtained by extrapolating (using the equation) the temperatures measured at points 10 and 11 to the ends of the standards S_3 and S_1 , respectively.

The thermal conductivity stack was surrounded by a series of individually controlled guard heaters. Bubble zirconia was used as an insulating media between the guard heater wall and the thermal conductivity stack. The guard heater wall was held within $\pm 1^\circ\text{C}$ with respect to the adjacent stack to ensure no appreciable radial heat loss or gain from the thermal conductivity stack.

The Pyroceram 9606 standards were 1-3/4 in. long and 1 in. in dia. Thermocouples 11 and 13 in standard S_1 and 8 and 10 in standard S_3 were 1 in. apart. Thermocouples 10 and 11 were located 1/8 in. from the end surfaces of the standards. The end surfaces of the standards and the sample were polished flat and parallel. A 1-mil platinum sheet was placed between the sample and the standards to reduce the thermal resistance at the interfaces. The sample was $\sim 1/2$ in. high and 1 in. in dia.

Calibration of Apparatus

The apparatus as received from the manufacturer was evaluated using the procedure recommended in the manual² supplied with the apparatus. The apparatus was tested with the standards and the sample of identical material whose thermal conductivities had been precisely measured. Tests were made using thermal conductivity stacks of Pyroceram 9606 and Armco Iron. In both cases, the samples were 1 in. in dia by 1 in. high with thermocouples installed as recommended by the manufacturer. Thirteen measurements were made at temperatures in the range of 65 to 900°C. The maximum deviation from the standard value was 5.3%. The average deviation for the 13 values was 2.3%. No tests were made using standards and samples whose thermal conductivities differ a few percent, since such standards were not available.

The apparatus was modified to measure 1/2-in.-high samples by increasing the length of the standards to 1-3/4 in. (Fig. 1). The samples were uninstrumented. Test runs were made using Pyroceram 9606 samples and Pyroceram 9606 standards whose thermal conductivity is standardized³ and similar to the thermal conductivities of the compounds to be measured. Empirical constants were obtained which were used to adjust the data taken using material of similar but unknown thermal conductivity.

Sample Preparation

A composition typical of material which results from process waste of fission-product fuel was chosen since the purpose of this work was to infer the thermal conductivity of ^{90}Sr heat sources. The composition of the three compounds SrO , Sr_2TiO_4 , and SrTiO_3 are given in Table 1. Samples prepared with these compositions represent the ^{90}Sr sample at the time of preparation (before radiation damage and accumulated decay product, zirconium, occur).

Table 1. Composition of SrO , Sr_2TiO_4 , and SrTiO_3

Source materials	Wt % constituents				
	SrO	CaO	BaO	MgO	TiO ₂
SrO	91.69	4.34	3.46	0.51	-
Sr_2TiO_4	65.56	3.10	2.47	0.37	28.50
SrTiO_3	51.03	2.42	1.92	0.28	44.35

The compounds were prepared⁴ by adding the desired amount of TiO_2 in the form of a fine slurry to the solution containing the strontium and precipitating the alkaline earths as carbonates. The alkaline-earth carbonate- TiO_2 mixture was calcined at 1200°C to decompose the carbonates to the oxides and, at the same time, to react the resulting oxides with the TiO_2 to form the titanates.

Samples of strontium fuel were prepared by hot pressing⁵ at 1200°C (SrO at 1300°C) in a graphite die at 4000 psi for 1 hr. Hot pressed samples of compounds which contain TiO_2 were slightly substoichiometric in oxygen (the oxygen deficiency was not >0.006 mole of oxygen per mole of compound). The samples were oxidized to stoichiometry by heating in air at 1200°C for 24 hr. Substoichiometric samples were black; stoichiometric samples were white. The samples were ground to size, and the top and bottom of the samples were ground flat and parallel before polishing.

The sample specimens were prepared without instrumentation (i.e., no thermocouples were inserted in the sample specimen) in order to simulate the radioactive samples as closely as possible. It is infeasible to instrument radioactive samples.

EXPERIMENTAL RESULTS

Variation in Interface Conductivity

Since samples without instrumentation were used for this work and are planned for future work with radioactive samples, the variation in the conductivity of the interface between the sample and standards and the resultant error were determined. Data were taken in a series of runs in which ΔT and Q were measured across the interface between two pieces of Pyroceram 9606. The temperatures were measured by two thermocouples placed 1/8 in. from the interfaces. The temperatures at the surfaces of the Pyroceram were obtained by extrapolation using known values of thermal conductivity and experimental values of Q and temperatures. The temperature drop across the interface was normalized by dividing by Q . The average $\Delta T/Q$ is shown as a function of the interface temperature in Fig. 2.

Measurement of the Thermal Conductivity

Titanium Dioxide

The thermal conductivity of a hot pressed specimen of 99.9% pure TiO_2^* (98.5% theoretical density) was measured to evaluate the operation

*The TiO_2 used to obtain the data was slightly substoichiometric in oxygen (see Experimental section this report).

Fig. 2. Error Due to Variable Interface Conductivity.

of the modified apparatus. The thermal conductivities obtained are shown in curve 1 of Fig. 3; curve 2 gives the thermal conductivities of 99.5% pure TiO_2 of 98% theoretical density recommended by the Bureau of Standards.⁶ The experimental values have been corrected using the calibration constant obtained using Pyrocera 9606 standards and sample.

Strontium Oxide

The thermal conductivity of a hot pressed SrO pellet of 95.9% theoretical density (4.44 g/cm^3) was measured. The pellet was handled in such a manner as to prevent CO_2 and moisture of the air from reacting with SrO before the measurement. Due to the reactivity of SrO in air, it was not metallographically examined. The thermal conductivity of the pellet is given in Table 2; the thermal conductivity values (Figs. 4 and 5) have been corrected to theoretical density using a simplified form of Loeb's⁷ equation.

Table 2. Thermal Conductivity of SrO

Temp., °C	Thermal conductivity, watts/cm°C	
	Experimental	Corrected ^a
405	0.02298	0.0240
491	0.02257	0.0235
592	0.02155	0.0225
682	0.02036	0.0212
747	0.01956	0.0204

^aTheoretical density.

Strontium Orthotitanate

The thermal conductivity of $\text{Sr}_2\text{TiO}_4^*$ (density, 4.92 g/cm^3) is given in Table 3 and Figs. 4 and 5. A photomicrograph of a typical sample is shown in Fig. 6. The photomicrograph shows what appears to be shallow

*These data were taken using a fully oxidized sample. Other data given in Table 4 show that within the limits of error for this apparatus, no difference was observed between stoichiometric and substoichiometric samples.

Fig. 3. Thermal Conductivity of TiO_2 .

Fig. 4. Correlation of Thermal Conductivity Data for SrO , Sr_2TiO_4 , and SrTiO_3 .

Fig. 5. Thermal Conductivity of SrO , Sr_2TiO_4 , and SrTiO_3 .

Fig. 6. Photomicrograph of Sr_2TiO_4 Sample.

holes or a clear phase. Since the density is so similar to the theoretical density, >99.5%, it was assumed that the slightly darker areas are optically clear phases in the gray matrix material.

Table 3. Thermal Conductivity of Sr_2TiO_4

Temp., °C	Thermal conductivity, watts/cm°C	
	First run	Second run
359	0.03400	-
384	-	0.03195
465	0.03267	-
495	-	0.03063
577	0.03293	-
592	-	0.03001
652	0.03219	-
692	-	0.02937
728	0.03199	-
749	-	0.02982

Strontium Titanate

The thermal conductivity of SrTiO_3 (density, 5.11 g/cm³) is given in Table 4 and Figs. 4 and 5. A photomicrograph of the theoretically dense pellet is shown in Fig. 7.

Table 4. Thermal Conductivity of SrTiO_3

Temp., °C	Thermal conductivity, watts/cm°C	
	First run	Second run ^a
394	0.0415	-
408	-	0.0409
421	-	0.0410
489	-	0.0388

Fig. 7. Photomicrograph of SrTiO₃ Sample.

Table 4. (Continued)

Temp., °C	<u>Thermal conductivity, watts/cm°C</u>	
	First run	Second run ^a
497	-	0.0392
515	0.0383	-
650	0.0367	-
696	-	0.0335
736	0.0347	-
813	0.0341	-
816	-	0.0313
849	-	0.0317
861	-	0.0313

^aUsing slightly substoichiometric sample.

DISCUSSION

Calibration Constant

The calibration constants (defined as the ratio of the measured value to the standard value of the thermal conductivities of Pyrocera^m) obtained varied as much as 15% at a given temperature (Fig. 8). However, the data obtained during a single calibration run fitted a straight line with a maximum deviation of <2%. Several sources of error which could contribute to the variation in the calibration constant can be identified: (1) variation in the particle size of the bubble zirconia, (2) changes in the composition of thermocouples due to contamination, and (3) variance in the interface conductivity. By reproducing as closely as possible the exact setup for an experimental run as for a calibration run, the errors resulting from factors other than the variance in the interface conductivity were held to a small fraction of the error due to the interface. This conclusion is supported by the excellent agreement of data taken with a material using different values for the calibration constants (Fig. 4 and Tables 3 and 4). Also, the data obtained for TiO₂ (Fig. 3) is in good agreement with published values.

Fig. 8. Calibration Constant Using Pyroceram 9606.

Interface Conductivity

The data for 30 values of ΔT are given in Fig. 2. A straight line was fitted to the data by using the method of least mean squares. The estimated variance of the points in Fig. 2 from the line is 0.832°C/watt , from the true line, 0.95°C/watt . Since there are two interfaces in each determination, the total estimated variance for the two interfaces by propagation of errors is $\sqrt{2} \times 0.95^\circ\text{C/watt}$ or 1.3°C/watt . A band which will include 95% of the points is obtained if twice the total variance is added to and subtracted from the mean; this band is $\pm 2.6^\circ\text{C/watt}$. Since the average $\Delta T/\text{watt}$ for the sample is 9°C the error due to the interface will be greater than $\pm 29\%$ for 5% of the thermal conductivity determinations. Fifty percent of the thermal conductivity determinations will have interface conductivity errors of less than $\pm 10\%$.

Thermal Conductivity

The data for the thermal conductivity of SrO , Sr_2TiO_4 , and SrTiO_3 were correlated under the assumption that the predominant mode of conduction in the range investigated is by phonons. Under this assumption, the reciprocal of the thermal conductivity is proportional to the absolute temperature. This function is linear and fits the data well. The function was fitted by least mean squares. The confidence limit (95%) is shown for the curves in Fig. 4. The functions for SrO , Sr_2TiO_4 , and SrTiO_3 were used to calculate the curves for the range of experimental values and for extrapolation outside that range. The thermal conductivity versus temperature curves are shown in Fig. 5. It is believed that the extrapolation is useful for the range shown.

Systematic Error

Thermal conductivity of TiO_2 was determined in order to evaluate the systematic error. Since the TiO_2 data agreed well with the weighted average of 36 sets of data analyzed for the National Bureau of Standards,⁶ it is believed that the systematic error does not greatly exceed the

precision. Systematic error can result when the thermal conductivity of the standard differs significantly from the sample. For this reason, the systematic error should be less for Sr_2TiO_4 than for SrO or SrTiO_3 .

FUTURE WORK

The work described in this report was undertaken for two purposes: (1) to obtain the thermal conductivity of strontium fuels under the condition of no radiation damage or accumulated decay product within the strontium fuel and (2) to develop a procedure for the determination of the thermal conductivity of radioactive fuels. In particular, it was desired to determine the error resulting from thermal conductivity measurements made on small uninstrumented samples. The data described in this report show that the thermal conductivity using 1/2-in.-high by 1-in.-dia uninstrumented disks can be determined with acceptable errors.

The thermal conductivity of radioactive fuel will be measured using essentially the same apparatus with the same type of sample. However, with radioactive samples the method will be absolute rather than comparative. The heat flow, Q , will be supplied by the sample; Q can be accurately determined by calorimetric methods.⁸ The top heater, which is not required, will serve as a guard heater. The standard S_1 (see Fig. 1) will be replaced by a temperature monitor of low thermal conductivity. The top guard will be adjusted to give zero ΔT (and zero heat flow) for the two thermocouples in the temperature monitor. The temperature of the monitor will be the same as the temperature at the top of the sample. The temperature at the bottom of the sample will be determined using the bottom monitor (which will replace the bottom standard). The bottom monitor will also measure the heat flow from the sample and indicate the correctness of the adjustment of the guard heaters. The sample height for several radioactive fuels for a ΔT of 50°C is given in Table 5.

The major source of error in the thermal conductivity measurements is expected to result from the variation of the conductivity of the bottom interface. The error for the interface conductivity of a nonradioactive sample has been discussed previously in the report. The error resulting from the variation of a single interface is expected to be greater for

radioactive samples due to the difficulty of preparing smooth parallel surfaces. However, since only one interface is involved, the errors are expected to be approximately the same as reported for nonradioactive samples. The overall error should be less since an absolute method will be used.

Table 5. Sample Height for ΔT of 50°C

Radioactive fuel	Sample height, in.
^{90}SrO	0.40
$^{90}\text{Sr}_2\text{TiO}_4$	0.55
$^{90}\text{SrTiO}_3$	0.68
$^{170}\text{Tm}_2\text{O}_3$	0.20
$^{244}\text{Cm}_2\text{O}_3$	0.14

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