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Thermophysical Properties of Materials

by
E. Fitzer

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NORTH ATLANTIC TREATY ORGANIZATION

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NORTH ATLANTIC TREATY ORGANIZATION

ADVISORY GROUP FOR AEROSPACE RESEARCH AND DEVELOPMENT

(ORGANISATION DU TRAITE DE l'ATLANTIQUE NORD)

THERMOPHYSICAL PROPERTIES OF MATERIALS

by

Erich Fitzer

Karlsruhe
Germany

MARCH 1967

FOREWORD

The Structures and Materials Panel of the Advisory Group for Aerospace Research and Development (AGARD) is comprised of scientists, engineers and technical administrators, from industry, government and universities throughout NATO, concerned with advancing the status of aerospace research and development and with developing technical means and data for optimizing the vehicles and equipment of interest to NATO. The Panel, therefore, provides a discussion forum, a mechanism for exchanging information, and a means for establishing and conducting cooperative laboratory programs in selected technical areas.

One of the technical areas of prime concern to this Panel is that of the behaviour of materials and structures at high temperatures. The range between about 1000°C and 2500°C is of particular interest, partly because of the incidence of this range in powerplants, very high speed flight, re-entry from space, propulsion devices such as rockets, etc. Several projects have been undertaken by this Panel on the development and behavior (mechanical and chemical) of materials in the lower part of this range but the Panel also recognized the dearth of necessary data on thermophysical properties, the lack of agreement, or in some cases knowledge, on methods of determining these data and the inadequacy of theory and understanding. It was also recognized that considerable work was underway on this subject in various countries.

Accordingly, in early 1966, the Panel agreed to initiate a project (1) to examine current activities and to evaluate the present stage of knowledge of thermal physical properties, (2) to examine techniques for their determination at elevated temperatures and encourage improvement in these techniques or development of new ones, (3) to promote fundamental research of the type that might provide a reliable theoretical basis for the calculation of these properties, and (4) possibly to define and organize a cooperative experimental program.

As a means of initiating action responsive to the above objectives, Prof. Dr Erich Fitzer, of the Institut für Chemische Technik, Karlsruhe, Germany, was engaged to visit a number of sources in NATO countries in order to prepare a survey of measurement techniques, a related bibliography and a limited compilation of data and theory on selected properties. This publication is Professor Fitzer's first report based primarily on his first series of visits and his related studies. The Panel, in the meantime, has engaged in a supplementary small symposium and is contemplating a limited cooperative laboratory program to test the reliability of measurement of thermal conductivity.

N.E.Promisel

Chairman,

AGARD Structures and Materials Panel

SUMMARY

This paper is the first report on a series of visits to organisations in NATO countries, undertaken by the author under the sponsorship of the Structures and Material Panel of AGARD. It contains a survey of measurement techniques, a related bibliography and a limited compilation of data and theory on selected thermophysical properties of high temperature materials. The visits confirmed that knowledge of thermal conductivity is the key point of all further research in this field, and the report deals mainly with its measurement and theory; also considered are temperature measurement, diffusivity, thermal expansion, emittance and specific heat, and electrical properties. Research activities and programmes within the NATO nations are summarised and proposals for collaboration within an AGARD research group are discussed.

RESUME

La présente communication constitue le premier compte rendu d'une série de visites faites par l'auteur sous l'égide de la Commission "Structures et Matériaux" de l'AGARD à divers organismes des pays de l'OTAN. Elle comporte une étude des différentes techniques de mesure utilisées, une bibliographie y relative et un recueil réduit de données expérimentales et théoriques sur certaines propriétés thermophysiques des matériaux pour hautes températures. Les visites ont permis de confirmer que des connaissances concernant la conductivité thermique constituent le point clef de toutes les nouvelles recherches à faire dans ce domaine; le compte rendu porte essentiellement sur la mesure et la théorie de cette propriété. Sont également examinés les problèmes de la mesure des températures, de la diffusivité, de la dilatation thermique, de l'émittance et de la chaleur spécifique, et des propriétés électriques. L'auteur décrit brièvement les activités et les programmes de recherches en cours dans les pays de l'OTAN et étudie des propositions de collaboration dans le cadre d'un groups de recherche de l'AGARD.

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Erich Fitzer

1. INTRODUCTION

1.1 The Aims of AGARD Project MGPM 315

During the 22nd Structures and Materials Panel session, held in Delft in April 1966, a new AGARD Project (MGPM 315) was discussed: it was agreed that a study should be made of the most important thermophysical properties of solid materials at high temperatures, especially heat conductivity and thermal expansion.

First, a survey of techniques, particularly to establish the temperature ranges of their application, and of suitable theoretical approaches was planned, particularly for higher temperatures. Furthermore the adequacy of published data for a limited number of materials, such as "super alpha" titanium alloy, an "alpha-beta" titanium alloy, a heat resistant nickel alloy, tungsten and graphite would be discussed. This would be with particular reference to the identification of the temperature range over which the measurement of these values begins to lose accuracy and reliability. The very pure metals gold and platinum would be taken as a standard for heat-conductivity measurements.

The aim of this study would be to promote fundamental research of the type that might provide a reliable theoretical basis for the calculation of these and other properties.

1.2 Overall Survey and Comments

Several visits were made to those NATO countries that are engaged on studies of the thermophysical properties of high temperature materials, and this report is the result of these visits. The visits confirmed the assumption that knowledge of the thermal conductivity is the key point of all further research in this field. Precise measurements of thermal conductivity are difficult and the theory of thermal conductivity is complicated and not very well developed. This report therefore deals mainly with the measurement and theory of thermal conductivity. One basis of all experimental work in measurements of thermal conductivity is the exact knowledge of the real temperature and that of small temperature differences within the specimens. Therefore the status of temperature measurement is discussed first.

Diffusivity is discussed in detail, not only as a means of determining the thermal conductivity but also as a material property which is important in engineering calculations

The thermal expansion behaviour of high temperature materials is treated more briefly, since its determination does not present so much of a problem and because experimental efforts within the countries visited are not as extensive. In addition, the theoretical treatment is very difficult.

Other properties, like emittance and specific heat, are only discussed insofar as they are important for the execution and interpretation of the thermal conductivity measurements.

The electrical properties, like electrical resistance, Seebeck coefficient and residual electrical resistance, are necessary for the theoretical analysis of the thermal conductivity and are therefore briefly considered.

The status of the measurement of all these properties is described in Section 2.

Sections 4 and 5 are shorter, because sufficient published literature is available on these subjects.

Section 4, dealing with thermophysical properties of some high temperature materials, is based on the excellent reports of the TPRC (Thermophysical Properties Research Center) at Purdue University, West-Lafayette, Indiana, USA. The discussion of the theory (Section 5) follows closely the work of P.G.Klemens, Pittsburgh, and that of Berman and Ziman of the Oxford group. In the last section (6) the research activities and programmes within the NATO nations are summarised and proposals for a possible collaboration with a research group of AGARD are discussed. The visits showed that all future work on this project will be of great interest in Europe as well as in America.

1.3 Literature on Thermophysical Properties

Individual reports are not quoted in this section. Those quoted in later sections and listed in Section 3.2 are numbered with (Lit.No.). The results of visits to laboratories and discussions with specialists are referred to separately, with the term (Vis.No.) in the list in Section 3.1.

Only literature in book form and monographs, on the problem outlined, are summarised in this section; they are grouped as follows:

1.3.1 Phenomenology and Theory

1.3.2 Experimental Methods and the Mathematical Treatment of Heat Transfer

1.3.3 Data on Thermophysical Properties

1.3.4 Proceedings of Special Conferences.

This division shows up the lack of monographs dealing with experimental methods so far, which means that special meetings are necessary. Such meetings, dealing with thermal conductivity, have been organised successfully in the United States and in England during the past six years (see Section 1.3.4). Also, a new monograph on thermal conductivity by American, British and Swiss specialists is now in preparation. Entitled "Thermal Conductivity", it is edited by R P Tye and will be available from Academic Press Inc. (London) in 1968. Table I on p.87 shows the projected chapters and the authors.

On the other thermophysical properties there are no special monographs, although chapters can be found in several materials handbooks.

1.3.1 *Phenomenology and Theory*

Klemens, P. G.	1956	Handbuch der Physik, Bd. 14	Springer, Berlin
Jones, H.	1956	Handbuch der Physik, Bd. 19	Springer, Berlin
Drabble, J.R. Goldsmid, H.I.	1961	Thermal Conduction in Semiconductors	Pergamon Press, Oxford, New York, Paris
Ziman, J.M.	1963	Electrons and Phonons, the Theory of Transport Phenomena in Solids	University Press, Cambridge
Ziman, J.M.	1964	Principles of the Theory of Solids	University Press, Cambridge

1.3.2 *Experimental Methods and Mathematical Treatment of Heat Transfer*

Schneider, P.J.	1955	Conduction, Heat Transfer	Addison-Wesley, Reading, Mass.
Eder, F.X.	1956	Moderne Messmethoden der Physik. Teil II, Thermodynamik	VEB Deutscher Verlag der Wissenschaften, Berlin
Campbell, I.E.	1956	High Temperature Technology	J.Wiley, New York, Chapman and Hall, London
Kingery, W.D.	1959	Property Measurements at High Temperatures	J.Wiley, New York, Chapman and Hall, London
Carslaw, H.S. Jaeger, J.C.	1965 (2nd Ed.)	Conduction of Heat in Solids	Clarendon Press, Oxford
Arpaci, Vedat S	1966	Conduction, Heat Transfer	Addison-Wesley, Reading, Mass

1.3.3 *Data on Thermophysical Properties*

WADC TR 58-476	1961-63	Handbook of Thermophysical Properties of Solid Materials	Pergamon Press, New York, London, Paris
TPRC	Beginning 1962 is continuing	Data Sheets on Thermophysical Properties. (Up to now thermal conductivity, diffusivity, radiation properties, viscosity, specific heat)	Thermophysical Properties Research Center, Purdue University, Lafayette, Indiana, USA

NSRDS - NBS 8	1966	Thermal Conductivity of Selected Materials. (Metals like Al, Cu, Au, Fe, Pt, W; oxides like Al_2O_3, BeO, MgO, ThO_2 and some organic compounds)	National Standards Reference Data Series, US Government Printing Office. Washington D.C. 20402, USA
NBS	Beginning 1965	Reference Tables on Thermocouples. Thermometry Project, NASA Contract: H - 92152	National Bureau of Standards, Cryogenic Data Center, Boulder, Colorado, USA
Landolt - Börnstein	In preparation	Thermal Conductivity of Pure Metals, Vol. II/5	Springer Verlag - Berlin, Göttingen, Heidelberg, New York
TPRC	1967	Thermophysical Properties of High Temperature Materials	McMiller Corporation, New York, NY, USA

1.3.4 Proceedings of Specialist Conferences

Thermal Conductivity Conference	1964 July 15-17	National Physical Laboratory, Teddington, Middlesex, England
American Conferences on Thermal Conductivity	1961 Oct. 26-28	Battelle Memorial Institute Columbus, Ohio, USA
	1962 Oct. 10-12	Division of Applied Physics, National Research Council, Ottawa, Canada
	1963 Oct. 16-18	Gatlinburg, Metals and Ceramics Division, Oak Ridge National Laboratory, Tennessee, USA
	1964 Oct. 13-16	US Navy Radiology Defense Laboratory, San Francisco, USA
	1965 Oct. 20-22	Denver University Dept. of Metallurgy, College of Engineering, Denver, Colorado, USA
	1966 Oct. 9-21	US Air Force Material Laboratory, Wright-Patterson Air Force Base, Dayton, Ohio, USA
Conference on "Technische Temperaturmessung"	1965 March 11	VDI - Bericht, Vol.112, 1966 Düsseldorf, Germany
Conference on "Temperature, its Measurement and Control in Science and Industry"	1962	Vol 3, Parts 1 and 2. Reinhold, New York, 1962

2. THE MEASUREMENT OF THERMOPHYSICAL PROPERTIES

What is meant by thermophysical properties? They are not well defined and could be interpreted as specific data for the particular materials relating to thermodynamic factors and coefficients of transfer phenomena. All measurements of these properties must be correlated to the measuring temperature, the determination of which is especially difficult above 1000°C. The problems are, for example disturbance of the temperature distribution by heat loss effects due to thermocouples or by radiation, or the impossibility of measuring the true temperature in holes by pyrometers, etc.

A further difficulty is the heating of the samples. In all cases this is achieved by direct or indirect electrical heating. For radiation heating, arc furnaces, electron guns and laser sources are generally used. The experimental difficulties of heating are not treated here, but aspects of furnace construction are outlined in the books by Kingery (Lit.1)* and Campbell (Lit.2), and in a paper by Rasor and McClelland (Lit.3). Further references are quoted later.

2.1 Temperature Measurement

The devices required to reduce temperature measurement errors to tolerable limits are usually those which constitute the major limitations on the design of an apparatus for the determination of thermophysical properties. The lack of agreement between the measurements of different scientists results primarily from this fact.

For thermal conductivity measurements another limiting factor is the accuracy with which the real heat flow in the specimen can be determined. Furthermore, it is more important to detect small temperature differences than to know exactly the real temperature, i.e. some tenths of a degree at 2000°C.

In general thermocouples and pyrometers are used in all types of apparatus for the measurement of thermophysical properties. Resistor thermometry is very seldom used directly (Flynn, O'Hagan, Lit.4; R.L.Powell, Vis.38; A.D.Little, Vis.35; Kuhlman, Vis.33; Berman, Vis.20). It is used mainly for calibration of thermocouples. According to R.L.Powell (Vis.38) the standard deviation at the triple point of helium is about 0.1 μV corresponding to 20 μ°K.

According to Rahlfs, of PTB (Vis.10) the calibration is performed between the boiling temperature of liquid oxygen, -183°C, and the freezing point of antimony, +630°C. Extension of the temperature range up to 1063°C (gold point) is in hand.

2.1.1 Thermocouples

Thermocouples have the following advantages: simple and cheap equipment, point measurements possible, short response time, good contact for welding or soldering to the test specimen, practically unaffected by mechanical forces, high stability for long duration, high sensitivity and accuracy, direct measurement of the test temperature, emittance negligible

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* For an explanation of Lit. number and Vis. number, see the first paragraph of Section 1.3 (p.2). The actual references are listed in Sections 3.1 and 3.2 (pp.43 and 55).

The likely sources of error are: electrical leakage of the insulation, inhomogeneities of the wire material, additional e.m.f. in the circuitry, disturbance of the temperature distribution by heat conduction, contamination, difficulty of precise positioning.

The balance of advantages and disadvantages is mainly affected by the particular temperature. Up to 1000°C most difficulties can be overcome. At higher temperatures the problems of electrical insulation and of chemical and physical stability become critical. Table II, on p.88, summarises insulators for high temperature (Lit.5)

The latest results of Kuhlman of GEC (Vis.33) have shown that BeO is the best electrical insulator up to 2300°C. A recently found transition at 2000°C can cause cracks. For higher temperatures HfO_2 is the best material. No advantages are found in using ThO_2. The commonly used thermocouples, their temperature range and their accuracy are listed in Table III on p.88.

The high accuracy and sensitivity of the thermocouples shown in Table III have only been achieved in the last few years by using selected wire materials and by appropriate calibration. According to Rahlfs of PTB (Vis.10), for the calibration of Pt vs Pt/10 Rh thermocouples an accuracy of ±0.3°C at 1063°C is claimed. Up to 1000°C Chromel-Alumel and similar iron alloy thermocouples are generally used because of their high e.m.f. But the experience of PTB has shown that, even for this temperature range, the thermocouples made of noble metals are more accurate because of their greater homogeneity in structure. Yet the mechanical preparation of the sheathed thermocouples causes deviations in e.m.f. (Vis.10, PTB). Thermocouples for very precise measurements have generally been used at temperatures below about 1500°C up to now. But the newer thermocouples Pt30Rh/Pt6Rh (e.g., Pt/RhEL18 from Degussa), Pt20Rh/Pt5Rh and Pt40Rh/Pt20Rh (Vis.25), have been introduced more and more with success for precise measurements up to 1800°C. The NBS in Washington (Lit.12) and NRC in Ottawa (Lit.167, 168) have recently published reference data. In addition, the first of these new thermocouples has a minimum e.m.f. at room temperature. Therefore the temperature of the cold junction is not critical and it is not necessary to thermostat it, if contamination is excluded. The e.m.f. changes with time by diffusion of rhodium through the hot junction. In PTB (Vis.10) an error of 0.5 to 1°C at 1000°C is observed after about 100 runs. NRC in Ottawa has found a change in calibration for their Pt/Rh thermocouples of about 5°C in 500 hours at 1500°C (Lit.167, 168). The refractory metals are extraordinarily sensitive, due to impurities, diffusion on the junctions and recrystallisation. Contamination with carbon can be avoided by sheets of tantalum (Kuhlman, Vis.33). The tungsten-rhenium thermocouples have been tested by Feith (Lit.11) and by Cunnington (Vis.36) up to 2300°C.

In obtaining measurements of high accuracy with thermocouples, great care must be taken of the electrical arrangement and the measuring procedure. All additional e.m.f.'s must be avoided. Berman (Lit 6, 8, Vis.20) uses super-conducting switches at low temperatures ($T < 40^{\circ}K$) for this purpose. The influence of electromagnetic fields can be prevented by using specially twisted bundles as compensating leads, shielded additionally by iron tubes (R L Powell, Vis.38). In high temperature apparatuses with high voltage supplies all measuring circuits must be separated from power circuits (McElroy, Vis 41). To minimise heat loss by conduction in the thermocouple leads, the wires are positioned along an isotherm around the specimen. Another way of preventing heat losses is to connect the measuring thermocouple to another one, heated separately (Goldsmid, Vis.23; Hopkins, Vis.34)

A special discussion on thermocouple positioning in apparatus for the measurement of thermal conductivity has been given by Tye (Lit. 158).

2.1.2 Pyrometers

Visual pyrometers can only be used for temperature measurements above 600°C, but their use is not limited at high temperatures. In general the brightness of the target at a specified wave length is visually compared with the brightness of an electrically heated tungsten strip lamp, acting as a reference.

The accuracy is improved and the range of application extended by the use of photoelectrical detector systems. Wheeler (Vis. 21) uses photometrical pyrometry above 300°C. In Table IV (p.89) data are given on both methods.

For the visual methods, data on commercially available pyrometers are listed in Lit. 13; for the photoelectrical system, data from one of the best laboratory developments are given in Vis. 10 (PTB); see also Table V (p.89) on commercially available compact photoelectric pyrometers. These are chiefly used in the United States, while in Europe only some privately developed ones were seen during the visits (Wheeler, Vis. 21; Schmidt, Vis. 7).

In all measuring systems used in practice, the brightness of the object is compared with a tungsten filament lamp. The lack of agreement given in Table V is caused by these lamps. Therefore much effort has been devoted to developing reference lamps with better reproducibility.

A new source of quasi-black-body radiation for accurate pyrometry up to 2700°C has been developed by the NPL at Teddington, England (Vis. 22, Lit. 14). Close approximation to a black-body source is achieved by electrically heating a tungsten tube in vacuo (to 1800°C) or in argon (to 2700°C). This tube is 2 mm in diameter and 45 mm long. The radiating surface is formed by a bundle of fine tungsten wires placed in the middle of the tube. Within the limits of ±0.2% no difference in emissivity over the wave length range of 0.5 to 0.7 μm was detected. The absolute value of the emittance is higher than 0.95. The constancy of the vacuum lamp is ±0.02°C per 100 hr at 1800°C. With argon filling there is a constancy of about ±0.05°C per hour at 2700°C. This new lamp will be used in recording pyrometers. Since the temperature is below 1250°C in this application, a stability better than ±0.1°C can be expected.

The absolute calibration of all standard lamps is performed by comparison with the radiation in the hollow space at the gold-point (Vis 10, PTB).

A pyrometer with a very low response time (0.22 msec) has been developed by Bollenrath (Vis 11, Lit 158). A germanium photocell is used for temperature measurement. The instrument is used for temperatures around 1000°C and an accuracy of ±1% is claimed.

Furthermore a new pyrometric system has been developed in the "Istituto Termometrico Italiano" at Turin by G. Ruffino and his co-workers (Vis. 18).

Apart from the development of the new pyrometers the practical side of the pyrometrical measurements must be considered. An important point is the emittance of the object and this difficulty is best overcome by measuring in small diameter holes. Where this is not possible, the principle of two colour pyrometers can be used (Schmidt, Vis.7).

Further errors can arise when the radiation from the source is absorbed by dust particles and smoke in the high temperature furnaces. This is a severe problem in pyrometric measurements, especially with graphite furnaces, but controlled gas flow can help to alleviate it (Pears, Vis.42).

2.2 Measurement of Thermal Conductivity

If a one-dimensional homogeneous heat flux is passed through an isotropic material, the thermal conductivity λ_T is defined by Fourier's law

$$\lambda_T = \frac{\dot{q}\,l}{\Delta T s}\,, \tag{1}$$

where $\dot{q}$ is the rate of heat flow per unit time, s is the cross-sectional area at right angles to the direction of heat flow and T the temperature difference along a distance l in the direction of heat flow.

The thermal conductivity is a function of temperature and therefore only small temperature gradients are allowed in precise measurements. On the other hand this requirement complicates the experimental determination of temperature differences.

For the calculation of the thermal conductivity, knowledge of the heat flux $\dot{q}/s$ is necessary. It can be measured directly or it can be derived from the temperature drop in materials with known thermal conductivity, through which is passed the same heat flux. Absolute methods and comparative methods are now considered separately.

2.2.1 Absolute Methods

The heat flow can be determined in practice by measuring the electrical energy dissipated in the heater or by using a calorimeter as a heat sink. The electrical method is more generally used because of its greater accuracy at high temperatures.

The different absolute methods, using indirect heating of the samples, will be discussed within the following classifications, referring to the boundary conditions assumed in the solution of the general heat-transport equation:

2.2.1.1 Longitudinal Methods
2.2.1.2 Radial Methods
2.2.1.3 Forbes Bar Methods

Direct heating methods for electrical conductors are treated in a separate section, the boundary conditions being usually the same, or similar to, those just given

2.2.1.1 *Longitudinal Methods*

(a) *Principles of the method and design of the apparatus*

In the longitudinal method, long cylindrical specimens are used which are in contact at the ends with a heat source and a heat sink respectively.

Under the assumption of unidirectional heat flux from heat source to heat sink the thermal conductivity can be calculated from the temperature gradient along the specimen axis, if the quantity of heat flowing from the heater to the sink and the cross section of the sample are known. Because of the unidirectional heat flux, the longitudinal method can be used equally well for isotropic as for anisotropic materials.

THE GUARD

To prevent radial heat losses the specimen rod is surrounded by a guard tube, matched as well as possible to the temperature distribution along the sample by cylindrical heaters wound on its outer surface. Very often as many as four separately regulated guard heaters are used. The thermal conductivity of the sample and the guard should be of the same order of magnitude. Ideal matching of the temperatures of the specimen and the guard is impossible.

To minimise radial heat loss effects, thick specimens should be used and the temperatures should be measured near the rod axis. In practice nearly all measurements are made with long thin samples, to allow more accurate measurement of temperature gradients over the long distances (Laubitz, Lit.15). Additionally Laubitz recommends matching the temperature of the environment roughly to that of the guard. The matching points need not be in the immediate vicinity of the guard heater; as long as the matching point is outside the heater region, the fractional temperature deviation from the ideal is constant along the whole length of the specimen. The space between sample and guard should be kept small and is preferably evacuated Nevertheless radiation to the colder walls cannot be sufficiently suppressed at higher temperatures. In practice, this limits the application of this method to temperatures of 850-1000°C.

THE HEAT FLUX

The heat flux is measured either electrically, by the energy input to the heater, or with a calorimeter in the heat sink. In either case proper contact between the specimen, the heater and the sink is required. When the heat flux in the heater is determined electrically, heat losses by the contact wires of the heater must be kept very small by using thin leads. In many cases the heaters are arranged in a hole in the specimen (Lit.16, 17).

THE TEMPERATURE MEASUREMENT

In all cases temperatures are determined by thermocouples positioned in the direction of the heat flux. To avoid heat conduction along the thermocouple wires it is recommended to wind them once on an isotherm around the rod. A comparison of the thermocouples, in position, under isothermal conditions is recommended before starting the experiment

(b) *Status and accuracy of measurements*

ACCURACY

The definition of the measurement errors is first discussed

McElroy (Lit 18) divides the errors contributing to the inaccuracy of any system into two groups determinate errors and indeterminate errors The latter are difficult to predict. Therefore the uncertainty quoted for most measurements is only a summation of the determinate errors. For measurements made as a function of a continuous variable, imprecision can be defined as the maximum deviation of any data from a smooth curve. The precision is quite different from accuracy, since an apparatus could yield remarkably precise data but, because of systematic errors, can be extremely inaccurate. The precision is composed of two terms, repeatability and reproducibility. Repeatability is the data spread associated with repeated observations without any imposed system change. Reproducibility is the data spread associated with repeated observations with intermediate imposed system changes. According to McElroy the reproducibility is 5 to 10 times the repeatability for good measurement systems. The absolute accuracy is 20 to 30 times this repeatability.

According to R.L.Powell (Vis.38) the precision is the standard deviation of repeated measurements with different samples, and the accuracy is about 10 times the precision in all thermophysical measurements.

It will be shown later that, in R.L.Powell's most exact measurements of thermal conductivity, using the longitudinal method, the precision was found to be 0.1%; that means an accuracy less than 1%. Unfortunately the measurement errors of most data given in literature, as well as those discussed during the visits, are not in line with the definitions just given. Very often the standard deviation, i.e. precision according to Powell, is used as accuracy. It was impossible in this report to refer all comments to a uniform definition. This could be a programme for a working group of specialists.

In the following a summary is given of the accuracies of different factors determining thermal conductivity measurement in a longitudinal apparatus.

Geometric dimensions can be usually measured to 5×10^{-4} cm (D.R.Flynn, Lit.4; R.L.Powell, Lit.19). Occasionally, with very soft materials, the accuracy is worse; seldom, however, does this contribute significantly to the overall inaccuracy (M.I.Laubitz, Vis.25).

Temperature measurements: as far as the accuracy of these is concerned (see also Section 2.1), the probable uncertainty in the gradient due to inaccuracies in effective thermocouple positions is ±0.2% (Lit.19).

Flynn (Lit.4) reports that with careful calibration and using the "isothermal test", the errors introduced by the conversion of thermocouple e.m.f. to temperature are less than ±0.2% of a 40°C temperature drop in the specimen up to 1000°C. The errors in reading the e.m.f.'s are not more than ±0.05% (for this 40°C drop). Errors due to heat conduction along thermocouple leads can certainly be made less than ±0.05%. Summarising, this gives a maximum uncertainty in the temperature gradient of ±0.59%, while the probable uncertainty is ±0.29%.

Heat flow: errors in the measurement of heat flow derive from many sources, too. At present, at least the following accuracies can be achieved: calibration of potentiometer, voltbox and shunt box < ±0.01%, e.m.f. of the standard cell < ±0.01% (Flynn, Lit.4).

Potential differences of about 0.1 µV can be detected, corresponding to temperatures of 0.02°K at liquid helium temperatures and 0.002°K at room temperature.

Applying corrections to the voltage drop in the potential leads and for the current passing through the voltbox (±0.2 - 0.4%) the accuracy of the measured power input can be ±0.05%. Heat flow along the current leads can be held below ±0.05%. Errors due to shunting of heat by the thermocouples and by the guard can be rather large, but can be restricted to below 0.3% at 1100°C. By proper arrangement of the system the accuracy in heat flow measurements can be better than ±0.35% even at 1100°C.

STATUS OF THE LOW TEMPERATURE APPLICATION

The longitudinal method is best for measuring the thermal conductivity at low temperatures, where heat losses by radiation can be neglected. Therefore the very accurate equipment of R.L.Powell (Vis.38, Lit.19) will be discussed in some detail, to outline the best accuracy achieved up to now by the longitudinal method.

This apparatus is used in the temperature range from 4°K to 300°K. The cryostat consists of concentrically mounted specimen, thermal shield, vacuum container, glass Dewar and outside metallic Dewar. The heat sink is maintained at a constant low temperature by a refrigerant, usually a liquefied gas. Metal specimens are 0.367 cm in diameter and 23.2 cm long; plastic specimens are 2.54 cm in diameter and 20.5 cm long. The diameter has a tolerance of 0.0005 cm and 0.001 cm respectively. Gold-cobalt versus copper thermocouples are used. Potential differences of about 0.1 μV can be detected. The thermocouple holders have a thin interior edge that gives line contact between the holders and the sample. The actual distances are measured by a vernier height gauge with an accuracy of about ±0.0005 cm.

For improvement of the contact between the specimen and the heater, mercury is used for metals and a large copper washer for plastics. The guard is divided into three zones; the bottom, centre and top of the shield are used as matching points. The thermocouple error is estimated to be less than ±1% over most of the temperature range. The precision is ±0.1%.

R.L.Powell is now working on an improved device and hopes to achieve far better results. He will use a guard with four heaters and the same size of specimen will be used. Temperature measurements are provided at eight points, avoiding any heat losses by the thermocouples.

The apparatus will have a floating heat-sink for achieving every temperature between liquid helium and liquid nitrogen. In this way temperature differences of only 10-15°K can be realised over the whole length of the specimen.

Electrical measurements will be done on the specimens at the same time.

These measurements of thermal conductivity will define a new limit for practical accuracy in thermal conductivity measurements.

All methods of determining thermal conductivity at high temperatures should be regarded critically on the basis of this error analysis for low temperature measurements.

STATUS OF THE HIGH TEMPERATURE APPLICATION

Because of the impossibility of compensating completely heat losses due to radiation, the longitudinal method can only be used up to about 1000°C. For high temperature application Flynn's equipment (Lit.4) is found to be the most exact. It is used from -150°C to 1100°C and an accuracy of ±1.1% at 100°C and ±1.0% at 900°C is claimed. For temperature measurement Pt versus Pt 10Rh thermocouples are employed. The specimen is a bar of 2 cm diameter, about 18 cm long. This method is combined with another using direct heating. Therefore the specimen is machined with a neck (see

Section 2.2.1.4 and Vis.40). The guard is regulated by three narrow heaters, one at each end and one in the centre. In addition, the guard is placed in an isothermal furnace. The space between the specimen and the guard is filled with alumina powder of very low thermal conductivity and low density.

(c) *Alternative design of the longitudinal method*

In a longitudinal method with compensated heat flux, H.E.Schmidt (Vis.7) has developed a modified apparatus without a guard. The method uses two miniature furnaces attached to the faces of a cylindrical sample, about 6 mm in diameter and 1 mm thick. The conductivity of the specimen is determined by measuring the electrical power necessary to compensate for a given temperature gradient across the sample. The temperature is measured directly at the interfaces between the specimen and the heaters. This method can be used from 20 to 1000°C with an accuracy better than ±5%. The application is restricted to materials with low conductivity ($\lambda < 0.2$ W/cm °C) (Lit.20).

(d) *Application of the longitudinal heat flow methods*

No further details on conventional longitudinal heat flow apparatuses are reported here, but a critical analysis of the longitudinal method is given by Laubitz in Lit.15. Table VI (p.90) gives a survey of institutes using longitudinal heat flow methods.

(e) *Summary*

The longitudinal heat flow method is commonly used for low temperature thermal conductivity measurements, but even for temperatures up to 1000°C a surprising accuracy of ±1% can be obtained. This method gives, at low temperatures, the most accurate values of all thermal conductivity devices.

The higher the temperature the greater are the difficulties, because of the radial heat flux which cannot be sufficiently controlled by guarding. Therefore the temperature range of practical use is below 800 to 1000°C. Nevertheless corrections for radial heat flow are necessary at temperatures greater than 400°C. The use of specimens with large diameters is suggested.

Because of the one-dimensional heat flux this method can be used for both isotropic and anisotropic materials. For determination of the temperature only, thermocouples are used.

Longitudinal methods are not applicable in the range above 1000°C which is of special interest, but they are valuable for really low temperature measurements, which are of fundamental importance in theoretical studies of thermal conductivity (see Section 5).

2.2.1.2 *Radial Methods*

(a) *Principles of the method and design of the apparatus*

In the radial methods a known radial heat flow is passed through a thick-walled hollow cylindrical specimen, in either the outward or the inward direction, and the temperatures in the specimen are measured at different radii. The heat flux is determined either by measuring the electrical power dissipated by the central heater (radial outflow method) or by measuring the amount of heat absorbed in a central calorimeter in a given time (radial inflow method).

The solution of the heat transport equation for radial heat flow is

$$\lambda = \frac{\dot{q} \log_e (r_2/r_1)}{2\pi(T_1 - T_2)} , \tag{2}$$

where $\dot{q}$ is the rate of heat flow per unit length of a cylindrical specimen and r_1, r_2 are the radii where the temperatures T_1 and T_2 are measured (at the same cross-section).

From Equation (2) it follows that only the ratio of distances is necessary for the calculation of the thermal conductivity, not the absolute values. Therefore no corrections for thermal expansion are necessary. See also D.L.McElroy (Lit. 18).

The measurement of heat flux is done in two different ways.

For "radial outflow", refractory metals are used as electrical heaters and their melting points limit the upper temperature of application of this method. In materials that are good conductors, difficulties arise at high temperatures from overcharging the heater in maintaining sufficient temperature gradients within the specimen.

In the "radial inflow" method the external electrical heater is not so liable to overcharging; temperatures up to 3000°C can be measured while maintaining a sufficient temperature gradient in all materials. The main disadvantage of this method results from difficulties with the calorimeter and its exact positioning within the sample hole. This is the reason for the diminished reproducibility and accuracy of the determinations. With larger temperature gradients, a further problem may arise from the thermal stress within the specimen, which is of particular concern with brittle materials.

In both methods, inflow and outflow, heat losses from axial heat flux must be kept small. Therefore the specimen is normally composed of a pile of discs, with axial guards at the ends of the pile. The high contact resistance at the disc interfaces prevents axial flow, which Pears (Vis. 42, Lit. 21) estimates to be only 0.08% of the radial flow. The axial flow is further reduced by the use of insulating discs in the pile and the proper choice of specimen dimensions (Feith, Vis. 33, Lit. 11).

Temperature measurement is achieved with thermocouples or optical pyrometers, and gives rise to the same problems as those mentioned in Section 2.1.

A severe problem is the deviation from concentric isotherms, and therefore several temperature holes must be used. Disturbances of the heat flow by the temperature holes can be made very small by the use of surface grooves for the thermocouples extending radially from each hole (Fulkerson, Vis 41, Lit 22). A very accurate determination of the radii is possible when the sight holes are drilled radially inward and by measuring the temperatures of the bottom of the holes pyrometrically (F. Patrassi, Vis 8, Lit 159). The corresponding temperature holes are usually placed on straight lines radiating from the centre. Laubitz (Vis 25) suggests that they be positioned randomly around the centre, resulting in less disturbance of the heat flow.

(b) *Status and accuracy of measurements*

Much effort is being put into the further improvement of radial methods for high temperature use, mainly in the United States. Three typical examples of application are now given.

(i) The outflow method for medium temperatures

The most accurate application of the outflow method for medium temperatures seems to have been realised by McElroy's group (ORNL, Vis.41, Lit.22). Samples of 75 mm diameter, with a central hole of 15 mm and a thickness of 13 mm, are used. The internal heater is made of Pt/Rh wire wound on an alumina tube. Temperature measurements are made with Pt/PtRh and WRe5/WRe26. This method has been used successfully for several materials (Si, Ni/Cr, C, Fe, Al, UO_2) up to 1000°C. The heat input can be determined very precisely (i e. ±0.4%) and the accuracy of the experiments is ±2% at 1000°C.

(ii) The outflow method for very high temperatures

The GEC group in Cincinnati (Feith, Vis.33, Lit.11) has used the outflow method with a tungsten heater for measuring thermal conductivity in refractory metals and oxides up to 2300°C. The samples have a diameter of 50 mm. If molybdenum is used as a heater material a better heat transfer to the sample is needed because of its lower melting point. For the highest temperatures, up to 2500°C, rhenium heaters are proposed. The temperature gradient can be measured within ±3% in a distance of 10 mm. The precision of the thermal conductivity measurements is given as ±6%.

(iii) The inflow method for the highest temperatures

The radial inflow method is used in several establishments, based on the concept of McClelland's apparatus (Lit.3). As a typical example the method of Pears and Pyron (SRI, Vis.42, Lit.21) is described. They use a graphite tube furnace of about 60 mm diameter and samples about 25 mm diameter and 25 mm long with a central hole of 7 mm diameter. The calorimeter of stainless steel is provided with a spirally wound thermocouple, producing turbulence in the water flow. A good contact between specimen and calorimeter is achieved by copper or graphite powder. An accuracy of ±7 to ±12% is claimed for temperatures from 600°C to 2750°C. Temperatures are determined by optical pyrometry. This apparatus enables measurements on pyrolytic carbon plates to be performed.

(c) *Application and limits of radial methods*

Radial methods can be used to very high temperatures. Radial inflow apparatuses are operated to 2800°C, while radial outflow apparatuses will work up to 2500°C (Lit.11). The upper temperature limit of the outflow method is determined by the maximum load that the central heater can carry. Higher temperatures can be reached with normal wires as heaters, using the apparatus of Moeller, who improved the emittance of his heater, a tungsten tube, by grooving it (Vis.30, Lit 23).

Inflow methods are limited by the heat transfer to the calorimeter and to the water flow respectively.

Some special ideas found in various laboratories for improving reproducibility are now listed. Among these are coating the tube with sand (Lit.3), a water turbulator by Hansen and Roisen (UCC, Vis.44) and rolling the thermocouple wires in the tube into a spiral (Pears, Vis.42). But so far the best way of getting good values is by training the laboratory assistant.

Radial methods are not only used for the highest temperatures. For medium temperatures, that is up to 1000°C, the outflow method is nearly as accurate as the longitudinal methods. The ORNL-group is now developing a further apparatus for vacuum up to 2200°C with tantalum heaters.

The temperature of application of the inflow method with carbon heaters can be increased by using pressure vessels up to 33 atm. as shown by UCC (Vis.44).

The accuracy is better with large samples but in this case many hours are needed to obtain steady-state conditions.

All radial methods require isotropic materials in the radial plane.

Table VII, on p.91, gives a survey of the institutions where radial methods were seen.

(d) *Summary of radial methods*

Summarising all the information, it can be stated that this method is used up to very high temperatures. To get precise values large samples should be used, which need a long time to reach steady-state conditions (at least a few hours). Concerning accuracy, the outflow type seems to be more precise achieving, in the medium temperature range at about 1000°C, almost the accuracy of longitudinal methods. For the highest temperatures (above 2300°), the inflow technique is preferred because of the danger of overheating of the internal heater in the outflow technique; however, the accuracy achieved is lower.

2.2.1.3 *Forbes Bar Methods*

The nomenclature follows M.J.Laubitz's proposal (Lit.15) in the chapter on linear and quasi-linear heat flow methods. According to Laubitz, in Forbes bar methods there is a transverse heat flow in the specimen in addition to the longitudinal flow. This radial heat flow is taken into account in the analysis of the heat flow problem. A variety of methods is described in the literature, but only a few modern ones are discussed here. These may be classified as follows:

(a) Methods which employ essentially guarded linear heat flow with a slightly mismatched guard.
(b) Methods based on "thin-rod" analysis.
(c) Methods which depend on a full two-dimensional analysis.

(a)

The method of Watson and Robinson (Vis.40, Lit.24) is mentioned as an example. It uses a guarded linear heat flow apparatus. The radial heat transfer coefficient is calculated from the distributions of the temperature in the specimen at two different temperatures of the guard, both similar to the temperature of the specimen. The correction for the radial heat flow is always less than 5% of the longitudinal one. Heat losses to the cold ends and to the surroundings are not compensated by this correction. Peavy's paper (Lit 160) gives an improved calculation procedure which does correct for all heat exchanges between the specimen and the surrounding insulation. This method is only described for the temperature range from -160 to 600°C, without comment on the accuracy obtainable.

(b)

The method of M J Laubitz (Vis.25) is typical for this group. The specimen, of 2 cm diameter and 20 cm length, with the heater in its centre is connected with two additional heaters at the ends, only to maintain the high temperature for the measurements, and is placed within a nearly isothermal furnace as a guard.

For the analysis of the heat flux equation two experiments are necessary. The first gives the temperature distribution caused by the central heater. In the second experiment, performed under similar conditions, the central heater is switched off and the temperature distribution due to a deliberate temperature mismatch between the furnace and the specimen is measured as a function of time, to determine the radial heat losses from the specimen.

This method works up to 1000°C. An accuracy of about ±1.5% is claimed. It was used for basic research on the validity of the present theories on thermal conduction in monovalent metals (M.J.Laubitz, Vis.25, Lit.25).

(c)

Based on a full two-dimensional analysis, Laubitz has described two methods, the "unmatched guard method" (Lit.26) and the "guardless method" (Lit.27).

In both of these methods central heaters in long cylindrical specimens are used and the temperature distribution is measured along the specimen under different steady-state conditions. In one of these methods the temperature profile within an axial hole of the specimen is determined by a movable thermocouple. Laubitz has simplified the experimental arrangement more and more, bearing in mind that the mathematical analysis becomes increasingly involved. In his two versions, the main advance is the rapid achievement of steady-state conditions. They were used up to 1100°C and an accuracy of ±1.9% and ±2.3% respectively is claimed.

Although the second method is used later for rapid measurements, the principle of the "thin rod" (see (b)) seems to be more promising for application at higher temperatures.

Summary

The Forbes bar methods use quite a simple experimental arrangement, but they require a complicated mathematical analysis for the evaluation of the thermal conductivity from measured quantities.

They include methods with a full two-dimensional analysis, and those that are very similar to the guarded longitudinal arrangement. Up to 1000°C, the same accuracy as with the longitudinal method is claimed for the best methods. Extension to higher temperatures should be possible.

2.2.1.4 *Direct-Heating Methods*

In this section only steady-state methods with direct heating by current of the electrically-conducting specimens are reported. For their analysis it seems adequate to discuss them briefly in three groups.

(a) Heated filament methods.

(b) "Necked-down" methods.

(c) Methods in which the temperature profile of radiating surface areas is measured.

Flynn (Vis.40, Lit.161) discusses, in his chapter of Tye's book on thermal conductivity, only those methods with a temperature drop in the direction of the current flow, that means only methods in groups (a) and (b). He divides the electrical heating methods into three categories depending on how the temperature rise in the conductor was measured.

(i) Kohlrausch's Method.
The temperature distribution along the conductor was measured by means of directly attached thermocouples or other thermometers.

(ii) Worthing's Method.
The temperature distribution along the conductor was measured by means of an optical pyrometer.

(iii) Callendar's Method.
The temperature rise in the conductor was computed from the change in electrical resistance of the electric current.

For a detailed discussion of the thermal and electrical potential distribution in the specimens and for the mathematical development, see Flynn's chapter (Lit. 161) in Tye's book.

(a) *Heated-filament methods*

These methods are characterised by the fact that the specimen has the shape of a long cylindrical rod or a filament, and are based on the well-known Kohlrausch analysis, assuming that all heat generated within the specimen is transported to its ends, both having the same temperature.

Kohlrausch's solution is restricted to the case where the temperatures of the specimen are not very different from that of the surroundings. Kohlrausch's method is usually used without a guard at room temperature or only slightly above. This method has been modified by Russian scientists (Mikrynkow, Lit. 28; Lozinskii, Lit. 29) for use at higher temperatures up to 700°C and 1500°C, respectively, using a furnace. This method produces the same difficulties as the use of a guard in the longitudinal methods. Within the NATO nations, this modification has been used up to 2000°C at the Central Laboratory of Sud-Aviation (Vis. 5) for the determination of the thermal conductivity of graphite, refractory metals and light metals.

The Method of Jain and Krishnan (Lit. 30) has been developed for high temperatures without using a guard. As in Kohlrausch's method, the temperature distribution along a long rod, whose ends are at ambient temperature, is measured. The radial heat loss by radiation is taken into account by comparing the temperature in the middle of a short rod with that of a very long rod of the same material. Both are heated by the same current. If the emittance of the sample material is known, its thermal conductivity can be calculated.

The method can be used up to the melting temperature of the specimen. Unfortunately, up to now the results do not agree well with those obtained by other methods. It is necessary to consider whether the boundary conditions are fulfilled (T_{max} of an infinitely long rod) and how the result is affected by uncertainties in the determination of the emittance.

In a Russian paper dealing with an analogous design, Lebedev (Lit. 31) uses a short and a long filament of the same material and measures the current necessary to reach the same central temperature in both wires. The emittance does not enter into the calculation if it is the same for both filaments.

A promising method has been developed by Bode (Vis. 10, Lit. 32). In contrast to the other methods mentioned, the ends of his specimens are heated by additional heaters to match them to the central temperature of the filament. The thermal conductivity is determined from the dependence of the electrical resistivity of the wire on temperature and current. In Bode's method the thermal conductivity at a distinct temperature, and not within a temperature interval, can be determined. The experimental arrangement ensures that the re-radiation to the specimen is kept small. The emittance can be determined, in addition. This method also offers the possibility of measuring the emittance if there is no back radiation to the specimen. The error caused by the Thomson effect and by radial heat flux can be neglected. The method is used up to 1100°C with a total error of a few per cent.

This method is being further developed for use in the temperature range 800°C to 2000°C. An essential improvement is achieved by maintaining a constant stress in the specimen wire. The new apparatus is constructed in such a way that it is also applicable to other direct-heating methods (Kohlrausch; Jain and Krishnan; Holm and Störmer).

In all these filament methods an error may be caused by a volume Peltier effect which is observed when a direct current flows through regions of inhomogeneous composition (Birkholz, Vis. 12A). The use of alternating current is therefore recommended.

The method of Powell and Schofield (Lit. 33) uses the radial temperature distribution in an electrically heated wire or rod for the calculation of the thermal conductivity. In this case the electrical energy dissipation within a given radius r is set equal to the heat flowing radially out of the cylindrical surface of radius r. The precision indicated is about 5 to 10%. This principle was also used by Angell, Rasor, McClelland and Scott (Lit. 18).

Although Powell and Schofield reported the best values of the thermal conductivity of graphite up to 2700°C, determined with this method twenty years ago, I could not find anybody who was using this method today. The influence of the emittance on the accuracy of temperature measurement seems to be the main problem.

According to Rasor and McClelland (Lit. 3) the error caused by the emittance can be neglected above 3000°C when using graphite specimens.

(b) *"Necked-down" methods*

The principle of the "necked-down" methods is the use of short specimens with a narrow constriction in the mid-section. In this way, heat losses by radiation and convection become negligible compared to the heat flow to the cold ends. According to Hopkins this is not true with hydrogen as the surrounding gas (Lit. 34).

Assuming the electrical and thermal potential fields to be equivalent, the Lorenz factor can be calculated directly. With the knowledge of the electrical resistivity, the thermal conductivity can also be obtained.

This method was first used by Holm and Störmer in 1930 (Lit. 35). Nowadays there are two developments in this field, using somewhat different techniques.

Hopkins (Vis. 24, Lit. 34) uses the voltage drop across the neck and the maximum temperature of the bridge (T_{max}) measured pyrometrically, to determine the Lorenz number. This method can be used up to temperatures above the melting point, because surface tension will keep the material of the neck in place even if it is molten.

Flynn (Vis. 40, Lit. 4) determines the neck temperature by using the neck of the specimen as a resistance thermometer. He used this method up to 1100°C and claimed the following accuracy: ±1.9% at 100°C and ±2.35% at 900°C. Compared with the results of the longitudinal apparatus (see Section 2.2.1.1), the values obtained by the "necked-down" method within the same specimen show an error of ±0.4%.

(c) *Methods in which the temperature profile of radiating surface areas is measured*

The rectangular bar method, proposed by Longmire, is critically reviewed and used in the Parma Research Laboratories of the Union Carbide Corporation (Vis.44, Lit.36).

The thermal conductivity is determined from measurements of the temperature profile across the surface of the directly-heated specimen in the shape of a rectangular bar. This method is especially apt for the determination of the thermal conductivity of graphite because the important anisotropy ratio can be obtained additionally by recording the temperatures of both the square cross-sections. The measured power input allows determination of the total hemispherical emittance. In the mathematical treatment of the method the longitudinal heat flux to the electrodes in the measuring section is neglected. The method is used up to 2800°C. The precision, as indicated by the data spread, is ±5 to ±10%.

For the "Hoch" method (Vis.30, Lit.37) the equation of heat conduction has to be solved for a finite cylinder, heated in a vacuum by induction from the outer side-wall surface. The heat will flow into the interior by conduction and dissipate by radiation from the planar faces of the specimen. In steady-state conditions, the temperature profile in the planar surfaces is measured. For calculation of the thermal conductivity it is necessary to know the emittance. The ratio of specimen specific heat to emissivity can be determined from the rate of cooling after switching off the power input. From this measured ratio and values for the specific heat from the literature the emittance is calculated. Most data are accurate within ±10 to ±20%, but the overall accuracy is claimed to be ±6%. The method can be used up to the highest temperatures (the temperature of destruction of the specimen).

Concluding remarks on the direct-heating steady-state methods

Considering that these direct heating methods can be used only for electrically conducting materials, there are so many advantages, especially in the high temperature range, that it is surprising that they have not yet found greater application.

The advantages are: the heat generated in the specimen is well known, there are no difficulties with thermal contact between heat source and specimen, no chemical reaction with the environment, only a few specimen materials are needed, steady-state conditions are reached rapidly, limited only by the melting point of the material, and there is the possibility of measuring additional properties using the same experimental set-up.

For a detailed discussion on the pros and cons of electrical heating methods see, for example, Flynn's chapter in Tye's book (Lit 161). Briefly, Flynn states that, with the possible exception of very low temperatures, where electrical heating methods have not been used, a properly designed electrical heating method can yield thermal conductivity values of an accuracy equal to or better than those obtainable by any other methods of measurement. In many cases electrical heating methods would be cheap and quick. He also points out that, up to 1000°C, direct-heating methods may be preferred while, for higher temperatures especially, Bode's arrangement seems to be promising.

If the materials can be made in the form of homogeneous rods or wires, an experimental study to compare all these electrical heating methods should be undertaken, in order to clear up the validity of the assumptions for the mathematical solutions and the effectiveness of the experimental arrangements.

A similar opinion was found at the Thermophysical Properties Research Center at Lafayette, USA (Vis.43). This centre has worked out a programme for the US Air Force with the aim of comparing the most important of the direct-heating methods. Also, at SERAI (Vis.1) in Belgium, a similar test programme has been developed and the various possibilities have been studied. Finally, at the PTB (Vis.10) at Braunschweig, Germany, further improvement of special direct-heating concepts is proposed.

All these activities should be sponsored and coordinated.

2.2.2 *Comparative Methods*

In a comparative heat flow apparatus a specimen of unknown conductivity is connected in series with a specimen of known conductivity and the temperature difference is measured under steady-state conditions in both specimens. Thus the heat flux can be calculated without directly measuring it. This is the essential advantage of the comparative methods. The accuracy of these methods depends on the availability of suitable standards, which limits their application. The problem of finding standards for all ranges of conductivity and of temperature is discussed in Section 6. So far no suitable standards for the higher temperature range are available. The long discussion that has taken place over the years on the thermal conductivity of Armco iron and platinum is an example. Comparative methods may become more important in the future for high temperature measurements if the necessary standards become available. The methods are now discussed briefly.

(a) *The comparative method of SNECMA, with longitudinal apparatus, applicable up to 2000°C (Vis.4, Lit.38)*

In this method a pile of specimens and standards is used in a longitudinal arrangement connected to a graphite heater on the top. This pile is surrounded by a tube made of the same material as the pile and connected to the same graphite heater on the top of the stack. The whole arrangement is insulated by an Al_2O_3 Pt guard. With this arrangement transverse heat losses are largely eliminated. All temperatures are measured by thermocouples using Chromel vs Alumel, Pt vs Pt/Rh and W vs W/Re with BN insulation.

Results of the thermal conductivity of UO_2, UC, BeO, C and W are available up to 2000°C. An accuracy of ±5% is claimed using the thermal conductivity values of Armco iron given by the American specialists (see Section 4).

The difficulties of thermal contact within the pile are overcome by "scooping" the interfaces of the specimen.

(b) *The Battelle self guarding method (Vis.29)*

The Battelle method can be used up to 2400°C with an accuracy of ±5%. Contrary to the SNECMA method, with a pile of specimens, the Battelle method uses specimens in the shape of discs with a longitudinal heat flow and Ta heaters. The reference sample is larger in diameter than the specimen. Thus, and because of the small height of the sample to be measured, the radial heat losses are minimised without using any guard. Only a thermal insulation of BeO powder is used.

(c) *The DEW longitudinal device (Vis.6, Lit.39)*

This method represents a classical longitudinal arrangement with a heated guard. It will work up to 700°C. Specimens in the shape of rods are used and the heat flux is determined across a nickel standard, placed between the specimen and the heat sink. The repeatability is ±5%.

(d) *The British Ceramic Research Association method (Vis.10)*

This method, using a longitudinal arrangement and a pile of specimens and standards in the shape of discs with a non-heated guard, is mentioned because of its peculiarity, that copper discs are placed between the specimen and the adjacent standard. The thermocouples are soldered to the copper discs and their temperature is measured. This procedure is very convenient when dealing with brittle or very hard materials, where thermocouples are difficult to attach. The sequence within the stack is heater, copper, standard, copper, specimen, copper, standard, copper, sink.

2.2.3 *Survey of Methods for the Measurement of Thermal Conductivity*

Summarising the steady-state methods for the determination of thermal conductivity it is useful to review the suitable temperature range of application.

The low-temperature range (up to 900°K)

Longitudinal methods are best for the lowest temperatures. The greatest accuracy is better than ±1%.

The medium-temperature range (900 - 1300°K)

Several methods can be used, with nearly the same accuracy. These are as follows:

(a) Longitudinal methods, accuracy ±1.1%, the temperature of 1300°K being the upper limit of application.

(b) The radial method, accuracy better than ±2%, becoming more and more superior at higher temperatures to longitudinal methods.

(c) The Forbes bar methods, accuracy ±1.5%, with the simplest experimental arrangement but the most complicated method of calculation. Forbes bar methods should also be applicable at higher temperatures.

(d) Direct-heating methods, with heated filament or "necked-down" arrangement, with accuracies of about 2 or 3%.

The high-temperature range (above 1300°K)

For this temperature range only radial methods and direct-heating methods are applicable. While much research is being done in developing radial methods for the highest temperatures (up to 3100°K), the development of direct heating methods seems to have been neglected up to now.

The accuracy in the determination of thermal conductivity at the highest temperatures is less than ±7%, perhaps ±10%.

For all these temperature ranges, comparative methods could be used if suitable standards were available. Therefore the programme on high temperature standards is of the greatest importance. Furthermore, the work on standards is important to permit comparisons of the results obtained using an absolute method with the work of other investigators.

2.3 Measurement of Thermal Diffusivity

The thermal diffusivity "a" is defined by the heat flow equation as follows:

$$\frac{\partial T}{\partial t} = a\left(\frac{\partial^2 T}{\partial x^2} + \frac{\partial^2 T}{\partial y^2} + \frac{\partial^2 T}{\partial z^2}\right), \tag{3}$$

where T is the temperature, t the time and x, y, z are Cartesian coordinates. The propagation of heat is described by the change of the temperature in the material as a function of time. The dimension of the diffusivity is $(\text{length})^2(\text{time})^{-1}$.

The diffusivity is correlated to the thermal conductivity by

$$\lambda = a \gamma c_p , \tag{4}$$

where γ is the density and c_p the specific heat of the material. If the density and the specific heat are known, the thermal conductivity can be obtained by measuring the diffusivity.

This simple equation (4) does not consider the effect of relaxation time. Therefore further oscillations of temperature can be caused by an impulse of heat. The well-known phenomenon of second sound in superfluid helium is an example of the effect of long relaxation times.

Finally, mass transport can be caused by temperature gradient (Soret effect), which ceases after reaching a certain concentration gradient. As a result the initial and the final thermal conductivity in the material can differ significantly.

As shown by the restrictions mentioned, the relation between conductivity and diffusivity can be more complicated than that given by Equation (4). As a consequence, conclusions drawn from steady-state measurements as to the behaviour in time-dependent processes, or from transient measurements to the steady-state behaviour, should be controlled by experiments with the same material

Generally, the measurement of thermal diffusivity in solids is more than an indirect way of determining the thermal conductivity. It is an additional means of studying transport phenomena, apart from its importance in technical applications.

Despite these restrictions, transient methods are widely used for the determination of the thermal conductivity because the specific heat and the density of solid materials can be measured more easily than thermal conductivity, even at high temperatures. The transient methods will be distinguished in this report as "non-steady-state" and as "periodic" ones. In transient techniques the response of a change in temperature in a sample exposed to a given transient heat flux or heat pulse is measured. For the evaluation of experimental results on diffusivity it is not necessary to know the absolute level of the temperature and the quantity of the heat flux

Small specimens may be used. The time required for measurement is very short

2.3.1 Non-Steady-State Methods

2.3.1.1 Flash methods

Generally non-steady-state methods are known as flash methods. The specimen, a thin plate (diameter $\simeq$ 10 mm, thickness = 0.5 - 5 mm), is heated indirectly and homogeneously in a tubular furnace. A short pulse of light is sent to the front of the specimen, which becomes additionally heated by absorbing this flash. The temperature increase at the rear face of the specimen is recorded by either thermocouples, cells of semiconductors or by multipliers. The recorded temperature change with time allows the evaluation of the thermal diffusivity.

Simple Xenon flash lamps are used as light sources for measurement up to 1600°C. For higher temperatures, laser sources with an output energy of about 20 joules must be used.

Mathematical solutions for the flash method have been given by Parker et al. (Lit.41), Taylor and Cape (Lit.42, 43) and Cowan (Lit.44). According to Parker and his co-workers, the thermal diffusivity can be calculated either from the time needed to reach half the maximum temperature at the rear face, $t_{1/2}$, or from the characteristic rise time, t_c (that is, the slope of the temperature with time), by the following equation

$$a = \frac{1.33\ d^2}{\pi^2\ t_{1/2}} = \frac{0.48\ d^2}{\pi^2\ t_c}\ , \tag{5}$$

where d is the thickness of the specimen.

This solution is valid if the following conditions are fulfilled:

(i) Heat flow is only in one direction (no radial heat flow).
(ii) Constant thermal properties within the material tested.
(iii) Homogeneous absorption of the heating pulse in an infinitely thin layer at the front of the specimen.
(iv) Infinitely short heating pulse.
(v) No heat losses by radiation.

In most cases these conditions cannot be realised completely, especially at high temperatures.

To satisfy the first condition the temperature is generally measured in the centre of the rear face of the specimen, where one-dimensional heat flux predominates.

The second condition holds in most cases, since the temperature rise caused by the pulse (only a few degrees) is only a negligible fraction of the temperature of the specimen.

As to the third condition, a coating at the front of the specimen may be useful if the material does not absorb the light in a thin layer.

Concerning the fourth condition, the error can be neglected if the pulse time is small compared with the characteristic thermal diffusion time t_c. This can be accomplished by increasing the thickness d of the specimen.

Cape and Lehman (Lit.42) have shown, that Equation (5) is valid within ±5%, without corrections for heat losses, if the condition

$$\frac{\lambda}{\epsilon d} > 1 \text{ cal/cm}^2\text{sec}^{\circ}\text{K} ,$$

is fulfilled, where ϵ is the emittance.

This means that, if materials with low thermal conductivity are tested, the thickness d should be kept as small as possible. For good conducting materials, the same becomes necessary at high temperatures, because their thermal conductivity decreases. Therefore most research organisations use very thin specimens for the flash methods. The thin specimens, however, are more sensitive to the effect of finite flash time (condition (iv)). Bollenrath (Vis.11) suggests solving this problem experimentally by using pulses of different duration and extrapolating to infinitely short time.

As to the fifth condition, several methods have been proposed to account for radiation losses from both surfaces of the specimen, leading to the derivation of a radiation loss factor.

According to Cape (Lit.42) the radiation factors are applied as perturbations to Equation (3), neglecting re-radiation from the surroundings.

Cowan (Lit.44) calculates the heat losses from both surfaces. The solution for the time-dependence of the temperature is given graphically. The following parameters R and A are introduced as corrections:

$$R \simeq \left(\frac{T_R}{T_F}\right)^3 , \tag{6}$$

where T_R is the rear face temperature and T_F the front face temperature.

$$A \simeq d_s(1 + R)\, \epsilon \left(\frac{T}{1000}\right)^3 \frac{1}{\lambda} . \tag{7}$$

The factor A can be determined experimentally from the cooling portion of the temperature history curve at the rear surface. According to Cunnington (Vis.36, Lit.45, 157) who uses the method described by Cowan, the effect of R is relatively small (less than ±4%).

With graphite or tungsten resistor-furnaces, a laser equipment as source for the flash and a multiplier as detector, this method can be used up to 2800°C. However, the exact correction of the experimental results for heat losses by radiation at these high temperatures has not yet been determined.

Table VIII, on p.93, lists institutions within the NATO nations using the flash method. The precisions given are not always well defined and are therefore not comparable directly. A critical analysis of the different methods, based on comparative measurements, would be a task for a team of specialists.

2.3.1.2 Other non-steady-state methods

For many years a variety of methods has existed for using temperature distributions in a heating or cooling process to calculate the diffusivity. Because of the development of the more elegant methods using small specimens, like flash methods and the periodic ones, these simple transient non-periodic methods are only rarely used. Two examples may be given:

(a) The transient radial inflow method of Cape, Lehman and Nakata (Vi.28) uses cylindrical specimens heated by induction in the outer sidewall surface. The temperature rise in the inner and outer part is recorded. The method is used up to 2500°C and an accuracy of ±5% is claimed.

(b) The same principle has been used by Huttinger (Vis.12B, Lit.46) in studying the baking of carbon bodies between room temperature and 1000°C. The specimen is slowly heated at a constant rate in a tubular furnace and the radial temperature distribution is measured as a function of the time. The recorded change of the radial temperature distribution allows the calculation of the thermal diffusivity as a function of temperature. A similar method has been described by H.W.Flieger for the investigation of moderately poor conductors (Lit.162). The accuracy claimed is ±1% in the temperature range from 70 to 1000°C.

2.3.2 Periodic Methods

In the periodic methods, a periodically varying heat flow is generated within the specimen. This is achieved by modulating the energy of a Joulean heater or of an electron beam or of an arc-image or by the re-radiation of the heat losses of the specimen. In most cases no additional heating is necessary. The variation of the temperature at different positions along the direction of heat flow within the specimen is observed and the thermal diffusivity can be calculated from these experimental results.

The different solutions of the general heat flow equations are dependent on the boundary conditions prescribed by the experiment.

The following cases will be discussed:

The Angstrom method, using direct resistance heating

The electron beam and arc-image methods, based on Cowan's principal calculation.

The periodic rate of cooling method, using heating by an arc-image

The disc-methods of Kaspar, with indirect heating of the specimen in a furnace

2.3.2.1 The Angstrom method

A hundred years ago Angstrom (Lit.47) developed his classical method. A thin long rod is heated sinusoidally at one end and the propagation of the temperature wave is measured at different points along the rod.

If the assumption of a semi-infinite rod and a sinusoidal temperature change is made, the thermal diffusivity may be determined either by measuring the ratio of the amplitudes (D) of the temperature wave at two points or by their phase difference [illegible]

If both $\Delta\phi$ and D are determined, the thermal diffusivity can be calculated at high temperatures without special allowance for radial heat losses.

$$a = \frac{\omega \Delta x^2}{2\,\Delta\phi \log_e D} \qquad (8)$$

ω is the angular frequency of the temperature variations and x the distance between the measuring points.

In practice is may be difficult to produce a pure sinusoidal temperature variation at the source, although it can be realised mechanically (Birkholz, Vis.12A). Goldsmid (Vis.23) uses the Peltier effect (see also Green and Cowoles, Lit.48) with advantage, because of the symmetry of the temperature waves.

If the initial wave is square or not truly sinusoidal, a Fourier analysis of the temperature variation at a point may be carried cut and attention can then be confined to the fundamental frequency. The higher harmonics can be neglected, for they are rapidly attenuated.

The amplitude of the temperature oscillation generally used is smaller than ±5°C; the frequency is about 1[min^{-1}].

Table IX (p.94) lists the institutions using the Angström method. As shown, this method has been used up to 900°C only.

According to Birkholz (Vis.12A) this method is suitable for higher temperatures and the theoretical conditions can be realised experimentally quite easily. The equipment is simple and cheap. This method should be considered and discussed by a team of specialists.

2.3.2.2 Periodic methods based on Cowan's analysis

Cowan (Lit.51) has presented a theoretical study on the modulation of the temperature in a specimen, when one face is periodically heated.

Based on this calculation, several methods of determining the thermal diffusivity have been developed. Generally, the specimen is a thin solid plate, a sheet or a disc, similer in shape to the specimens used in the flash method. The front face of the specimen is heated directly by electron bombardment or by the radiation from an arc-furnace. No additional indirect heating is necessary. The change in temperature of the faces during the modulated heating is recorded photoelectrically.

Cowan's treatment allows calculation of the thermal diffusivity from the phase shift between the temperature variations on the front and rear faces, between the beam modulation and the temperature variation on the front or on the rear face of the specimens. In addition thermal diffusivity may be calculated from the attenuation of the temperature wave within the specimen. In practice, these solutions are used in the form of nomograms.

For all these calculations of the temperature distribution within the specimen, the one-dimensional differential equation is used, neglecting heat flux in the direction parallel to the faces. Cowan further assumes that the difference in heat loss from the faces of the specimen is proportional to their difference in temperature and that the energy is absorbed in an infinitely thin layer. A correct calculation of the diffusivity of a specimen from the measured attenuation or phase shift is possible only when the heat loss parameters - in the same way as for the flash method - are known. Cowan has shown how these parameters can be determined.

Cowan's solution seems to be correct, but the accuracy for the experimental determination of the loss parameters is only a few per cent so far.

(a) *Heating with an electron-gun*

This method is practised in only three research establishments (see Table X, p.95). By using an electron-gun, the samples can be heated up to the temperatures of melting or vaporisation. This means that the temperature is limited only by the temperature of destruction of the specimens. The heating must be carried out in a high vacuum ($\leqslant 10^{-4}$ mmHg). This is a disadvantage for materials with high vapour pressure. Modulated heating can be attained very easily and accurately by modulating the electron beam power. The reproducibility is very good and scientists using this method are convinced that, after further development, determination of the thermal diffusivity should be possible within about 2%, even at very high temperatures.

(b) *Heating with an arc-image or plasma*

An arc-image equipment for the periodic heating of carbon and graphite specimens has been used with success for many years in the USA (UCC, Vis.44). No vacuum is necessary. The modulation of the heating power is realised by intermittent light, with rotating shutters between light source and specimen. In France (SNECMA, Vis.4) new equipment has been developed in which a plasma (5900°C) is used as a source of radiation for the additional heating of one end of a small rod specimen. The sinusoidal modulation is done by a "persienne". The specimen is heated basically over the whole length, as well as at the end, by a tubular graphite furnace The temperature wave is measured at two points along the rod through a hole in this furnace (the Angström arrangement).

2.3.2.3 *Rate of cooling method*

This special method was developed in the UCC Research Laboratories, Parma, by Null and Lozier (Vis.44, Lit.52).

An arc-image furnace is used for heating. The small cylindrical specimens, a few mm in diameter and 10 mm in length, are heated periodically by interrupting the beam of light by means of an electromagnetically controlled shutter No additional heating is used. The surface temperature of the specimen is recorded during the cooling interval Analyses of the problem, with the assumption of one-dimensional heat flow along the axis of the sample and short cooling intervals, show that the heat flow equation can be solved as follows

$$\frac{\partial T}{\partial t} = -2\frac{H_s}{r(\rho c_p)}, \qquad (9)$$

where γ is the density, c the specific heat, λ the thermal conductivity, Δt the decrease in surface temperature, t the cooling time and H_0 the flux absorbed at the surface of the specimen, which is measured radiometrically.

As the temperature decrease is only a few degrees within a cooling time of only 30 msec the use of Newton's law seems to be justified. This method gives only values of the product $\lambda c \gamma$ but, in combination with Cowan's phase shift method, where $\lambda / c \gamma$ is determined, the specific heat may be eliminated. The method is used for graphite in the temperature range of 2400°K to 3300°K. The reproducibility reported is ±14%.

2.3.2.4 *The "disc methods" of Kaspar*

As mentioned earlier the main problem in the measurement of the thermal diffusivity at high temperatures results from uncontrolled heat losses by radiation. Kaspar (Vis.26) avoids the radiation losses by an appropriate experimental arrangement. He has developed two methods, the "double disc method" (Lit.53) and, as an improved technique, the "single disc method" (Lit.40).

The specimen is heated indirectly in a tubular furnace. At the end of the tube mirrors reflect radiation from the specimen back to the specimen. This reflected energy is modulated by means of rotating blades.

In the double disc method a pair of discs is exposed at their outside faces to identical periodic flux variations. The thermal diffusivity is determined from the amplitude ratio of the temperature variations on the front and back of one of the discs. The main difficulty of this method is the detection of the very small temperature differences of some tenths of a degree at 2000°C or more. According to Kaspar a sensitivity of about 10^{-3} °C may be achieved.

In the double disc method, the amplitude and phase of the periodic temperature variations on the front of both discs must be identical. This is difficult to achieve, and so the single disc method was developed. In this method a shield with a tolerably low thermal load impedance is used to avoid heat losses in the specimen and Kaspar used stacks of very thin tantalum carbide elements with success.

These methods have been used so far in the temperature range 1500°C to 3000°C, with a graphite tube furnace and an inert atmosphere. The sample dimensions are: diameter 25 mm and length 2 mm. The accuracy seems to be about ±10%.

2.3.2.5 *Summary of periodic transient methods*

The periodic transient methods seem to be very promising for application at high temperatures. First developed by Angstrom one hundred years ago, more modern methods are available to-day such as the direct heating by electron bombardment, by radiation from an arc-image or a plasma, or by re-imaging the radiation of the indirectly heated specimen itself. All these new modifications with periodic heating and cooling are applied in practice for the measurement of refractory materials up to the highest temperatures, i.e. up to the temperature of destruction of the specimens.

In comparing the advantages of the special modifications, the electron gun technique offers advantages because it permits, by simple means a perfect sinusoidal modulation of the heating on one hand, and has a wide range of experimental possibilities for varying the heating and measuring conditions on the other hand. The users of this technique are convinced that they are able to develop this method further into a high precision technique. The disadvantage is in the necessity for high vacuum, which makes application of the method impossible for materials with high vapour pressures and high evaporation rates. For such materials the periodic heating by radiation using an arc-image or a plasma is advantageous. In these cases the modulation must be performed mechanically.

Since Kaspar's method does not necessitate heat loss corrections, it seems the most appropriate for very high temperature work.

2.3.3 *Summary of Thermal Diffusivity Measurements*

With modern transient methods, measurement of the thermal diffusivity may be performed on small specimens and in a short time. For most non-stationary methods the experimental set-up is simpler than for thermal conductivity apparatus, but the source of energy is very expensive in most transient methods. However, the accuracy achieved is not yet as high as with the steady-state methods. With further development of the measuring techniques an accuracy of better than ±5% might be reached, even at very high temperatures. Only for the Angström method has a reproducibility of better than ±2% been claimed. The Angström method should be highly promising for high temperature application, too. Generally, the transient methods can be used up to the temperature of destruction of the specimen. A great difficulty is the exact determination of the heat loss parameters, knowledge of which is necessary to calculate the diffusivity of the sample material from measured data.

Another method is that of Kaspar, who suppresses radiation losses experimentally. This method is highly promising, but further development is necessary.

Apart from their unique application at very high temperatures, some of the methods can be used down to low temperatures, too, resulting in a wide range of application temperatures.

This is true for the Angström method as well as for the electron-gun method, when IR detectors are used (Wheeler, Vis 21).

When thermal diffusivity measurements are used indirectly for the determination of the thermal conductivity, the specific heat must be known as accurately as possible (see Section 2.4).

2.4 Measurement of Specific Heat

As shown in Section 2.3, knowledge of the specific heat is necessary for the calculation of the thermal conductivity from thermal diffusivity measurements. Therefore, techniques for determining the specific heat, especially at high temperatures, are considered.

The specific heat is temperature-dependent. The method most used is the "drop-calorimetry" method, which essentially gives values of the enthalpy of the specimen at the temperature of the heated specimen relative to that at the temperature of the calorimeter. By keeping the same calorimeter temperature, but making runs at different furnace temperatures, the relative enthalpy can be found as a function of temperature.

The adiabatic calorimeter is adequate for the determination of the specific heat at very small temperature intervals, but it is more complicated. Furthermore, some special techniques, such as the pulse heating method, exist.

2.4.1 Adiabatic High-Temperature Calorimeters

Generally, the specimen, heated under adiabatic conditions, serves as its own calorimeter. The specimen, sometimes enclosed in a close-fitting capsule of non-reacting material is heated by an internal electrical heater in a sensitively controlled environment and the temperature rise in the sample caused by an additive energy input is measured.

The specimen and its container are surrounded by an adiabatic shield, the temperature of which is matched to the temperature of the specimen as closely as possible by additional heaters. This is accomplished by monitoring the temperature of the specimen at different points, and again at equivalent points of the adiabatic shielding, by thermocouples and by using the differences of the emfs of corresponding thermocouples to control the power of the shield heaters.

This method looks very simple in principle but is very complicated in practice if high accuracy is required. Only four establishments within the NATO nations were found to use this method.

2.4.1.1

The University of Tennessee (Stansbury, Vis 45, Lit.54, 55) has been working for many years on the development of adiabatic calorimeters of high accuracy. So far temperatures of 1000°C can be realised. The accuracy claimed (±0.5%) is the best found in high temperature calorimetry. It should be mentioned that the short range order in Cr-Ni alloys could be detected by this technique (Lit.56), showing that these alloys are unsuitable as standards for thermal conductivity measurements.

2.4.1.2

The NPL Metallurgy Division (Vis.22) has developed a high temperature adiabatic calorimeter for the determination of the heat of reaction in metallic systems. This calorimeter can be used for measurements of the specific heat, too. Temperatures up to 1100°C can be achieved so far. In this high temperature range an accuracy of ±1% is claimed (Lit.57). Kubaschewski thinks that it is possible to extend the temperature of application up to 1700°C.

2.4.1.3

R Kohlhaas and his collaborators (Vis 13) have developed an adiabatic calorimeter and used it from room temperature up to 1600°C (Lit 102, 103). The cylindrical specimens of 22 mm diameter and 60 mm height hang on a molybdenum wire within a thermal insulation of alumina. The adiabatic conditions in this arrangement are realised by a directly heated molybdenum tube, the temperature of which is matched to the temperature of the specimen. Kohlhaas and Braun have studied systematically (Lit.101) the errors in such an adiabatic calorimeter system.

For this method an accuracy of ±2% at 1500°C is claimed. This calorimeter has been developed for studies on specific heat of iron alloys during the transition between the solid and liquid states, i.e. for 1600°C as the upper temperature limit. Using other materials for heaters (tantalum) and a different insulation instead of alumina, this system, using a tubular furnace, could be developed for application at higher temperatures. Kohlhaas corrects his measurements for heat losses and calls his method "quasi-adiabatic".

2.4.1.4

According to Flynn, the NBS also has an adiabatic calorimeter, used up to 500°C. Further details are not known.

2.4.2 Drop Calorimetry

In this technique the encapsulated specimen is heated in a separate tube furnace up to the measuring temperature and then dropped into a calorimeter, where the temperature increase is measured.

The arrangement seems very simple, but special care must be taken to account for heat losses from the capsule during the drop and to avoid heat transfer from the furnace to the calorimeter. The precautions become more and more important, the higher the temperatures.

The furnace tube is connected to the calorimeter by a drop tube which normally contains two shutters. These are only opened during the drop and are supposed to prevent heat exchange between furnace and calorimeter. The temperature of the capsule is measured pyrometrically. Heat losses can be minimised by using a hydrogen atmosphere, for in this case thermal equilibrium is reached very quickly, on account of the high thermal conductivity of hydrogen (Lit.58, Vis.33).

A critical study of the different types of drop calorimeters and their respective advantages and errors is given by Douglas (Lit.59). For temperatures near room temperature a precision of ±0.04% and an accuracy of ±0.5% are claimed. At very high temperatures (2800°C) an accuracy of ±1% has been reached. The overall accuracy of a drop calorimeter is comparable with that of an adiabatic calorimeter. Because the measured specific heat is calculated from the difference between large numbers, further improvement in accuracy is very difficult and also expensive.

The drop calorimeter is widely used in the NATO nations.

Problems in drop calorimetry arise if the sample undergoes a transition during the experiment or remains in an ill defined state after the quenching.

Because of the large number of places using drop calorimetry, no special list is compiled. The institutions can be found in the list of visits (Section 3.1).

2.4.3 *Pulse-Heating Calorimetric Techniques*

These methods are limited to electrical conductors but can be used up to the melting point of the material. The specimen behaves as its own calorimeter. The specific heat is calculated from the rate of temperature increase resulting from uniform electrical heat dissipation within the specimen. Two different methods are used in the institutes visited:

(a) ORNL-method of Kollie (Vis.41, Lit.60)

The predetermined specimen temperature is achieved by Joule heating of the rod with a constant current and recording the rod temperature and the power dissipation in a known length of the specimen. From these the electrical resistivity and the total emittance are determined. By suddenly increasing the current and measuring the temperature and power of the test section as a function of time, using a high speed digital voltmeter, the specific heat is calculated. The rod is suspended in a black-body vacuum chamber of constant temperature. Radiation losses occurring during the pulse ($\simeq$ 4 sec) are accounted for by using previously measured total emittance values. This method is used up to 1400°C. The accuracy is ±1% from 100 to 800°C and somewhat less at higher temperatures (±5%).

Black-body conditions in the chamber were attained by spray-coating all surfaces inside the chamber with a pure colloidal synthetic graphite. It is assumed that this coating has an emittance of approximately 1.0.

The method was improved recently by connecting the output of the fast digital voltmeter, for current, voltage and temperature, directly to the ORNL computer system.

(b) Atomics International method (Taylor, Finch, Vis.28, Lit.61)

This method uses pulses of the order of milliseconds and heating rates from 1000°C to 60,000°C per second. The specimens have the shape of filaments and the temperature of the sample is determined from the electrical resistance of the filament. A dual beam oscilloscope is used to record the resistivity as a function of time. The samples are heated additionally by means of a furnace, which determines the upper limit of the temperature of application.

Because of the high heating rates and the small temperature variation (1°C) the radiative losses amount to only a few per cent of the dissipated energy. With a correction which uses only an approximate value for the emittance, this error in the specific heat due to radiation is reduced to less than ±1%. The overall accuracy was estimated to be better than ±5%.

According to Finch (Vis. 28) the same pulse-heating technique has been used in France by Lallement and Affortit (Lit.62).

2.4.4 *Summary of Measurements of the Specific Heat*

Drop calorimetry is the most frequently used method for measuring specific heat in all temperature ranges up to the upper limit of about 2800°C. No other method is more accurate. This method, yielding only mean values between a wide range of temperatures, is not applicable for materials which undergo transformations in the interval between the temperature of the furnace and the temperature of the calorimeter

The adiabatic calorimeter, which avoids this disadvantage, becomes very complicated in the experimental arrangement if great accuracy at high temperatures is required.

The pulse technique is not very accurate, but it can be used up to the temperature of destruction of the specimens. A list of institutions using adiabatic and pulse methods is given in Table XI (p.96).

2.5 Measurement of Emissivity

In the preliminary report the nomenclature of Worthing (Lit.63) and Richmond (Lit.64) was used. According to this, all properties of a material itself should have the ending "ivity", whereas properties relating to the surface should end in "ance". In this sense we can define

emittance:	ratio of energy radiated by a surface, per unit area, per unit time, to the energy radiated by a black body at the same temperature;
emissivity:	emittance of a material having an optically smooth surface and a thickness sufficient to be opaque.

In a comment on this proposal, Lozier (Vis.44) said that international standardisation requires the use of "emittance" to describe surface-dependent emissive properties to be abandoned in favour of "emissivity". The nomenclature in the report was therefore adapted to this principle.

Knowledge of the emissivity of a specimen is necessary for a correct pyrometrical measurement of temperature, as well as for the calculation of heat losses by radiation.

For the first problem, the temperature is measured in holes whenever possible, which means that near black-body conditions are realised. Direct-heating methods using filaments and transient methods have to measure the true temperature of the surface. In this case knowledge of the spectral emissivity is important because the optical pyrometers usually use a wavelength of 0.665 μ.

2.5.1 *Experimental Determination of the Emissivity*

2.5.1.1 *Measurement of reflectivity*

The emissivity of a surface is related to its reflectivity by Kirchhoff's Law. Since reflectivity is easier and more accurate to measure than emissivity, the National Bureau of Standards (Lit.65) has developed an apparatus for reflectivity measurements at elevated temperatures. It uses a continuous gas laser as the illuminating source in an integrating sphere reflectometer. The incident radiation is cut off so that the reflected radiation can be distinguished from the emitted flux. By use of the laser source the reflected flux is intensified to many orders of magnitude and can be detected very accurately. The disadvantage that only a few wavelengths, determined by the characteristics of the laser, can be measured, may be overcome by interpolating between the measured values when the whole spectrum at room temperature is known.

The reflectivity at room temperature is measured by an ellipsoidal mirror reflectometer, where a narrow beam of radiation from the monochromator is focused through a small hole in the ellipsoidal mirror on to the specimen, which is positioned at the first focus of the ellipsoid. The ellipsoidal mirror focuses the radiation reflected by the specimen on to the detector, which is positioned at the second focus of the mirror system, some 40 cm below the specimen. The flux losses are considerable and an error analysis has not yet been given (Lit.65).

This reflectometer measures the directional-hemispherical reflectivity of specimens from room temperature up to 2500°C, either absolutely or relatively. The error in absolute reflectivity varies from about +0.7% for a perfectly diffuse specimen to about -0.02% for a perfectly specular specimen.

2.5.1.2 *Measurement of total emissivity*

In all methods a specimen in the form of a thick-walled hollow cylinder is heated and the emissivity of the specimen is compared with that of a standard. The various methods differ in the arrangement of the specimen and the standard.

In the "center post technique" (Vis.27, Lit.66) the specimen is positioned in a susceptor having a centre post. The specimen fits over the centre post and is heated by the susceptor. The emissivity of the specimen is taken as the ratio of the radiated energy of the surface of the specimen to that of the centre post, multiplied by the known emissivity of the susceptor material.

In the "deep cavity technique" (Vis.27, Lit.66) the susceptor has no centre post; it forms the bottom of the central cavity and the specimen forms the side walls. To increase the effective emittance of the cavity it is lined with a high-emittance material (NiO). The emittance of the specimen is taken as the ratio of the radiance of the flat top of the specimen to that of the cavity.

The susceptor was made of tungsten. The temperatures were measured with an optical pyrometer and the radiated energy with a total radiation pyrometer. Corrections were made for optical scattering error and temperature difference error.

It was found that the "center post technique" is more accurate than the "deep cavity technique". Data obtained by the shallow cavity technique and by integration of normal spectral emissivity measured by the rotating cylinder method are less accurate, too. The centre-post data were estimated to be in error of about 0.05 in emissivity (Lit.66).

2.5.1.3 *Measurement of spectral emissivity*

For the determination of the spectral emissivity the "hole-in-tube method" can be used. The specimen, in the form of a thin sheet, is rolled into a 1.25 mm diameter tube, 280 mm long. A 1 mm hole is drilled through one wall near the centre of the tubular specimen, thus forming a black-body cavity for pyrometric temperature measurements. The emissivity is calculated from the ratio of the temperatures measured in and by the side of the hole. The apparatus was operated to 1700°C. An error analysis is not given.

This method is also used at the Battelle Memorial Institute (Vis.29) for the determination of the total emissivity.

At the Southern Research Institute (Vis.42, Lit.67), the radiance of a specimen is compared with the radiance of a black-body cavity at the same temperature. The surface temperature of the specimen is determined with thermocouples up to 1300°C and up to 2750°C by optical pyrometry. For the determination of the true temperature, a method of iterating between the readings of the optical pyrometer and a total radiation detector (a 160-junction thermopile) is used. A hole, 1.25 mm diameter and 150 mm long, in graphite serves as a black body. The apparatus operates from 400°C up to 2750°C and an accuracy of about ±12% is claimed.

The method used at the PTB for emissivity measurements (Vis.10, Lit.68) measures the polarisation of the emitted radiation as a function of the angle of emissivity. This method can be used for metals up to 2500°K with an accuracy of ±2%. The emissivity of hafnium and tungsten were determined in this way.

2.5.1.4 *Summary of measurements of emissivity*

Although many efforts have been made to measure precise values of emissivity the problem of using these values for the calculation of heat losses has not been solved satisfactorily because the emissivity is highly dependent on the surface smoothness, the degree of oxidation or chemical reaction, and the surface structure.

Therefore all methods of determining thermophysical properties at high temperatures try to avoid corrections in which numerical values of the emissivity have to be used. Institutions measuring the emissivity are listed in Table XII on p.97.

2.6 Measurement of Thermal Expansion

Thermal expansion is another important thermophysical property of thermal expansion behaviour. The following coefficients are very often used to describe ....

The *mean coefficient* of linear thermal expansion, defined as

$$\alpha_m = \frac{L_2 - L_1}{L_1} \frac{1}{T_2 - T_1} , \qquad (10)$$

where L_1 and L_2 are the lengths of the specimen at temperatures T_1 and T_2.

The *coefficient* α_T *at temperature* T (instantaneous coefficient of linear thermal expansion), defined as

$$\alpha_T = \frac{dL}{dT} \frac{1}{L} \qquad (11)$$

The thermal expansion is easier to measure than the thermal conductivity, even at high temperatures.

2.6.1 *Interferometers*

The interferometric method has been recommended by the ASTM. Its upper temperature of application is determined by the optical parts of the interferometer: 1000°C for vitreous silica, 1500°C for sapphire.

2.6.1.1 *Principle of the method*

Generally the interferometers consists of two flats with reflecting surfaces, placed parallel to each other a short distance apart. The specimens are arranged between the two flats. If monochromatic light impinges on the surfaces from one side, interference fringes are formed. When the two surfaces are moved by a dilatation of the specimen, the fringes move too, and this movement is used to determine the thermal expansion. In vacuo, the linear thermal expansion is given by

$$\frac{\Delta L}{L_0} = \frac{\lambda N}{2L} , \tag{12}$$

where L and ΔL are the specimen length and its increment, λ is the wavelength of the light used and N is the number of fringes which have passed the reference mark.

Polished flats made of vitreous silica of optical quality are used as reflecting surfaces. The bottom of the lower flat is ground so as to be non-reflective, while the surfaces of the top flat should have an angle of about 15 minutes of arc between them, to reduce and eliminate extraneous reflections and fringes. A reference point or line is etched in the surface of the upper flat. Monochromatic light of known wavelength is used for illumination. Kirby (Vis.39) is developing a laser as the source of light. To produce a convenient number of fringes in the field of vision, the specimens must be of exactly the same length, to within several wavelengths of light.

2.6.1.2 *The Fizeau and the Newton's rings method*

There are several types of interferometer, such as the Fizeau interferometer, the Abbe-Pulfrich interferometer, the Priest interferometer and the UCC interferometer using Newton's rings.

The expansion of a specimen measured with the Fizeau interferometer is determined absolutely, but the expansion as measured with the Priest and Abbe-Pulfrich interferometers is determined relative to the expansion of some reference material, such as vitrous silica. The interferometer using Newton's rings also gives absolute data.

In the Fizeau interferometer proposed by the ASTM (Lit.69), two vitreous silica flats are used. The specimens, of the order of a few mm long, are placed between the two flats in such a way that one of the three points of support bears most of the weight of the top flat. This position, directly over the support, is chosen as the reference point of the fringe system. The whole arrangement is placed in a furnace. For plate separation up to 5 mm, a discharge lamp filled with helium is convenient. For separations up to 20 mm another wavelength is adequate and a mercury lamp may be used.

The sensitivity of this method is about 2×10^{-5} mm, since a fringe separation corresponds to one-half a wavelength of light and 1/10 of a fringe can be estimated.

The Newton's rings method has been developed by the UCC (Vis. [illegible], Lit.70). Measurement of thermal expansion by the interferometer using Newton's rings is rather similar to the Fizeau interferometer method. One of the optical flats is replaced by a plano-convex lens, which rests on the specimen with the plane side. Newton's rings are formed between the curved surface of the lens and the lower surface of the optical flat, positioned closely above. For the determination of the thermal expansion a portion of the area of the first Newton's ring is viewed by a photomultiplier. The output of this photomultiplier is received by a xy-recorder in addition to the emf of the thermocouple attached to the specimen. The apparatus operates in a vacuum. Specimens about 12.5 and 25 mm long and of 25 mm diameter are used. For illumination a mercury arc lamp was chosen.

The sensitivity of the system should be equal to that of the other interferometers. As the apparatus is not yet finished, an error analysis is not given. The Newton's rings method is superior to the Fizeau method if automatic recording is used.

Interferometry methods were seen in use during visits to the establishments shown in Table XIII (p.98).

2.6.2 *Push-Rod Dilatometers*

In a push-rod dilatometer, the specimen is heated indirectly by a controlled furnace. The thermal expansion of the specimen is conveyed out of the heated zone by an extension rod and compared with the expansion of a known material.

For measurement of the differential expansion between the specimen and the reference material, various arrangements are used, such as dial gauges (Adamel, Vis.2; Pears, Vis.42, Lit.73), differential transformers (Linseis, Vis.12B; Netzsch, Vis.31, 44), mirror systems (Bollenrath, Leitz, Vis.11, 28), etc. The sensitivity of these measuring devices is satisfactory, but the accuracy of the results is affected by a number of doubtful factors, such as the thermal expansion of the reference material, the slipping and deformation of the specimen, inhomogeneous heat distribution in the furnace, and so on.

Many different ways of overcoming these difficulties have been proposed. An unusual solution, with the specimens inclined at 30^0 to the horizontal axis, deserves mention here (Gatto, Vis.15).

In most cases the temperature of the specimen is measured by a thermocouple alongside or above it. The only accurate way is to measure the specimen temperature in an axial hole of the specimen as is done at the CNR (Vis.15) and Aacken (Vis.11).

The upper temperature limit of application is determined by the stability of the material of construction, especially of the push rod. In most cases vitreous silica is used up to 1000°C, and Al_2O_3 up to 1500°C. At higher temperatures zirconium, tungsten or graphite parts must be used.

Push-rod dilatometers are more widely used in Europe than in the United States. They are commercially available (Adamel, Linseis, Netzsch, Leitz, etc.)

For all these apparatuses an accuracy of about ±1% up to 1000°C can be assumed (Lit.71, 72). Gatto (Vis.15) claims a sensitivity better than 0.5%. For high temperature measurements the SRI apparatus (Vis.42, Lit.73) is suitable and is now described.

The high temperature dilatometer of the SRI uses graphite extension rods and can be operated up to 2750°C. The specimen required is about 18 mm in diameter and a length of 75 mm is desirable for accurate results. Movement of the specimen is measured by a dial gauge, which indicates total movement of 2×10^{-4} mm. When required, tungsten pads are inserted at the ends of the specimen to eliminate diffusion of graphite from the dilatometer parts into the specimen. Either a helium or an argon atmosphere is employed, but the equipment can also be operated in a vacuum.

An overall accuracy of ±5% is claimed. Errors resulting from temperature gradients are estimated to be less than ±1%. Because of the general application of the push-rod method, no separate table of institutions using this method is given; they can be seen from the list of visits, in Section 3.1.

2.6.3 The Baudron Dilatometer

The Baudron dilatometer (Lit.131) is a modification of the push-rod system. The principal idea is to eliminate the effect of the dilatation of the rod system on the recording system. This is done by arranging the push-rod system at right-angles to the specimen axis. The dilatation of the specimen is transferred to a differential transformer by a pivoted lever.

The Baudron dilatometer was seen in use at the British Ceramic Research Association (Vis.19). There sintered alumina is used as the material for the rod system. The lever bearings are positioned in an Invar block, outside the furnace, to minimize errors caused by the variation of the distance between the bearings. The method gives a reproducibility within ±1% and an overall accuracy > ±2%.

The creep of the rod system could be a very serious disadvantage and no experience at very high temperatures is available.

2.6.4 Twin Microscope Comparators

If mechanical systems for measuring the thermal expansion cannot be applied because of excessive temperatures, optical systems may be used with advantage. Such a system consists of a furnace containing the sample and two long-focus (tele-) microscopes.

This method seems to be simple but some difficulties arise in practice. Suitable marking of the specimens is one difficulty, because the optical observation requires the brightness of the marks to differ from the background. A sufficient contrast between marks and background has been achieved by using small holes drilled in the sample (Vis.33, Lit.74) or by using thin wires as marks.

Another difficulty arises because the optical measurements are subject to error caused by refraction of the light beams in the medium surrounding the sample. Therefore a correction factor must be applied to obtain accurate results.

Similar problems occur in modern creep-testing apparatuses. Some laboratories use recording comparators for measuring the elongation (GEC, Vis.33). With the "Optron" device a sensitivity in automatic optical length measurement of $2.5 \cdot 10^{-3}$ mm ($1 \cdot 10^{-4}$ inch) is achieved.

Some examples are now given to illustrate the measurement of thermal expansion by twin microscopes.

2.6.4.1 *Kirby's newly developed apparatus* (Vis.39)

Kirby uses a vertically mounted tubular vacuum furnace of 0.5m length and a rhenium heater with five separately controlled heater units. The sample length is 100 mm Pt/6Rh vs Pt/30Rh and W/13Rh vs W/25Rh thermocouples are used for the temperature measurements. A standard deviation of ±23 ppm is claimed; that means a precision in length measurement of 2.3×10^{-3} mm.

Exchange of specimens with GEC Laboratories (Vis.33) has produced excellent agreement.

2.6.4.2

The *GEC group* uses twin microscopes for measuring the thermal expansion up to 2500°C (Vis.33, Lit.74, 90).

The specimens, about 60 mm long, are heated in a tungsten furnace operating in a helium atmosphere. Rod and sheet material can be used and the specimen is positioned horizontally in a tungsten block supported within the furnace. Small holes of 0.25 mm diameter, 50 mm apart, are used as the reference marks. Above 1000°C, the temperature of the specimen is measured pyrometrically; below this temperature Pt vs Pt/10Rh thermocouples are used. The temperature uniformity of the entire specimen is within ±10°C, achieved by the massive tungsten holder and additional radiation shields. The microscope is calibrated before each test by using a standard linear scale etched on a glass slide. The measured expansion values are corrected for the refraction occurring in the helium atmosphere. The reproducibility is ±2% at 2500°C.

2.6.4.3 *Collins's measurement of the thermal expansion of graphite* (Lit.75)

Optical methods have been used for some years to measure the thermal expansion of graphite and Collins's apparatus is an example. He uses 30 cm long samples and obtains a reproducibility of the length measurement of 1×10^{-2} mm. This corresponds to 1% of the total change in length of a specimen which is heated from 1000°C to 2500°C.

2.6.5 *X-ray Techniques*

Lattice expansion can be measured directly by means of high temperature X-ray techniques. Systems using films as well as direct-reading arrangements are employed. Before detailed consideration of the systems, the relation between lattice expansion and bulk expansion must be discussed.

For isotropic non-porous materials an agreement between lattice expansion and bulk expansion can be expected. Kirby (Vis.39) has compared his latest measurements of thermal expansion of platinum up to 1000°C with the X-ray data of Edwards, Speiser and Johnston (Lit.76) and found good agreement. On the contrary there is disagreement between lattice expansion and bulk expansion of graphite (cf.Lit.77, 78) and differences can be expected with alloys, too.

High temperature X-ray research is undertaken at the following establishments.

2.6.5.1 *UCC Research Laboratories, Parma* (Vis.44)

These laboratories use a Debye-Scherrer technique. The specimens may be heated indirectly or directly. When the electrical conductivity of the sample does not undergo marked changes during prolonged heating, the direct method is used. In other cases heating is by radiation of the sample from tungsten or tantalum strips. In addition a graphite heater tube can be used to achieve higher temperatures.

The upper temperature limit is the melting point of the specimens, which are directly heated. Indirect heating can be used up to 2400°C. An accuracy of ±0.1% in the measurement of the lattice parameters is claimed.

2.6.5.2

SNECMA (Vis.4, Lit.79) has developed a high temperature X-ray technique with a very strong heating system, using a U-shaped tantalum heater. This chamber will work up to 2350°C. Systematic research on the refractory metals and oxides is in hand.

2.6.5.3

Karlsruhe (Vis.12B) is studying the lattice expansion of several graphites up to 1500°C with different chambers (Lit.78).

2.6.6 *Summary on Thermal Expansion*

As mentioned in the Introduction, the measurement of thermal expansion at high temperatures is not as difficult as that of thermal conductivity, because optical as well as X-ray methods can be used. The interferometric methods are the most precise. However, because of the lack of transparent materials for high temperatures their application is usually limited to temperatures below 1000°C; by using expensive sapphire optics they can be operated up to 1500°C. Push-rod systems can be used up to 2700°C with appropriate construction materials. However, in general they are not very precise. Baudron's modification is only rarely used. The latest twin-microscope methods give very precise values; sensitivity in length measurement of 2×10^{-3} mm for samples 100 mm long has been achieved.

The possibility of replacing methods measuring bulk thermal expansion by X-ray methods should be investigated. As a basis for theoretical considerations, the X-ray methods should be used.

Table XIV, on p.99, lists institutions which use optical and X-ray methods.

2.7 Measurement of Electrical Properties

As outlined in Section 5 it is also necessary to measure electrical properties such as the electrical resistivity and the Seebeck coefficient, as functions of temperature as well as the residual resistance for theoretical assessment of experimental results on thermal conductivity.

The electrical resistivity is composed of a term arising from the electron-lattice interactions and a term dependent on imperfections. The first term is dependent on the temperature and dominates at higher temperatures. This intrinsic electrical resistivity is used in relation to the thermal conductivity, expressed by the Wiedemann-Franz-Lorenz law. The second term is independent of the temperature, becomes important at very low temperatures and is called the residual resistivity. It is a measure for the impurity level. In practice, instead of using the residual resistivity directly, the ratio of resistance at room temperature to that at liquid helium temperature (4.2°K) is used. This minimises extraneous factors in the measurement such as sample geometry and shape (Lit.80) and effects due to anisotropy.

The Seebeck coefficient is used for calculation of the theoretical Lorenz number, which is necessary to separate the total thermal conductivity into an electronic and a lattice portion.

It seems to be very important that these electrical properties should be determined on the specimen whose thermal properties are measured. It should be recommended that all establishments engaged in measuring thermal conductivity should measure these electrical properties simultaneously (Vis.25, 38, 41).

2.7.1 *Measurement of Electrical Resistivity*

Electrical resistivity can be determined by methods based on Ohm's law or by measuring the inductive resistance with alternating current. The latter method is not suitable for very accurate results or for measuring the temperature dependence at high temperatures. They are mentioned at the end of this section.

If specimens of well-defined shape like rods, tubes or filaments are available the voltage drop method can be used. This is especially suitable for methods with direct heating at high temperatures. For such arrangements see Lit.80 and 81.

An accuracy of about ±0.1% up to 1100°C (Lit.25) is claimed, in which the largest contribution to the error arises from the uncertainty of the cross-sectional area.

The electrical resistivity has been measured on metals up to 2500°C in, for example, the GEC laboratories (Vis.33). Results on molybdenum and on the independence of grain structure and grain size are reported in Lit.11.

The electrical resistivity of graphite has been measured up to 2500°C by McClelland (Lit.3), who includes a discussion of the experimental difficulties.

To avoid difficulties arising from the requirement of well-defined shapes for the specimens, the newly developed measuring methods, generally employed in the field of semi-conductors can be used. The so-called "four-point potential-drop technique" can use specimens of unknown dimensions, provided they are large enough, with respect to the probe spacing, to be assumed as infinite (Lit.80 and 81).

The four probes are arranged in one line and the tips are pressed against the sample. The current is passed between the outer two probes, and the voltage is measured between the inner two. The resistivity is given by

$$\rho = \frac{2\pi l E}{I} . \qquad (13)$$

where l is the spacing between each pair of adjacent electrodes, E is the voltage and I is the current.

Probes with very small spacings can be made. The contacts can be of 0.07 mm (0.0003 in.) diameter tungsten wires with sharp points, and the spacing l can be 0.02 mm.

No discussion on the accuracy for specimens of highly-conducting materials or on the upper temperature limit could be found in the literature.

The residual resistivity at the temperature of liquid helium by this "four-contact potential-drop method" was measured by Weisberg (Lit.80) for similar specimens with a precision of a few percent using a nanovoltmeter (sensitivity 10^{-9} V).

For semi-conductors a precision of only 10% is claimed.

As already mentioned, the electrical resistivity can also be measured by alternating current methods without contacting the specimen. The measurement is based on the determination of the change of either the mutual inductance between two coils or the impedance of one coil when the specimen is placed in the centre of the coil (Lit.117, 118). These methods are less accurate than the methods using direct current (Lit.80) described earlier. They are therefore only used when current and potential leads cannot be attached to the specimen or when its size and shape are unsuitable for the direct current method.

Such methods are widely used for determination of the homogeneity of large specimens, as for example in the US Air Force standard programme for thermal conductivity (Vis.35).

2.7.2 *Measurement of the Seebeck Coefficient*

The differential thermoelectrical power with dimension [μV/°C] between a pair of metallic conductors is called the relative Seebeck coefficient. Using a reference metal with known absolute Seebeck coefficient, the absolute Seebeck coefficient of the measured material can be calculated by subtraction. Generally platinum, employing the absolute Seebeck data given by Cusack and Kendall (Lit.97), is used.

The experimental arrangement is combined with the electrical resistivity measurements, using direct-heated specimens and an additional indirect heater on the thermal insulation, providing a temperature gradient of some degrees (Vis.41, Lit.82).

The Seebeck coefficient can be determined fairly accurately. Laubitz (Vis.25, Lit.25) gives a maximum accuracy of ±0.1 μV/°C, which is about ±0.4% up to 1100°C. McElroy (Vis.41, Lit.82) claims an absolute error of ±0.9% up to 1000°C.

3. LISTS OF VISITS AND BIBLIOGRAPHY

3.1 List of Establishments Visited

This section summarises visits made to various establishments as follows:

(a) Scientists contacted during the visits.
(b) Main field of interest of the institution in relation to this AGARD project.
(c) Equipment for studying thermophysical properties.

EUROPE

Vis.No.

Belgium

1. SERAI
Société d'Etudes, de Recherches et d'Application pour l'Industrie,
Bruxelles, 1091 Chaussee d'Alsemberg.
(a) W.R.Ruston, Dr E.Votava.
(b) Basic research on direct-heating methods for thermal conductivity measurements projected.
(c) X-ray equipment for high temperatures, standard equipment for metallurgical research.

France

2. IRSID
Institute de Recherches de la Sidérurgie Francais,
St.Germain-en-Laye, 185 Rue Président Roosevelt.
(a) C.A.Constant, Prof. Dr Kozakevich, Dr G.Urbain, Dr Lukas.
(b) Testing of metallic materials at high temperatures. Basic research on liquid metals and on the transition from the solid to the liquid state.
(c) α: (push-rod, Adamel) up to 1000°C.
c: (drop calorimetry).
ρ: up to 1400°C (projected).

3. ONERA
Office National d'Etudes et de Recherches Aérospatiales,
Chatillon-sous-Bagneux (Seine), 29 Avenue de la Division Leclerc.
(a) J.Poulignier, M. Lignon, M. Guyot.
(b) Basic and applied research in Co and Nb alloys, carbides and coatings.
(c) α: (push-rod), c, ρ, thermo-analysis.

Vis.No.

France (continued)

4. **SNECMA** Societe Nationale d'Etude et de Construction de Moteurs
d'Aviation, Usine de Suresnes,
92 Suresnes, 22 Quai Gallieni.

(a) M. Wellard, R.Deimas.

(b) Testing of high temperature materials, such as tungsten, graphite, carbides and oxides.

(c) λ : (comparative methods) up to 2000°C.
a : (periodic arc image).
c : up to 1000°C.
α : (X-ray) up to 2850°C.
ρ : up to 1800°C.

5. Sud-Aviation Societe Nationale de Construction Aéronautique,
Division Engins Spatial et Electronique,
92 Courbevoie, 55 rue Victor Hugo.

(a) G.Kamoun.

(b) Testing of high temperature materials (tungsten, graphite, beryllium).

(c) λ : (direct heating, Kohlrausch) up to 2000°C.
a : up to 750°C.
α : (push rod) up to 1500°C.
c : (drop calorimetry) up to 2400°C.
ϵ : (total).

Germany

6. **DEW** Deutsche Edelstahlwerke AG, Zentrallaboratorium,
415 Krefeld, Oberschlesienstrasse 16.

(a) Prof. Dr K.Bungardt, Dr W.Spyra.

(b) Development and testing of alloy steels for high temperatures.

(c) λ : (longitudinal apparatus) up to 100°C
(longitudinal comparative method) up to 700°C.
α : (push-rod) up to 1000°C.

7. **Euratom** Euratom Institut für Transurane,
Kernforschungszentrum Karlsruhe-Leopoldshafen,
7501 Leopoldshafen.

(a) Dr H.E.Schmidt

(b) Studies on thermal conductivity at high temperatures (UO_2, PuO_2, refractory metals).

(c) λ : (method of compensated flux) up to 1000°C.
a : (electron beam), no temperature limit.

Vis.No.

Germany (continued)

8. Kernforschungszentrum, Karlsruhe

Institut für Material- und Festkörperkunde,
Kernforschungszentrum Karlsruhe-Leopoldshafen,
7501 Leopoldshafen.

(a) Prof. Dr F.Thümmler, Dr Ondratschek, Dr E.Patrassy.

(b) Development of nuclear fuels, based on oxides; powder metallurgy.

(c) λ: (radial method) up to 2000°C
(longitudinal method) in development.

9. Krupp

Friedr. Krupp, Widia-Fabrik, Versuchsanstalt,
43 Essen 1, Münchner Strasse 125/127.

(a) Prof. Dr O.Rüdiger, Dr J.Hartwig, Dr W.Michel

(b) Development and testing of high melting carbides at high temperatures.

(c) λ: (longitudinal apparatus) up to 1000°C.

10. PTB

Physikalisch-Technische Bundesanstalt,
33 Braunschweig, Bundesallee 100.

(a) Dr U.Schley, Dr K.H.Bode, Dr P.Rahlfs, Dipl.Phys.H.Kunz.

(b) Calibrating of measurement techniques of thermophysical properties; control of standards for thermal conductivity; development of new methods for the determination of thermophysical properties.

(c) λ: (longitudinal apparatus) up to 500°C, for plates up to 300°C.
(longitudinal comparative method) room temperature.
(direct-heating method) up to 1100°C.
α: (push-rod) up to 1000°C.
ϵ: from -180°C up to 2000°C.
T: (recording pyrometer) in development.

11. TH Aachen

Institut für Werkstoffkunde, Rheinisch-Westfälische Technische Hochschule, Aachen.

(a) Prof. Dr F.Bollenrath, Dipl.Phys.H.E.Evers.

(b) Applied research on thermophysical properties of high temperature materials as a basis for solving aeronautical engineering problems.

(c) a: (flash or electron gun) being planned.

12A. TH Karlsruhe

Institut für angewandte Physik,
Technische Hochschule, Karlsruhe,
75 Karlsruhe, Kaiserstrasse 12.

(a) Prof. Dr F.Stöckmann, Dr U.Birkholz.

(b) Basic research in solid state physics, especially semi-conductors.

(c) a: (Angstrom method) up to 600°C.

Vis.No.

Germany (continued)

12B. TH Karlsruhe

Institut für Chemische Technik,
Technische Hochschule Karlsruhe,
75 Karlsruhe, Kaiserstrasse 12.

(a) Prof. Dr E.Fitzer, Dr K.Hüttinger, Dipl.Chem.H.Böder.

(b) Applied research on high temperature materials; development of artificial graphites, silicides, borides, composites, coatings.

(c) λ : (comparative method) room temperature.
a : (cooling-down method) up to 1000°C.
α : (push-rod) up to 1800°C
(X-ray) up to 1500°C.

13. Uni Köln

Institut für theoretische Physik,
Universität Köln.

(a) Prof. Lange, Dr R.Kohlhaas.

(b) Basic research in thermal and magnetic properties of metals and alloys up to their melting temperatures.

(c) λ : (longitudinal method) up to 100°C
(radial method) up to 1000°C.
c : (quasi-adiabatic calorimeter) up to 1500°C.

Italy

14. CNRN

Concilio Nazionale delle Ricerche Nucleari
Casaccia, Roma, Via Anguillarese.

(a) Ing.Franco, Dr S.Morretti, Dr Fizzotti, Ing.Bianci, Ing.Evangelisti.

(b) Applied research on nuclear materials, vanadium alloys, steels, UO_2; heat transfer between oxides, steels and molten metals.

(c) λ : (of molten metals) up to 1000°C.
a : (Xe-flash) up to 1500°C.
α : (push-rod, Adamel).

15. CNR

Concilio Nazionale delle Recerche
Gruppo di Recerche sulla Technologia dei
materiali non traditionali
Milano, P. le Rodolfo Morandi, 2.

(a) Prof. Dr Ing.F.Gatto.

(b) Applied research on high temperature materials and coatings.

(c) α : (push-rod) up to 1500°C.

16. Euratom

CCR Ispra, Servicio Chimico Fisico.

(a) Ing.C.Mustacchi, S.Giuliani.

(b) Measurement of thermophysical properties of reactor materials.

Vis.No.

Italy (continued)

Euratom (continued)
(c) λ: (longitudinal method) up to 600°C
(radial outflow method) 500 - 1000°C
(comparative method) room temperature.
a: (electron gun) no temperature limit.
ρ: up to 1700°C.

17\. **FIAT**
Central Laboratory, Sezione energia nucleare
Torino, Corso G.Agnelli 200

(a) Prof. Dr J.L.Locati, Dr C.P.Galotto, V.Gallina, M.Omini, Dr Rosatelli, Dr I.Amato, Dr G.Frigerio.

(b) Basic research in solid-state physics and metallurgy, applied research and development of nuclear materials, testing of engineering materials.

(c) α: (push-rod, Netzsch) up to 1500°C.

18\.
Istituto Termometrico
Torino, Via delle Acacie,

(a) Dr G.Ruffino.

(b) Calibration of temperature measurements, development of new pyrometric instruments.

(c) α: (interferometric) 90° - 273°K.
T: (recording pyrometer) under development.

United Kingdom

19\.
British Ceramic Research Association,
Physical Measurements Section,
Queens Road, Stoke-on-Trent, England.

(a) P.Popper, D.B.Binns.

(b) Developing and testing of ceramic materials.

(c) λ: (longitudinal comparative method) up to 400°C.
a: (Xe-flash) room temperature.
c: (drop calorimetry) up to 1000°C.
α: (push-rod, Baudron dilatometer) up to 1000°C.

20\.
Clarendon Laboratory, University of Oxford,
Department of Physics, Parks Road, Oxford, England.

(a) Dr R.Berman

(b) Basic research in thermal conductivity of non-metallic materials at very low temperatures.

(c) λ: (longitudinal method) at temperatures of liquid and solid He up to 90°K.
c: (drop calorimetry) in the range of very low temperatures.

Vis.No.

United Kingdom (continued)

21. GEC General Electric Company,
Hirst Research Centre. Wembley, Middlesex, England.

(a) H.W.Davidson, H.H.W.Losty, M.I.Wheeler.

(b) Basic and applied materials research for electrical and nuclear application, development of a diffusivity apparatus using electron bombardment.

(c) a : (electron beam) from 300°C, no upper limit.

22. NPL National Physical Laboratory, Teddington, Middlesex, England.
Metallurgy Division, Division of Applied Physics,
Standards Division.

(a) Dr Hopkins, Dr O.Kubaschewski, C.R.Barber, Dr T.Quinn, W.A.Dench.

(b) Calibration of standards; development of measurement techniques; thermochemical research.

(c) λ : this department is closed.
c : (adiabatic calorimeter) up to 1500°C.
T : (recording pyrometer) under development.

23. Bath University of Technology, School of Physics,
Ashley Down, Bristol 7, England.

(a) Prof. S.H.Ayliffe, Dr H.I.Goldsmid.

(b) Basic research on thermal conductivity of semi-conductors and thermoelectrical alloys; studies of the change of thermal conductivity with magnetic field (Maggi-Righi-Leduc effect).

(c) λ : (longitudinal method) from -90°C to +400°C, magnetic field up to 10,000 gauss.
a : (laser flash) up to 1200°C (in collaboration with Windscale (Reactor Development Laboratories, Windscale, Sellafield, Seascale, Cumberland)).

24. University of Wales, Department of Physics,
Singleton Park, Swansea, Wales.

(a) Dr M.R.Hopkins

(b) Basic research on thermal and electrical properties of metallic materials in solid state and during melting; thermal conductivity in semi-conductors.

(c) λ : (direct-heating) no temperature limit.
ρ : no temperature limit.

AMERICA

Vis.No.

Canada

25. NRC National Research Council, Division of Applied Physics,
Montreal Road, Ottawa, Ontario

(a) Dr H.Preston-Thomas, Dr M.I.Laubitz, Dr R.E.Bedford.

(b) Basic research on thermal conductivity of metals;
temperature measurements.

(c) λ: (longitudinal and Forbes bar) up to 1000°C.
ρ: (residual electrical resistance at 4°K) up to 1000°C.
S: up to 1000°C
T: development of high temperature thermocouples.

United States

26. Aerospace Corporation, Materials Sciences Laboratory,
2400 El Segundo Boulevard, Los Angeles, California.

(a) I.D.McClelland, Dr J.Kaspar, E.H.Zehms, I.Richardson, Mr Wolten.

(b) Materials research for aerospace purposes; thermophysical
properties of graphite at very high temperatures.

(c) a: (Kaspar's periodic re-radiation technique) up to 2800°C.
α: (X-ray) up to 1500°C.

27. AFSC Air Force Materials Laboratory,
Materials Engineering Branch,
Wright-Patterson Air Force Base, Ohio, 45433.

(a) A.M.Lovelace, M.L.Minges, G.L.Denman, Mr Stevenson.

(b) Basic and applied research on materials for aerospace application. Basic research on heat transfer and the influence
of roughness.

(c) λ: (radial inflow apparatus) up to 2300°C
(guarded hot-plate) up to 1500°C.
a: (laser flash) up to 2500°C.
α: (optical method) up to 2300°C.
ϵ: laser source integrating sphere reflectometer, up to 2500°C,
accuracy $\simeq$ 1%; ellipsoidal mirror reflectometer, only at
room temperature, centre post technique and deep cavity
technique for determination of the total emittance up to
2000°C.

28. AI Atomics International, Research Groups on Metallurgy,
Solid State Physics and Material Science,
P.O.Box 309, Canoga Park, California.

(a) E.V.Kleber, M.M.Kakata, R.A.Finch, C.A.Smith, C.C.Weeks.

(b) Basic and applied research on nuclear materials, especially
Zr-hydrides and U-oxides.

Vis.No.

United States (continued)

AI (continued)
(c) λ : (radial inflow apparatus) up to 2700°C.
a : (laser flash Method (Cape and Lehmann)) up to 1800°C
(transient radial inflow method) up to 2500°C.
c : (drop calorimetry) up to 1650°C
receiver 750 - 800°C
hydrogen pressure apparatus
(pulse calorimeter) no temperature limit.
α : (optical) up to 3000°C.

29. Battelle
Battelle Memorial Institute, Columbus Laboratories,
505 King Avenue, Columbus, Ohio.

(a) D.Dingee, J.F.Lagedrost, R.McCann, C.F.Lucks.

(b) Contract research in measuring thermophysical properties.

(c) λ : (longitudinal comparative method) up to 850°C
(self-guarding disc method) up to 1800°C
(radial inflow method) up to 2500°C.
a : (laser flash) up to 2000°C.
c : (drop calorimetry) up to 2000°C.
α : (push-rod method. SiO_2 and graphite).
ε : (total emittance) up to 2000°C.

30. Sixth Conference on Thermal Conductivity, Dayton, Ohio, October 19 - 21, 1966.

(Only the scientists not mentioned in a separate Vis.No. are listed here.)

(a)	(b)	(c)
P.G.Klemens University of Connecticut, Storrs, Connecticut	Basic research in solid state physics and transport mechanisms.	-
C.E.Moeller Midwest Research Institute, 425 Volker Boulevard, Kansas City 10	Measurements of thermal conductivity at high temperatures.	λ : radial outflow apparatus up to 2700°C.
M.Hoch Ohio State University, Department of Chemistry, Columbus, Ohio	Basic research in thermal conductivity measurement at high temperatures	λ : steady-state method using direct heating up to 2800°C.
B.H.Morrison Los Alamos Scientific Laboratories, Box 1663 NU, Los Alamos, New Mexico	-	-

Vis.No.

United States (continued)

31. Dynatech
Dynatech Corporation, 17 Tudor Street,
Cambridge, Massachusetts 02139.

(a) R.P.Tye, L.C.Hoagland.

(b) Development and production of apparatus for measuring thermophysical properties.

(c) λ : (radial outflow) up to 2700°C
(radial inflow) in development, now up to 900°C
(longitudinal) up to 1000°C
(hot plate method).
c : (drop calorimetry) up to 1200°C.
α : (push-rod, Netzsch) up to 1000°C.

32. GA
General Atomic Division, General Dynamics Corporation,
Research Department, Metallurgical Department,
San Diego, California 92112.

(a) Dr W.L.White, M.T.Simnad, C.L.Meyers, Dr W.C.Bokros, G.B.Engle, W.V.Goeddel.

(b) Basic and applied research in reactor materials, especially graphite.

(c) λ : up to 1000°C.
α : (push-rod, X-ray).

33. GEC
General Electric Company, Nuclear Material and Propulsion
Operation Materials Evaluation and Analysis,
Cincinnati, Ohio 45215.

(a) W.E.Niemuth, J.B.Conway, Mr Flagella, A.D.Feith, W.C.Kuhlmann.

(b) Basic research on ceramic nuclear fuels and refractory materials.

(c) λ : (radial outflow) up to 2500°C.
a : (laser flash) up to 1000°C.
α : (optical method) up to 2500°C.
c : (drop calorimetry) up to 2500°C.
T : (thermocouples) up to 2300°C.

34. JPL
Jet Propulsion Laboratory. California Institute of Technology,
4800 Oak Grove Drive, Pasadena, California 91103.

(a) D.B.Fischbach, M.H.Leipold.

(b) Research on graphite and refractory ceramics.

(c) α : (optical) up to 2000°C
(X-ray) up to 1500°C
(push-rod) up to 1000°C.

Vis.No.

United States (continued)

35. A.D.Little — A.D.Little Incorporated,
20 Acorn Park, Cambridge, Massachusetts 02140.

(a) H.McMahon, Dr Glaser, A.E.Wechsler.

(b) Development and production of cryogenic equipment, contract research in thermophysical properties, coordinator of the USAF programme for high temperature standards for thermal conductivity.

(c) λ: (longitudinal) in development
(radial outflow apparatus) up to 2700°C.

36. Lockheed — Lockheed Missiles and Space Company, 3251 Hanover Street,
Building 205, Palo Alto, California.

(a) R.Perkins, G.R.Cunnington, J.Smith, Mr Bragg, A.J.Funai, Dr Goetzel.

(b) Applied research on materials for aeronautics.

(c) λ: (radial inflow apparatus) up to 2700°C
(guarded hot-plate method) up to 500°C
(longitudinal comparative method) up to 500°C.
a: (Xe flash) up to 800°C
(laser flash) up to 2400°C.

37. MIT — Massachusetts Institute of Technology,
Cambridge, Massachusetts 02139.

A. Heat Transfer Laboratories

(a) Prof. W.M.Rohsenow.

(b) Research on heat transfer.

(c) Measurements of thermophysical properties in collaboration with Dynatech (Vis.31).

B. Laboratory for Material Science

(a) Prof. W.D.Kingery.

(b) Research in ceramics. No further interest in thermophysical properties of refractories.

38. NBS — National Bureau of Standards, Cryogenic Division,
Boulder, Colorado 80302.

(a) R.L.Powell.

(b) Basic research in low temperature thermal conductivity, calibration and development of standards.

(c) λ: (longitudinal apparatus, floating heat sink) 4°K - 300°K.
ρ, S: at low temperatures.

Vis.No.

United States (continued)

39. NBS National Bureau of Standards,
Gaithersburg, Maryland.

A. Length Section

(a) R.K.Kirby

(b) Measurement of thermal expansion, collection of data.

(c) α: (optical method) up to 1600°C
(interferometric method) up to 1000°C.

B. Office of Standard Reference Data

(a) Dr E.R.Johnson.

(b) Collection of thermodynamic and transport data.

(c) No experimental work.

40. NBS National Bureau of Standards,
Washington, DC, 20234.
Environment Engineering Section,
Building Research Division, IAT.

(a) D.R.Flynn.

(b) Basic research in thermal conductivity up to 1000°C. Engineering measurements of thermal conductivity up to 600°C.

(c) λ: (longitudinal heat flow method combined with necked-down direct-heating method in one apparatus) from 100°C up to 1100°C
(longitudinal apparatus utilising Forbes bar method) two similar apparatuses with a combined temperature range from -160°C up to 850°C
(longitudinal apparatus) from -180°C up to +50°C
(absolute cut bar apparatus) from 100°C up to 1200°C
(longitudinal steam calorimeter apparatus) from 200°C up to 1400°C
(guarded hot-plate apparatus) from 0°C up to 60°C
(guarded hot plate apparatus) from -50°C up to +300°C in development.

41. ORNL Oak Ridge National Laboratory,
Metals and Ceramics Division,
Oak Ridge, Tennessee 51831.

(a) D.L.McElroy, W.Fulkerson, R.K.Williams, I.P.Moore, T.G.Kollie.

(b) Thermophysical properties of reactor and refractory materials.

(c) λ: (radial outflow apparatus) up to 1000°C
(radial outflow apparatus) up to 2200°C in development
(longitudinal apparatus) -190°C up to +80°C
(longitudinal apparatus) up to 100°C.
c: (pulse heating method) 100 - 1400°C.
ρ, S: up to 1000°C.

Vis.No.

United States (continued)

42. **SRI** Southern Research Institute,
2000 Ninth Avenue, South Birmingham, Alabama.

(a) C.D.Pears, C.M.Pyron.

(b) Contract research and measurement of thermophysical properties at high temperatures.

(c) λ: (radial outflow apparatus) up to 800°C
(radial inflow apparatus) up to 3000°C
(guaraded hot plate method) up to 540°C.
c: (drop calorimetry) up to 2800°C.
α: (push rod method) up to 3000°C.
ϵ: from 400 up to 2750°C.

43. **TPRC** Thermophysical Property Research Center,
West-Lafayette, 2595 Yeager Road, Indiana 47906.

(a) Prof. Y.S.Touloukian, R.W.Powell, D.P.Dewitt, C.J.Ho

(b) Collecting of literature on thermophysical properties, selecting most probable values, experimental control of the data of thermal conductivity.

(c) λ: (longitudinal method) -90°C to room temperature
(longitudinal comparative and absolute method) from 50°C to 400°C
(longitudinal comparative method) from 200°C to 1000°C
(direct-heating methods) from 200°C to 1000°C
all techniques are in preparation.

44. **UCC** Union Carbide Corporation, Carbon Products Division,
Parma Technical Center, 12900 Snow Road, Parma, Ohio.

(a) J.Bowman, W.W.Lozier, J.Meers, Mr Rittersbusch, Mr Weinard, Mr Weber.

(b) Research and development of carbon and graphite products. Thermophysical studies up to the highest temperatures.

(c) λ: (radial inflow method) up to 2700°C, pressures up to 40 atm
(direct-heating, rectangular bar method) up to 2500°C
(guarded hot plate method) up to 500°C.
a: (Xe flash) up to 900°C
(periodical arc-image method) up to 3000°C
(cooling-down method) up to 3000°C.
α: (interferometric method)
(X-ray) to 2700°C
(push-rod) to 1000°C.
ρ: up to 2500°C.

Vis.No.

United States (continued)

45. University of Tennessee,
Department of Chemical and Metallurgical Engineering,
Knoxville, Tennessee.

(a) Prof. Dr E.E.Stansbury.

(b) Basic research in metallurgical calorimetry.

(c) c : (adiabatic calorimeter) 500 - 1000°C.

46. Westinghouse Electric Corporation,
Astronuclear Laboratory,
Thermophysical Properties Laboratory,
Pittsburgh, Pa.15236, Box 10864.

(a) P.S.Gaal.

(b) Investigations of nuclear fuels and structural materials.

(c) λ : (comparative cut bar) 20 - 200°C
(radial outflow) 1000 - 2000°C.
a : (flash) 20°C.
α : (recording push-rod)
-200 to +1000°C.
(twin microscopic) for testing 10 samples concurrently,
20 - 2700°C
(interferometer) -200 to 20°C.
c : drop calorimetry to 600°C.
ρ : -200 to 3000°C.

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McClelland, J.D. — Review of Scientific Instruments, Vol.31, 1960, p.595.

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4. DATA AVAILABLE ON THERMOPHYSICAL PROPERTIES

The conclusion of the AGARD Structures and Materials Panel during the 22nd session (see also Section 1.1 of this report) was that the adequacy of published data for a limited number of materials, such as titanium alloys, heat-resistant nickel alloys, tungsten and graphite, as well as gold and platinum, should be studied and recommendations for further study should be made.

As shown in Section 1.3.3, some new literature exists in WADC Technical Report 58-476 (Lit.83). The comprehensive collection of the TPRC *Data Sheets on Thermophysical Properties* (Lit.84) has been issued continuously since 1962, and is supplemented each year. In addition two volumes with data on the thermal conductivity of the most interesting high-temperature materials (Lit.85, 86) have been published by the same institution. These publications, sponsored by the National Bureau of Standards will be available after revision within the National Standard Reference Data Series from the Superintendent of Documents, US Government Printing Office, Washington, DC, 20402. In January 1967 the first volume was published as NSRDS-NBS 8 (Category 5 - Thermodynamic and Transport Properties), wherein the thermal conductivity of the following metals is reported: Al (solid and liquid), Cu, Au, Fe, Mn, Hg, Pt, Pt/Rh, Ag, W as well as some non-metallic materials, such as Al_2O_3, BeO, borosilicate glass, diamond, MgO, Pyroceram, fused and crystalline SiO_2, TiO_2 and numerous liquids and gases (Lit.87).

A new comprehensive data collection on thermophysical properties of high-temperature materials has recently been issued by Y.S.Touloukian from the TPRC and it replaces the WADC report (Lit.83), which has become out of date. It is printed by the McMiller Corporation of New York, and is commercially available.

4.1 Data on Thermal Conductivity

In the following, parts of the two cited reports of the TPRC on the thermal conductivity of selected materials are discussed in more detail. The first one, (Lit.85), now replaced by (Lit.87), deals with the thermal conductivity of the metals Al, Cu, Au, Fe (pure and Armco), Mn, Hg, Pt, Pt/Rh, Ag and W. That of the metals Cd, Co, Pb, Mg, Mo, Ni, Nb, Ta, Sn, Ti, Zn and Zr, and of a number of types of graphites as well as the thermal conductivity of some gases, is summarised in the second report (Lit.86).

These two reports contain, not only the complete references in detail and diagrams of all these data as functions of temperature, but also a recommendation of the most probable values. Copies of the diagrams for some of the materials are included in this section as a basis for discussion on the adequacy of the available literature within the framework of an AGARD specialist group.

The following elements are represented:

Pure metals:	Au (gold)	(Fig.1)
	Cr (chromium)	(Fig.2)
	Fe (Armco iron)	(Fig.3)
	Ni (nickel)	(Fig.4)
	Pt (platinum)	(Fig.5)
	Ti (titanium)	(Fig.6)
	W (tungsten)	(Fig.7)
Non-metal:	graphite	(Fig.8, 9)

As can be seen from the diagrams (Figs.1-9), some of these materials such as Armco iron (Fig.3) and graphite (Fig.8) have been studied intensively, while on other metals only single values are available, such as for gold at temperatures above 100°C (Fig.1). It can also be seen that the various measurements on some distinct metals, for example on Armco iron (Fig.3), agree well, while on others, such as tungsten, they differ so widely that they are not comparable. The reason for these differences is firstly in the incomparability of the specimens caused by their different quality and their inhomogeneity, secondly in the different methods of measurement and finally in the differing skill and care of those carrying out the measurements.

While the collection of references and compilation of data by the TPRC is universally acceptable, recommendations on most probable values differ according to different specialists.

It is impossible to dispute, in principle, the arguments of the critics that the TPRC recommendations are influenced too much by subjective judgement. (A practical example is the thermal conductivity of platinum, which seems to have been clarified at the last conference on thermal conductivity (Vis.31); the agreed value differed from the values recommended at this time (compare Figure 13 with Figure 5).)

A visit to the Thermophysical Properties Research Center at work, and discussion with scientists there, gave the impression of a very critical selection and balancing of the individual results as objectively as possible. As a first estimate these recommended values are very useful and great efforts have been made to find recommended values, based very often on highly unreliable results. But for fundamental research, the study of the literature referred to in these compilations should be done by the research scientist himself in relation to his special problems. The collection of data in Figures 1 - 9 shows very clearly the need for a comprehensive programme of collaboration and fresh comparisons between the work of specialised institutions all over the world.

With regard to measurement accuracy, it seems premature to discuss in detail the thermal conductivity of alloys, because the thermal history of the specimens is an additional source of error. The short range order in the area of solid solutions of Cr-Ni alloys, apart from segregations, recrystallization etc. in complex high temperature alloys should be mentioned.

Besides these American efforts in collecting data, the independent data collection in the well-known Landolt-Börnstein book should be noted. The chapter on thermal conductivity was written by K.H.Bode (Lit.88).

4.2 Data on Thermal Diffusivity

The TPRC data sheets on thermophysical properties (Lit.84) have so far included the diffusivity of the following materials:

Metals:	Al, Be, Cr, Mo, Ni, Pt, Ta, Ti, W, Zn.
Alloys:	Ti/Mn, Ti/Al, Co/Cr, Ni/Cr, Ti/Nb.
Non-Metals:	TiC, TaC, graphite.

A further up-to-date collection of thermal diffusivity data appears in Y.S.Touloukian's recent book on "Thermophysical Properties of High Temperature Materials" (Lit.166).

4.3 Data on Thermal Expansion

Newly revised data on thermal expansion of interest to the Panel are given in Y.S.Touloukian's book (Lit.166) and by R.K.Kirby (Vis.39, Lit.89).

Thermal expansion data are not included in the TPRC "Data Sheets on Thermophysical Properties".

Many experimental results in the study of the thermal expansion of refractory materials have come from Conway's group (GEC, Vis.33, Lit.90). For example Figures 10 and 11 show his latest results in which an analogous behaviour of all these materials is shown.

According to Kirby (Vis.39), the use of a logarithmic plot of expansion versus temperature is questionable since there is no reason to expect the plot to be linear. The approach to line[illegible] apparently due to the fact that most materials expand about the same amount [illegible] from room temperature to the melting point.

The bulk thermal expansion of graphite bodies is a special problem. It has been shown in several papers that the bulk expansion behaviour is not connected with the lattice expansions (see Figure 15 and Lit.78). This is caused by anisotropy, porosity, bulk density and distortions (see Figure 16 and Lit.105).

4.4 Data on Emissivity

Data on radiation properties are reported in Touloukian's recent book on thermophysical properties of high temperature materials (Lit.166) and in the TPRC data sheets (Lit 84).

So far emissivity values of the following materials of interest to the AGARD Panel have been published:

Metals:	Cr, Co, Au, Fe, Mn Mo, Ni, Pt, Rh, Ag, Ta, Ti.
Alloys:	Ni/Cr, Ti/Mn, Ti/Nb, Ti/Al.
Non-Metals:	TiC, SiC, graphite.

A team of specialists is collecting data on emissivity for the new edition of the Landoldt-Börnstein book (Lit.88). Those interested may contact C.Tingwald, PTB (Vis.10).

5. SURVEY OF THE THEORY

It is not the purpose of this report to enter into a profound discussion of the theory of thermophysical properties. In this section only a brief survey of the most important topics is given in relation to further development of the technical application of materials, especially at high temperatures, where measurements are difficult and unreliable. A better knowledge of present theories and their future development enables one to understand the atomic mechanisms and at least the phenomenology. Furthermore, understanding the theory enables one to estimate the possibility of extrapolating data to other temperature regions, or as a future aim, of calculating thermophysical properties theoretically, without experiment. Discussion is mainly of the theory of thermal conductivity; the theories of thermal expansion and of specific heat are mentioned only briefly.

5.1 Theory of Transport Phenomena

Although thermal conductivity is the main subject, a general survey of electrical conductivity is given as well. General treatments of the theory of thermal conductivity are given by P.G.Klemens (Lit.137, 138), H.Jones (Lit.139) and J.M.Ziman (Lit.140, 141).

The mechanism of heat transport is a highly complicated phenomenon, through the combination of many related effects which cannot be considered separately. The main contributions to heat transport come from phonons and free electrons, but ambipolar heat flow, photons, spin waves and excitons also contribute.

With the concept of a solid as a system of coupled oscillators which transmit the thermoelastic waves as a basis, it is convenient to write, by analogy with the kinetic theory of gases:

$$\lambda = \frac{1}{3} \sum_j c_j v_j l_j , \tag{14}$$

where c is the specific heat, v the mean velocity of particles and the mean free path of particles; j describes the conducting processes.

The heat flow by these carriers is limited by a variety of scattering mechanisms, the most important of which are

phonon - phonon scattering,
phonon - point defect scattering,
phonon - electron scattering,
phonon - boundary scattering,
electron - phonon scattering,
electron - point defect scattering.

The main problem in the understanding of thermal conductivity is the determination of the mean free path of the different scattering mechanisms.

Because, in non-metallic solids, only phonon scattering occurs, this group of materials will be discussed first. In metals electron scattering only is dominant and this group will be discussed later.

5.1.1 *Conductivity in Insulators*

Heat conduction in solids without free electrons is due to lattice waves. In this so-called lattice thermal conduction, λ_L, the heat is transported by directional, cooperative quantized vibrations (called phonons) of the thermally-excited interacting lattice ions. The same phonons are responsible for specific heat and thermal expansion in metals.

The phonon distribution is disturbed by a temperature gradient and the interaction processes tend to restore the equilibrium distribution. The balance of these two trends causes a steady-state non-equilibrium distribution and a resulting heat current, which therefore defines the thermal conductivity.

The process of obtaining an expression for the lattice thermal conductivity starts with the Boltzmann equation for phonons. The solution is generally obtained either by the use of a variational procedure with trial functions for the phonon distribution, or by the use of the relaxation-time concept, in which it is assumed that the disturbed occupation number of each phonon mode returns exponentially with time to its Planck distribution value. Within the relaxation-time framework, the simplest solution of the Boltzmann equation yields an expression for the conductivity,

$$\lambda = \frac{1}{3} \int c(\omega)\, v^2(\omega)\, \tau(\omega)\, d\omega , \tag{15}$$

where $c(\omega)$ is the contribution to the specific heat per unit volume from phonons of angular frequency ω, $v(\omega)$ is their group velocity and $\tau(\omega)$ their effective relaxation time. The expression for the mean free path of phonons l can similarly be written

$$\lambda = \frac{1}{3}\int c(\omega)\, v l(\omega)\, d\omega . \tag{16}$$

In insulators without point defects the mean free path is limited by departures of the lattice vibration from the simple harmonic. In the resulting interactions, the energy of the interacting phonons is conserved, while the vector sum of the wave numbers may either be conserved or changed by a reciprocal lattice vector. The interactions may involve any number of phonons, but those involving three phonons are probably the most important. The conservation conditions may then be written

$$\hbar\omega_1 + \hbar\omega_2 = \hbar\omega_3 \tag{17}$$

$$q_1 + q_2 = q_3 + g . \tag{18}$$

ω is the frequency, q the wave number and g the reciprocal lattice vector. If $g = 0$ the interaction is a Normal process (N process), while if g is a reciprocal lattice vector there is an Umklapp process (U-process). N- and U-processes influence the thermal conductivity in a different way. N-processes do not change the heat flow directly, but are means for energy exchange among the phonons. U-processes change directly the energy and the direction of phonons and in this way the heat flux is limited by the efficiency of the process in restoring the equilibrium phonon distribution.

Regarding the temperature dependence of the conductivity in non-metals, three regions can be distinguished. In region 1, at sufficiently high temperatures, when $T \gg \theta_D$ (θ_D = Debye temperature) the heat flow through the crystal is determined by this mutual scattering of phonons, in particular by U-processes. The conductivity is an intrinsic property of the substance and is inversely proportional to the number of phonons N_{ph}. N_{ph} is given by Bode (Lit.88) as

$$N_{ph} \propto \left(\frac{\hbar\omega}{kT} - 1\right)^{-1} \tag{19}$$

and for

$$T \gg \frac{\hbar\omega}{k} , \quad N_{ph} \propto \frac{kT}{\hbar\omega} . \tag{20}$$

From these results, the mean free path l_{ph} is proportional to the reciprocal of the absolute temperature T :

$$l_{ph} \propto \frac{1}{T} \tag{21}$$

or, for high temperatures,

$$\lambda \propto \frac{1}{T} . \tag{22}$$

This temperature dependence is always observed for crystalline dielectric materials at high temperatures and can be used for a careful extrapolation to higher temperatures if the temperature dependence of the lattice part in region 1 is known.

At increasing temperatures the thermal conductivity becomes more and more complicated because of additional conducting mechanisms, for instance scattering processes of higher order and conduction by photons and electrons. The behaviour is only understood qualitatively.

Of the proposed special theories for isolators, that of Leibfried and Schlöman (Lit.145) is used most. The theory was developed with the assumption of an infinite ideal crystal, having no lattice defects and impurities, and permits the calculation of the number of U-processes and their influence on the thermal conductivity. Some corrections have been made by Steigmeier and Kudman (Lit.146), relating to the temperature and material dependence of the non-harmonic parameters and to the consideration of higher order processes (4-phonon-processes). The differences between experiment and theory are considerable and the quantitative use of these theories is restricted.

With decreasing temperature, the probability of U-processes is diminished. According to the theory of Peieris (Lit.142) the temperature-dependence of the thermal conductivity in this temperature range 2 is given by

$$\lambda \propto \left(\frac{T}{\theta_D}\right)^n \exp\left(\frac{\theta_D}{bT}\right) \tag{23}$$

and should be infinite at the lowest temperatures.

According to Klemens, Berman and Ziman (Vis.20), the scattering on point-defects become dominant in this region. This scattering process is only effective for phonons with high frequency. With decreasing temperature the number of phonons with high frequency diminishes. Now the influence of N-processes becomes important; the frequency spectrum is altered and phonons can be changed from low to high frequency.

By this mechanism the exponential increase of conductivity with decreasing temperature is inhibited, resulting in a reversed temperature-dependence at the lowest temperatures of thermal conductivity (temperature region 3), which passes through a maximum at about

$$\frac{T}{\theta_D} = \frac{1}{10} - \frac{1}{30} .$$

This maximum can only be observed for quite pure materials. A systematic study of the influence of point-defects in this area between the temperature ranges 3 and 2 has been made by Berman and his collaborators (Vis.20, Lit.143) with isotopes of light elements, originally LiF now He^3/He^4. In such well defined mixtures of isotopes the alteration of the relaxation time for N-processes can be calculated.

Callaway (Lit.144) has developed a relaxation time theory which facilitates the calculation of the low temperature behaviour of thermal conductivity, including the maximum region. He succeeded in introducing a combined relaxation time for the various scattering processes effective in this temperature region. His expression for the thermal conductivity consists of two terms λ_1, normally the main contribution, and λ_2, which may be regarded as a correction term related to the treatment of N-processes. The Callaway theory has been used successfully to describe the thermal conductivity of a wide variety of crystals. With some reservations the behaviour of lattice thermal conductivity at low temperatures is well understood and can be treated in a partly quantitative manner. An exact theory based on theoretical calculation of the lattice forces does not exist at present.

Within the region $T \ll \theta_D$, the mean free path of the phonons reaches the magnitude of the crystal dimensions, and then the thermal conductivity is determined by the crystal diameter (Casimir, Lit.147) and also by the specific heat, for the wave velocity is approximately constant. We may write

$$\lambda \propto c_0 d \propto T^3 d \; , \tag{24}$$

where d is the crystal diameter.

In this temperature region the thermal conductivity is no longer an intrinsic material property. The simple Equation (24) does not express the completely different mechanism of scattering. A property on a macroscopic scale, such as the thermal conductivity [$\mathrm{watt}^{-1}\mathrm{sec}^{-1}\mathrm{degree}^{-1}$], is no longer significant, if this property depends only on the geometry of the specimen considered. Taking this into account, it is not surprising that the specimens of bad-conducting glass with no crystalline long-range order show a better conductivity than crystalline graphite, which is a much better conductor at higher temperatures.

For the temperature range near absolute zero, the thermal conductivity is directly proportional to temperature. This is because the thermal resistance is entirely caused by small-scale defects with dimensions smaller than the dominant phonon wave length.

5.1.2 *Thermal and Electrical Conductivity in Metals*

Heat transport in a metal is principally achieved by the motion of conduction electrons. The lattice thermal conductivity, λ_L , becomes significant in less pure metals and in alloys, though it is still small compared with the electronic contribution (λ_e). Both conduction mechanisms are limited by various scattering processes, each process acting as a separate resistance in series. The measured conductivity λ_m is approximately the sum of both parts,

$$\lambda_m \simeq \lambda_e + \lambda_L \; . \tag{25}$$

The lattice part of the thermal conductivity amounts only to a few per cent. This conduction mechanism has been discussed in Section 5.1.1 for non-metallic materials. Therefore, in the following, only thermal conductivity by electron is considered.

There are two main scattering processes which limit the electronic conductivity in the expression Equation (25). The first is the scattering of conduction electrons by thermal vibrations of the lattice (the phonons), as represented by the electron-phonon resistivity, W_p , a characteristic property for a given metal. This scattering is most important at intermediate temperatures (about 40 to 80°K) and higher. The second process is the scattering of conduction electrons by imperfections (both impurity atoms and lattice defects), as represented by the electron-defect resistivity, W_0 . This scattering is most important at the lower temperatures. The reciprocal of the total electronic thermal conductivity, λ_e , is the total electronic thermal resistivity, W_e , which is assumed to be the sum of the two resistivities, W_p and W_0 , plus a small deviation term W_{p0} , that is,

$$\frac{1}{\lambda_e} = W_e = W_0 + W_P + W_{P0} \quad (26)$$

This equation is analogous to the one used in electrical circuit theory for the total resistance when the resistances are in series.

The deviation term has been studied theoretically by Kohler (Lit.149) and independently, in experiments, by Powell et al. (Lit.150). It is of the form

$$W_{P0} = \frac{\alpha W_P W_0}{(\beta W_P + \gamma W_0)} \quad (27)$$

where α, β, and γ are first-order constants and can be determined experimentally. Though theoretically significant, the term is numerically important only for very pure metals.

Whenever the interaction term W_{P0} is negligible, the thermal equivalent of Matthiessen's rule for electrical resistivity,

$$W_e = W_P + W_0 , \quad (28)$$

is approximately correct.

Powell (Lit.151) has given a graph for this relation and its equivalent for conductivity (Fig.12). With knowledge of the two separate terms of Equation (28), prediction of the total electronic thermal conductivity becomes feasible.

Both theoretical and experimental research have led to expressions for magnitudes and temperature-dependences of the electron-phonon and electron-defect resistivities:

$$W_P = AT^n \quad (29)$$

($n \simeq 2$ to 3, $T < 40^0K$); $W_P \simeq$ a constant (near room temperatures); $W_0 = B/T$ (at all temperatures). The constant A in the electron-phonon resistivity term is related to the intrinsic properties (including the characteristic temperature, θ_D) of a given metal and will not change for minor additions of chemical impurities or physical imperfections; B in the electron-defect resistivity term is related to the given amount of imperfection and residual electrical resistivity of the actual specimen. Above 40^0K, the electron-phonon resistivity approaches a constant value, often labelled W_∞ .

In Figure 12 the Lorenz number L is also shown. It is defined according to the Wiedemann-Franz-Lorenz law as follows:

$$L = \frac{\lambda_e}{\sigma T} = 2443 \times 10^{-8} . \quad (30)$$

Sommerfeld related the Lorenz number L to the Boltzmann constant k and to the elementary charge e :

$$L = \frac{\pi^2}{3} \times \left(\frac{k}{e}\right)^2 . \quad (31)$$

As seen in Figure 12, the Lorenz ratios for a high conductivity specimen extrapolate to approximately the Sommerfeld value at 0°K but fall considerably below it at higher temperatures. The behaviour of the low-conductivity alloys is different: the values between about 10 and 60°K are higher, but above 60°K the values are again lower. The regions where the ratios are above the Sommerfeld value indicate temperature ranges where lattice conductivity is important.

The Lorenz ratio should be constant if the conduction electrons are scattered elastically. This is approximately true at high temperatures, where there is a large amount of thermal vibration giving rise to large electron-phonon scattering, and at low temperatures, where the residual term is predominant in the electrical resistivity. At intermediate temperatures, the condition of elasticity no longer holds, the Lorenz ratios decrease considerably from the Sommerfeld value, if the lattice thermal conductivity is negligible. Any significant amount of lattice thermal conductivity will raise the Lorenz ratio above the value it would have had if only the electronic term in the thermal conductivity were considered.

A more detailed expression for the Lorenz number has been derived by Ziman (Lit.140, 141):

$$L_0 = \left[\frac{\pi^2}{3} \frac{k^2}{3e^2} - S^2 + \frac{8T^2\pi^4k^4}{45\, e^2\sigma(\zeta)} \left(\frac{\partial^2\sigma(\epsilon)}{\partial\epsilon^2} \right)_{\epsilon=\zeta} + \dots \right] . \tag{32}$$

Here the Seebeck coefficient S enters into the second term. Up to now the third term has been neglected but, according to McElroy (Vis.41), it should be taken into account in the future and used to explain deviating results.

Finally, a simple correction of the Wiedemann-Franz-Lorenz relation given by Bäcklund (Lit.152), which is widely used by experimental researchers, should be mentioned.

Bäcklund separates the electronic thermal resistivity W_e into three parts:

W_{eT} due to thermal scattering of electrons,
W_{eS} due to spin-disorder scattering,
W_{eJ} due to impurity scattering.

He writes

$$W_e = W_{eT} + W_{eS} + W_{eJ} \tag{33}$$

$$\rho = \rho_T + \rho_S + \rho_J \; . \tag{34}$$

with $\rho_T = -\bar{\rho}_0 + bT$.

where $\bar{\rho}_0$ is the residual electrical resistance and b a constant. The Wiedemann-Franz-Lorenz law can then be written as

$$W_e = \frac{1}{\lambda_e} = \frac{\rho + \bar{\rho}_0}{LT} . \tag{35}$$

This relation holds even for the transition metals and is valid in the region where $\rho \propto 1/T$. $\bar{\rho}_0$ is determined by the temperature-dependence of the electrical resistance at intermediate temperatures.

Bäcklund reports a good approximation to the real behaviour for iron between 90°K - 1300°K and McElroy confirms this.

5.1.3 Comparison Between Experiment and Theory

Better experimental methods and better defined specimen materials have become available in the last few years and some research teams have started basic research to correlate these new experimental results with the present theories. The work of the following teams will be discussed:

The research team at the Clarendon Laboratory, Oxford (Vis.20), studying lattice conductivity at low temperatures.

The research by R.L.Powell on the low temperature behaviour of metals (Vis.38).

The work of Laubitz (Vis.25), on testing the validity of present theories for monovalent metals up to 1000°C.

The studies of the ORNL group (Vis.41) to explain the conductivity behaviour of several materials up to 1000°C and to try an extrapolation to higher temperatures.

5.1.3.1

Berman (Vis.20) has shown that the low-temperature thermal conductivity of dielectric crystals may be explained very satisfactorily by Callaway's relaxation-time theory. However, when $T \simeq \theta_D$ the agreement is only qualitative in most cases.

5.1.3.2

R.L.Powell is experimenting in the low temperature region. With a set of copper specimens with various impurity contents and with various lattice defects, he found that the intrinsic thermal resistance and the residual resistivity are additive only if the latter is caused by differing physical states, and not if it is caused by different chemical impurities, which influence the intrinsic resistivity additionally.

5.1.3.3

Laubitz and his group are trying to calculate theoretically the electronic part of the thermal conductivity of monovalent metals, using experimental results on the electrical resistivity and a theoretical calculation of the Lorenz number. The lattice part of the thermal conductivity is calculated theoretically, using Leibfried and Schlömann's approach (Lit.145).

As shown recently, the measured values agree quite well with the theory at temperatures higher than $T/\theta_D \simeq 2$. The agreement could be extended to $T/\theta_D \simeq 1.5$, if it is assumed that the number of free electrons per atom is about 0.6 instead of 1.0, which is compatible with present-day knowledge.

5.1.3.4

The main aim of McElroy's research group (Vis.41) at Oak Ridge is to predict the temperature behaviour of interesting materials at higher temperatures with the help of accurate measurements in the range of about 0 to 1000°C. On the same specimen the thermal conductivity, the electrical resistivity and the thermoelectric power are measured. Using Ziman's expression for the Lorenz number, separation of the measured thermal conductivity into electronic and lattice parts is attempted. A lattice contribution of about 25% is found for tungsten. A prediction of the thermal conductivity up to 1400°C by extrapolation seems to be possible, using this research.

5.1.3.5 Practical use of the theories for predicting thermal conductivity

As pointed out in the previous sections, a theoretical prediction of thermal conductivity behaviour seems to be possible for well-defined pure monovalent metals in the medium temperature range, as shown by Laubitz on copper between 300 and 1000°C.

For temperatures below 300°C the calculated values agree with the experimental data only if arbitrary assumptions are made. Future work on other monovalent metals should determine whether the calculation of thermal conductivity from electrical data is justified.

For temperatures above 1000°C no experimental comparison with theory exists. Additional difficulties in the calculation can be expected because of further scattering processes.

For alloys the effect of point-defects can be estimated roughly, if these are distributed homogeneously. (See the latest contributions of Klemens (Lit.136).)

There is no chance possibility of calculating the thermal conductivity in complex alloys if thermal instability can be expected. It is also impossible to derive the thermal conductivity of heterogeneous materials from the theory.

The extrapolation of exactly measured conductivity data to higher temperatures, using electrical data additionally, seems most promising, although the complexity of the scattering mechanism has often not been understood completely so far.

The conductivity behaviour of pure non-metallic conductors at very low temperatures is beginning to be understood.

5.2 Specific Heat Theories

Debye's theory on specific heat is generally considered to be sufficient for the consideration of this thermophysical property. Debye has assumed a simple isotropic three-dimensional frequency distribution and characterises the temperature-dependence of the specific heat at constant volume by the limiting frequency ν_{max}, which is a characteristic of each material. The specific heat, according to Debye, is related to ν_{max} by

$$c_v = \frac{9 F h^2}{k T^2 \nu_{max}^3} \int_0^{\nu_{max}} \frac{\nu^4 e^{h\nu/kT} d\nu}{(e^{h\nu/kT} - 1)^2} \qquad (36)$$

and the Debye temperature is defined as

$$\theta_D = \frac{h\nu_{max}}{k} . \tag{37}$$

At very high temperature $c_v = 3R$, because $e^{h\nu/kT}$ becomes small when $ht \gg h\nu$, whereas at low temperatures $c_v = aT^3$, which is the same dependence as is shown for the phonon part of the thermal conductivity at very low temperatures in Equation (24).

The real frequency spectrum can be determined by neutron scattering measurements (see J.A.Krumhansl, Lit.155). Knowledge of the frequency distributions enables the thermodynamic properties of the crystals to be calculated by means of statistical formulae. This field of research is important for discussion of the theory of thermophysical properties.

Further efforts in solid-state physics have been directed towards a separation of the various parts contributing to the specific heat, which is determined experimentally, using Equation (38). A discussion of new results has been given by Braun and Kohlhaas (Vis.13, Lit.103).

$$c_p = (c_p - c_v) + c_D + c_E , \tag{38}$$

where c_p is the experimentally determined value, c_v the specific heat at constant volume, c_D the Debye part and c_E the electronic part. Difficulties arise when the specific heat at constant pressure is to be corrected to constant volume, using the expression

$$c_p - c_v = \frac{T V_0 \beta^2}{\kappa} , \tag{39}$$

where β is the volume expansion coefficient, α the isothermic compressibility and V_0 the atomic volume at the standard temperature T_0. β can be found from $\beta = 3\alpha$, where α is the linear coefficient of thermal expansion. However the temperature-dependence of κ is usually unknown, since the empirical relation (40) of Nernst and Lindemann (Lit.154), with a constant A to be determined by experiment, is not generally valid.

$$c_p - c_v = A c_p^2 T . \tag{40}$$

Braun and Kohlhaas therefore propose to divide the experimental c_p versus T curve directly into the lattice part, the electron part and the magnetic part. By this procedure the $(c_p - c_v)$ correction is needed only for the lattice part.

The lattice part $c_{p\,latt}$ is composed of the harmonic part c_H and a sum resulting from the non-harmonic portion of the lattice vibration, consisting itself of the part $(c_p - c_v)_{latt}$ and c_{vA}.

For the harmonic approximation, the crystal expansion does not have to be considered:

$$c_{vH} = c_{pH} = c_H . \tag{41}$$

According to Debye's theory

$$c_H = c_D \frac{\theta_D}{T} = 3R \left(1 - \frac{1}{20}\right) [(\theta_D/T)^2 \ldots\ldots] . \tag{42}$$

For the correction of $(c_p - c_v)_{latt}$ the Grüneisen equation (Lit. 153) can be used:

$$(c_p - c_v)_{latt} = c_D \beta G T , \tag{43}$$

where the Grüneisen coefficient G , given by

$$G = \frac{\beta V_0}{\kappa c_D} , \tag{44}$$

is nearly independent of temperature.

The non-harmonic part of the specific heat has so far only been approximated by

$$c_{vA} = AT , \quad A \text{ being } -3 \ldots\ldots -5 \times 10^{-3} . \tag{45}$$

The electronic part of the specific heat can be expressed as

$$c_E = \gamma T[1 + F(\xi_0)T^2] = \gamma T + bT^3 , \tag{46}$$

where $F(\xi_0)$ is a function of the Fermi energy ξ . For free electrons

$$F = -3\pi^2/10\,T_0^2 .$$

γ can be calculated from specific heat measurements at low temperature.

The magnetic part of the specific heat in ferromagnetic materials can be expressed as

$$c_{pM} = c_p - c_D G T + c_D + c_{pE} + c_{vA} . \tag{47}$$

Using this expression Braun and Kohlhaas (Lit. 103) have separated the experimental results on specific heat measurements of the metals Fe, Co and Ni. They have used the adiabatic calorimeter (see Section 2.4.1.3) and the results are shown in Figure 13. One can recognise the interesting consequences of modern research in the field of specific heat, showing the deviation of c_D from c_p .

5.3 Remarks on the Theory of Thermal Expansion

Thermal expansion is caused by the asymmetry of the atomic lattice vibrations.

Originally the theoretical treatment began from consideration of the potential energy of two lattice components as a function of the asymmetric elongation:

$$\varphi_{(r)} = -A r^{-m} + B r^{-n} . \tag{48}$$

Grüneisen has given two rules: firstly

$$\alpha = \text{constant} \times c_v , \tag{49}$$

which means that the temperature-dependence of the thermal expansion will follow that of the specific heat c_v. Or inversely: by exact measurement of the lattice expansion the temperature-dependence of the specific heat c_v can be controlled.

Grüneisen's second rule, expression (50), enables one to estimate the constant in expression (49).

$$3\alpha = \frac{c_v \kappa}{V_0} \, \frac{m + n + 3}{6} = \frac{c_v \kappa}{V_0} G . \tag{50}$$

m and n, the exponents of expression (48), are independent of temperature within a wide temperature range.

As the compressibility κ is nearly temperature-independent, the cubic coefficient of expansion 3α ($= \beta$ for isotropic materials) will follow Debye's T^3 law also at low temperatures.

In recent years a careful experimental investigation of the Grüneisen parameter G has been made for some monatomic crystals by several authors. The general conclusion is that G remains almost constant over a wide range of temperatures near the Debye temperature θ_D of the solid, while in the lower temperature region a marked deviation from the Grüneisen rule occurs. For copper this deviation results in a reduction in G of about 15%. A qualitative explanation of the effect has been given by Bijl and Pulian (Lit.156) on the basis of a continuum model of the crystal; however, the theoretical range of temperatures over which deviation would occur is much higher than the one experimentally observed.

In Gallotto's research group (Vis.17) basic considerations of thermal expansion behaviour of solid argon are treated, resulting in a new theory by Gallina and Omini (Lit.135). Argon is used because only of this material are the intermolecular forces known with sufficient accuracy.

6. CURRENT ACTIVITY ON THERMOPHYSICAL PROPERTIES IN NATO COUNTRIES AND PROPOSALS FOR FUTURE COLLABORATION

6.1 Current Activity

In the course of my visits through the NATO countries I found a large number of institutions and specialists interested in, and working intensively in, the field of thermophysical properties of solid material at high temperature (see Section 3.1).

In Europe the specialists work mostly in establishments or nuclear energy centres or in industry. It seems that most of them do not have sufficient contact with other specialists. In many cases they even do not know each other, nor are they aware of the research problems of others. The initiative of R.W.Powell in gathering all the specialists together for a discussion in Teddington in 1964 is significant (Lit.106).

but now all activity in the field of thermophysical properties has been discontinued at Teddington.

On the other hand, specialists in the USA are collaborating and the Canadians have joined them. This led firstly to special meetings, the most important of which are the annual Thermal Conductivity Conferences which have been held since 1961, sponsored in turn by the leading laboratories. A limited number of copies of the proceedings, containing all the reports presented, are then issued (Lit.107-112, see also Section 1.3.4).

Secondly, this personal contact between specialists resulted in cooperative tests on interesting specimens (see also Section 6.2). The first one was started by Battelle on the thermal conductivity of iron, and last year the differences in the published data on the thermal conductivity of platinum were clarified (Vis.30, Lit.91, 92).

Furthermore, some leading specialists are trying to prove the validity of the present theories on thermal conductivity by precise measurements. Such fundamental research is done in Canada (Vis.25) and in the USA (Vis.41), (see also Section 5).

For applied research, an extended technical programme, supported by the US Air Force, with the aim of developing high-temperature thermal conductivity standards, was initiated in 1965 (see Section 6.2).

The efforts made to create specimen banks for standards deserves mention. The National Bureau of Standards at Boulder, Colorado, is preparing standards for thermal conductivity measurements at low temperatures. Similar efforts are under way in Germany, where thermal conductivity standards for the medium temperature range can be bought from the PTB (Vis.10).

A central institution for studies in thermophysical properties of materials is the TPRC in West-Lafayette, Indiana (Vis.45), supported by the NBS, the US Air Force, American industry and other institutions. The task of this centre is to gather all available data on thermophysical properties (cf. Section 4), to select well-proved data and to control their validity by experiments at the centre.

Although a large number of laboratories are equipped for research on thermophysical properties, not all of them seem to be able to make full use of their capacity and suitable research projects are awaited.

6.2 Standards for Thermal Conductivity Measurement

6.2.1 General

The first step towards achieving comparable results in thermal conductivity measurements is to make suitable, well-defined standards available. Many efforts have been made to find such standards for high temperature work.

A.D.Little Inc. (Vis.36, Lit.93, see also Section 6.2.2) have established the following criteria for an ideal thermal conductivity standard:

(a) There shall not be any phase change.

(b) The radiative contribution to the heat transfer must be negligible.

(c) The material should be isotropic, for many types of thermal conductivity equipment require this.

(d) The material should have a homogeneous microstructure.

(e) The microstructure should remain stable, regardless of the heat treatment.

(f) The vapour pressure must be low, for many instruments work in vacuum.

(g) The sample materials must have sufficient mechanical strength to withstand the loading applied during the measurements and they must not creep or distort during the test period.

(h) The thermal shock resistance should be good.

(i) The material should have good machinability and good fabricability.

(k) The melting point must be sufficiently high.

(*l*) The material must be chemically stable.

(m) The material should be easily available and should not be too expensive.

Recrystallisation has not been considered, in the study referred to, as a severe disadvantage, since grain growth can be minimised by prior annealing.

The use of very pure metals is not favourable because their physical properties are highly sensitive to small amounts of impurity. Only in the region below room-temperature is this aspect important. Thus only a relatively small group of materials may be appropriate.

At ambient temperatures, and up to about 1000°C, the following metals have received attention from several investigators:

Armco iron and high purity iron,
nickel,
Inconel 702,
platinum.

Iron was found to be an excellent thermal conductivity standard below 910°C (phase change to γ-iron). High purity iron is a better reference material than Armco iron, which is rather sensitive to heat treatment. The temperature-dependence of the thermal conductivity can be controlled by calculating the thermal conductivity from the electrical resistivity and the lattice portion (Lit.92).

Because of the chemical instability of iron the more stable nickel is proposed as a standard. According to W.Spyra (DEW, Vis.6) it can be used between -100 and +350°C, where the accuracy of the smoothed curve is within about ±1.5% (Lit.94).

To overcome the chemical instability of iron at high temperature, Inconel was proposed as a standard. But, as recent studies have shown (Lit 95, 96), this complex alloy is unsuitable because of its sensitivity to heat treatment.

Platinum is chemically stable over the whole temperature range, but very different results on its thermal conductivity have been reported (see Figure 5). According to the last conference on thermal conductivity (Vis.31, Lit.91), agreement between all those scientists present was achieved. The thermal conductivity of platinum of not extremely high purity is now known up to 1000°C within ±2% and, although the thermal conductivity is affected by purity, platinum seems to be a suitable standard up to 1000°C (see Figure 14).

6.2.2 The US Air Force Programme for High Temperature Standards

The US Air Force has given a contract to A.D.Little Inc. to develop standards in the temperature range between 1000 and 2700°C (Vis.27, 35). Within this programme materials within a wide range of conductivities and readily available in consistent quality are considered.

The materials are distributed by A.D.Little to ten institutes using different methods for comparison of the measurements. The first results were expected in the spring of 1967 and the programme was due to be finished by the end of 1967. At the time of the visit to A.D.Little Inc. (October 1966) the following materials had been selected:

ceramics:	Al_2O_3, ThO_2,
intermetallics:	sintered TiB_2,
metals:	sintered and arc molten W,
graphites:	the types CER and RVD of the UCC and POCO 1924-H.

It would be of great interest for research workers in Europe to compare these American test results with their own.

6.3 Proposals for Future Collaboration

In discussing the special problems in this report, several proposals for collaboration within the AGARD framework have already been made. In this section some ideas on the necessity and priority of research in different fields is discussed.

6.3.1 Literature

The literature has been collected by the TPRC (Vis.42) and is universally available on request and no further individual activity seems to be necessary. A team of specialists within AGARD could seek methods of direct collaboration with this well organised research centre.

A critical discussion of these references by a team of specialists within AGARD would be helpful to the TPRC in proposing recommended values.

6.3.2 Standards

As already mentioned, the availability of standards is the first step towards a comparison of methods and equipment. The standards should be tested by as many specialists as possible.

The best procedure for AGARD specialists would be to join one of the existing American programmes, eventually enlarging or supplementing them from more general points of view.

Such collaboration would be of advantage not only to European specialists, giving them a good contact with this organised research, but also to the American programmes themselves, by using the European research capacity for comparison beyond the framework of the research contract.

6.3.3 Specialist Conferences

As the annual American Conferences have shown, personal contact and continued discussion promote the development of this science in an unique way. The improvement in accuracy, to ±2%, of experimental results on thermal conductivity at 1000°C is due to interchange of experience during these well-coordinated conferences. The best proposition is for AGARD specialists to enlarge these conferences through participation by the European nations, provided that the difficulties of travelling and expense can be overcome. European conferences on thermal conductivity, or more generally on "thermophysical properties at high temperatures", should be arranged.

6.3.4 Comparative Testing of Different Methods

The activities of scientists in the measurement of thermophysical properties develop from their special research interests, such as solid-state physics, nuclear technology, aeronautical research, general engineering aspects, etc. Every scientist uses methods most suitable for his own purposes and, since the setting-up of the apparatus and the achievement of precise measurements is very time-consuming, all scientists try to find experimental solutions only for their own problems. The use of different methods for comparing results seems to have been neglected. In the further development of a standard programme, a critical comparison of the different methods, and perhaps development of newer, better methods, should be considered. This is especially important for the electrical direct-heating steady-state methods and the periodic non-steady-state methods. Neither group has found as much favour as seems to be necessary for a critical examination.

Part of this problem is to obtain a uniform definition and use of such terms as repeatability, reproducibility, precision and accuracy.

6.3.5 Development of the Theory

The validity of existing theory on thermal conductivity should be proved experimentally by exact measuring methods and it might then be extended to simple alloys. However, the development of methods should be undertaken before starting new experimental work on verifying the theory. Although theoretical pre-calculation of the thermal conductivity does not seem possible, the extrapolation of precise experimental results to higher temperatures, as well as the estimation of the effect of alloying elements in simple alloys, does seem possible.

6.3.6 Research on Thermal Expansion

Bulk thermal expansion is caused by lattice expansion. Although apparatuses for the determination of lattice parameters at high temperature are well developed, no systematic research on lattice expansion for refractory materials has been done. Theoretical considerations of thermal expansion behaviour should be based on X-ray measurements and this could provide a programme of work for a team of specialists.

The situation for graphite is more complicated, as already mentioned (Section 4.5). The bulk expansion behaviour is determined not only by lattice expansion but is influenced to a high degree by structural parameters of the artefact (see Figures 15 and 16). The correlation of lattice expansion and bulk expansion on different high-temperature materials should be clarified.

6.3.7 Concluding Remarks on the Proposals

As mentioned in the Introduction, all the scientists contacted during the visits were very interested in collaboration. This is true of European research, where so far the work has been done mostly by individuals, as well as for the American research workers, who have good contacts with one another and want to collaborate with their European colleagues as well. If this collaboration could be promoted, a series of problems could be studied within an AGARD project. Some specific ones are summarised as follows:

1. Development of standards for thermal conductivity measurements.
2. Comparison of measuring techniques and their most suitable application.
3. Development of new measuring techniques, especially the steady-state direct-heating methods and the periodic transient ones.
4. Comparative error analysis of all methods and critical comparison of available results, using a uniform nomenclature.
5. Development of better equipment for temperature measurements, especially recording pyrometers.
6. Testing the validity of present theories of thermal conductivity for pure metals and simple alloys, using additional measurements of electrical properties.
7. Measuring of the phonon-part in metals, in high magnetic fields or by alloying techniques.
8. Promotion of new theoretical approaches in thermal conductivity, based on exactly defined metals and simple alloys.
9. Studies on thermal expansion behaviour, using high temperature X-ray techniques as a basis for developing older theories.

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TABLE I

List of Chapters and Authors in the Monograph "Thermal Conductivity" (edited by P.R.Tye)

Name	*Address*	*Topic*
Dr P.G.Klemens	Westinghouse Electric Corp., Pittsburgh 35, Penn., USA (now University of Connecticut, Storrs, Connecticut)	General introductory theory to the thermal conductivity of solids.
Dr G.K.White	Bell Telephone Laboratories, Murray Hill, New Jersey, 07971, USA (until Oct.1967)	Measurement of solid conductors at low temperatures.
Dr M.J.Laubitz	National Research Council, Ottawa 2, Ontario, Canada	Measurement of good conducting solids at high temperatures.
Dr D.L.McElroy	Oak Ridge National Laboratory, Oak Ridge, Tennessee, USA	Measurement of thermal conductivity by radial flow methods.
Prof. G.K.Danielson	Iowa State University, Ames, Iowa 50010, USA	Thermal diffusivity and other non-steady-state methods.
Dr E.Fried	General Electric Company, PO Box 8555, Philadelphia, Penn., USA	Thermal conduction contribution to heat transfer at contacts.
Dr R.W.Powell	Thermal Properties Research Centre, West Lafayette, Indiana, USA	The thermal comparator method for thermal conductivity measurement.
Dr E.Steigmeier	RCA Laboratories, Zurich 5, Hardturmstrasse 169, Switzerland	Thermal conductivity of semiconducting materials.
Prof. A.W.Pratt	Dept. of Building Sciences, University of Aston in Birmingham, England	Theory of thermal conductivity of insulating materials.
Mr M.J.Hickman	Basic Physics Division, National Physical Laboratory, Teddington, Middlesex, England	Measurement of thermal conductivity of insulating materials.
Dr E.McLaughlin	Dept. of Chemical Engineering, Imperial College, Kensington, S.W.7, England	Theory of thermal conductivity of fluids.
Dr H.Ziebland	Explosives Research and Development Establishment, Waltham Abbey, Essex, England	Measurement of thermal conductivity of fluids.
Mr D.F.Flynn	National Bureau of Standards, Washington, DC, USA	Electrical heating methods.

TABLE II

Insulators for Thermocouples

Material	*Maximum Temperature ($^{\circ}C$) (helium, 1 atm)*
Al_2O_3	1800
Bn	2000
BeO	2100
HfO_2	2200
ThO_2	2300

TABLE III

Properties of Commonly Used Thermocouples

Temperature of Application	*Thermocouple*	*E.M.F. ($\mu V/^{\circ}K$)*	*Accuracy*	*Remarks*
about $40^{\circ}K$	Au/0.03Fe vs Ag/0.37Au Au/0.03Fe vs Nb	10 – 20 10	±0.2% ±1-2%	Lit.6-8 Lit.6-8
$300^{\circ}K$	Au/0.03Fe vs 90Ni/10Cr	10 – 20	±0.5%	Good sensitivity at $1^{\circ}K$ Lit.6-8
$4^{\circ}K$ to $300^{\circ}K$	Au/2.1Co vs Av/0.37Au	5 – 40	±0.5%	Lit.9-10
$-250^{\circ}C$ to $350^{\circ}C$	Cu vs Constantan	16 – 60	$\pm 0.1^{\circ}C$	
$-200^{\circ}C$ to $1200^{\circ}C$	Chromel vs Alumel	16 – 40	$350^{\circ}C$ $\pm 0.1^{\circ}C$ $350^{\circ}C$ $\pm 1^{\circ}C$	Changes in vacuum
Room temperature to $1500^{\circ}C$	Pt vs Pt/10Rh	5 – 10	$1100^{\circ}C$ ±0.2% $1100^{\circ}C$ ±0.5%	Operates very well up to $1100^{\circ}C$
$1000^{\circ}C$ to $1800^{\circ}C$	Pt/6Rh vs Pt/30Rh	10	about $\pm 2^{\circ}C$ at $1500^{\circ}C$	for exemple, "EL 18" Degussa Hanau, Germany (Vis. 10)
$400^{\circ}C$ to $2400^{\circ}C$	W/13Re vs W/25Re	0.02 – 35	±2%	Feith (Lit. 11) gave a sensitivity of $\pm 0.3^{\circ}C$ at $2000^{\circ}C$ (Vis. 33)

TABLE IV

Data for a Visual and a Photoelectrical Pyrometer

	Visual	*Photoelectrical*
Temperature of application	T > 600°C	600 - 3700°C
Sensitivity	at 1000°C, ±1.5°C at 2000°C, ±5°C at 3000°C, ±10°C	±0.04°C
Accuracy	-	±1°C
Response time	-	10 sec
Stability	-	0.002°C/h for a radiating area of 0.3×0.3 mm^2

TABLE V

Data on Automatic Pyrometers

	Leeds and Northrup	*Pyro. Inc.*
Sensitivity	±0.2°C at 1063°C	0.14% (peak-to-peak noise)
Stability	0.0017°C/h	0.005°C/h at 1063°C
Response time	1 sec	0.1 - 0.2 sec
Minimum object diameter	1.4 mm	0.7 mm

TABLE VI

Institutions Using Longitudinal Heat Flow Methods

Institutions	*Remarks*	*Vis.No*	*Lit.No*
Canada			
NRC, Ottawa, Ontario	up to 1000°C	25	-
Germany			
DEW Zentrallaboratorium, Krefeld	RT - 100°C accuracy ±2.5%	6	17
Europ. Transuranium Inst., Karlsruhe	RT - 1000°C accuracy ±5% only for bad conductors	7	20
Krupp, Widia-Fabrik, Versuchsanstalt, Essen	RT - 700°C	9	-
PTB, Abt. III, Wärme, Braunschweig	RT - 500°C, accuracy 2-3% thick samples used	10	101
University of Cologne, Institut für theoretische Physik	-180 - 100°C accuracy ±3.5%, precision ±2%	13	120 121
Italy			
Euratom. Inst. Servizio Chimico Fisico Ispra	RT - 600°C flat discs, 80 - 120 mm dia. 10 - 25 mm thick	16	-
United Kingdom			
University of Oxford Clarendon Laboratories	1°K - 90°K accuracy ±5%	20	6
Bath University of Technology	-90°C - 400°C, magnetic field 10.000 gauss	23	113
USA			
NBS, Cryogenic Division, Boulder, Colorado	4°K - 300°K accuracy ±1%	38	19
NBS, Building Res. Division, Washington	(a) -180°C - +50°C	40	-
	(b) RT - 600°C in air RT - 750°C in vacuum		16
	(c) RT - 1100°C platinum approximate accuracy ±1%		1
	(d) 100 - 1200°C cut bar approximate accuracy ±2.5%		114
ORNL, Oak Ridge, Tennessee	-195°C - +100°C accuracy ±1.8%	41	22
TPRC, West-Lafayette, Indiana	50°C - 400°C in construction	43	-

TABLE VII

Institutions Using Radial Heat Flow Methods

Institutions	*Remarks*	*Vis.No*	*Lit.No*
Germany			
Inst.f.Material- u. Festkörperkunde, Kernforschungszentrum, Karlsruhe	500°C - 2000°C radial outflow high temperature gradients	8	159
University of Cologne, Institut für theoretische Physik	RT - 1000°C accuracy ±6% radial outflow	13	119
Italy			
Euratom. Inst. Servizio Chimico Fisico Ispra	500°C - 1000°C radial outflow	16	-
USA			
AFSC, Materials Engineering Branch, Wright Patterson Air Force Base, Ohio	up to 2300°C radial inflow	27	-
Atomics International Metallurgy, Solid States, Physics and Material Science Groups, Canoga Park, California	up to 2700°C radial inflow accuracy ±16%	28	122
Battelle Mem. Inst. Columbus, Ohio	1000°C - 2500°C accuracy ±5% to 1800°C ±8% above radial inflow	29	-
Dynatech Corp. Cambridge, Massachusetts	(a) up to 2700°C accuracy +10% radial outflow commercially available (b) up to 900°C radial inflow in development	31	123

TABLE VII (Continued)

Institutions	*Remarks*	*Vis.No*	*Lit.No*
USA (continued)			
GEC, Nuclear Mat. and Prop. Operation Cincinnati, Ohio	800°C - 2500°C radial outflow	33	11 124
Lockheed, Missiles and Space Company, Palo Alto, California	up to 2700°C accuracy ±20% radial inflow	36	-
Midwest Research Institute, Kansas City, Kansas	up to 2500°C radial outflow in construction	30	23
Nat. Beryllia Corp. Haskell, New Jersey	up to 2500°C accuracy ±10% radial outflow commercially available	-	125
ORNL, Metals and Ceramics Division, Oak Ridge, Tennessee	-156°C - +1000°C accuracy ±2% radial outflow up to 2200°C in development	41	22 96
Southern Research Institute, South Birmingham, Alabama	(a) -140°C - +2250°C radial outflow accuracy ±5 - 15%	42	
	(b) -140°C - +2750°C radial inflow accuracy ±7 - 12%		21
UCC, Carbon Products Division, Parma Technical Center, Parma, Ohio	up to 2800°C accuracy ±20% at 1000°C ±8% at 2600°C pressures up to 33 atm radial inflow	44	126

TABLE VIII

Institutions Using Flash Methods

Institutions	*Remarks*	*Vis.No*	*Lit.No*
France			
Sud Aviation, Div.Spat. el elec., Courbevoie	up to 750°C	5	-
Italy			
CNRN, Casaccia, Roma	up to 1500°C, accuracy ±6%, Xe-flash, laser in development up to 1000°C	14	127
United Kingdom			
British Ceramic Research Association, Stoke-on-Trent, England	only at room temperature, Xe-flash, thermoccuples as detectors	19	-
Reactor Development Lab., Windscale, England	laser flash up to 1200°C	23	-
USA			
AFSC, Mat. and Eng. Branch Wright-Patterson Air Force Base, Ohio	up to 2500°C, Ta-furnace InAs as IR-detector	27	115
Atomics International Canoga Park, California	up to 1800°C, laser detector to 1500°C thermocouples, above multipliers	28	43
Batte'le Memorial Inst. Columbus, Ohio	up to 2000°C, laser (20 joules), Ta-furnace, PbS-detector, multiplier in construction	29	-
GEC, Nucl. Mat. and Space Operation, Cincinnati,Ohio	up to 1000°C, laser (20 joules), Pt-furnace up to 1600°C, PbS-detector	33	-
Lockheed, Missiles and Space Company Palo Alto, California	from 150°C to 800°C accuracy ±4.5%, Xe-flash, with laser to 2400°C	36	45 157
UCC, Carbon Prod. Div. Parma Techn. Center Parma, Ohio	up to 900°C, Xe-flash, thermocouples as detectors, accuracy ±2%	44	

TABLE IX

Institutions Using the Angström Method

Institutions	*Remarks*	*Vis.No*	*Lit.No*
France			
SNECMA, Usine de Suresnes	in preparation: radiation heating by a plasma source and a "persienne"	4	-
Germany			
Institut für angewandte Physik, Technische Hochschule, Karlsruhe	working at room temperature, reproducibility ±2 - 3%, in construction up to 600°C, mechanically regulated resistor heating	12A	-
United Kingdom			
Bath University of Technology	up to 400°C, use of a semiconductor as heater	23	-
USA			
Iowa State University, Ames, Iowa	up to 900°C, accuracy ±2% direct resistant heat	-	49
Radio Corporation of America, Princeton, New Jersey	up to 1000°C accuracy ±2%	-	50

TABLE X

Institutions Using Periodic Transient Techniques

Institutions	*Remarks*	*Vis.No*	*Lit.No*
France			
SNECMA, Usine de Suresnes	in preparation: radiation heating by a plasma source and a "persienne"	4	–
Germany			
Euratom Inst. f. Transurane, Kernforschungszentrum Karlsruhe	electron beam, accuracy about ±5%	7	20
Italy			
Euratom Inst. Servizio Chimico Fisico, Ispra	electron beam, from 300°C with IR-detector	16	127
United Kingdom			
GEC, Hirst Research Centre, Wembley, Middlesex	electron beam, from 300°C with IR-detector	21	–
USA			
Aerospace Corp. Mat. Sciences Laboratory, Los Angeles, California	indirect heating, periodic disc methods using re-radiation by mirrors, up to 2800°C	26	40 53
American Machine and Foundry Company, Alexandria, Virginia	periodic electron beam, 1000°C - 1100°C accuracy ±2.5%	–	116
UCC, Carbon Prod. Div. Parma Techn. Center, Parma, Ohio	(a) periodic arc image up to 3000°C (b) periodic cooling down up to 3000°C	44	36

TABLE XI

Institutions Measuring Specific Heat by Adiabatic and Pulse Methods

Institutions	*Remarks*	*Vis.No*	*Lit.No*
France			
Centre d'Etudes Nucleaires Fontenay-aux-Roses	pulse heating	-	62
Germany			
University of Cologne, Institut für theoretische Physik	quasi-adiabatic up to 1600^{o}C accuracy ±2%	13	101 102 103
United Kingdom			
NPL, Metallurgy Division	adiabatic up to 1500^{o}C accuracy ±1%	22	57
USA			
Atomics International Metallurgy, Solid States, Physics and Material Science Groups, Canoga Park, California	pulse heating no temperature limit	28	61
ORNL, Metals and Ceramics Division Oak Ridge, Tennessee	pulse heating 100 - 1400^{o}C accuracy ±1% reproducibility ±0.5%	41	60
University of Tennessee Dep. Chem. Met. Eng. Knoxville, Tennessee	adiabatic 500 - 1000^{o}C accuracy ±0.3%	45	54 55 56

TABLE XII

Institutions Measuring Emissivity

Institutions	*Remarks*	*Vis.No*	*Lit.No*
France			
Sud Aviation, Div. Spat. et Electron, Courbevoie	determination of the total emittance	5	-
Germany			
PTB, Braunschweig, also - Inst.Berlin Abbestr.1-12, Dr Sauerbrey; Inst.f. Elektrowerkstoff.d.FHG, Prof.Dr Mecke, Freiburg, Eckerstr.4; Elektrowärme- Inst.Dr K.Herold, Essen, Beethovenstr.32	up to 2500°C accuracy ±2% (other addresses are from information given by PTB (Vis.10)	10	68
USA			
AFSC, Mat. and Eng. Branch, Wright-Patterson Air Base, Ohio	laser source integrating sphere reflectometer 4 up to 2500°C, accuracy α 1%, ellipsoidal mirror reflectometer (room temperature) centre post technique, deep cavity technique, up to 2000°C	27	65 66
Battelle Memorial Institute, Columbus, Ohio	hole-in-tube method up to 2000°C	29	
Southern Research Institute, South Birmingham, Alabama	determination of spectral emittance, up to 2750°C, accuracy ±12%	42	67
National Bureau of Standards, Washington	nearly the same equipment as at the AFSC, mentioned above	40	163 164 165

TABLE XIII

Institutions Using Interferometry Methods

Institutions	*Remarks*	*Vis.No*	*Lit.No*
Italy			
Inst. of Thermometry Turin	temperature range 90 - 273°K	18	128
USA			
NBS Gaithersburg	Fizeau method, laser as light source, up to 1000°C (under development)	39	69
UCC, Parma	Newton method up to 1000°C	44	70

TABLE XIV

Institutions Using Optical and X-ray Methods for Measuring Thermal Expansions at High Temperature

Institutions	*Remarks*	*Vis.No*	*Lit.No*
Belgium			
SERAI, Bruxelles	X-ray, up to 1200°C U-shaped Ta heater	1	-
France			
SNECMA, Usines de Suresnes	X-ray, up to 2850°C	4	79
Germany			
Institut für Chem. Technik, Karlsruhe	X-ray, up to 1500°C	12B	-
USA			
Aerospace Corporation, Los Angeles, California	X-ray, up to 1500°C	26	-
AFSC, Wright-Patterson Air Base, Ohio	optical, up to 2300°C	27	-
Atomics International, Canoga Park, California	optical up to 3000°C accuracy 1% up to 1000°C 1.5% from 1000°C - 2000°C	28	130
GEC, Cincinnati, Ohio	optical, up to 2500°C	33	74
IPL, Pasadena, California	(a) X-ray, up to 1500°C (b) optical, up to 2000°C	34	132 133 134
NBS, Gaithersburg, Maryland	optical, up to 1600°C	39	-
UCC, Parma, Ohio	direct to indirect heating up to 2400°C, accuracy ±1%	44	129

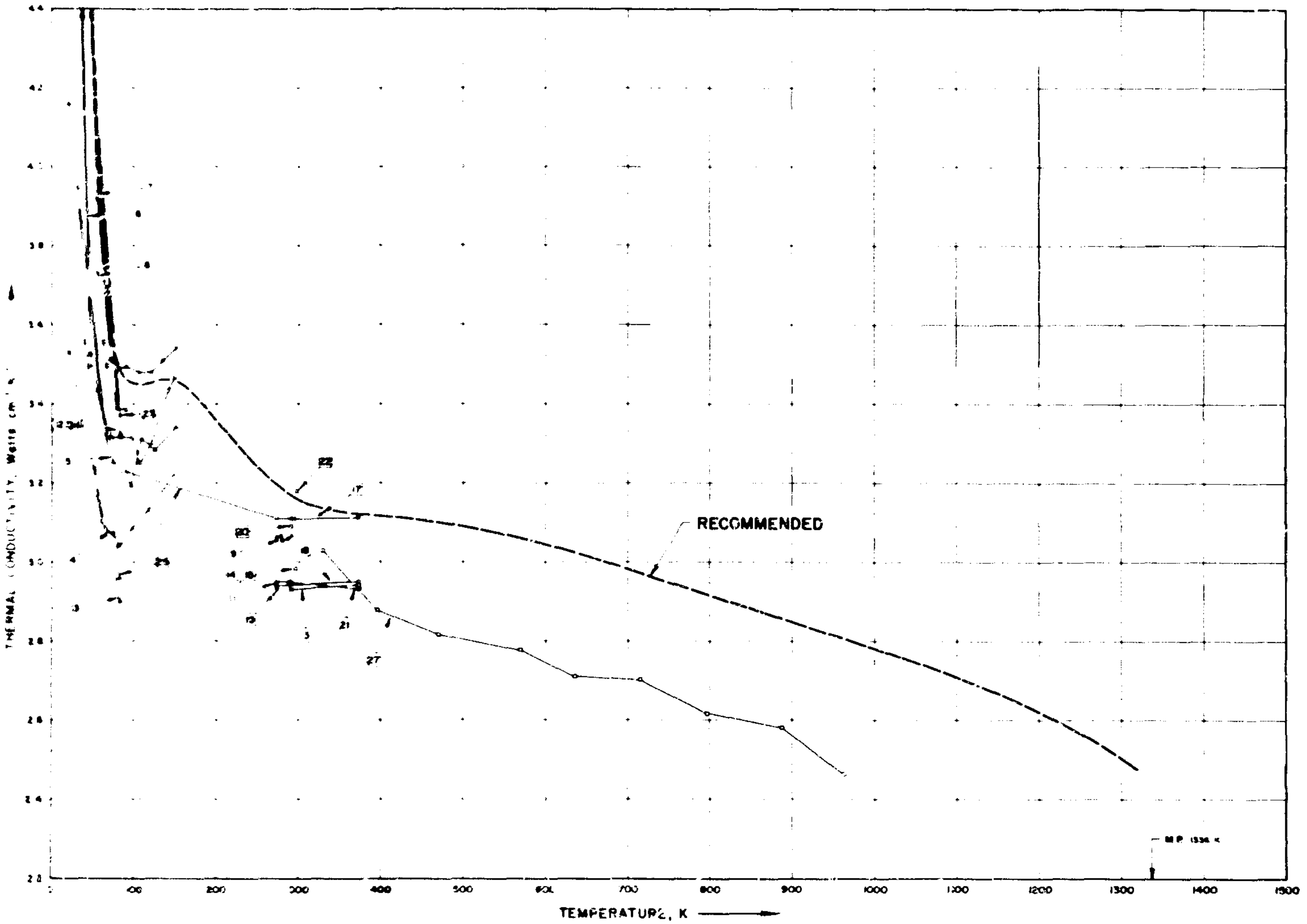

Fig. 1 Thermal conductivity of gold

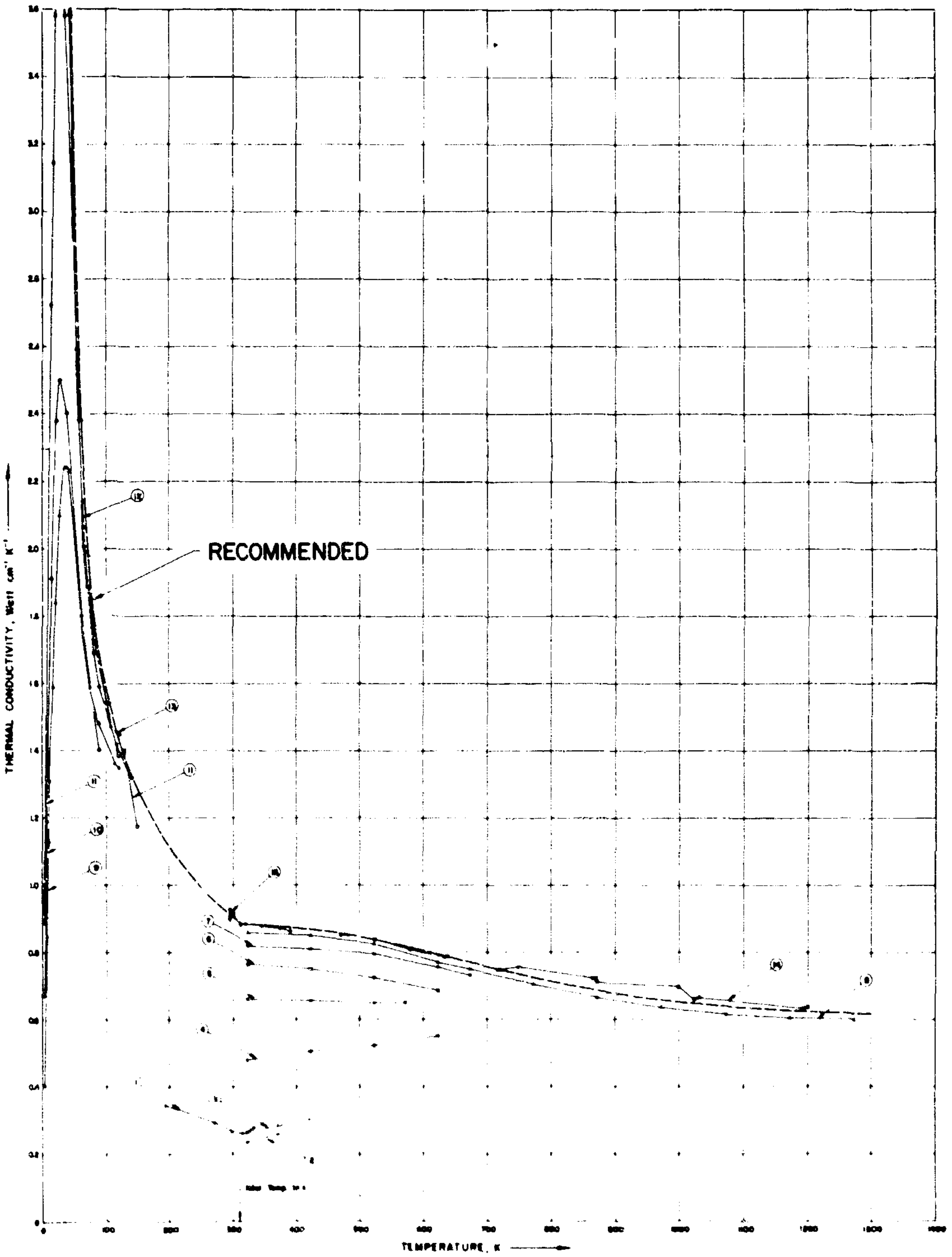

Fig.2 Thermal conductivity of chromium

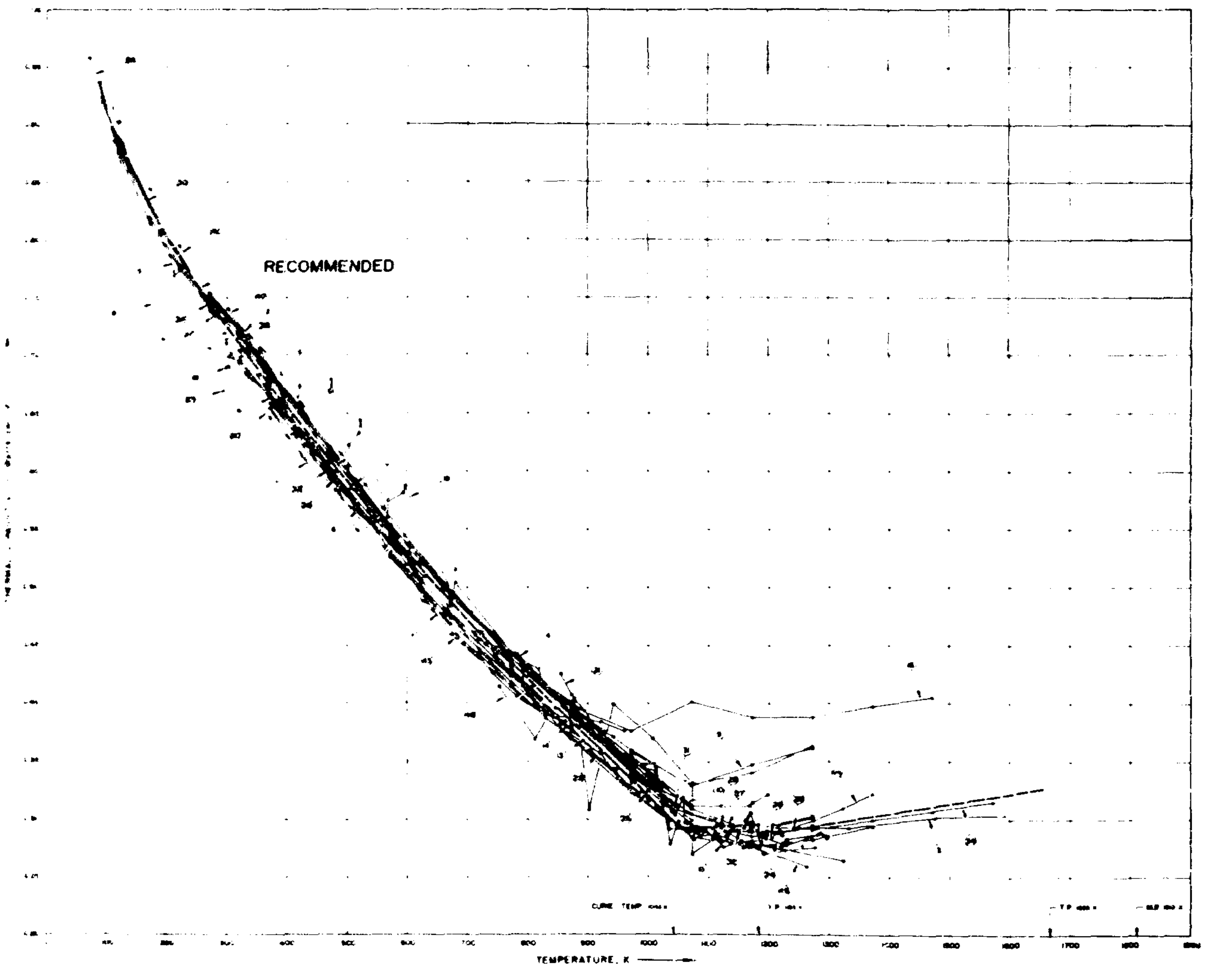

Fig.3 Thermal conductivity of iron (Armco)

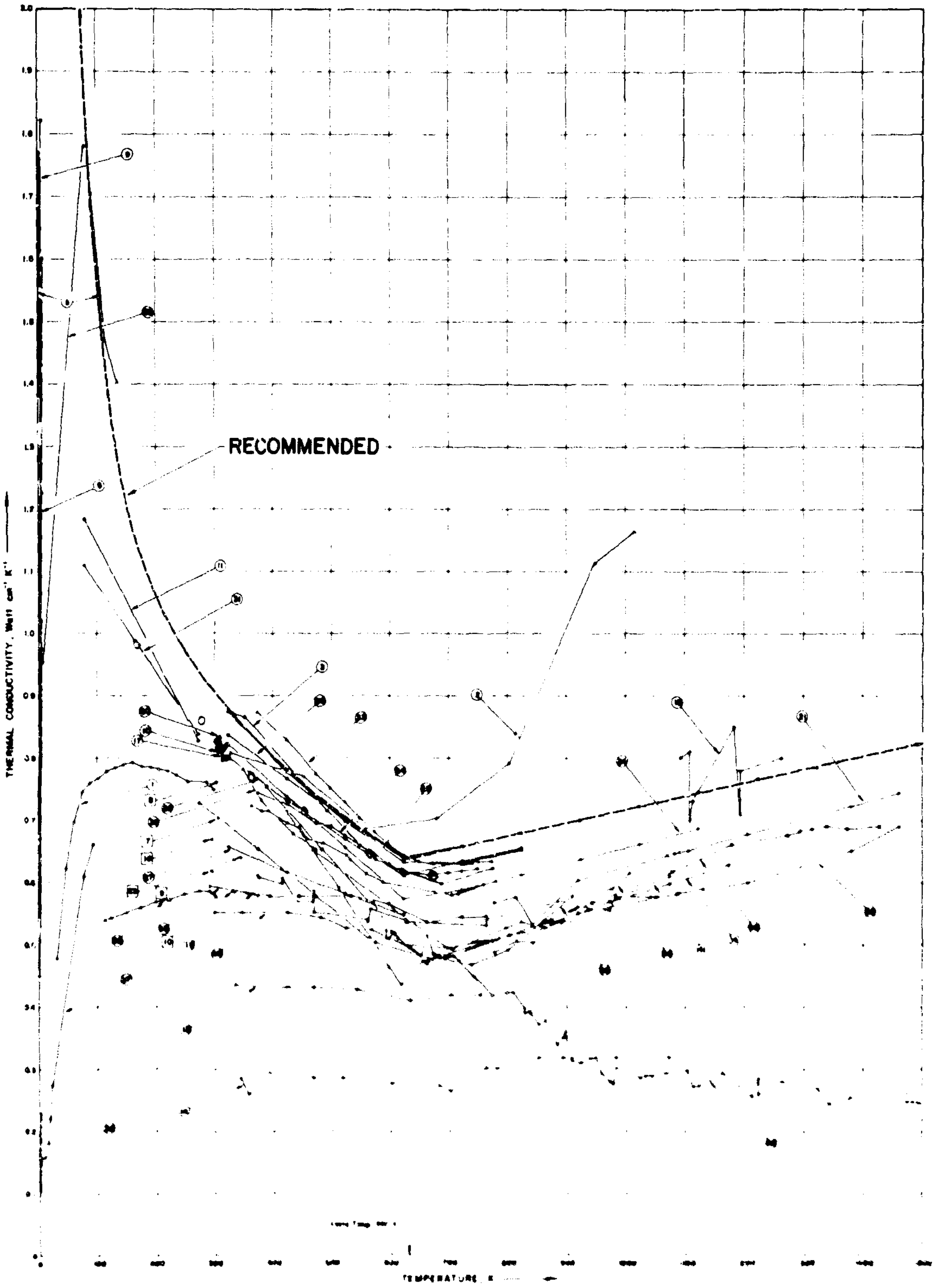

Fig. 4 Thermal conductivity of nickel

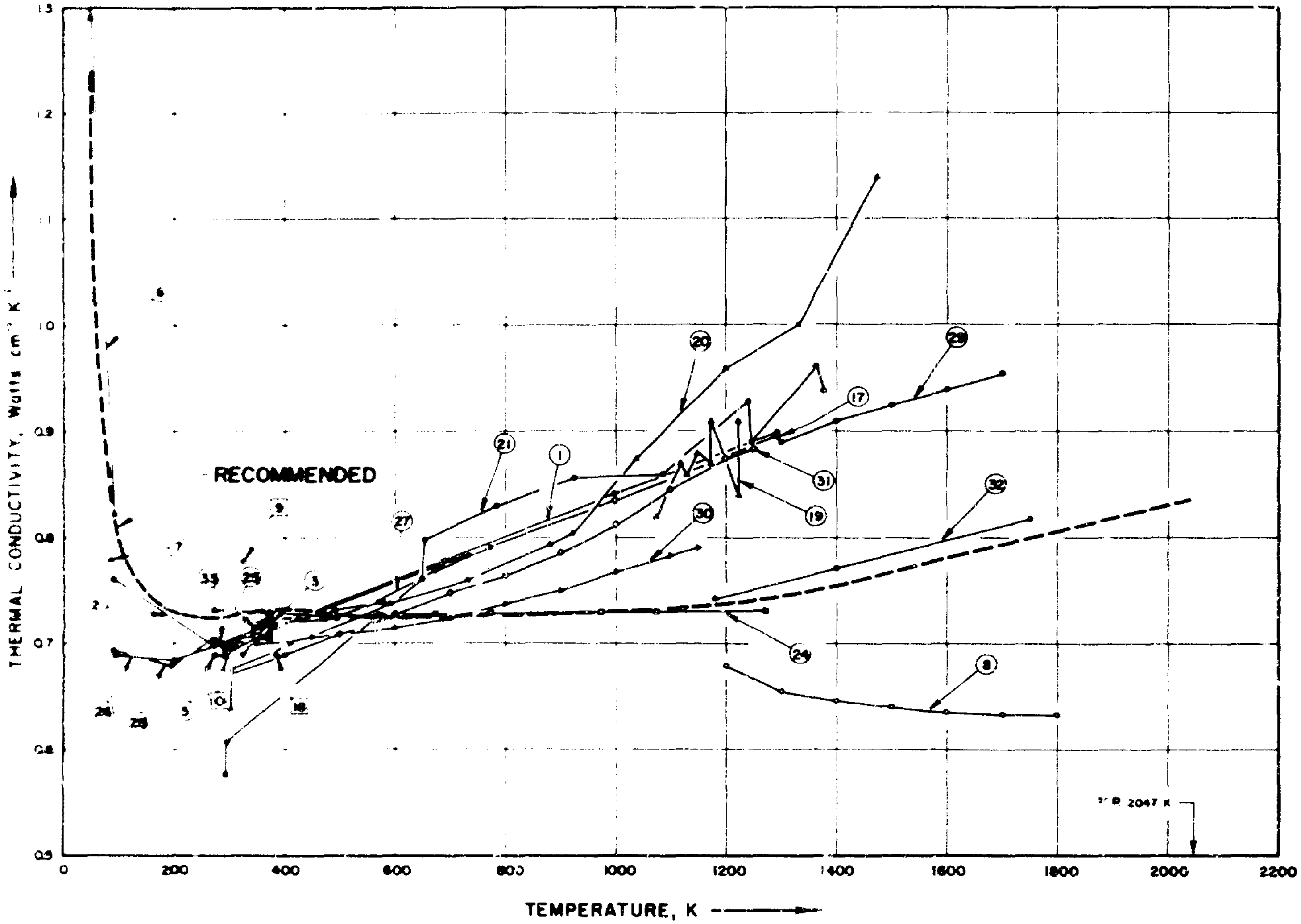

Fig.5 Thermal conductivity of platinum

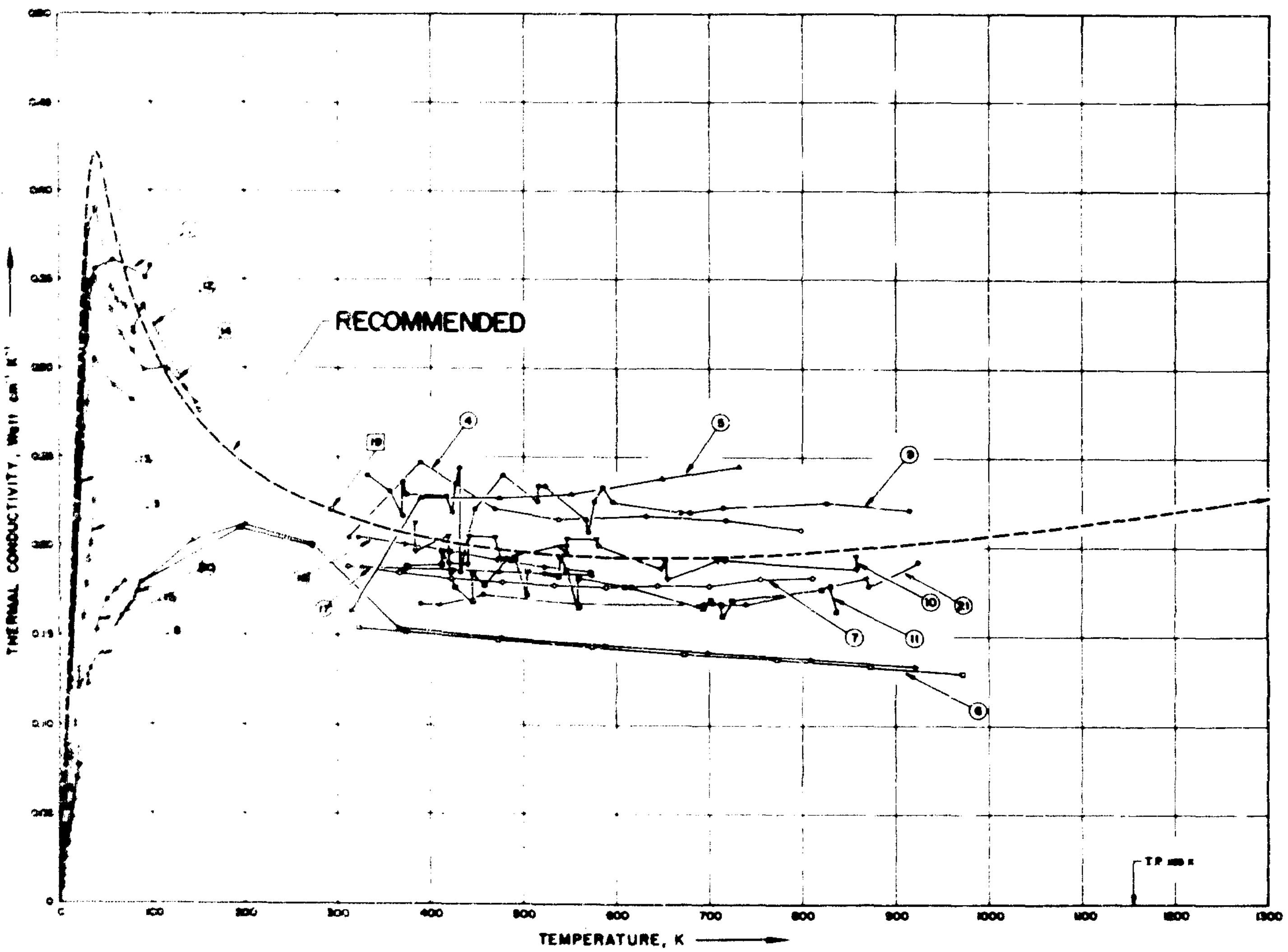

Fig.6 Thermal conductivity of titanium

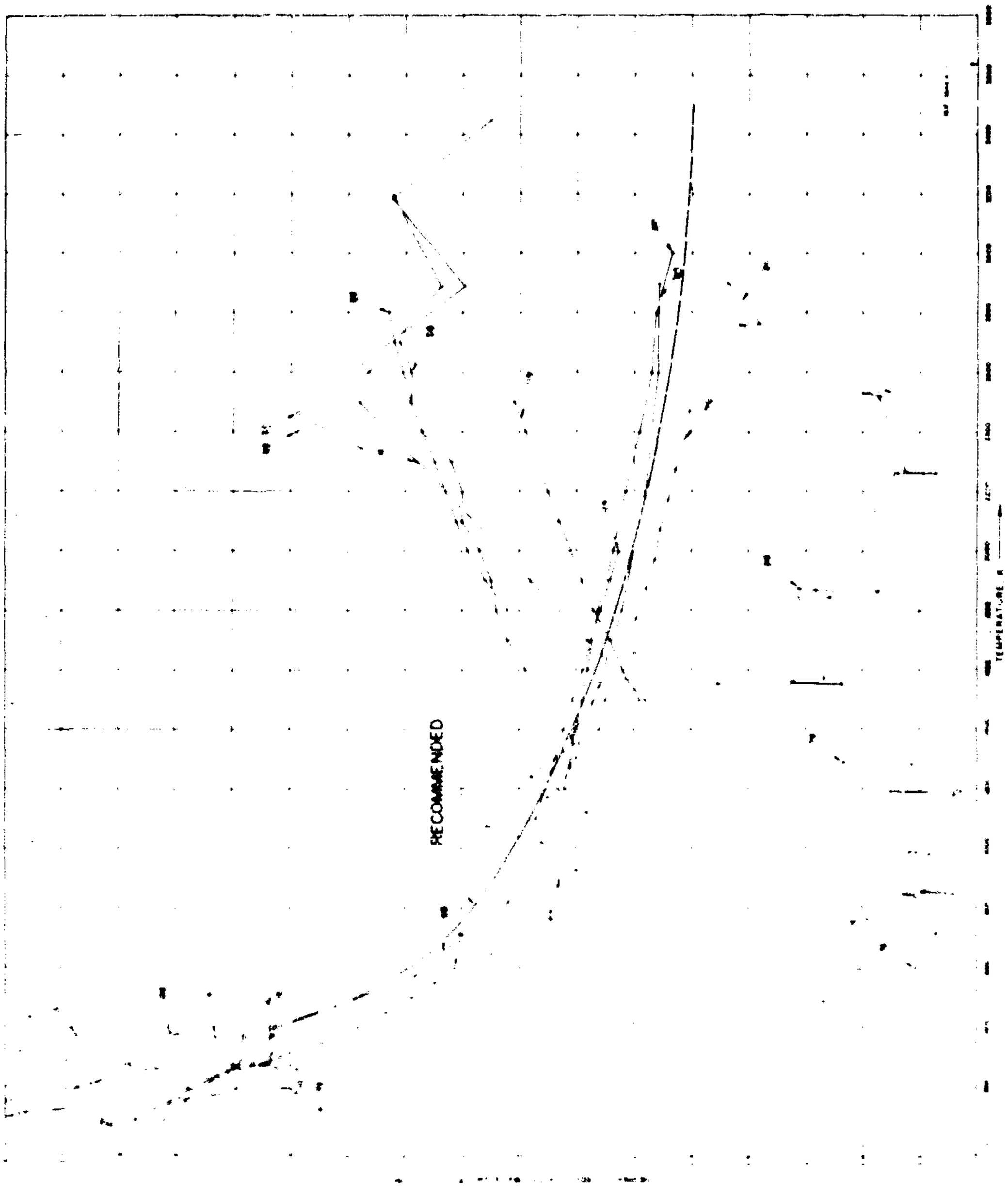

Fig.7 Thermal conductivity of tungsten

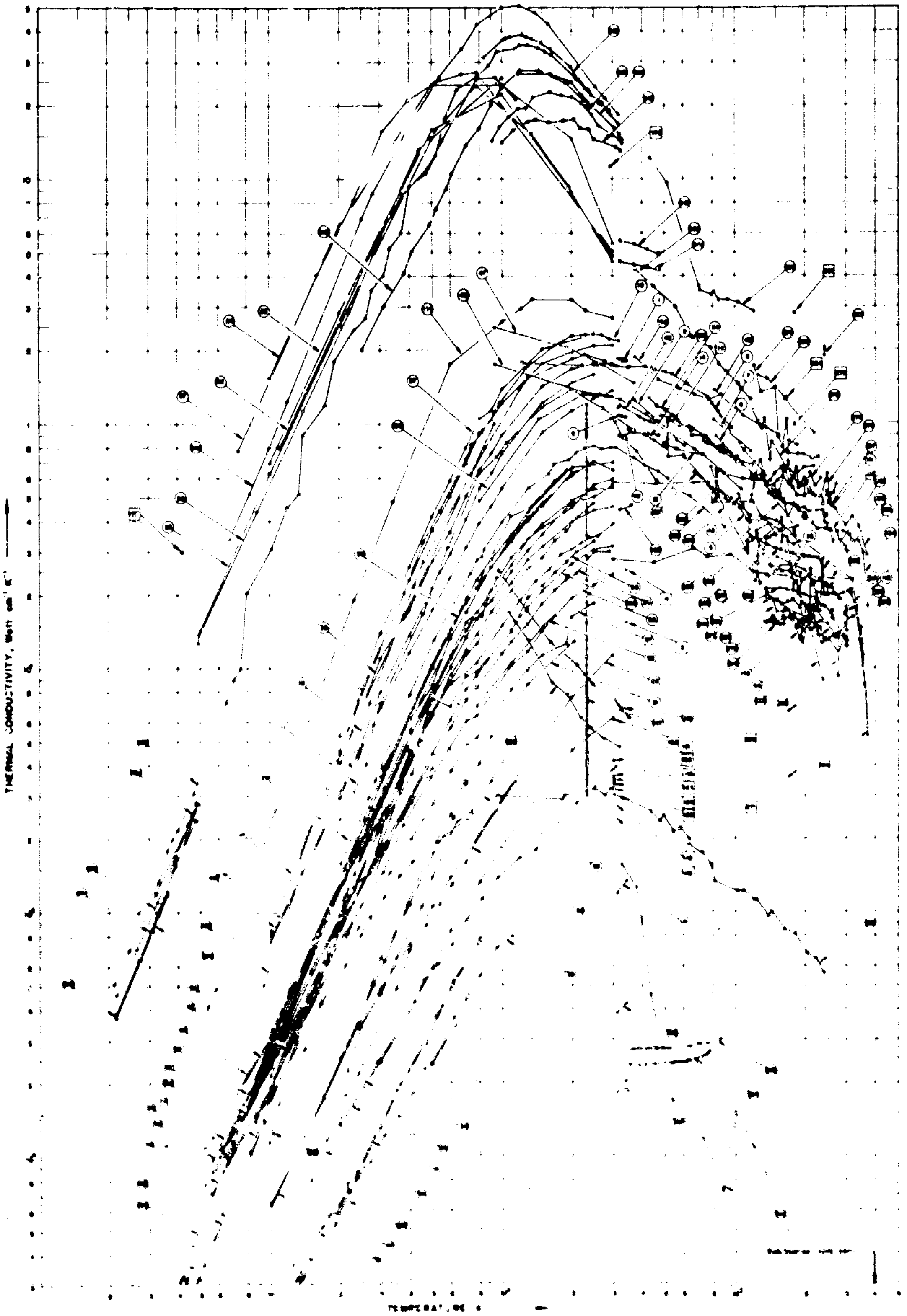

Fig.8 Thermal conductivity of graphite

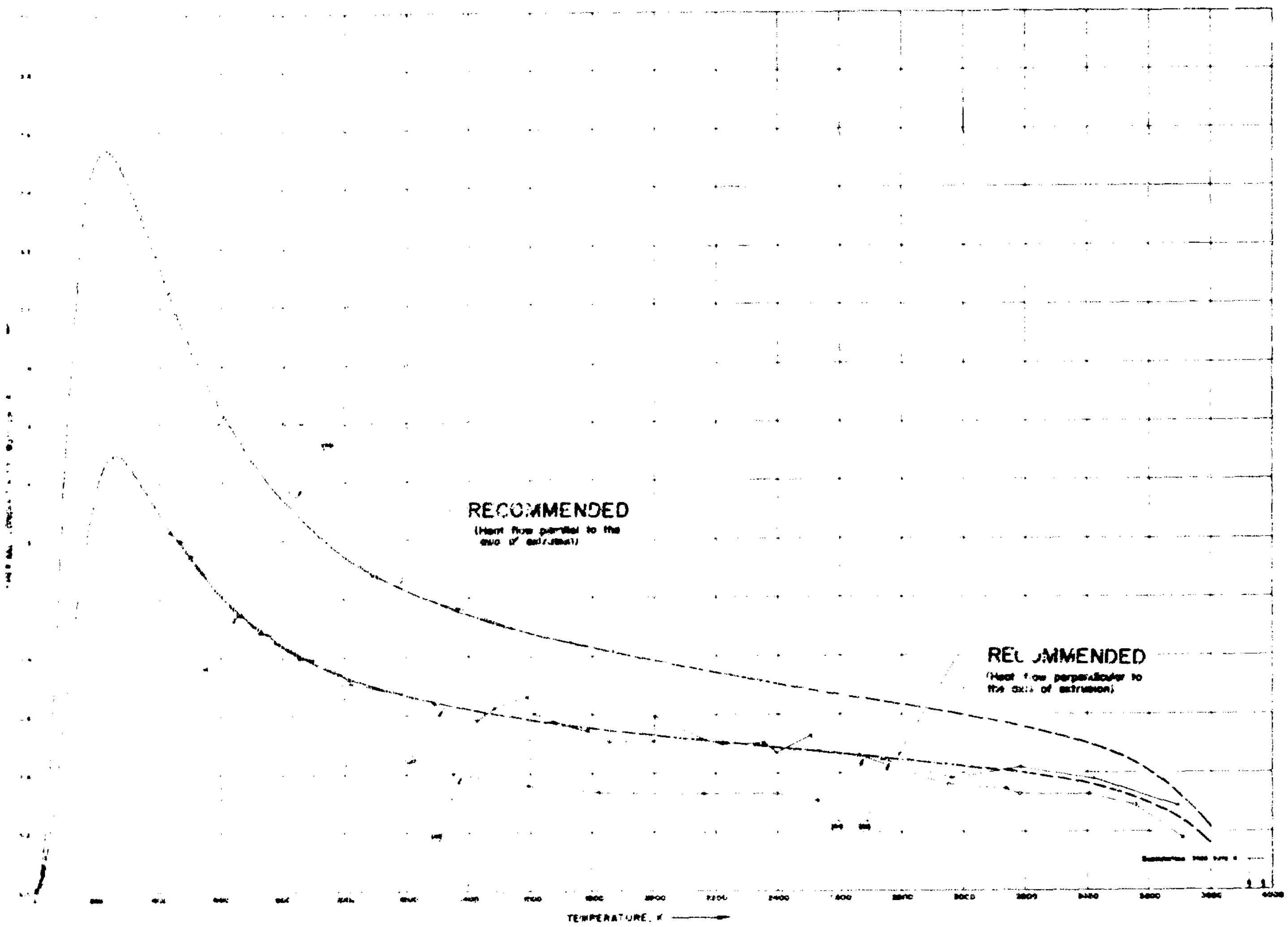

Fig. 9 Thermal conductivity of 875S graphite

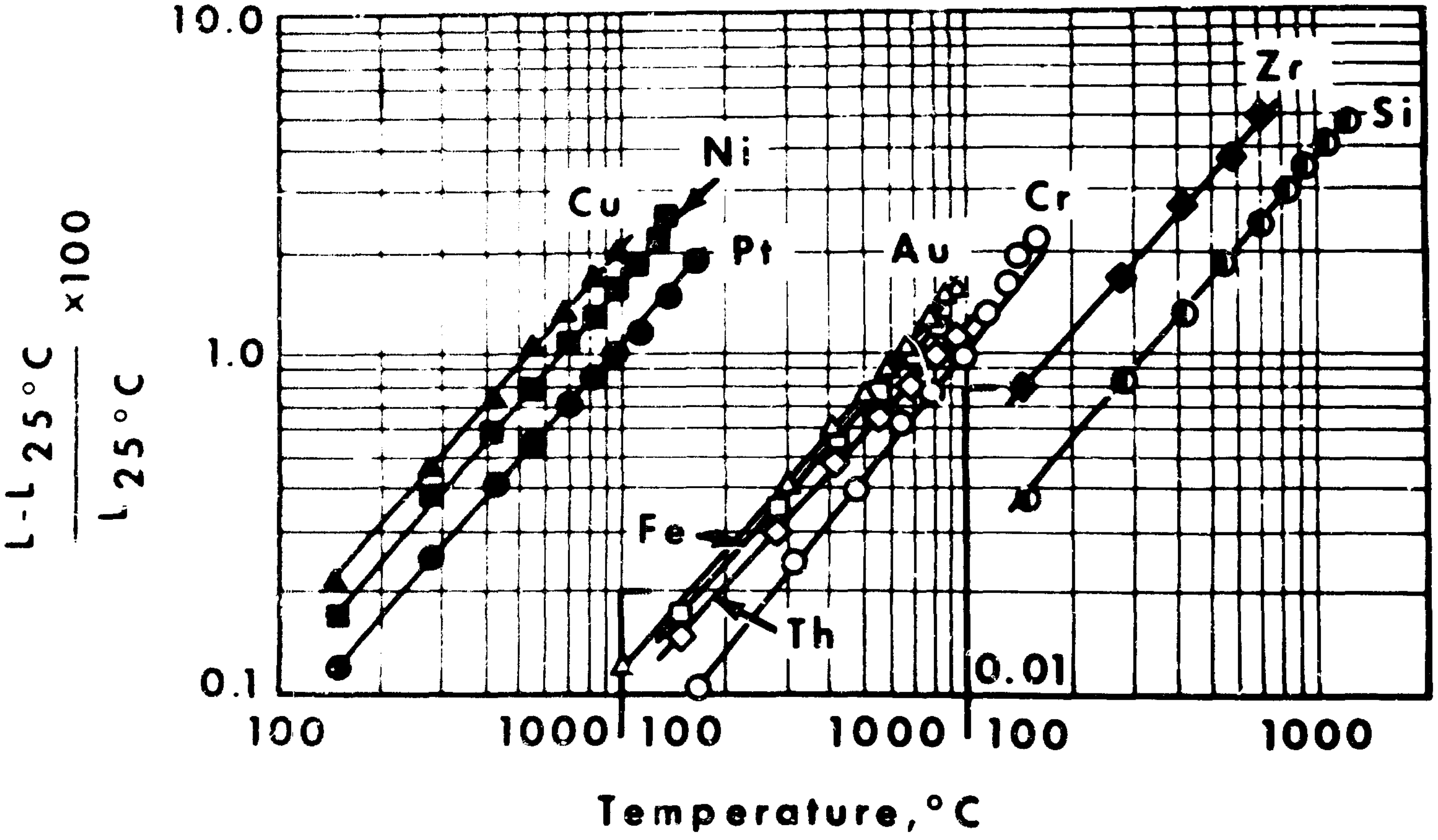

Fig.10 Thermal expansion data of several metals plotted to yield a linear relationship

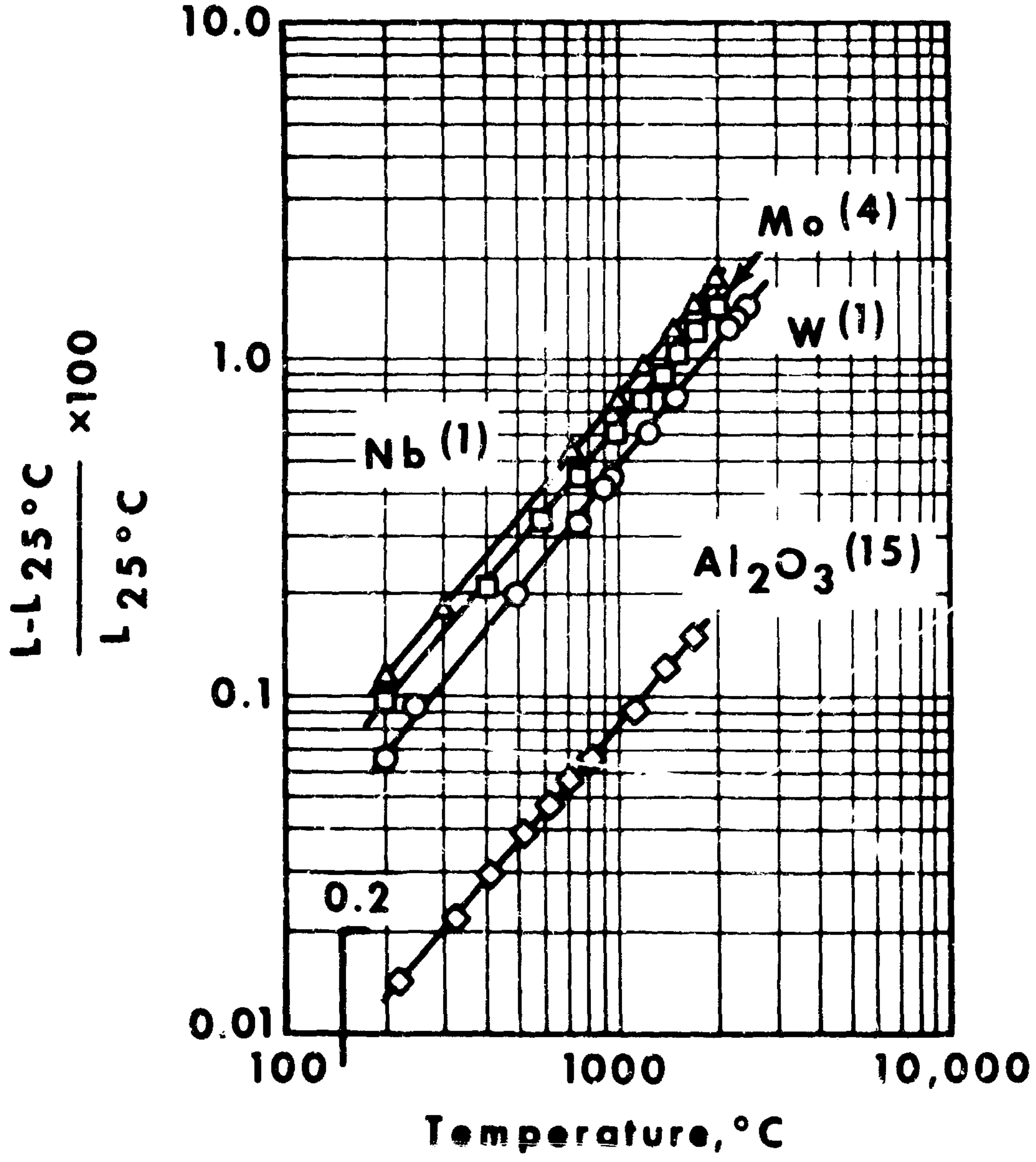

Fig. 11 Plots of $\Delta L/L$ versus T °C for W, Mo, Nb and Al_2O_3

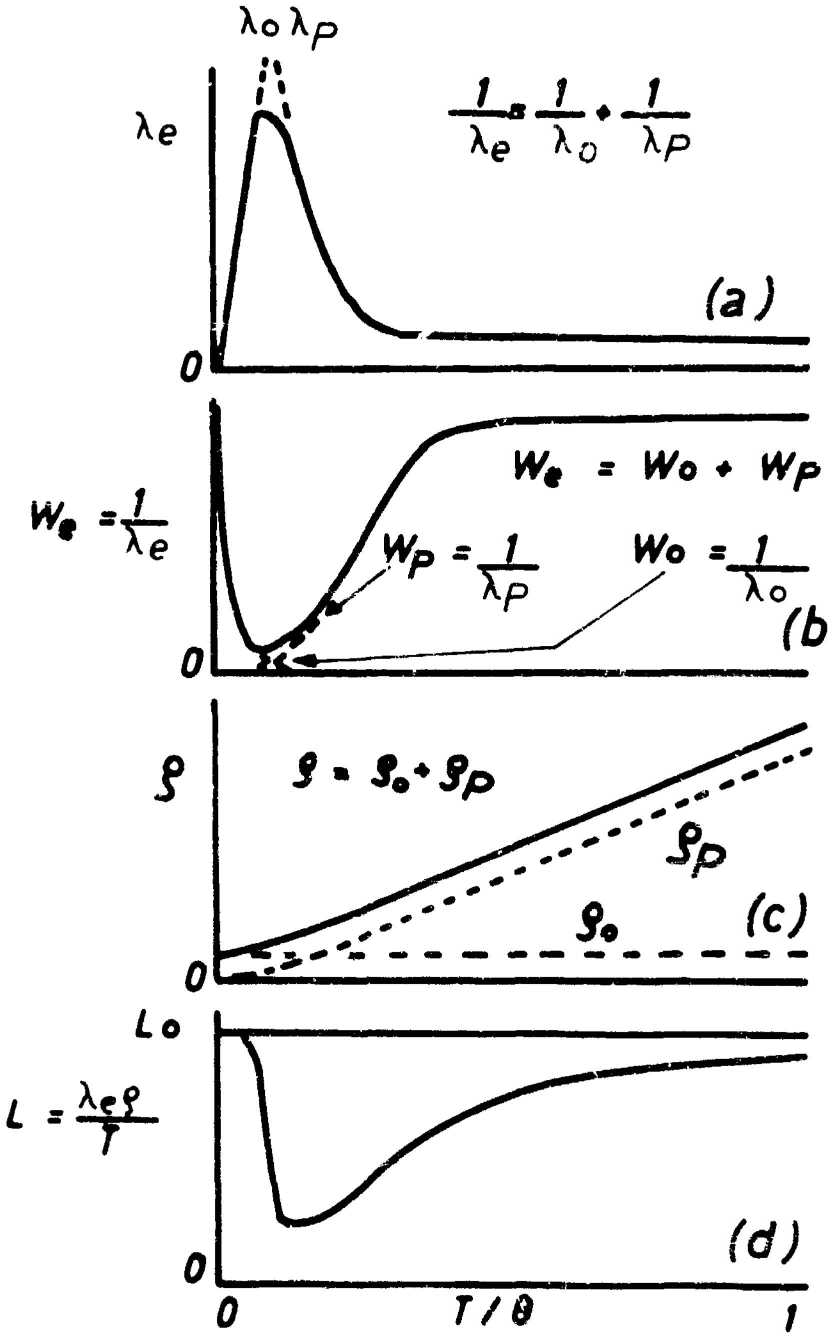

Fig. 12 (a) Thermal conductivity
(b) Thermal resistivity
(c) Electrical resistivity
(d) Lorenz ratio

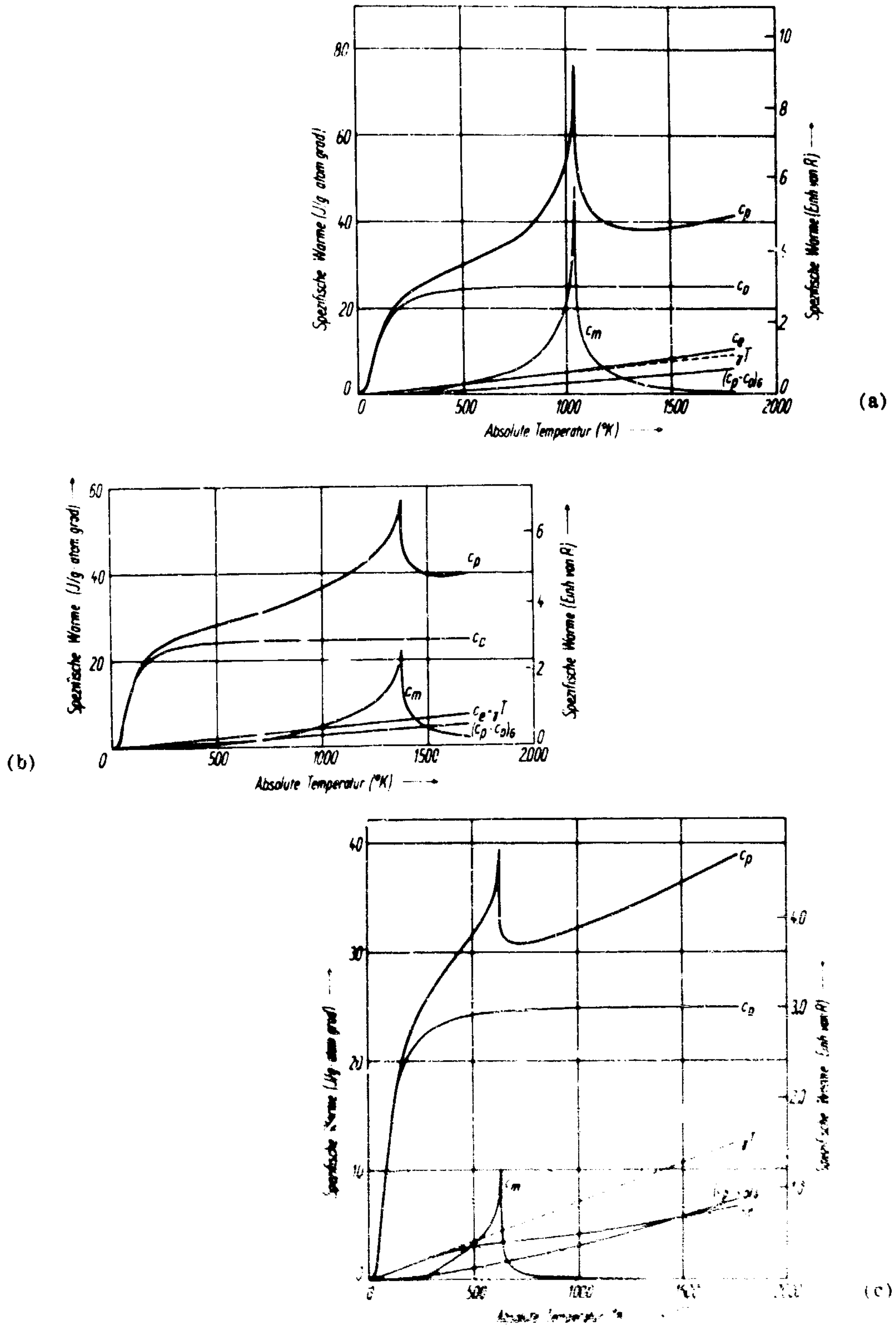

Fig.13 The various contributions to the specific heat of (a) iron, (b) cobalt and (c) nickel as a function of temperature

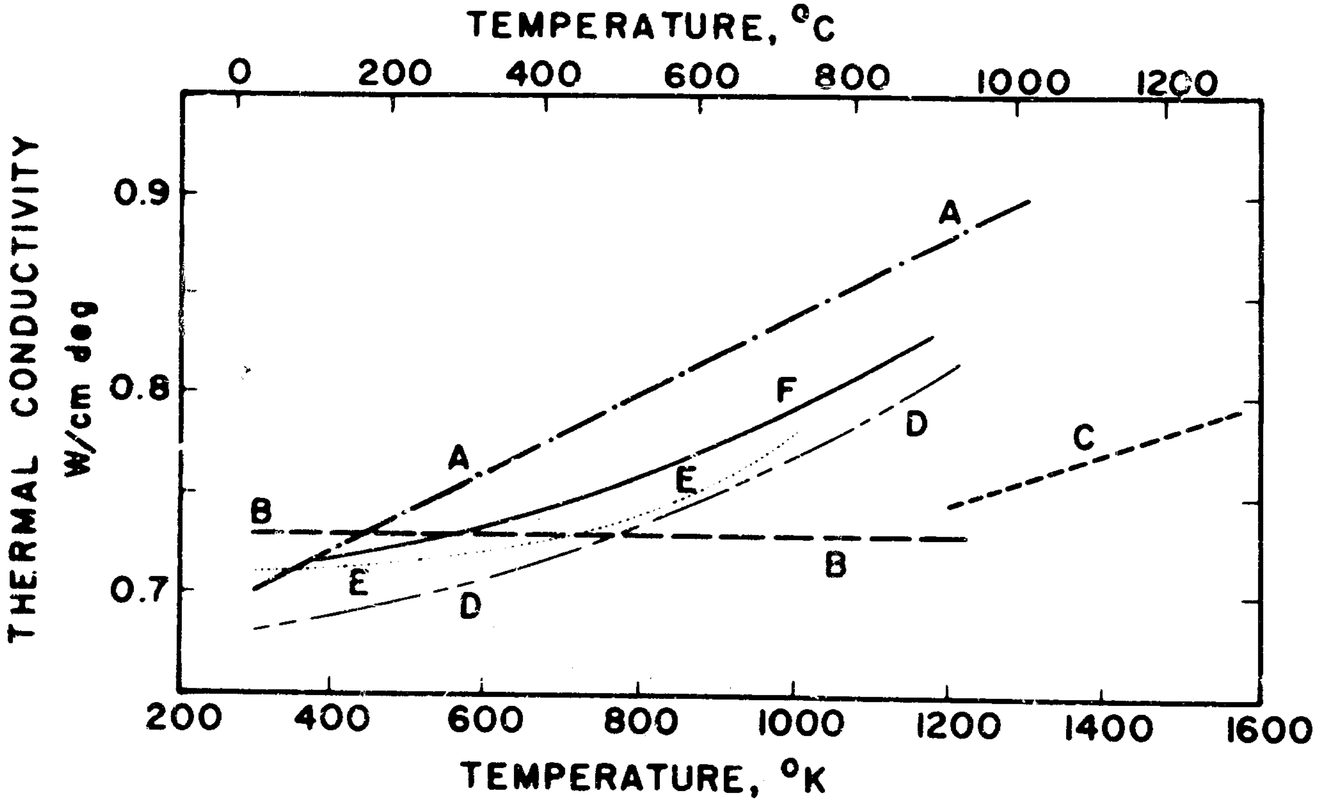

Fig. 14 Thermal conductivity of platinum above room temperature as reported by several investigators

A. Holm and Stormer (15)
B. Powell and Tye (18)
C. Wheeler (19)
D. Martin, Sidles and Danielson (2); the values shown correspond to their sample D
E. Laubitz and van der Meer (20)
F. Present Investigation; the values shown were obtained by the longitudinal flow method in the NBS platinum apparatus

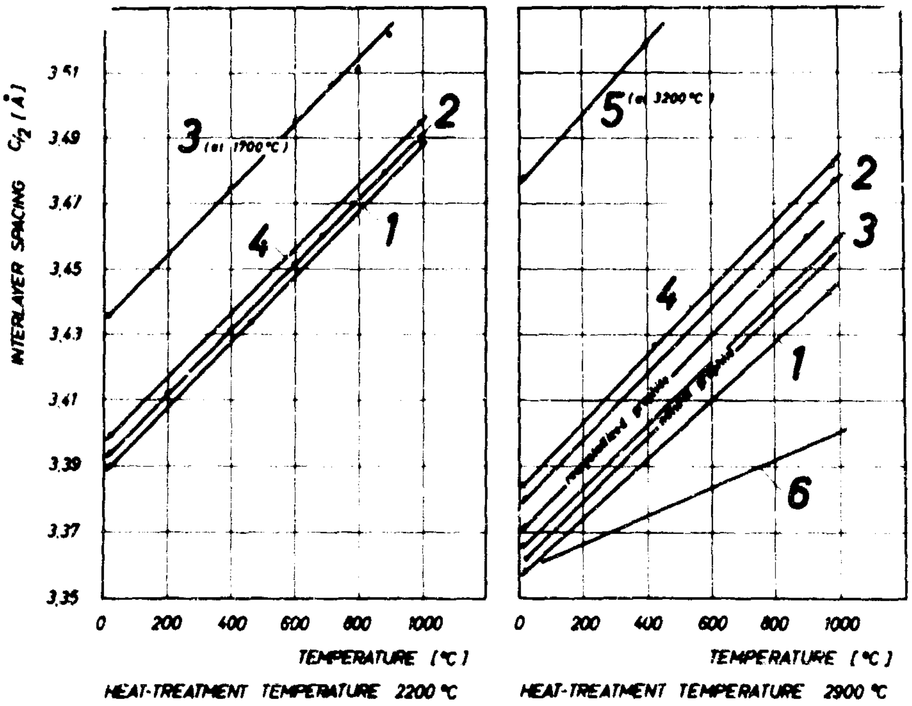

Figure 15

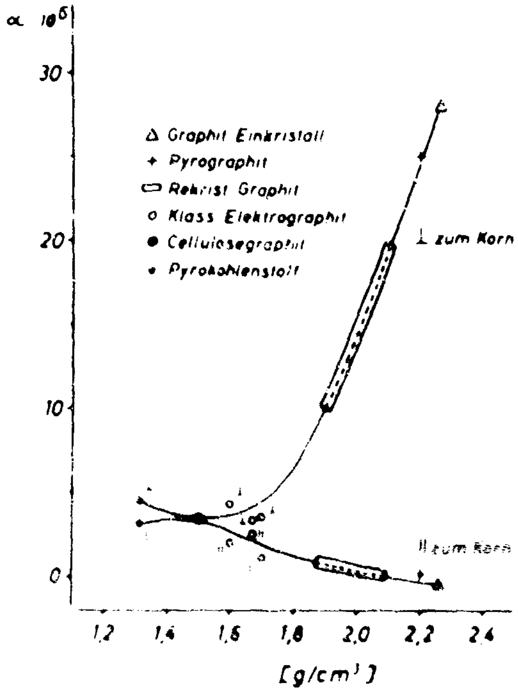

Figure 16

APPENDIX

Working Party on Thermophysical Properties of High Temperature Metals of the Structures and Materials Panel of AGARD.

The first discussions took place at the 24th Panel meeting on 17 to 26 April 1967, in Turin, Italy and the final discussions at the 25th Panel meeting on 24 to 30 September 1967, in Ottawa, Canada.

Chairman: Professor F. Bollenrath
Vice-Chairman: Dr A.M. Lovelace
Members: Mr H.V. Kinsey
Professor L. Locati
Professor F. Niordson
Specialist: Professor E. Fitzer

AGARD Structures and Materials Panel
List of Members as at the 24th Panel Meeting

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Helle	Mr E.	Nor.	
Incarbone	Dr Ing. G.	It.	
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Kjøllesdal	Mr H.	Nor.	Chairman of the Materials Group
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Küssner	Prof. Dr Ing. H.G.	Ger.	
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Miller	Mr W.B.	US	
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Niordson	Prof. F.	Den.	

			Functions
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Pouligmier	Mr J.P.	Fr.	
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Members: Dr A.J.Barrett
Mr W.Barrois
Mr H.V.Kinsey
Mr H.Kjøllesdal
Mr N.H.Mason
Mr R V.Rhode

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Planning Committee

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Mr H.V.Kinsey
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Mr N.H.Mason
Mr R.V.Rhode

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