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THERMAL CONDUCTIVITY OF SOLID MATERIALS

BY

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A THESIS

PRESENTED IN PARTIAL FULFILLMENT OF

THE REQUIREMENTS FOR THE DEGREE

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ABSTRACT

A literature search on the methods of determining the thermal conductivity of solids has been made.

A compilation of the various methods of determining the thermal conductivity of solids presented in the literature is given in the report. In general, the literature is lacking in correlations of thermal conductivity with other properties of solid materials. A review of the correlations found is presented. A summary of data presented in the literature is presented in the text.

A method of determining the thermal conductivity of solid materials without measuring the heat flow through the test specimen is presented. The use of a reference material of known thermal conductivity enables the experimenter to determine the thermal conductivity of the test specimen without measuring any properties of the test material other than its physical dimensions. The use of two samples of test materials of different lengths enables the determination of the thermal conductivity to be corrected for contact resistance between the test and reference samples without determination of the contact resistance. The experimental procedure and the design of the apparatus for the proposed method are presented. The design of the apparatus and method of correction for contact resistance has not been previously reported in the literature.

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PREFACE

Thermal conductivity is one of the elusive properties of solid materials. Determination of the thermal conductivity has been attempted for many years.

The purpose of this thesis is to provide a summary of experimental procedures, apparatus and data that have been presented in the technical literature, and to present a composite of the best features of apparatus used in the past, together with any simplifications, modifications, or new ideas, as a method of determining the thermal conductivity of solid materials. The method includes a detailed apparatus design and experimental procedures.

The thesis is presented in four sections. The first section discusses the thermal conductivity and its relationships to heat flow and properties of solid materials. A review of literature correlations of the thermal conductivity with other properties of solid materials is presented. Included are the Lorenz relationship, a proposal by Thornton⁴⁰ for correlating thermal conductivity, elasticity, density, and the velocity of sound in the material, and the modern theory of electron and lattice thermal conductivities.

The second section is a synopsis of the literature experimental apparatus and procedures, including steady-state linear and radial flow methods, and transient methods based on variable temperatures and periodic temperatures in the sample.

The third section is a summary of literature data on the thermal conductivity and thermal diffusivity of solid materials.

The fourth and final section presents the author's proposed design of apparatus and experimental procedure for determining the thermal conductivity of solid materials. A steady-state longitudinal heat flow apparatus is proposed.

Three designs are presented for use in the temperature ranges -200°C. to 40°C. , 20°C. to 300°C. , and 200°C. to 1000°C. Armco Iron, an extremely uniform composition iron, is used as the reference standard. A novel method of calculating the thermal conductivity to eliminate the effect of contact resistance between reference and test samples is presented. The detailed design of apparatus and the method of calculating the thermal conductivity from experimental data are the original work of the author.

All literature references are written in English. They may be obtained from the Engineering Societies Library in New York City by those who wish to examine a particular procedure or apparatus in greater detail.

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NOMENCLATURE AND ABBREVIATIONS

A	area
AWG	American Wire Gauge
B.P.	boiling point at atmospheric pressure
c_p	specific heat at constant pressure
C°	centigrade
d	sign of differential quantity
D	diameter
e	elasticity of material
E	voltage
f	mean free path of lattice
g	radiation heat loss
h	coefficient of convective heat transfer
I	electrical current
J	Joule conversion factor
k	thermal conductivity
k_e	electron thermal conductivity
k_g	lattice thermal conductivity
K	constant used in thermal conductivity - temperature relationship
l	length
L	Lorenz number = $k/6T$
m	mass of electron
M	slope of line
q, Q	quantity of heat
r	radius
R	molecular gas constant
R_o	electrical resistance
R_x^o	thermal resistance where x = subscript
S	surface area
t	temperature
T	absolute temperature
u	velocity of sound in crystal lattice
U	overall heat transfer coefficient
V	velocity of sound
W	power in watts
x	distance
α	thermal diffusivity = $k/c_p e$
Δ	difference
ϵ	volume fraction of one material in another
θ	time
λ	latent heat of evaporation
π	ratio of circumference of circle to its diameter
ρ	density
δ	electrical resistivity
τ	average time between molecular collisions
ω	period of wave

NOMENCLATURE AND ABBREVIATIONS (Cont'd)

Subscripts

a	apparent
c	continuous
e	contact resistance
i	insulation
m	mean
o	observed
p	plastic
r	reference
t	test
T	true
Σ	sum

SECTION I - THERMAL CONDUCTIVITYDefinition of Thermal Conductivity

Fourier's law is the fundamental differential equation for the flow of heat through a solid by conduction. It is symbolically stated as follows:

$$\frac{dQ}{d\theta} = -k A \frac{dt}{dx}$$

$dQ/d\theta$ = quantity of heat flow per unit time

dt/dx = is the rate of change of temperature with distance in the direction of heat flow

A = the area at right angles to the direction of heat flow

k = proportionality factor dependent upon the material through which the heat is flowing and the temperature of the material.

The proportionality constant k is known as the thermal conductivity of the material.

Use of the Thermal Conductivity

The thermal conductivity must be used for exact design work involving the flow of heat in solid materials. Applications include the design of heat exchangers, design of systems operating at high temperatures such as nuclear reactor cores and missile exhaust systems, insulation of cryogenic storage systems, and all systems which may exchange heat with their surroundings. It is important to determine the thermal conductivity of new materials precisely since their application will in part be dependent upon their thermal behavior. It is not the scope of this paper to justify or indeed attempt to illustrate the multitude of applications of thermal conductivity data. A glance at the bibliography of this report will suffice in showing that the research being done, and that which has been done in the past, is only beginning to scratch the surface of the field of measurement of the thermal conductivity of solids. Note that only American and English literature of the period 1900 to 1963 has been evaluated.

Factors Affecting the Thermal Conductivity of Solid Materials

The thermal conductivity of solid materials is a unique intensive property. It is not related to other properties of the material by any simple relationships. Several relationships have been proposed.

The best known is the Lorenz relationship which is as follows:

$$\frac{k}{\sigma} = LT$$

where k is the thermal conductivity, σ is the electrical resistivity, T is the absolute temperature and L is a proportionality factor called the Lorenz number.

However, the factor L is found to vary with materials and even with temperature³⁴ for one material as is shown in Table 1 for Armco Iron.

Another relationship has been proposed by Thornton⁴⁰. This is $k = e \rho = v^2 \rho$, where e is the elasticity of the material, ρ is the density and v is the velocity of sound in the material. Data are presented in the reference paper which appear to prove this theory for non-conductive materials. No later papers have been found which mention this relationship.

One basic relationship for the thermal conductivity has become popular in the last twenty years. The availability of cryogenic materials has made possible very low temperature determinations. Basically, it is believed that the thermal conductivity of a solid is equal to the sum of two factors, the free electron conductivity k_e and the lattice conductivity k_g . These are defined as follows:

$$k_e = \frac{\pi^2}{3} n \frac{\mathcal{J} R^2 T}{m} \qquad k_g = 1/3 \rho C_p f u$$

where n = number of free electrons per unit volume

\mathcal{J} = average time between collisions

R = molecular gas constant T = absolute temperature

m = mass of an electron ρ = density of material

C_p = specific heat of material at constant pressure

f = mean free path of lattice

u = velocity of sound in lattice

This relationship has been proposed by Bidwell⁵ and substantiated at low temperatures by Toxen⁴¹, Garber, Scott and Blatt¹⁰, and White, Woods and Elford.⁴⁶ The latter authors propose and substantiate by measurements that the lattice conductivity is proportional to the square of the absolute temperature below 10°K.

An earlier paper by Bialobjeski³ proposed that the thermal conductivity of metals is due entirely to free electron heat transfer. Bidwell⁵ also proposes that the following relationship holds true:

$$\frac{k}{c_p \ell} = K \frac{1}{T} + K^1$$

where K and K^1 are constants for a particular material.

Data are presented in the paper for lead, tin and zinc. Plots of $1/T$ vs. $k/c_p \ell$ are made which are straight lines. The quantity $k/c_p \ell$ is known as the thermal diffusivity of the material and has units of square length per unit time.

Other less esoteric factors are known to have considerable effects upon the thermal conductivity of a material. These include: 1) the past history of a particular material, e.g., steel will undergo metastable phase changes with temperature. (The thermal conductivities of the phases are different.) 2) impurities in the material, and 3) anisotropy of the material, e.g., the thermal conductivity of graphite has been measured⁴⁴ along the various axes and found to differ.

Thermal conductivities of materials have been measured by many experimental methods and experimenters. Agreement among the data of these experimenters is not always found, even when they use the exact same sample for the determination.^{33, 34}

TABLE ILorenz Number of Armco Iron as a Function of Temperature³⁴

<u>Lorenz Number x 10⁸</u>	<u>Temperature °C.</u>
2.36	-150
2.52	-100
2.62	- 50
2.74	0
2.78	50
2.87	100
2.95	150
3.02	200
3.02	250
3.02	300
3.06	350
3.06	400
3.02	450
3.00	500
3.00	550
3.01	600
3.00	650
3.00	700
2.99	750
2.88	800
2.76	850
2.70	900
2.68	950
2.67	1000

SECTION 2 - MEASUREMENT OF THERMAL CONDUCTIVITY

Introduction

There are many different methods of measuring the thermal conductivity of solids found in the literature. They may be grouped into one or more of the following categories: steady-state methods, transient methods, exact methods, comparative methods, electrical heat generation methods, "quick and dirty" methods, and bar, rod, slab or wire methods. This paper will use the nomenclature of two previous summaries, one by Carslaw and Jaeger⁷ and the other by Ingersoll.¹⁹

Steady-State Linear Flow - Slab Method

This method is used to determine the thermal conductivity of materials having a low value of thermal conductivity, i.e., insulators. A sample is prepared having a large surface width to thickness ratio. The heat flow through the thickness of the sample is measured by calorimetric or electrical means. The heat flow is assumed to be unidirectional because of the low thermal conductivity of the sample.

An apparatus of this type is described by Hager.¹² A thin rectangular stainless steel foil is used as a heating source by passing a measured current through it. Slab shaped samples of the material to be tested are placed on both sides of the foil to prevent heat loss to the surroundings. The entire test specimen (samples and foil) are encased in plastic and immersed in a constant temperature bath. The temperature difference across the slabs is measured using differential thermocouples. The thermal conductivity of the material is calculated from the one dimensional Fourier heat transfer equation using the current to

determine the heat flow. Equilibration time in the bath is 15 minutes and an accuracy of 3% is claimed.

Steady-State Longitudinal Flow - Bar and Rod Methods

This method is used to determine the thermal conductivity of materials which may be shaped into a rod or bar. The entire sample except for the ends must be encased in an insulating material to prevent heat loss to the surroundings. Heat may be supplied either qualitatively or quantitatively to one end of the sample, i.e., electrically measured or by a source whose input to the sample is unknown. In addition to insulation, guard heaters may be used to supply heat to the insulation to prevent lateral heat transmission to the surroundings. Temperatures on different points of the sample are measured and the physical characteristics of the sample are measured. If heat is supplied qualitatively the heat flow through the sample is determined by using a reference sample or calorimeter. The thermal conductivity of the sample may be calculated from the one dimensional Fourier heat flow equation. This method is the easiest to use experimentally providing that lateral heat loss is eliminated and exact physical measurements and determination of heat flow may be made.

This type of apparatus was one of the first used for experimentally determining the thermal conductivity of solids. Refinements of techniques have been made continuously and are still in progress.

Konno²² and Livermore²³ describe the basic apparatus and use it for determining the thermal conductivities of many materials. Konno's²² apparatus consists of a water bath and furnace as heat sinks, differential thermocouples for measuring the temperature difference across the specimen and copper specimens on either end of

the sample. The heat input is determined by electrical measurements. The thermal conductivity is calculated from $Q = kA \, dt/dx$. Livermore²³ uses a similar apparatus consisting of an electric heater, guard ring, two continuous calorimeters and three thermocouples. Manipulative details, a diagram of the apparatus and actual determinations are presented.

Bidwell^{4,5} uses a similar apparatus and measures the radial temperature drops across the silocel insulation around his sample. The gradient along the specimen was found to change with distance at a constant rate.

"This means that the radial temperature drops across the silocel are constant from top to bottom. This was checked experimentally."⁴

Silverman³⁷ uses this type of apparatus, but includes a bar of known thermal conductivity silver soldered to the test specimen. The heat flow through the specimen is not determined. Measuring the temperature difference between points a known distance apart on the sample of material of known thermal conductivity and the temperature difference between points on the specimen gives a relative value of the specimen's thermal conductivity. The actual value of the sample specimen is determined from that of the known material.

Francl and Kingery⁸ use the same type of comparative apparatus as Silverman but use a silver foil and a compression load on the sample to reduce contact resistance.

Weeks and Siefert⁴⁴ adapted this type of apparatus for introduction of the sample by remote control. Their apparatus is placed in an evacuated chamber to eliminate convection heat loss. A heated radiation shield reduces radiation heat loss from the sample. The remote sample handling enables samples of nuclear

reactor materials to be evaluated while still radioactive.

Garber, Scott and Blatt,¹⁰ Powell and Hichman,³³ Powell et al,³⁴ Steele and Rosi,³⁹ Toxen,⁴¹ and White, Woods and Elford⁴⁶ all describe the use of the longitudinal heat flow method of measuring thermal conductivity. These papers were presented within the last 6 years.

Harman, Cahn and Logan¹³ utilize Peltier heat to maintain a temperature gradient along the specimen. Straight-forward measurements allow the calculation of absolute values of thermoelectric power, thermal conductivity, and electrical resistivity.

Schroder describes an apparatus of this type but simplifies the heat flow and temperature difference measurements immensely. The heat source is a refluxing liquid in contact with a silver foil on one end of the sample. The cold end is in contact with a liquid having a boiling point several degrees below that of the heater liquid. The boiling liquid at the cool end of the specimen is condensed and either weighed or collected in a graduated cylinder. The heat flow through the test specimen is equal to the weight of distillate times its latent heat of evaporation. The temperature difference across the sample is equivalent to the difference in the boiling points of the liquids used, e.g., an illustrative example uses monobromobenzene B.P. 155.6°C. and orthodichlorobenzene B.P. 179°C. Proper guarding of the sample against lateral heat loss is used. Since the properties of the fluids used may be determined exactly this procedure eliminates experimental uncertainty due to temperature measurements and heat flow measurements.

Steady-State Radial Flow - Hollow Cylinders

This method is used for materials similar to those used in steady-state longitudinal rod and bar measurements. The test specimen is prepared in the shape of a hollow cylinder. Heat flow is measured either from the wall to the center or from the center out along the lateral direction. The area through which heat may be lost at the ends is minimized by proper insulation. Convection heat loss is minimized by placing the test specimen in a vacuum chamber. The thermal conductivity is calculated from $Q \ln \frac{r_1}{r_2} = 2 \pi k l (t_2 - t_1)$, where Q is the heat supplied per unit time, r_1 and r_2 are the radii at which t_1 and t_2 are measured, and l is the length of the specimen.

Powell and Hickman³³ and Powell et al³⁴ have used the radial flow method for determination of thermal conductivities of metals and for comparison of thermal conductivities determined by this method with those determined by longitudinal heat flow methods.

Neel and Pears²⁷ determined the thermal conductivity of materials at temperatures to 5000°F. or the destruction point of the sample. High temperature furnaces with helical graphite heaters are used as the heat source. Temperatures are measured by thermocouples and/or pyrometers. The heat flow is measured by a calorimeter with water flowing through the center of the sample. Thermal insulation and guarding of samples is used.

Sehr³⁶ proposes the use of a radial heat flow method for determining the thermal conductivity of catalyst particles embedded in plastic.* The heat input is measured as an electrical quantity.

*The techniques of determining the thermal conductivity of particles is discussed on Page 17 of this report.

Steady-State - Electrical Methods

O'Day²⁸ derives equations for the case of a long thermally insulated bar, with ends kept at the same constant temperature and carrying an alternating current to make the temperature distribution parabolic. This is only possible in materials where the temperature coefficient of electrical conductivity is greater than three times the temperature coefficient of thermal conductivity. The thermal conductivity at the center of the bar is equal to $J I^2 R_o l^2 / 2A t_m$ where J is the Joule conversion factor, I is the current flowing in bar, R_o is the electrical resistance at 0°C ., l is the length from end to center of bar, A is the cross sectional area of bar, and t_m is the mean temperature above the ends.

Bode⁶ derives an exact solution for the one dimensional heat conduction equation for a current carrying wire placed in vacuo. The relationship for thermal conductivity follows:

$$k(t_o) = \left(\frac{X_2 - X_1}{A} \right) t_o \frac{E_o}{2} \frac{dI_o}{dT_o} \frac{\left[1 - \frac{dR/dI}{dR_o/dI_o} \right]^2}{1 - 1 - \left[\frac{(dT_m/dI) I_o}{dT_o/dI_o} \right]^2}$$

where $K(t_o)$ is the thermal conductivity at no electrical load, $X_2 - X_1$ is the distance between measuring points, A is the cross section of the wire at no electrical load, E , R , I and T are voltage, resistance, current and temperature. Subscripts o and m refer to no load and mean values.

The apparatus consists of a wire sample, and electric heaters in a vacuum chamber with water cooled walls to maintain a constant temperature surrounding. The current, voltage and temperature of the system and sample are measured under equilibrium conditions. The experimental procedure is involved and the calculations that follow are tedious.

Other Steady-State Methods

Hoch and Blackburn¹⁴ have solved the heat conduction equation for the case of a finite cylinder in a vacuum heated radially by means of high frequency induction and losing heat only by radiation at the ends. In the steady state the cylindrical surface is at a constant temperature and the flat circular end surfaces exhibit a temperature gradient along the radius. This gradient has been correlated to the thermal conductivity. Experimental procedure and calculation methods are presented. The experimental procedure is discussed further by Vardi and Hoch.^{43, 15}

Loeb²⁴ discusses an envelope type of steady state thermal conductivity apparatus. The use of a spheroidal sample circumvents the end effects in a cylinder of finite length. The suitability of the spheroidal shape is discussed. Heat flow equations are derived showing the effects of the variable distance between any two isotherms on the amount of heat crossing any unit area of an isotherm. It is proven that the amount of heat flowing through a spheroidal isotherm slice perpendicular to the axis of the spheroid is proportional to the width of the slice and independent of the position of the slice with respect to the spheroid. The theorem is applied to find the heat distribution necessary to keep the sample core at a uniform temperature. Experimental apparatus is not described.

Hogan and Sawyer¹⁶ describe an apparatus that incorporates facilities for measuring radial heat loss with the same apparatus used to determine the temperature gradient along the axis of a bar. The test specimen is placed in a furnace after a heater coil has been placed on one end and heater lead wires placed on each end

of the specimen. The specimen is allowed to come to thermal equilibrium with the furnace. The specimen is raised to $\sim 1^\circ\text{C}$. above the furnace temperature by use of a current through the specimen. The temperature profile in the specimen along the axis is measured, as well as the power input necessary to maintain the temperature. The power to the specimen is turned off and the furnace and specimen allowed to re-equilibrate. The heater coil is used to supply heat at one end of the specimen. The temperature gradient along the specimen is measured and a plot of temperature against distance is made. This should be a straight line of slope M . The thermal conductivity k is equal to R/M where $R = \frac{EI}{lJ\pi r^2 \Delta T}$, EI is the power input to specimen necessary to maintain it above furnace temperature, l is the specimen length, r is the specimen radius, and ΔT is the temperature difference between specimen and furnace in the first part of the experimental procedure. Data are presented that were obtained using this apparatus.

Van Dusen⁴² describes a simple method of determining the thermal conductivity of poor conductors in terms of the thermal conductivity of a reference material having a high thermal conductivity. A reference cylinder is cut into two equal halves perpendicular to its axis. A heat supply is placed at one end and a cooling source at the other. The cylindrical halves are contacted and the temperature is measured at distances along the cylinder. The contact surface is moistened and the temperature profile made again. A very thin sample of the test specimen is placed between the halves of the cylinder and the temperature profile is again determined. The thermal resistance of the contact and of the sample is determined from a plot of temperature against distance. A typical test result is given for a brass cylinder as dry contact = 1 cm brass, wet contact = 0.5 mm brass.

Transient Methods - Variable Temperature State

Transient temperature measurements are used to determine the thermal conductivity or the thermal diffusivity of a sample. The thermal diffusivity is equal to the thermal conductivity divided by the product of density and heat capacity at constant pressure.

Frazier⁹ fully develops the theory of this method and describes apparatus and procedure for meeting the conditions of the theory, together with methods for eliminating or minimizing errors. The results of a test on a nickel specimen are given and show a precision of 0.1%.

Beatty, Armstrong and Schoenborn² describe an apparatus utilizing a steam heated Platen press with a sandwich made from a block of copper between two test specimens. The thermal conductivity of the specimens must be much less than that of copper, because it is assumed that the copper block is at a uniform temperature. A procedure for determining the thermal diffusivity of the specimen without using a copper block is also presented. The thermal diffusivity is equal to $-0.932 l^2 M$ where l is the sample thickness and M is the slope of the straight line portion of a plot of $\ln \frac{T_0 - t}{T_0 - t_0}$ against θ , T_0 is the surface temperature of the Platen, t is the temperature of the middle of the sample at time θ , and t_0 is the initial temperature of the specimen.

Hsu¹⁸ proposes the measurement of the temperature variation with time at two stations inside a semi-infinite solid, after one face of the solid has been suddenly raised to a certain high temperature. The temperature-time variation is recorded by means of thermocouples, a string galvanometer and synchronized

photographic equipment. The measurement time is only 30 seconds. The result is estimated to be accurate to 2% for certain metals.

Hsu¹⁷ also proposes another method in which two samples that are cylindrically shaped with cylinders of a material of known conductivity at one end of each sample are heated to temperatures T_1 and T_2 . They are brought into contact quickly and the temperature at the end of one sample is recorded with time. The relationship for determining conductivity is given as a function of the specific heats, densities, temperatures before contact and temperature as a function of time.

Sehr³⁶ proposes a method in which a sample is heated to an initial temperature and then placed in a well-stirred bath. The rise in temperature of the bath as a function of time is proposed as a method of determining the thermal conductivity of the sample.

Powell³¹ has designed a simple comparator which consists of two spheres of small diameter (1/8 to 5/16 inch) in an insulated block. The balls are completely covered except for one surface. One part of one ball is slightly above the plane of insulation while the other ball is below the surface, but has an equal area exposed to the atmosphere. The balls are initially heated to a known temperature. The specimen is brought in contact with the exposed ball, the other ball is allowed to cool by convection. The ball temperatures are measured by thermocouples. By comparing the cooling rate of the sample against that of a known material the thermal conductivity is determined. Factors affecting this apparatus are roughness and physical condition of the sample surface. A detailed analysis of the theoretical basis for the Powell Comparator is given by Ginnings.¹¹

Transient Methods - Periodic Temperatures

An original paper on this method is presented by King.²⁰ A heat wave is impressed in a wire. The temperature time lag at points of the wire are measured. The thermal diffusivity of the sample is calculated from the data as follows:

$$\alpha = \frac{1}{q} \sqrt{\frac{-g + \sqrt{g^2 + \omega^2}}{2}}$$

where α is the thermal diffusivity, g is the rate of heat loss by radiation, ω is the period of the impressed heat wave and q is the quantity of heat input.

An improvement on this method was presented by Starr.³⁸ By using a sinusoidal temperature impression at one end of the wire and determining the decrement of the temperature wave traveling along the specimen, elimination or determination of heat loss is made unnecessary.

A discussion of the concepts of transient state measurements is presented by Kingery and McQuarrie.²¹

A later apparatus and technique has been developed by McIntosh, Hamilton and Sibbitt.²⁵ However, in their own words:

"The necessary apparatus is complex and expensive and the detailed analysis of the data is difficult."²⁵

The accuracy claimed was 6.8%.

An apparatus for use in the temperature range of 30 to 1000°C. is described by Abeles, Cody and Beers.¹ It employs a method in which the attenuation and dispersion of a thermal wave, propagated through a solid, are measured. The theory underlying this method is presented as well as experimental data.

Parker et al²⁹ describe a method in which the heat pulse is supplied to the sample by a flash lamp. The temperature at the other end of the sample is measured by a thermocouple connected to an oscilloscope. The oscilloscope reading is taken using a Polaroid camera. This procedure is done to try to eliminate experimental errors due to poor measurement of temperature-time relationships and contact resistance between sample and heat source.

Quick and Dirty Methods

There is one method described in the literature in which a spot of a wax having a known melting point is placed on one end of a cylindrical sample. A sample of known thermal conductivity and having the same dimensions as the test specimen is similarly prepared. The samples are simultaneously placed on a hot plate or other heat source. The time that it takes for the wax on each sample to melt is measured. This is relative to the thermal conductivity of the sample.

Weeks and Siefert⁴⁵ used this method to determine the thermal conductivity of synthetic sapphire against a lead standard.

McLaren²⁶ proposes the use of Templestik crayons (color coded for melting point) to enable the determination of thermal conductivity to be made at different temperatures by this method.

Methods for Small Particles

There have been methods proposed for the evaluation of the thermal conductivity of small particles. The particles are imbedded or mixed with another material of known thermal conductivity to give a homogenous sample. The apparent thermal

conductivity of the sample is obtained by one of the standard methods. The thermal conductivity of the particles are then calculated using a relationship between that of the observed and known materials.

Schröder³⁵ recommends the use of the following relationships to determine the thermal conductivity of the test material embedded in plastic:

$$R_t = \frac{R_o R_p}{R_p - R_o} \quad R = \frac{l}{kS} \quad S_t = S_o \left(\frac{\rho_o - \rho_p}{\rho_t - \rho_p} \right)$$

where R is the thermal resistance of the material, l is the length of sample, S is the cross section of sample, k is the thermal conductivity and ρ is the density of the material. Subscripts o refers to observed, t to test material, and p to the plastic.

Sehr³⁶ theoretically derives the following relationships for the test material and an encasing material having thermal conductivities of the same order of magnitude:

$$\frac{k_o - k_c}{2k_o - k_c} = \epsilon \frac{k_t - k_c}{2k_t + k_c}$$

where k is the thermal conductivity and ϵ is the volume fraction of test material in the sample. Subscripts o refer to observed, t to test material and c to the continuous phase.

SECTION III - THERMAL CONDUCTIVITY DATA

A complete listing of thermal conductivity values of materials has recently been published as part of the American Institute of Physics Hand book. The entire section on solids is edited by Powell.^{32A}

Data have been abstracted from other literature and are presented on the following pages.

TABLE 2

THERMAL CONDUCTIVITIES OF VARIOUS MATERIALS

<u>Material</u>	<u>k cal./cm.sec.°C.</u>	<u>α^* cm²/sec.</u>	<u>t°C.</u>	<u>Reference</u>
Alumimum		0.86	25	29
	0.504		18	22
	0.471		273	
	0.425		430	
	0.394		470	
	0.360		605	
Armco Iron		0.17	25	29
	0.749		0	32
	0.608		200	
	0.487		400	
	0.389		600	
	0.300		800	
	0.286		900	
	0.292		1000	
0.308		1200		

<u>Material</u>	<u>k cal./cm.sec.°C.</u>	<u>α^* cm²/sec.</u>	<u>t°C.</u>	<u>Reference</u>
antimony	0.0442		0	22
	0.0386		182	
	0.0456		469	
	0.0575		610	
beryllium oxide	0.435		70	44
bismuth	0.0194		18	22
	0.0170		160	
	0.0177		222	
	0.0177		233	
	0.0183		256	
copper (OFHC alloy)		1.14	25	29
germanium	0.133		27	39
graphite	0.012		25	40
(L axis extrusion)	0.367		70	44
(ll axis extrusion)	0.605		70	44
Hastelloy A	0.0252		50	37
	0.0354		300	
	0.0438		500	
	0.0565		800	
Inconel	0.0266		50	
	0.0326		200	
	0.0405		400	
	0.0484		600	
	0.0563		800	

<u>Material</u>	<u>k cal./cm.sec.°C.</u>	<u>α^* cm²/sec.</u>	<u>t°C.</u>	<u>Reference</u>
lead		0.0011	0	5
(melting point)	0.083		50	8
	0.077		222	22
	0.074		298	
	0.069		326	
magnesium		0.56	25	29
molybdenum	0.161		1927	14
monel	0.0414		50	37
	0.0452		100	
	0.0610		300	
	0.0744		500	
	0.0927		700	
	0.1088		900	
A nickel		0.16	25	29
	0.146		50	37
	0.126		200	
	0.112		300	
	0.107		400	
	0.107		500	
	0.122		600	
	0.141		800	
D nickel	0.103		50	37
	0.084		200	
	0.072		300	
	0.080		400	
	0.096		600	
	0.109		800	

<u>Material</u>	<u>k cal./cm.sec.°C.</u>	<u>α^* cm²/sec.</u>	<u>t°C.</u>	<u>Reference</u>
quartz	0.016		25	40
(optic axis)	0.014		70	44
sapphire	0.071		70	44
	0.067		100	45
silicon	0.299		27	39
silicon carbide	0.489		70	44
silver		1.70	25	29
spinel	0.028		70	44
steel 1020		0.15	25	29
steel 4340		0.091	25	29
stainless steel	0.0294		50	37
302	0.0363		200	
	0.0446		400	
	0.0530		600	
	0.0613		800	
stainless steel	0.0527		50	37
430	0.0541		200	
	0.0558		400	
	0.0575		600	
	0.0594		800	
stainless steel	0.0422		50	37
446	0.0446		200	
	0.0474		400	
	0.0503		600	
	0.0532		800	

<u>Material</u>	<u>k cal./cm.sec.°C.</u>	<u>α^* cm²/sec.</u>	<u>t°C.</u>	<u>Reference</u>
tin		0.38	25	29
	0.1575		25	28
	0.151		108	22
	0.143		209	22
titanium	0.0364		50	37
	0.0354		200	
	0.0335		400	
	0.0319		600	
uranium	0.035		70	44
uranium dioxide	0.023		70	44
vanadium	0.0635		1500	14
titanium carbide	0.0234		966	
	0.0243		1424	
zinc		0.41	25	29
	0.30		-100	4
	0.28		0	4
	0.263		97	22
	0.250		242	
	0.241		313	
	0.220		400	

$$* \alpha = \text{thermal diffusivity} = k/c_p \rho$$

SECTION IV - PROPOSED METHOD FOR MEASURING
THE THERMAL CONDUCTIVITY OF SOLID MATERIALS

Introduction

A careful review of the literature methods for the determination of the thermal conductivity of solid materials has led to the following design criteria:

1) the heat flow through the sample must be determined accurately if a single sample is used or by a comparative method, 2) the use of test samples of the exact same physical dimensions will enable the apparatus to be calibrated to eliminate deviations due to physical shape, 3) if possible temperature measurements should not be made within the test material so that thermocouples may be used to measure temperatures at a similar point for all tests, and 4) the contact resistance between a test and reference material must be considered.

With the above factors in mind one experimental procedure based on the longitudinal heat flow, steady-state method and a basic apparatus modified for three temperature ranges is proposed. The three temperature ranges covered are -200°C. to 40°C. , 20°C. to 300°C. , and 200°C. to 1000°C. It is the prerogative of the experimenter to choose the apparatus specifications to be used at the overlapping temperature ranges.

Sample Preparation

Two cylindrical shaped specimens of the material to be tested should be prepared. Each one should be exactly 1.00 inches in diameter. One sample should be 3.00 inches long, and the other 1.50 inches long. The use of the two specimens will eliminate the need for determining any quantity other than the physical dimensions of the test specimens. By determining the apparent thermal conductivity of the test material at the same temperature for each sample length, the true thermal conductivity may easily be calculated, if the contact

resistance between the test and reference materials is assumed to be constant. This calculation is shown in Appendix B.

Reference Material Preparation

It is proposed to use cylinders of reference material on each side of the test specimen as shown in Figure 1. Armco Iron is recommended for use as the reference material because of the wide use in the literature and because of the many exact determinations made of its thermal conductivity³². Armco Iron is the trade name of an extremely uniform composition iron manufactured by the Armco Steel Corporation, 703 Curtis Street, Middletown, Ohio. Two identical reference cylinders should be machined from a single sample of Armco Iron. Each should be 3.00 inches long and 1.00 inches in diameter. Each cylinder should be drilled with 1/32 inch diameter holes for encasement of thermocouples as shown to scale in Figure 1. The radial position of the off-axis thermocouples, 2 and 6, is immaterial. The holes should be drilled parallel with the axis of the cylinder to depths of exactly 0.50 and 2.50 inches from the planar faces of the cylinder. The thermocouple holes should be backfilled with a solder or a nonconductive material after insertion of the thermocouples. The small diameter of the holes and thermocouples will have a negligible contribution to heat transfer, i.e., $(1/32)^2 \times 100\%$. The thermocouples should be protected from contacting the surfaces of the reference sample by suitable electrical insulation, such as asbestos spaghetti. 30 gauge thermocouple wire is recommended for use. The types of thermocouples to be used are discussed in another part of this section.

Preparation of Test Apparatus

The test material, prepared as previously described, is wet with a small droplet of liquid mercury at the surface to be contacted with the reference sample

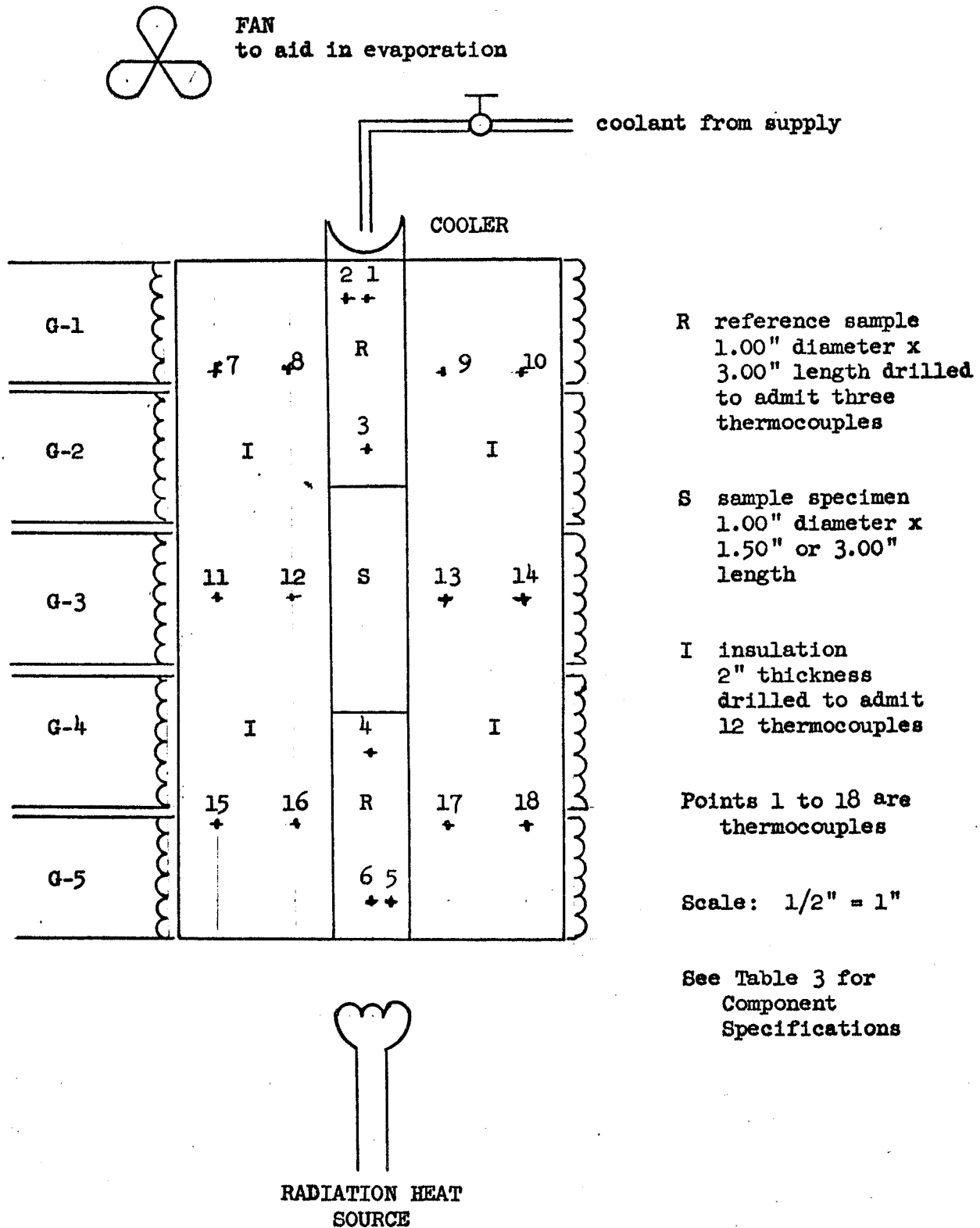


FIGURE 1

PROPOSED EXPERIMENTAL APPARATUS

to reduce contact resistance between the test and reference materials. Mercury is used because it will not damage the iron reference standards by amalgamation. Any surface reaction between mercury and the test material is compensated for by the experimental procedure.

A two-inch thick layer of insulation is used to prevent lateral heat transmission with the surroundings. The insulation is further protected from heat transmission by guard units. Table 3 shows the recommended materials for use depending upon the temperature at which the thermal conductivity is to be measured.

Standard 3/4 inch IPS pipe covering may be used since it is approximately 1.05 inches inside diameter. The insulation layer is drilled, as shown to scale in Figure 1, to admit ten thermocouples which will be used to measure the temperature difference across the insulation and in turn regulate the heat flow between the guard units and the insulating layer. The type of thermocouples used will be dependent upon the temperature conditions of the determination as is shown in Table 3. Thermocouple construction may be the same as for the thermocouples imbedded in the reference samples. The 1/32 inch holes for the thermocouples should be drilled perpendicular to the axis of the cylindrical insulation. A separate hole must be drilled for each thermocouple. The direction of entry of the thermocouple is unimportant as long as the thermocouples will be spaced one inch apart on the same radius of the cylinder.

A Leeds and Northrup K-3 potentiometer is recommended for measuring the thermocouple output. Measurements of 0.01% of 1°C. may be made using this instrument.

TABLE 3

RECOMMENDED EQUIPMENT SPECIFICATIONS

Temperature	-200°C. to 40°C.	20°C. to 300°C.	200°C. to 1000°C.
Thermocouple mv/°C.	copper-constantan ~ 0.03	iron-constantan ~ 0.05	chromel-alumel ~ 0.04
Insulation k BTU/hr.ft. ² F/in. at °F.	Styrafoam 22 0.28 40	Kaylo Standard 0.51 300	Kaylo 20 0.62 1000
Manufacturer	Dow Chemical	Owens Corning	Owens Corning
Potentiometer	- - - Leeds and Northrup Type K-3 Model #7553-5 - - - - - + 0.01% of 20 microvolts, full scale 1.6 volts		
Guard Unit 5 of each type required	10 feet 1/8" dia. nickel tubing made into 2" high 5" ID coil 8 loops per coil	20 ft. length nickel- chrome wire #18 made into 2" high 5" ID coil 16 loops per coil	10 ft. length Kanthal #15 wire made into 2" high 5" ID coil 8 loops per coil
Manufacturer	Sweeco Tube Corp. Clifton, N. J.	Lewis Engineering Co. Naugatuck, Conn.	Kanthal Corporation Bridgeport, Conn.
Guard Unit Power Supply	pressure liquid nitrogen max. 7.8 lbs./hr.	5 type 2PF-136 Powerstat Variable Autotransformer 110 volt input, 20 amperes maximum output Fischer Scientific Company, Union, N.J.	
Coolant	0 to 10 cc./min. liquid nitrogen	either liquid nitrogen or water	0 to 2 cc./min. water
Heat Source	General Electric 250 watt Infrared Industrial Lamp Type PLG 401 W Surface temperature 500°F.		GE Quartzline High Intensity Lamp 1500 watts.

The insulation materials recommended in Table 3 have been chosen because of their availability from standard commercial suppliers. All insulating materials recommended are available in 3/4" IPS 2 inch thicknesses.

The guard heaters used for determinations above room temperature have been sized to maintain the insulation layer at essentially the same temperature as the test materials. Nichrome (nickel-chrome) wire is not suitable for high temperature work. Kanthal wire is a molybdenum base material which is suitable for use to 2500°C. Kanthal wire does not undergo any change in its electrical properties at high temperatures with time in an air atmosphere. It does become very brittle on extended use or temperature cycling. The high temperature guard heater coils will have to be changed periodically or whenever the apparatus is disassembled. A layer of asbestos insulation should be placed over the guard heater units to protect personnel from the heat and electrical dangers. The heat input to the guard heaters is changed by changing the voltage to the heaters by use of "Variac" autotransformers.

For the low temperature determinations a coil of nickel tubing is used for the guard unit in place of the electrical wire heater. Liquid nitrogen is passed through the coil at a pressure close to atmospheric. The boiling of the nitrogen will be used to prevent heat transmission from the surroundings to the apparatus. The flow rate of liquid nitrogen will be regulated by needle valves on each coil. The coils may be fed by one liquid nitrogen supply header connected to a liquid nitrogen storage tank.

Sample calculations for the sizing of the guard units are shown in Appendix C. All values have been taken in such a manner as to oversize the equipment, e.g., the coefficient of convective and radiant heat transfer from the apparatus to the surroundings has been taken as $20 \text{ BTU/hr.ft.}^2 \text{ }^\circ\text{F.}$ although it is known that the real value of this quantity will be on the order of 2 to 5 $\text{BTU/hr.ft.}^2 \text{ }^\circ\text{F.}$ The resistance of Kanthal wire #15 is essentially the same as that of the #18 Nichrome wire. The calculated amperages are at the limits of the current carrying capacity of the heater wires. Actual operation should be at much lower currents because of the large safety factor used in calculations.

Cooling of one surface of the reference sample is accomplished by means of a cooler pot in which a suitable liquid is boiled into the atmosphere. The pot is simply a $1/2$ inch deep 1.00 inch diameter cup having its lower surface planar. It is recommended that the pot be made of either Armco Iron or nickel. The coolant liquid is dripped into the pot through a $1/8$ inch stainless steel tube in such a manner as to assure a complete film of liquid over the inner surface of the pot. Coolant flow rate is controlled by operation of the needle valve in the coolant line. For operation from -200°C. to about 70°C. liquid nitrogen is recommended as the coolant, because it is readily available, inexpensive and nonflammable. Water is recommended for use at temperatures above 70°C. Care must be taken not to use any combustible material as the coolant in the apparatus. A fan is recommended to aid in the evaporation of the coolant from the cooling pot by removing the vapors above the surface into a moving air stream. The maximum quantities of each coolant required are calculated in Appendix C.

Two heat sources are recommended in Table 3. One is for high temperature work and the other is for moderate and low temperature work. The heat input to the sample is controlled by varying the distance between the heat source and the reference sample hot surface. The Quartzline Lamp heat input into the reference sample may also be controlled by varying the supply voltage to the lamp by means of a Variac Autotransformer connected so that it operates as a step-up transformer, and maintaining the lamp at a constant distance from the unit.

In making the electrical wiring for the apparatus care must be taken not to exceed allowable currents in rubber coated copper electrical wires. #18 wire is suitable for use with currents of not more than 4 amperes, and #14 wire may be used to 15 amperes.

Experimental Procedure

The test and reference samples are prepared as previously described and placed into the insulation - guard unit suitable for the temperature of operation. The heating and cooling sources are turned on. The guard units are adjusted so that the temperature difference between thermocouples on the same position longitudinally on the insulation but radially 1" apart is less than 2°C.* The system is allowed to equilibrate while maintaining the desired temperature differential in the insulation.

Readings of the thermocouple outputs of the thermocouples embedded in the reference material are made when it is determined that steady state has been achieved by noting that all temperatures in the system are constant and within specified values. An additional check that the lateral heat loss is negligible should be made by checking to see that the temperature difference between

*Sample calculations in Appendix A show that 2°C. through the insulation is equal to approximately 2% of the total heat input.

thermocouples 1 and 2, and 6 and 5 is essentially zero. The procedure is repeated with the test material sample of different length.

The test made on the second test specimen should be made under conditions that will make the extrapolated temperature values at the reference - test sample interfaces equal for both tests. This is done to simplify calculations of the true thermal conductivity of the test material.

Determination of the Thermal Conductivity From Experimental Data

The data points of temperature against length from the heat receiving surface, are plotted on graph paper. A sample plot for a material having a thermal conductivity less than that of the reference material is shown in Figure 2. Points 1 and 3, A and B, and 4 and 6 are connected by straight lines. Over the temperature range of the test it may be assumed that the thermal conductivity of the reference and test materials does not change significantly. The slope of lines 1, 3, and 4, 6 should be identical. Lines 1, 3 and 4, 6 are extended to the contact surface between reference and specimen samples. The slope of line A,B is a fictitious number which will be used to determine the apparent thermal conductivity of the test material. The apparent thermal conductivity (k_a) is easily shown to be equal to the slope of line 1,3 ($M_{1,3}$) multiplied by the thermal conductivity (k_r) of the reference material and divided by the slope of line A,B. * ($M_{A,B}$) or

$$k_a = \frac{M_{1,3}}{M_{A,B}} k_r$$

A simple algebraic derivation* will also show that the true thermal conductivity of (k_T) of the test material is equal to the product of the apparent thermal

* See Appendix B

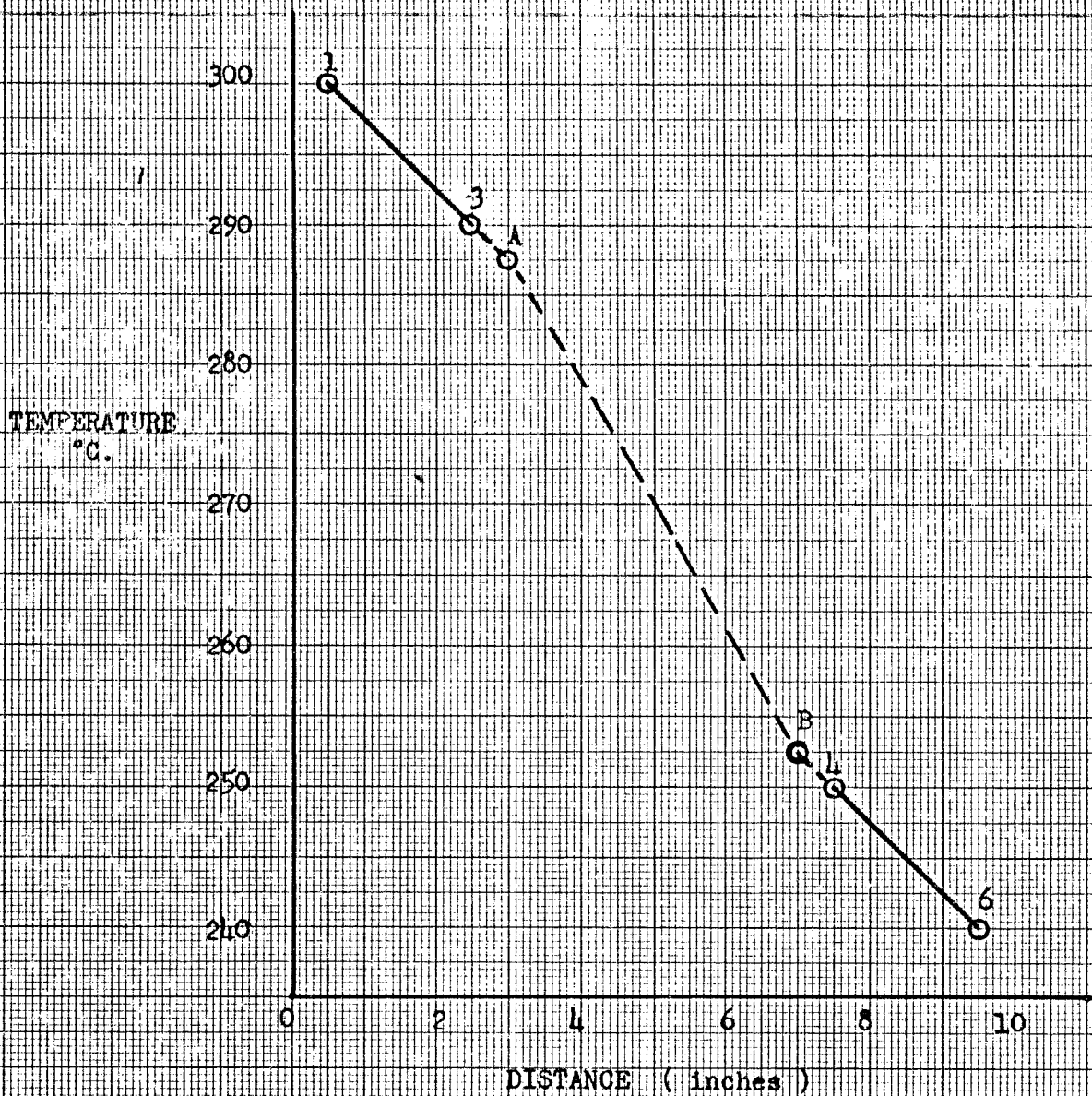


FIGURE 2

Temperature vs. Distance for 3.00 inch sample having a thermal conductivity less than that of the reference sample

Point 1 is the temperature measured at position 1 in the reference sample

Point 3 is the temperature measured at position 3 in the reference sample

Point 4 is the temperature measured at position 4 in the reference sample

Point 6 is the temperature measured at position 6 in the reference sample

Point A is the extrapolated value of the temperature at the hot side of the reference-test sample interface

Point B is the extrapolated value of the temperature at the cold side of the reference-test sample interface

conductivities of the two different lengths of test specimens, divided by twice the apparent thermal conductivity of the 1.50 inch (k_{a2}) specimen minus the apparent thermal conductivity of the 3.00 inch (k_{a1}) specimen or

$$k_T = \frac{k_{a2} k_{a1}}{2k_{a2} - k_{a1}}$$

The temperature at which the thermal conductivity is determined is taken as the mean value of the temperatures at points A and B.

Determination of the Thermal Conductivities of Particles Using the Proposed Apparatus

It is recommended that either the procedure of Schröder³⁵ or Sehr³⁶ be used to calculate the thermal conductivity of small particles embedded in another material. The thermal conductivity of the mixture of small particles and encasement material may be determined by experiments with the apparatus proposed in this thesis.

Calibration of the Apparatus

In order to check the reliability of all components and to determine the precision of the apparatus it is recommended that a calibration experiment be performed using Armco Iron test specimens as well as reference standards. Attempts should be made not to move or change the thermocouples embedded in the reference standards once the calibration has been completed as it is well known that individual thermocouples of the same materials may vary slightly in their response to temperature.

Discussion of Proposed Apparatus and Procedure

The proposed apparatus fulfills all the specifications stated in the introduction to this section. It has several advantages over other methods

proposed in the literature. Among these are: 1) simplicity of fabrication and operation, 2) no special techniques for operation are needed other than the ability to use a potentiometer and autotransformer, and 3) an uncomplicated method of calculating the thermal conductivity from test data is used to compensate for contact resistance between the test and reference samples.

The apparatus design and especially the method of determination of the thermal conductivity from test data are new and have not been presented in the literature to the knowledge of the author.

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APPENDIX A

CALCULATION OF THE PERCENTAGE HEAT LOST Laterally FOR AN X°C. TEMPERATURE

DIFFERENCE BETWEEN REFERENCE POINTS IN THE INSULATION

- (1) Assume a 10°C. temperature difference between points 1 and 3 in the reference sample at a mean sample temperature of 300°C.

Using the Fourier One Dimensional Heat Flow Equation:

$$Q = kA \Delta t / \Delta x$$
$$Q = 0.55 \times 5.15 \times 10 / 5.08 = 5.48 \text{ cal/sec}$$
$$k = 0.55 \text{ cal/cm sec } ^\circ\text{C.}$$
$$A = D^2/4 = 5.15 \text{ cm.}^2$$
$$\Delta t = 10^\circ\text{C.}, \Delta x = 2 \text{ inches} = 5.08 \text{ cm.}$$

- (2) For the insulation the lateral heat flow between thermocouple points 11 and 12 which are 1 inch apart is:

$$Q' = \frac{2\pi l \Delta t}{\ln r_2/r_1}$$
$$Q' = \frac{0.157 \times 10^{-3} \times 2 \times 5.08 \times \Delta t}{\ln 5.08/2.54}$$
$$k = 0.038 \text{ BTU/hr ft } ^\circ\text{F.} = 0.157 \times 10^{-3} \text{ cal/cm sec } ^\circ\text{C.}$$
$$r_2 = 5.08 \text{ cm.}$$
$$r_1 = 2.54 \text{ cm.}$$
$$l = 5.08 \text{ cm.}$$

$$Q' = 0.0145 \Delta t \text{ cal/sec } ^\circ\text{C.}$$

- (3) The percentage heat loss is:

$$Q'/Q \times 100 = 0.0145 \Delta t / 5.48 \times 100 = 0.264 \Delta t \text{ } \%/^\circ\text{C.}$$

- (4) For the 9 inch reference and test sample apparatus, the heat loss for a 2°C. temperature difference between points 11 and 12 is:

$$0.264 \times 2^\circ\text{C.} \times 4.5 = 2.16 \%$$

The 4.5 factor corrects the calculations from a 2 inch to a 9 inch basis.

A P P E N D I X B

Calculation of the Apparent Thermal Conductivity of the Test Material

The basic Fourier One Dimensional Heat Flow Equation is:

$$q = k A \Delta t / \Delta x$$

The slope of line 1,3 in Figure 2 is $M_{1,3} = \Delta t_{1,3} / \Delta x_{1,3}$

The slope of line A,B in Figure 2 is $M_{A,B} = \Delta t_{A,B} / \Delta x_{A,B}$

Since one dimensional heat flow through a constant area is assumed:

$$q/A = k_r M_{1,3} = k_a M_{A,B}$$

where k is the thermal conductivity and subscript r refers to the reference specimen, and a to the apparent value for the test material.

Therefore:

$$k_a = k_r M_{1,3} / M_{A,B}$$

Calculation of the True Thermal Conductivity of the Test Material

The overall resistance to heat flow between points A and B is

$$R_{\Sigma} = R_c + R_t$$

where resistance R is equal to x/kA , and subscripts refer as follows; Σ = total, c = contact, and t = true value for the test material.

The heat flow q is equal to $\Delta t/R = \Delta t k A / x$

For the 3.00 inch sample: $q_1 = k_{a1} A_1 \Delta t_1 / x_1$ (1)

For the 1.50 inch sample: $q_2 = k_{a2} A_2 \Delta t_2 / x_2$ (2)

where k_a is the apparent thermal conductivity of the system between points A and B.

But $x_1 = 2x_2$, $A_1 = A_2$, $\Delta t_1 = \Delta t_2$, as specified in the sample preparation and experimental procedure.

The heat flow between points A and B is $\Delta t/R$ in terms of resistance.

$$q_1 = \frac{\Delta t_1}{R_{t1}} = \frac{\Delta t_1}{R_{c1} + R_{t1}} \quad (3) \quad q_2 = \frac{\Delta t_2}{R_{c2} + R_{t2}} \quad (4)$$

Since the temperatures and materials at points A and B are similar for both tests it may be assumed that the contact resistances are the same, i.e., $R_{c1} = R_{c2}$

By inverting (3) and (4) and substituting:

$$\frac{1}{q_1} = \frac{R_{c1} + R_{t1}}{\Delta t_1} \quad \frac{1}{q_2} = \frac{R_{c2} + R_{t2}}{\Delta t_2}$$

Subtracting:

$$\frac{1}{q_1} - \frac{1}{q_2} = \frac{R_{t1} - R_{t2}}{\Delta t_1} = \frac{x_1}{A_1} \frac{(1/k_{t1} - 1/2k_{t2})}{\Delta t_1} \quad (5)$$

Similarly by inverting and subtracting (1) and (2):

$$\frac{1}{q_1} - \frac{1}{q_2} = \frac{x_1}{A_1} \frac{(1/k_{a1} - 1/2k_{a2})}{\Delta t_1} \quad (6)$$

Equating (5) and (6) and combining like terms:

$$\frac{x_1}{2k_t A_1 \Delta t_1} = \frac{x_1 (1/k_{a1} - 1/2k_{a2})}{A_1 \Delta t_1}$$

Simplifying:

$$k_t = \frac{k_{a1} k_{a2}}{2k_{a2} - k_{a1}}$$

APPENDIX C

Sizing of Guard Heater Units

Assume the insulation is at 300°C. and the coefficient of convective and radiant heat transfer to the surroundings is 20 BTU/hr ft² °F. and the ambient temperature is 30 °C.

$$\begin{aligned}Q &= hA \Delta t = h 2\pi r l \Delta t \\Q &= 2120 \text{ BTU/hr} \\Q &= 620 \text{ watts}\end{aligned}$$

$$\begin{aligned}h &= 20 \text{ BTU/hr ft}^2 \text{ °F.}, \quad r = 2.5 \text{ inches} \\l &= 2 \text{ inches}, \quad \Delta t = (300-30) \times 1.8 \text{ °F.} \\1 \text{ BTU} &= 0.293 \text{ watt hrs}\end{aligned}$$

How much #18 Nichrome wire can be spaced 1/8" apart on a 2" long 5" diameter cylinder?

$$\begin{aligned}\text{amount} &= \pi D l \text{ spacing} \\ \text{amount} &= 20 \text{ feet}\end{aligned}$$

$$\begin{aligned}\text{spacing} &= 8 \text{ loops per inch} \\ l &= 2 \text{ inches}, \quad D = 5 \text{ inches}\end{aligned}$$

The resistance of the wire is 0.4 ohms per foot. The total resistance of 20 feet is 20 x 0.4 = 8 ohms. At 100 volts how much current will the wire draw? $R_0 = E/I$ $I = 100/8 = 12.5$ amperes. This is equivalent to 100 x 12.5 = 1250 watts, which is more than the required amount. The guard heater specification for the Nichrome wire is therefore:

20 feet of #18 Nichrome wire in the form of a 5" diameter coil 2" long with 1/8" spacing between loops. The Variac must handle 12.5 amperes at 100 volts output.

For a 1000°C. apparatus temperature the heat loss that must be compensated for may be assumed to be directly proportional to the loss calculated above:

$$Q = 620 \times 970/270 = 2200 \text{ watts}$$

The resistance of Kanthal wire #15 is 0.38 ohms per foot or about the same as that of the Nichrome wire. By using 1/2 the length of wire and twice the spacing between loops the power output is raised to 2500 watts.

Sizing of Guard Cooling Unit

For the nickel tubing used in the guard coolant unit and the boiling liquid nitrogen an overall heat transfer coefficient of 300 BTU/hr ft² °F. is assumed. An 11°C. temperature difference between the apparatus and tubing is also assumed. The heat transfer with the surroundings may be assumed to be the same as for the 300°C. apparatus temperature since the apparatus is at -200°C.

$$\begin{aligned}Q &= 2120 \text{ BTU/hr} = UA\Delta t \\ A &= 0.35 \text{ ft}^2\end{aligned}$$

$$\begin{aligned}U &= 300 \text{ BTU/hr ft}^2 \text{ °F.} \\ \Delta t &= 20 \text{ °F.}, \quad A = \text{to find}\end{aligned}$$

For 1/8" tubing the area is equal to 0.033 ft²/ft of length.

The length required is therefore 0.35/0.033 = 10 feet.

The length used in one 5" diameter coil is $\pi D = \pi 5/12 = 1.3$ feet.

The number of coils required is 10/1.3 = 8 loops each being 1/4" apart center to center. The specification for the cooler guard unit is:

10 feet of 1/8" diameter nickel tubing made into a 2" high 5" diameter coil having 8 loops spaced 1/4" apart, center to center.

The maximum amount of coolant needed in the guard cooler unit is determined from the latent heat of vaporization of liquid nitrogen and the heat load on the guard cooler:

$$Q = \text{amount} \times \lambda = 2120 \text{ BTU/hr} \quad \lambda = \text{latent heat of vaporization of liquid nitrogen}$$

$$\text{amount} = 7.8 \text{ lbs/hr}$$

Sizing of Heat Source

From the determinations in Appendix A the heaters must supply about 5 cal/sec to the reference sample. As a safety factor the radiation heaters will be sized to supply 20 cal/sec.

$$20 \text{ cal/sec} = 72 \times 10^3 \text{ cal/hr} = 83.5 \text{ watts} \quad 1 \text{ cal} = 1.16 \times 10^{-6} \text{ Kw-hr}$$

The lamps chosen have surface temperatures of 500°F. and above 2000°C. The watt rating of the lamps are also several times the calculated requirement.

Sizing of Coolant Requirements

Using a value of 15 cal/sec of heat flow through the reference sample the equivalent heat flow per hour is 215 BTU/hr. This heat will be used to vaporize either water or liquid nitrogen whose latent heats of vaporization are 1000 BTU/lb and 275 BTU/lb respectively. The amount of each required is therefore:

Nitrogen		Water	
$215/275 = 1 \text{ lb/hr}$	$\rho = 0.8 \text{ gm/cc}$	$215/1000 = 0.25 \text{ lb/hr}$	$\rho = 1 \text{ gm/cc}$
$1 \text{ lb/hr} = 10 \text{ cc/min}$		$0.25 \text{ lb/hr} = 2 \text{ cc/min}$	

The area of the coolant pot is fixed at $\pi r^2 = \pi(0.5/12)^2 \text{ ft}^2 = 0.0055 \text{ ft}^2$
The overall heat transfer coefficient would have to be:

$$U = Q/A\Delta t$$

$$U = 275/40 \times 0.0055 \quad \Delta t = 40^\circ\text{F. as conservative estimate}$$

$$U = 870 \text{ BTU/hr ft}^2 \text{ }^\circ\text{F.}$$

This calculated value is certainly small for film boiling on a plane surface.

The coolant pot will be sized to hold 5cc.

$$\text{Volume} = 5 \text{ cc.} = 0.31 \text{ in}^3 \quad A = \pi/4 \text{ in}^2$$

$$\text{Height} = \text{volume/area} = 0.31/0.79$$

$$\text{Height} = 0.4 \text{ inches}$$

The coolant pot will be 1/2 " high. The feed will be introduced through a 1/8" diameter pipe in such a manner as to cover the pot bottom with a liquid film to insure film boiling. The bottom of the coolant pot must be exactly the same dimension as the reference sample it covers, that is 1.00 inches in diameter and planar in shape.