10.007

NATIONAL BUREAU OF STANDARDS REPORT

7323

THERMAL CONDUCTIVITY OF SEMI-CONDUCTIVE SOLIDS;
METHOD FOR STEADY-STATE MEASUREMENTS ON
SMALL DISK REFERENCE SAMPLES

Technical Progress Report
for Period
April 1 to June 30, 1961

Report to the
Bureau of Ships
Department of the Navy
Washington, D. C.

NBS

U. S. DEPARTMENT OF COMMERCE

NATIONAL BUREAU OF STANDARDS

THE NATIONAL BUREAU OF STANDARDS

Functions and Activities

The functions of the National Bureau of Standards are set forth in the Act of Congress, March 3, 1901, as amended by Congress in Public Law 619, 1950. These include the development and maintenance of the national standards of measurement and the provision of means and methods for making measurements consistent with these standards; the determination of physical constants and properties of materials; the development of methods and instruments for testing materials, devices, and structures; advisory services to government agencies on scientific and technical problems; invention and development of devices to serve special needs of the Government; and the development of standard practices, codes, and specifications. The work includes basic and applied research, development, engineering, instrumentation, testing, evaluation, calibration services, and various consultation and information services. Research projects are also performed for other government agencies when the work relates to and supplements the basic program of the Bureau or when the Bureau's unique competence is required. The scope of activities is suggested by the listing of divisions and sections on the inside of the back cover.

Publications

The results of the Bureau's research are published either in the Bureau's own series of publications or in the journals of professional and scientific societies. The Bureau itself publishes three periodicals available from the Government Printing Office: The Journal of Research, published in four separate sections, presents complete scientific and technical papers; the Technical News Bulletin presents summary and preliminary reports on work in progress; and Basic Radio Propagation Predictions provides data for determining the best frequencies to use for radio communications throughout the world. There are also five series of nonperiodical publications: Monographs, Applied Mathematics Series, Handbooks, Miscellaneous Publications, and Technical Notes.

A complete listing of the Bureau's publications can be found in National Bureau of Standards Circular 460, Publications of the National Bureau of Standards, 1901 to June 1947 ($1.25), and the Supplement to National Bureau of Standards Circular 460, July 1947 to June 1957 ($1.50), and Miscellaneous Publication 240, July 1957 to June 1960 (Includes Titles of Papers Published in Outside Journals 1950 to 1959) ($2.25); available from the Superintendent of Documents, Government Printing Office, Washington 25, D. C.

NATIONAL BUREAU OF STANDARDS REPORT

NBS PROJECT		NBS REPORT
1006-20-10461	August 17, 1961	7323

THERMAL CONDUCTIVITY OF SEMI-CONDUCTIVE SOLIDS;
METHOD FOR STEADY-STATE MEASUREMENTS ON
SMALL DISK REFERENCE SAMPLES

Technical Progress Report
for Period
April 1 to June 30, 1961

Report to the
Bureau of Ships
Department of the Navy

INDEX NO. S-R007 12 01, Task No. 800
(BuShips 1700S-645-59). Code 342B

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

Thermal Conductivity of Semi-Conductive Solids; Method for Steady-State Measurements on Small Disk Reference Samples

by

D. R. Flynn

1. ABSTRACT

The salient features of the final model of an absolute cut-bar apparatus suitable for thermal conductivity measurements on small solids at temperatures up to 1200°C or higher are described. The high temperature portions of the apparatus which will contact the specimen are 60% platinum-40% rhodium alloy. The remaining high temperature structural portions are high purity (99+%) alumina. Construction of the apparatus was completed and initial check measurements started. A brief summary of initial measurements to be made with the apparatus is given.

2. STATEMENT OF PURPOSE

To develop a method and apparatus for steady-state thermal conductivity measurements at temperatures to 800°C and above, and suitable for solids in the form of small specimens (1/2-in. by 1-in. diameter disks), with the objective of providing samples for use by other laboratories as thermal conductivity reference specimens in connection with their measurements on solid semi-conductors.

3. WORK PERFORMED IN REPORTING PERIOD (April 1 to June 30, 1961)

During this quarter, construction of the high temperature model of the absolute cut-bar apparatus was completed. The design of this model was based on experience gained using the modified prototype apparatus described in previous reports.

3.1 Apparatus

The general assembly of the thermal conductivity apparatus is shown in Figure 1. The specimen is interposed between a hot bar and a cold bar, fabricated from 60% platinum-40% rhodium alloy. The bars are supported by an alumina tube from below and an alumina rod from above. Surrounding the inner assembly and concentric with it is an alumina guard cylinder, which is in turn surrounded by a stainless steel case. The space inside the lower alumina support tube, that between the central assembly and the guard cylinder, and that between the guard cylinder and the outer case are filled with finely divided alumina powder as thermal insulation.

The cold bar can be brought to a desired temperature by means of a small heater fabricated from 0.25 mm diameter platinum-20% rhodium wire wound around the alumina bar immediately above the cold bar.

The hot bar is raised to the desired temperature above the cold bar by means of a small heater between the hot bar and the lower alumina support tube. This heater was fabricated from 0.25 mm diameter platinum-20% rhodium wire and has a resistance of approximately 2 ohms (at room temperature).

In order to prevent heat losses down the support tube from the hot bar heater, a small platinum-20% rhodium heater was wound on the support tube at a position about 4 cm below the hot bar.

A helical groove on the outer surface of the guard cylinder was wound with two separate heaters of 0.5 mm platinum-20% rhodium. Between the heaters the guard cylinder wall thickness was reduced to permit a longitudinal temperature distribution along the guard cylinder corresponding to that along the inner assembly.

Temperatures in the system are determined by means of platinum/platinum-10% rhodium thermocouples. All thermocouples were fabricated from 0.38 mm reference grade thermocouple wire that was calibrated against a standard thermocouple at the NBS High Temperature Laboratory, with the exception of the thermocouples in the specimen, which will be fabricated from calibrated 0.20 mm reference grade wire.

Thermocouple positions are shown in Figure 1. There are two thermocouple stations in the hot bar and two in the cold bar. In the hot bar support tube there are two thermocouple stations and one differential thermocouple. Eight thermocouples were installed to define the temperature distribution along the guard cylinder. The number and location of specimen thermocouples will be determined by the nature and geometry of the specimen.

3.2 Instrumentation

Power for the two guard heaters and the cold bar heater is supplied by variable voltage a-c transformers, which in turn are fed by a voltage regulating transformer. A power relay activated by an electronic controller connects an external resistance in series with the appropriate heater when the platinum/platinum-10% rhodium control thermocouple indicates a higher temperature than that set on the controller. The controllers are commercially obtained instruments consisting of a self-contained d-c potentiometer, converter (chopper), amplifier, and relay.

The hot bar heater, which provides the power flowing through the specimen, is powered by a commercial 28-volt, 4-ampere transistorized d-c power supply. A small bank of power resistors can be switched into the circuit to attain the desired voltage drop across the heater.

The heater in the hot bar support tube must be closely regulated in order to prevent heat gains or losses from the hot bar heater. A bias is placed on the signal from a multiple junction differential thermocouple located in the hot bar support tube, using a small d-c potentiometer. The resultant signal is amplified by a breaker-type d-c amplifier and fed into a d-c recorder. The error signal from the control slidewide in the recorder is fed into a current-adjusting type proportional controller incorporating automatic reset control and rate control. The output of this unit regulates a magnetic amplifier which feeds power to the heater. The operation of a control system of this general type has been described in detail by West and Ginnings.[1]

The noble metal leads of the thermocouples are brought to an insulated zone box maintained at room temperature. A common cold junction placed in series with a double-pole selector switch is used to automatically reference each thermocouple against an ice bath.

Thermocouple voltages are read on a calibrated precision potentiometer, usually to 0.1 μv. The power to the hot heater bar is obtained by determining the d-c voltage drop across potential taps located midway between the differential thermocouple junctions in the hot bar support and by determining the d-c current through the heater. This is done by impressing the voltage on a calibrated volt box and passing the current through a calibrated shunt box. Voltages developed across the output of the shunt and volt boxes are measured on the precision potentiometer.

4. FUTURE ACTIVITIES

When initially installed, the hot bar and cold bar were not cut apart, in order to allow measurements to be made of the thermal conductivity of the platinum-40% rhodium alloy. With known conductivities the hot and cold bars may subsequently be used as heat flow meters. Upon completion of conductivity determinations of the bar it will be cut in two and the faces optically polished.

In order to check the apparatus when assembled, it was decided to conduct measurements on two materials of known thermal conductivity. The nickel-chrome alloy measured in

---

1. E. D. West and D. C. Ginnings, Rev. Sci. Inst., 28, 1070 (1957).

the modified prototype apparatus was selected as the high conductivity material. For a low conductivity specimen, a material having a thermal conductivity similar to that of many thermoelectric materials was preferred. Measurements made on Pyroceram indicate that it would be a satisfactory specimen material. A specimen of Pyroceram Code 9606 was fabricated for measurement in the NBS metals apparatus. A specimen from the same bar is being fabricated for measurement in the high temperature absolute cut-bar apparatus. The Heat Measurements Section, NBS, is currently measuring the thermal diffusivity of this material.

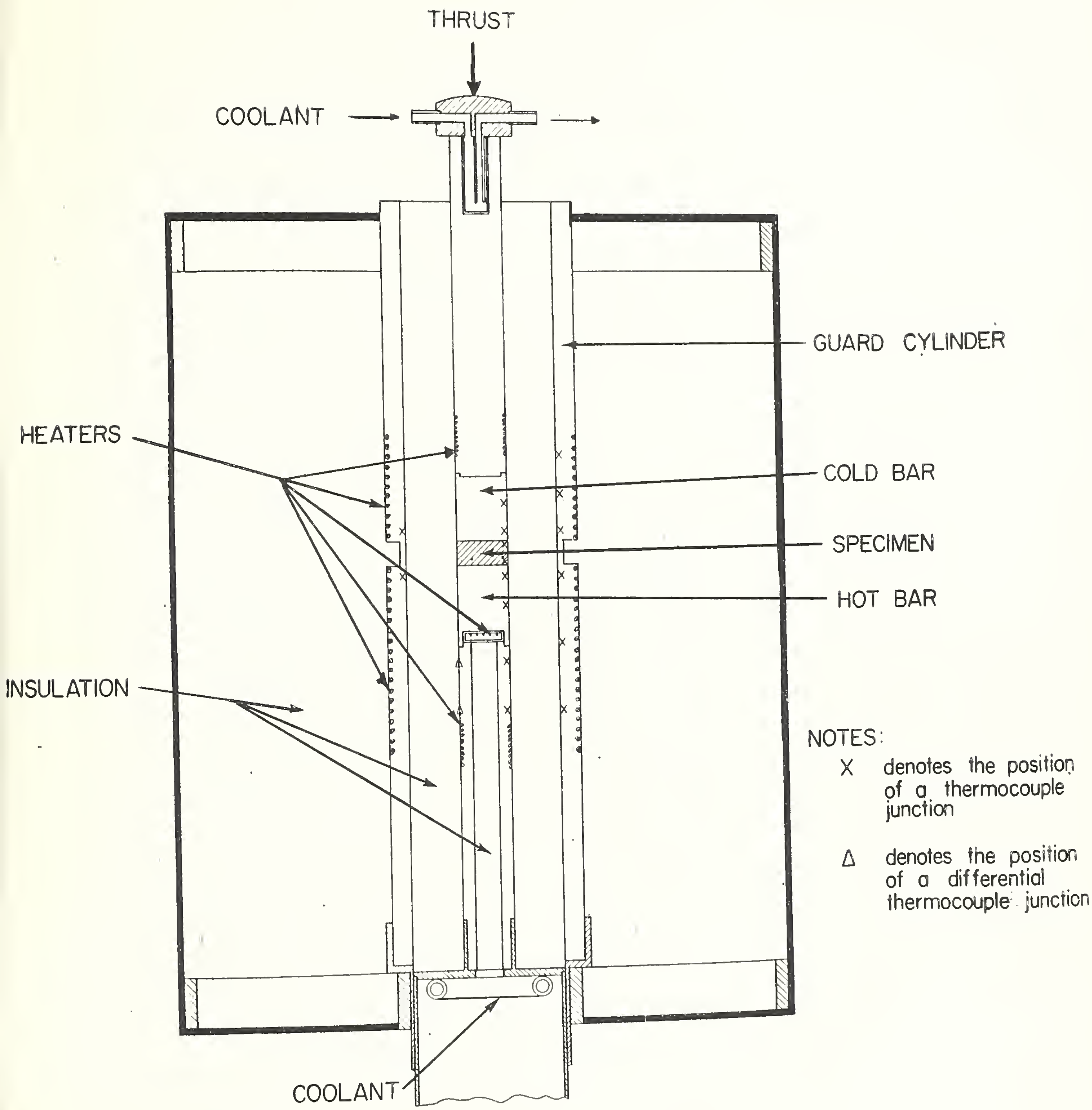
THRUST
COOLANT
GUARD CYLINDER
HEATERS
COLD BAR
SPECIMEN
HOT BAR
INSULATION
NOTES:
X denotes the position of a thermocouple junction
Δ denotes the position of a differential thermocouple junction
COOLANT

Figure 1.

U. S. DEPARTMENT OF COMMERCE
Luther H. Hodges, *Secretary*

NATIONAL BUREAU OF STANDARDS
A. V. Astin, *Director*

THE NATIONAL BUREAU OF STANDARDS

The scope of activities of the National Bureau of Standards at its major laboratories in Washington, D.C., and Boulder, Colorado, is suggested in the following listing of the divisions and sections engaged in technical work. In general, each section carries out specialized research, development, and engineering in the field indicated by its title. A brief description of the activities, and of the resultant publications, appears on the inside of the front cover.

WASHINGTON, D.C.

Electricity. Resistance and Reactance. Electrochemistry. Electrical Instruments. Magnetic Measurements. Dielectrics.

Metrology. Photometry and Colorimetry. Refractometry. Photographic Research. Length. Engineering Metrology. Mass and Scale. Volumetry and Densimetry.

Heat. Temperature Physics. Heat Measurements. Cryogenic Physics. Equation of State. Statistical Physics.

Radiation Physics. X-ray. Radioactivity. Radiation Theory. High Energy Radiation. Radiological Equipment. Nucleonic Instrumentation. Neutron Physics.

Analytical and Inorganic Chemistry. Pure Substances. Spectrochemistry. Solution Chemistry. Standard Reference Materials. Applied Analytical Research.

Mechanics. Sound. Pressure and Vacuum. Fluid Mechanics. Engineering Mechanics. Rheology. Combustion Controls.

Organic and Fibrous Materials. Rubber. Textiles. Paper. Leather. Testing and Specifications. Polymer Structure. Plastics. Dental Research.

Metallurgy. Thermal Metallurgy. Chemical Metallurgy. Mechanical Metallurgy. Corrosion. Metal Physics. Electrolysis and Metal Deposition.

Mineral Products. Engineering Ceramics. Glass. Refractories. Enameled Metals. Crystal Growth. Physical Properties. Constitution and Microstructure.

Building Research. Structural Engineering. Fire Research. Mechanical Systems. Organic Building Materials. Codes and Safety Standards. Heat Transfer. Inorganic Building Materials.

Applied Mathematics. Numerical Analysis. Computation. Statistical Engineering. Mathematical Physics. Operations Research.

Data Processing Systems. Components and Techniques. Digital Circuitry. Digital Systems. Analog Systems. Applications Engineering.

Atomic Physics. Spectroscopy. Infrared Spectroscopy. Solid State Physics. Electron Physics. Atomic Physics.

Instrumentation. Engineering Electronics. Electron Devices. Electronic Instrumentation. Mechanical Instruments. Basic Instrumentation.

Physical Chemistry. Thermochemistry. Surface Chemistry. Organic Chemistry. Molecular Spectroscopy. Molecular Kinetics. Mass Spectrometry.

Office of Weights and Measures.

BOULDER, COLO.

Cryogenic Engineering. Cryogenic Equipment. Cryogenic Processes. Properties of Materials. Cryogenic Technical Services.

Ionosphere Research and Propagation. Low Frequency and Very Low Frequency Research. Ionosphere Research. Prediction Services. Sun-Earth Relationships. Field Engineering. Radio Warning Services.

Radio Propagation Engineering. Data Reduction Instrumentation. Radio Noise. Tropospheric Measurements. Tropospheric Analysis. Propagation-Terrain Effects. Radio-Meteorology. Lower Atmosphere Physics.

Radio Standards. High Frequency Electrical Standards. Radio Broadcast Service. Radio and Microwave Materials. Atomic Frequency and Time Interval Standards. Electronic Calibration Center. Millimeter-Wave Research. Microwave Circuit Standards.

Radio Systems. High Frequency and Very High Frequency Research. Modulation Research. Antenna Research. Navigation Systems.

Upper Atmosphere and Space Physics. Upper Atmosphere and Plasma Physics. Ionosphere and Exosphere Scatter. Airglow and Aurora. Ionospheric Radio Astronomy.

NBS