NATIONAL BUREAU OF STANDARDS REPORT

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MEASUREMENTS OF THE THERMAL CONDUCTIVITY OF SEVERAL EXPLOSIVES BY THE CONDUCTIVE DISC METHOD

BY

H. E. Robinson, D. R. Flynn, and T. W. Watson

Report to the
Fire Protection Section
Building Technology Division
NBS

U. S. DEPARTMENT OF COMMERCE
NATIONAL BUREAU OF STANDARDS

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1. INTRODUCTION

In connection with determinations of the thermal properties of explosives and solid propellants, undertaken by the Fire Protection Section of the National Bureau of Standards for the Naval Ordnance Laboratory, measurements were desired of the thermal conductivity of these materials. Because of possible danger of ignition of the materials, it was decided that the tests should be made on samples of limited size (less than 1 lb) in an available small underground vault, with controls and observations at a station outside the vault.

The Heat Transfer Section was consulted concerning the possibility of providing a suitable reliable apparatus for making these thermal conductivity measurements. A new type of thermal conductivity measuring apparatus, which had been under mathematical consideration, appeared more promising for this application than several alternative methods that were considered. Accordingly, the Heat Transfer Section constructed an appropriate apparatus of the new type, the design and calibration of which are described below.

2. DESCRIPTION

The essential elements of the "conductive disc" apparatus consist of a circular disc of metal sandwiched between two specimens of the same diameter, which in turn are sandwiched between two water-cooled plates of the same diameter (see Figure 1). The conductive disc is uniformly heated at its circular edge by means of electrical resistance wire set in a groove. The heat generated at the disc edge tends to flow in the disc radially toward its center, and also to flow from the disc through the two specimens to the uniform-temperature cold plates. Thus, the steady-state temperature of the conductive disc decreases toward its center. By measuring the temperature of the disc at its center and at a

suitable radius, the effective conductance of the specimens can be calculated, if the temperature of the cold plates and the conductivity and thickness of the disc metal are known.

The apparatus constructed has a 6-inch diameter disc of Type 303 stainless steel, fine-ground to a uniform thickness of 0.7264 cm. In addition to one thermocouple at the center, three additional thermocouple junctions are located at 120° angular spacing at a radius of 5.080 cm, all thermocouple junctions being inset in metallic contact with the stainless steel at one surface of the disc. The cold plates are 6-inch discs of brass, 1 cm thick, with several turns of 3/8-inch copper tube soldered to their non-working sides for circulation of coolant. The working surfaces of the cold plates were turned flat in a lathe after the soldering was completed. A thermocouple junction is inset in the brass disc at its center for measuring its temperature. The dimensions selected for the conductive disc of the apparatus were predicated on measurements on specimens 1/2 inch thick by 6 inches in diameter, having thermal conductivities in the range from 0.5 to 5.0 mw/cm C.

The heater winding in the edge-groove of the conductive disc consists of approximately 13 ft of No. 30 constantan wire, bifilarly wound, insulated with alundum-saturated fiberglass sleeving. Its electrical resistance is approximately 60 ohms.

Associated equipment includes a 20 gal. tank with a laboratory immersion pump for circulating water through the cold plate coils at about 1 gpm, a voltage regulator and a variable a-c transformer for power supply to the disc heater, and a precision potentiometer for thermocouple readings. All thermocouples are made of No. 30 copper and constantan wires, and are used with the reference junctions at 0°C.

3. USE AND ELEMENTARY ANALYSIS OF THE APPARATUS

An approximate analysis of the conductive disc method is given below, adequate for showing its chief operating characteristics, for designing the apparatus for the measurements desired, and for enabling certain computations of the test results.

A more rigorous analysis of the method is being made to develop a comprehensive mathematical treatment of the conductive disc apparatus, but is not needed for the apparatus

described here. As used in the tests reported here, the apparatus was calibrated by means of a guarded hot plate thermal conductivity apparatus. The calibration curve thus established served as a means for translating the measurements made in the disc apparatus into conductivity determinations tantamount to those that would have been obtained in tests in the guarded hot plate.

For steady temperature conditions, an approximate differential equation for the disc apparatus (which neglects non-radial temperature gradients in the disc, and radial temperature gradients in the specimens) is

$$2\pi rmK\left(\frac{d^2v}{dr^2}+\frac{1}{r}\frac{dv}{dr}\right)dr-2\pi rdrv\left(\frac{k}{L}+\frac{k'}{L'}\right)=0 \qquad (1)$$

in which the first part represents the change in radially-directed heat flux in the disc in a radial distance dr at a radius r, and the second part represents the flow of heat from the disc toward the cold plates through both specimens, through the disc surface area between r and (r+dr). In equation (1), m is the thickness of the conductive disc, K is its thermal conductivity, k and k' are the thermal conductivities of the two specimens and L and L' their thicknesses, and v is the temperature of the disc surface referred to the cold plate temperature as 0. It is assumed in equation (1) that the conductivities of the disc and specimens are constant at values corresponding to their mean temperatures.

Equation (1) becomes, upon simplification,

$$\frac{d^2v}{dr^2}+\frac{1}{r}\frac{dv}{dr}-\mu^2v=0 \qquad (2)$$

where

$$\mu^2=\frac{1}{mK}\left(\frac{k}{L}+\frac{k'}{L'}\right) \qquad (2a)$$

Equation (2) is Bessel's equation of order zero, having the solution

$$v=AI_0(\mu r) \qquad (3)$$

Since $I_0(o)=1$,

$$\frac{V}{V_0}=I_0(\mu R) \qquad (4)$$

where V_0 is the temperature of the disc surface at radius 0, and V the disc surface temperature at radius R, and where I_0

is the modified Bessel's function of the first kind and of order 0.[a]

The mean temperature of the disc within the radius R, referred to the observed temperature of the cold plates as zero, is given by

$$V = \frac{1}{\pi R^2}\int_0^R 2\pi r V_o I_o(\mu r)dr = \frac{2V_o I_1(\mu R)}{\mu R} \qquad (5)$$

where I_1 is the modified Bessel's function of the first kind, of order 1. It is found, using tables of I_1, that

$$\frac{2V_o I_1(\mu R)}{\mu R} = \frac{V_o+V}{2} \text{ to within } 1/2\% \text{ for } V/V_o<1.4.$$

Thus for the tests reported, wherein $V/V_o<1.3$, it is adequate to take the actual mean temperature of the disc in a test as

$$t_d = 0.5(V_o+V) + t_c \qquad (6)$$

where t_c is the observed temperature of the cold plates.

Similarly, the mean temperature of the specimen in a test is calculated as

$$t_s = 0.5(t_d+t_c) = 0.25(V_o+V) + t_c \qquad (7)$$

4. CALIBRATION

If the value of μR were known accurately, equations (4) and (2a) would serve to determine $(k/L+k'/L')$, within the limit of exactness of the I_o solution. However, it was not feasible to obtain a piece of the metal used for the disc, of size adequate for a direct measurement of its thermal conductivity, K.

Further, it was desired to use in a test only one specimen of explosive, rather than two, and therefore a permanent "dummy specimen" was used in all tests in place of a duplicate of the principal specimen. This consisted of a 6-inch diameter disc of 1-inch thick semi-rigid glass-fiber

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a/ Values of $I_o(x)$ and $I_1(x)$ are available to 7 places in tabulated form for values of x from 0 to 10 in increments of 0.001 (Tablitsy znachenii funktsii Besselia ot mnimoio argumenta, ... 1950, Moscow).

insulating board of stable low thermal conductivity (0.348 mw/cm C at 50°C) maintained at a constant thickness of 0.991 inch during use by three small fiber pegs thrust perpendicularly through the board. The lower cold plate of the apparatus, the dummy specimen, and the conductive disc were fastened together with rubber cement to form a sub-assembly.

To calibrate the test apparatus, tests were made on six 6-inch disc specimens of thermal conductance from 0.3 to 4.4 mw/cm^{2}C, each in combination with the dummy specimen of thermal conductance of approximately 0.14 mw/cm^{2}C, and the steady-state value of V/V_0 was observed in each case. The conductances of the six principal specimens, and of the dummy specimen, were determined by means of previous tests conducted on them in the form of 8-inch squares in the Bureau's guarded hot plate apparatus (ASTM Designation C177-45).

The results of the calibration measurements are recorded in Table 1, and there are plotted in Figure 2 the hot plate values of conductance, k/L, of the principal specimens, versus the observed values of V/V_0 from the conductive disc measurements on them. A regular curve is faired amongst the six calibration points, to yield a smooth curve representing the calibration of the conductive disc apparatus. Departures of the experimental points and the curve do not exceed one per-cent.

The conductances in Figure 2 are values corresponding to the mean temperature of the specimen in the conductive disc test when the cold plate temperature was about 27C and the disc mean temperature was about 69C. In tests made with the conductive disc apparatus, using the calibration curve of Figure 2, the conductance obtained requires correction, as indicated below, to compensate for the change in disc conductivity if the disc mean temperature departed significantly from 69C. The corrected conductance corresponds to the actual mean temperature of the specimen in the conductive disc test.

The temperature coefficient of conductivity of Type 302 stainless steel (in the equation $K_d = K_{69}[1+ (t_d-69)]$) is 0.0012, approximately. Accordingly, the conductance obtained from the calibration curve, k/L(c.c.), should be corrected by the relation

$$\frac{k}{L}(\text{at } t_s) = \frac{k}{L}(\text{c.c.}) \times [1+0.001(t_d-69)] \qquad (8)$$

Taking the value of k/L (2.31 mw/cm^{2}C) from the calibration curve at V/V_o=1.15, and the value of k'/L'=0.137 at 48°C mean temperature, and using Equations (4) and (2a), the conductivity of the Type 303 stainless steel of the disc is calculated to be 0.150 w/cm C at 69°C. A measurement made here in 1956 on a sample of Type 303 rod 1 inch in diameter indicated its conductivity to be 0.141 w/cm C at 65°C. NBS Circular 556, "Thermal Conductivity of Metals and Alloys at Low Temperatures, a Review of the Literature" (1954), gives data (Figure 23) which indicate that the conductivity of S.S. 303 at 69°C is about 0.153; the Stainless Steel Handbook of the Allegheny Steel Corporation indicates a value of 0.155 w/cm C at 69°C. The value of disc conductivity obtained from the disc calibration measurements is seen to be intermediate between available values from other measurements.

5. PROCEDURE AND OPERATION

In the present use of the apparatus, the lower cold plate, the dummy specimen, and the conductive disc formed a sub-assembly, and it was necessary only to interpose the single test specimen between the conductive disc and the upper cold plate. For good thermal contact, the test specimen must be flat and of uniform thickness, and the contacting surfaces must be clean. To improve thermal contact between the working surfaces and the specimen, an 18-lb weight was centered on the upper cold plate, resulting in a pressure on the specimen of approximately 0.8 psi. Care was taken to assure that the several circular parts and the weight were positioned coaxially.

Ball bearings are set near the edges in the outward faces of both cold plates at angular spacings of 120°. Micrometer measurements of the axial distance across the ball bearings in the two cold plates, with and without the test specimen in place, enabled the thickness of the specimen to be accurately determined.

Thermal insulation of the working parts of the assembled apparatus was accomplished by pouring around it within a removable 12-inch diameter container a free-flowing powder insulation (silica aerogel). The powder insulation was readily drained from around the apparatus by opening a sliding gate at the bottom of the container.

To start a test, the disc heater winding was energized at a reasonable power input rate (an initial setting of about 6 watts input was used for these measurements), and if the power input and the cooling water temperature were constant, the apparatus reached a substantially steady temperature condition in from 2 to 3 hours. Readings of V and V_o were then made periodically for an hour or more to assure that steady-state conditions existed. The value of the ratio V/V_o does not depend on the rate of power input, but to determine this ratio with good precision, it is desirable that the difference between V and V_o be determinable with a precision of better than 1 part in 200, and therefore that the rate of power input be adjusted to yield an adequate difference of V and V_o. In testing explosives, however, it was desirable that the maximum temperature of the disc in contact with the test specimen be limited to some safe value. For this apparatus the maximum disc temperature, which occurs at the rim, is approximately equal to

$$t_{max} = t_c + V_o(V/V_o)^2 \qquad (9)$$

for values of V/V_o up to 1.3.

6. RESULTS OF MEASUREMENTS ON EXPLOSIVES

Table 2 summarizes the data obtained with the conductive disc apparatus on explosives.

The six materials were found to have thermal conductivities between 1.9 and 2.6 mw/cm C.

During the course of the measurements made in the underground vault on the explosives, two check measurements were made on the original 1.28-cm Neoprene specimen used in calibrating the apparatus. The first was made on initially setting up the apparatus in the vault prior to the first TNT measurement. Following the second TNT measurement, it was necessary to clear the vault for other work, and the second check test was made when the apparatus was reinstalled, just prior to the measurement of the QR specimen. The conductance of the Neoprene specimen, as determined by the hot plate and used in the calibration test, was 2.494 mw/cm² C at 48°C mean temperature. In the first check test, the conductance obtained was 2.50 at 45°C; in the second, 2.58 at 40°C. There was therefore an observed variation in results of 2.4 percent, to which the results on the explosives must be considered subject.

The two measurements made on TNT, at 33 and 38°C, indicate a negative temperature coefficient of conductivity (α = -0.011 per deg C). Because of the small difference in test temperatures, the uncertainty in this value of α is probably large. Hot plate tests conducted on paraffin (129°F M.P.) and Halloway at 70° to 90°F mean temperature have indicated temperature coefficients of -0.005 and -0.0043 per deg C, respectively, for these waxy materials.

TABLE 1 - CALIBRATION MEASUREMENTS

Principal Specimen	Hot Plate Data k/L* mw/cm² C	Heater Volts	V/V_o	Cold Plate Temp. t_o °C	Disc Temp. at r=o °C	Disc Temp. at r=R** °C	Disc Temp. at Rim °C	Mean Temp. (o<r<R) Disc °C	Mean Temp. (o<r<R) Specimen °C
Fiber glass board 1.11 cm	0.3093	25	1.0272	26.22	66.89	67.99	69	67.4	46.8
Gum rubber 1.10 cm	1.342	30	1.0900	26.76	64.36	67.75	72	66.0	46.4
Neoprene 91.7 pcf 1.28 cm	2.494	42	1.1614	26.29	66.27	72.73	82	69.5	47.9
Neoprene 94.0 pcf 1.32 cm	2.999	43	1.1936	28.49	65.55	72.72	83	69.1	48.8
Neoprene 97.3 pcf 1.32 cm	3.117	45	1.1976	26.73	65.41	73.05	84	69.2	48.0
Gum rubber 0.36 cm	4.379	55	1.2704	26.12	66.59	77.53	93	72.0	49.0

* k/L is corrected to correspond to the mean temperature of the specimen in the conductive-disc test. The conductance of the dummy specimen, k'/L', was 0.116 $(1+0.0039t_s)$ mw/cm² C, where t_s is its mean temperature.

** Average of three temperatures at R=5.08cm, at 120° angular spacing.

TABLE 2 - THERMAL CONDUCTIVITY, k, OF EXPLOSIVES

Specimen	Thickness (cm)	V/V_o	Cold Plate Temp. (°C)	Disc: Center Temp. (°C)	Disc: Ave. Temp. at R=5.08cm (°C)	Disc Mean Temp. (°C)	Specimen: Mean Temp. (°C)	Specimen: k (mw/cm C)
TNT	1.272	1.1094	24.72	40.18	41.87	41	33	2.04
"	1.272	1.1029	24.57	50.36	53.01	52	38	1.93
B	1.272	1.1316	18.89	44.25	47.21	46	33	2.50
CYCLOTOL	1.271	1.1358	6.74	18.17	19.72	19	13	2.51
DINA	1.270	1.1275	0.05	12.00	13.52	13	6	2.33
RDX	1.281	1.1083	21.97	51.19	54.35	53	37	2.05
[illegible]	1.273	1.1354	23.24	47.17	50.42	49	36	2.59

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