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.

NATIONAL BUREAU OF STANDARDS
U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS
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INDEX NO. S-R007 12 01, Task No. 800 
(BuShips 1700S-645-59). Code 342B

IMPORTANT NOTICE

Approved for public release by the 
Director of the National Institute of 
Standards and Technology (NIST) 
on October 9, 2015.

NATIONAL BUREAU OF STANDARDS
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 slidewire 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.

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

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.
Figure 1.

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THE NATIONAL BUREAU OF STANDARDS

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WASHINGTON, D.C.


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