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APPARATUS FOR MEASURING THERMAL CONDUCTIVITY OF METALS UP TO 600 C

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ABSTRACT

Apparatus for measuring the thermal conductivity of metals up to 600 C is described. The method employed consists in comparing the conductivity of a metal, either directly or indirectly, with that of lead. Lead was selected as the standard since previous measurements have established its conductivity within fairly close limits. Determinations are made by measuring the axial temperature gradients in two cylindrical bars soldered together end to end, one end of the system being heated and the other cooled, and the convex surfaces protected from heat loss by a surrounding guard tube. Data are given on the conductivity of commercial nickel, high purity zinc, high purity nickel, and several commercial nickel-chromium and other alloys widely used for heating elements and thermocouples.

CONTENTS

I. INTRODUCTION

The thermal conductivity of metals is of considerable theoretical and practical interest, and numerous publications on the subject have been made. Perhaps for no other definite thermal property are the published results so widely divergent and subject to such great uncertainty, particularly at high temperatures. This may be attributed in part to the inherent difficulties of measurement, but perhaps to an even greater extent to the fact that the conductivity of a metal or alloy is sensitive to heat treatment, crystal structure, and slight changes in composition. In fact the exact description of a polycrystalline specimen, necessary and sufficient for its reproducibility so far as conductivity is concerned, is in many cases about as uncertain as the value of the conductivity itself.

The Bureau of Standards has received numerous requests for determinations of the thermal conductivity of particular metals, primarily alloys for special industrial purposes, and it therefore seemed desirable to set up an apparatus with which such tests could be made from time to time, over a considerable range of temperature.

The great sensitivity of thermal conductivity to changes in composition of alloys, and to the physical condition of the constituents, suggests the possibility that such measurements may in some cases be useful in metallographic investigations.

II. METHOD OF MEASUREMENT

Methods used for determining the thermal conductivities of metals have varied widely, both steady heat flow and variable heat flow having been employed. In nearly all cases "absolute" determinations have been made, which involve measurements of quantities of heat. The comparative method of using two specimens, one of known conductivity, through which heat flows in series, appeared to offer great simplification and to be less subject to uncertainty, particularly at high temperatures. This method, which seems first to have been employed by Berget,[1] has been used in the present work.

In the measurements to be described lead was selected as the primary standard, since it appeared to be the most suitable for the purpose, and previous measurements had established its conductivity at ordinary temperature within fairly close limits. If it eventually proves necessary or desirable, it will be possible to determine the absolute conductivity of the lead actually used, under conditions most favorable to accuracy. Such a determination need be made at but one mean temperature. On account of the undesirable mechanical properties of lead, a specimen of nickel previously compared with lead was used as the working or secondary standard in routine measurements.

The method employed consists in measuring the axial temperature gradients in two cylindrical bars soldered together end to end, one end of the system being heated and the other cooled, and the convex surfaces protected from heat loss by a surrounding guard tube. When a steady state has been attained, the heat flux is the same in both bars, and the conductivity at any point in either bar is inversely proportional to the temperature gradient at that point. If the absolute value of the conductivity of the metal of one bar is known at some temperature within the experimental range, that of the metal of the other bar can be calculated at all points at which the gradient has been measured. Gradients cannot be directly observed, but can be calculated from direct measurements of temperature distribution along the bars. By using the same procedure and employing a bar of a single material, the temperature coefficient of conductivity of the material of this bar can be determined without reference to other materials.

III. DESCRIPTION OF APPARATUS AND EXPERIMENTAL PROCEDURE

Elaborate refinement of apparatus to obtain great precision was not attempted. Such refinements did not appear to be warranted, at least for measurements on polycrystalline materials. The aim was to construct an apparatus of comparatively simple design, with which a precision of better than 2 percent might be expected.

A cross section of the apparatus with test specimens in place is shown in figure 1. The specimen to be measured (*A*), 15 cm long and 2 cm in diameter is soldered to the copper cylinder (*C*) which serves as a heat collector from the surrounding heating coil. The standard bar (*B*), 5 cm long and 2 cm in diameter, is soft soldered between specimen (*A*) and the brass chamber (*J*), through which water at constant temperature is circulated. Tin is ordinarily used

[1] Berget, Comp. Rend. 105, p. 224, 1887.

for joining the two bars, and low melting point solder (Bi–Sn eutectic) for attaching the water cell. Tin is also used to provide thermal contact between specimen (*A*) and the copper cylinder. At higher temperatures the thermal contact is not impaired, since the copper alloys with the tin and produces a hard soldered joint.

The copper cylinder (*C*), 8.5 cm long and 3 cm in diameter, is surrounded by a heating coil of chromel A ribbon wound on a thin-walled alundum tube (*G*), 8 cm long and 4 cm in outside diameter.

The guard or shield tube (*D*), made of chromium-nickel stainless steel (18–8), is 23 cm long, 6.8 cm outside diameter, and has a wall thickness of 0.8 cm. The top is jointed and soldered to a nickel tube (*E*) of the same cross section and 5 cm long. A copper tube (*K*), 0.4 cm inside diameter, through which constant temperature water circulates, is soldered around the top of the nickel tube. The shield tube is heated at its base by a heating unit consisting of 6.3 ohms of chromel ribbon wound around alundum tube (*H*), 8 cm long and 5.6 cm outside diameter.

The copper ring-shaped disk (*N*), 12 cm in outside diameter, is soldered to the shield tube at the joint between the steel portion and the nickel portion. Spiral grooves are cut on both sides of this ring and a heating unit consisting of no. 22 chromel wire insulated with glass beads is cemented into the grooves. This auxiliary heating unit is necessary for accurate control of the temperature of the guard tube. The portion of the guard tube above the ring heater is made of nickel, a much better heat conductor than chromium-nickel steel, in order that proper adjustment of the temperature of the guard would always be possible by supplying heat at the joint between nickel and steel. If the guard tube consisted of the same metal throughout its length, it would be necessary in some cases to cool the region where the ring heater is located, a procedure which was considered less convenient than making the guard tube in two sections.

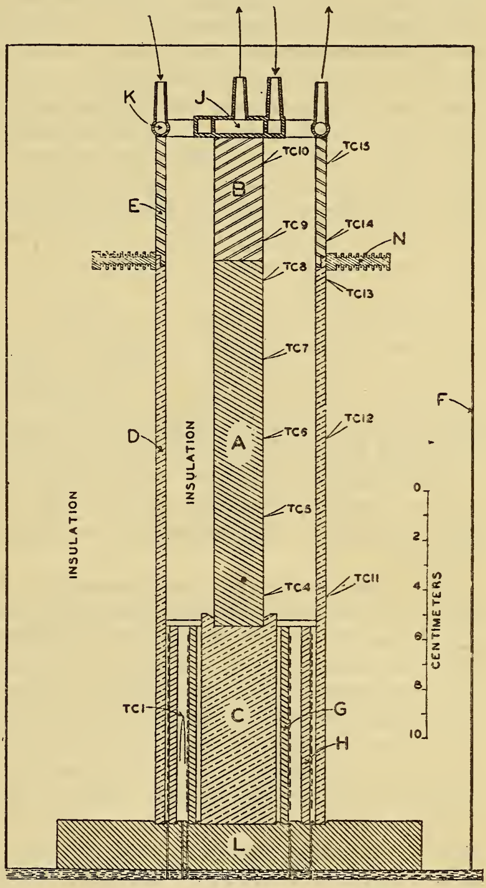

Figure 1.—*Cross section diagram of apparatus.*

The guard tube, heating coils, and copper cylinder rest on an alundum disk (*L*), 2.5 cm thick and 14 cm in diameter. The apparatus and alundum base are surrounded by a galvanized steel case (*F*), about 20 cm in diameter, split longitudinally into two halves for convenience. The entire assembly rests on asbestos board and is insulated underneath with insulating brick. The annular spaces between the test specimens and guard tube, and between the latter and the steel case, are filled with diatomaceous earth for thermal insulation.

Thermocouple 1 is placed between the heating coils (*G*) and (*H*) to facilitate temperature control. Thermocouples 4 to 10, inclusive, used for measurement of temperature distribution along the test specimens, are attached to the surface of the bar either by electric spot welding or by peening. The latter method was found to be more satisfactory in most cases. Very small transverse cuts slightly deeper than the wire diameter are made on the convex surface of the specimen, the junction laid in the cut and peened in by tapping with a hammer or punch. In this process the cut closes up on the wire and grips it firmly. The two wires forming the thermocouple are previously butt welded, and the junction reduced to about the same size as the wire. It was found that if the wires were merely peened in without welding, high contact resistances sometimes developed. All thermocouples on the test specimens are made of no. 36 chromel P and alumel wires.

Thermocouples 11 to 15, inclusive, of no. 22 chromel-alumel, are attached by peening to the guard tube directly opposite couples 6, 8, 9, and 10, respectively. All leads are insulated with short lengths of porcelain or glass tubing. The leads to thermocouple 1 and connections to the heating coils pass through the base of the apparatus. All other leads are brought out through the open top.

Constant temperature water from a thermostated tank is allowed to flow through chambers (*J*) and (*K*) through separate rubber tubes. Suitable valves are attached to control the rates of flow in each chamber.

The cold junctions of all thermocouples are contained in small glass tubes inserted in holes in a thermally insulated aluminum block. This arrangement assures substantial equality in the temperatures of the cold junctions, and is far more convenient than the use of ice baths. The temperature of the cold junction is observed with a mercury thermometer, and the drift in this temperature during the course of a set of readings is too small to be of any significance whatever. The actual temperature of the cold junction is of course easily measured with an accuracy far greater than is required.

Electric energy is supplied to the heating coils (*G*) and (*H*) by a 60-volt storage battery, and suitable rheostats are provided for adjustment of current. The disk heater (*N*) is supplied with alternating current from the switchboard, since constancy of power supply to this coil is not vital. In making an experiment, the system is heated up rapidly with ac to the desired temperature and then switched over to dc. Currents in the individual heating elements are then manually adjusted from time to time until an approximately steady state is attained and the temperatures at corresponding points on specimens and guard tube are approximately equal. This procedure requires several hours on account of the considerable quantity of lagging around the apparatus. At the time thermocouple readings are made, a certain amount of drift in the temperature of the hot end can be tolerated without significant error. This point as well as the question of required degree of equality of temperatures of tube and specimen will be discussed in a later section. To eliminate the effect of drift, thermocouple readings are taken at equal intervals of time (usually one half minute), beginning at the hot end, proceeding to the cold end, and repeating readings back to the hot end. The averages of the two readings at each point give a set of readings substantially the same as would be obtained if all thermocouples could be observed simultaneously. Several such sets of readings are ordinarily made in the course of a day.

The temperature distribution along the specimen (or specimens) is calculated from the thermocouple readings and the measured positions of the junctions along the bar. The temperature gradient at the mean temperature between two adjacent junctions is taken as the ratio of the temperature difference between the junctions and the distance between them. No appreciable error is introduced by taking finite differences, since the temperature as a function of distance along the specimen has in all cases only a small curvature. If a value for conductivity is assigned for some temperature within the experimental range, values at other temperatures can be calculated by the relation:

$$K\frac{dT}{dx}=K\frac{\Delta T}{\Delta x}=\text{constant}$$

where K = thermal conductivity
and $\frac{dT}{dx}=\frac{\Delta T}{\Delta x}$ = temperature gradient.

This relation obviously holds for a bar consisting of two specimens of the same diameter placed end to end, as well as for a specimen of a single material.

IV. TESTS TO DETERMINE PRECISION OF METHOD

To determine the precision of measurement attainable in the type of apparatus described, a series of experiments on three metallic specimens was made. In addition to lead, the primary standard, nickel and zinc were selected for this purpose. These metals will be designated as reference standards. The general plan was to intercompare these metals in all possible combinations and observe the consistency of the results obtained.

Determination of the temperature coefficients of thermal conductivity of the three metals was first made. This was accomplished as already noted by placing the same metal in the *A* and *B* positions (refer to fig. 1). A number of experiments of this kind were made on each metal, using various temperature gradients in each case. Comparisons of the three metals were then made by placing the various materials in a number of combinations of the *A* and *B* positions. All the results obtained were calculated relative to lead, the thermal conductivity of which was assumed to be 0.352 watts cm^{-1} deg^{-1} at 0 C (Int. Crit. Tables, vol. 5, p. 182).

A typical example of a single set of readings is given in table 1. Readings of the thermocouples on the guard tube were taken at approximately the same time as those on the specimen, but the exact time is not significant, since the drift in temperature difference between tube and specimen is negligible over a considerable period.

TABLE 1.—*Typical example of set of thermocouple readings*

TC no.	Thermocouples on specimen				Thermocouples on tube	
	Time	Emf	Time	Emf	TC no.	Emf
	min	*μv*	*min*	*μv*		*μv*
4	0.5	10,308	6.5	10,315	11	10,190
5	1.0	8,422	6.0	8,426		
6	1.5	6,572	5.5	6,574	12	6,500
7	2.0	4,829	5.0	4,830		
8	2.5	3,132	4.5	3,132	13	3,037
9	3.0	2,256	4.0	2,256	14	2,147
10	3.5	585			15	571

A skeleton table of calculations for a lead specimen is given in table 2. The quantity $\Delta x/\Delta T$, the reciprocal of the temperature gradient, is proportional to the thermal conductivity. The numbers in the last column were obtained by assigning the value 0.352 watts cm^{-1} deg^{-1} to the value of the conductivity at 0 C. This process required a slight extrapolation since the lowest mean temperature at which the gradient was observed was 58 C.

TABLE 2.—*Skeleton table of calculations*

Thermocouple	Mean	Temperature	Temperature difference between specimen and shield tube	Mean temperature between adjacent thermocouples	Difference in temperature between adjacent thermocouples ΔT	Distance between adjacent thermocouples ΔX	$\frac{\Delta X}{\Delta T}$	Thermal conductivity watts cm^{-1} deg^{-1}
	μv	°*C*	°*C*	°*C*	°*C*	*cm*		
TC_4	10312	277.3	3.0	254	45.7	3.14	687 (x10^{-4})	0.305
TC_5	8424	231.6		209	45.1	3.14	697	.310
TC_6	6573	186.5	1.8	165	43.5	3.14	722	.322
TC_7	4830	143.0		122	42.7	3.15	738	.328
TC_8	3132	100.3	2.3					
TC_9	2256	78.6	2.7	58	41.3	3.16	765	.340
TC_{10}	585	37.3	0.3					

V. EXPERIMENTAL RESULTS

Description of the three metals, nickel, lead and zinc, used in the experiments described in the previous section is given in table 3. Results of all measurements on these metals are graphically represented in figures 2, 3, and 4. It will be noted that the results obtained with all combinations of the metals in the *A* and *B* positions are mutually consistent within about 2 percent. The conductivity of zinc as a function of temperature shows a slight but distinct curvature. Those of the other metals do not, but the curve for nickel shows a very abrupt change in direction at about the temperature of the magnetic transformation point.

Measurements have been made on a number of commercial iron alloys, the results of which are given in the following paper (RP669). In the present paper are included data on the thermal conductivity of nickel of high purity, as well as nickel-chromium and other alloys widely used for heating elements and thermocouples. These materials are described in table 4, and the results of conductivity measurements are shown graphically in figures 5 and 6. Results on all materials described in this paper, interpolated from the experimental data, are given in table 5.

TABLE 3.—*Description of reference standards*

Designation	Material	Impurities	Description
(N.S.)	Nickel	Cu 0.14, Mn 0.09, Fe 0.60	Commercial malleable nickel. Specimen machined from hot rolled rod.
(L.S.)	Lead	Unanalyzed	Bureau of Standards melting point standard lead. Probably the purest lead available in usable quantity. Sample was cast in a bottom feed cast-iron mold and machined to proper dimensions.
(Z.S.)	Zinc	Pb 0.04, Fe 0.02 Cd, not detected	"Chemically pure" zinc. Sample cast in graphite mold and machined.

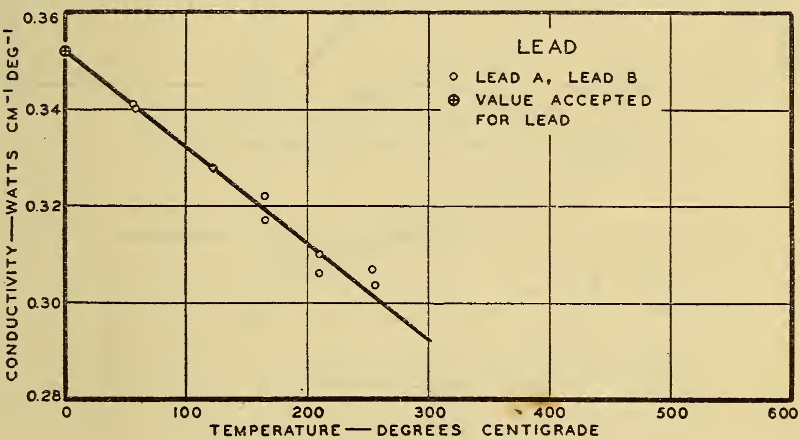

FIGURE 2.—*Results of measurements on lead.*

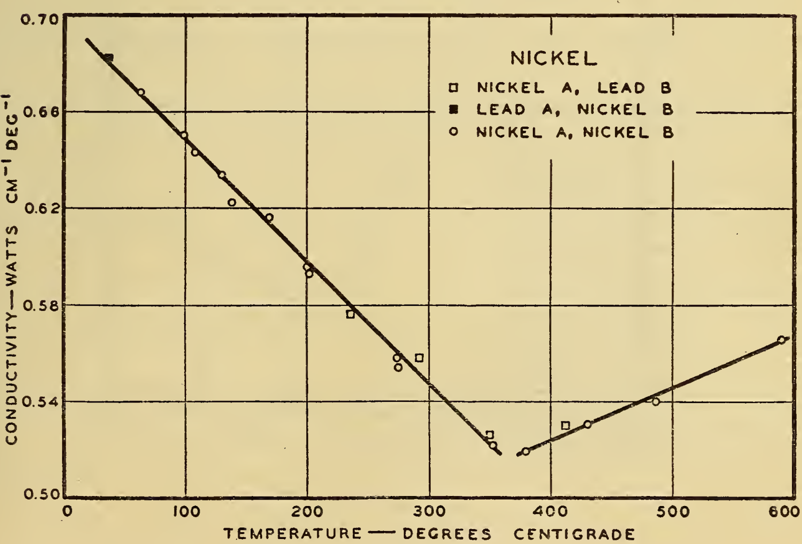

FIGURE 3.—*Results of measurements on commercial nickel.*

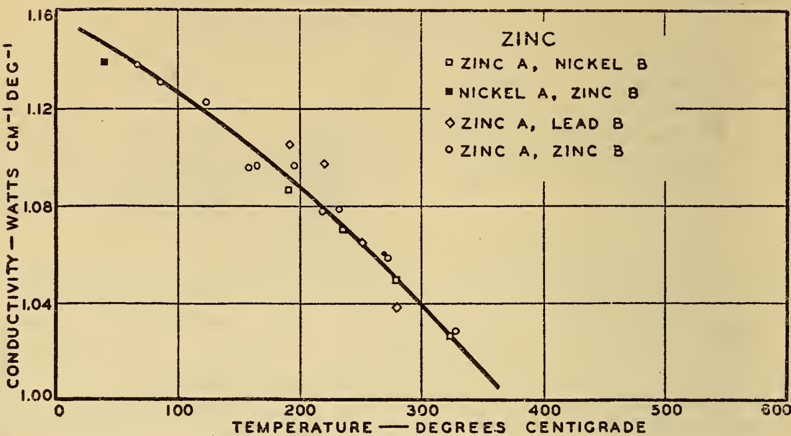

FIGURE 4.—*Results of measurements on zinc.*

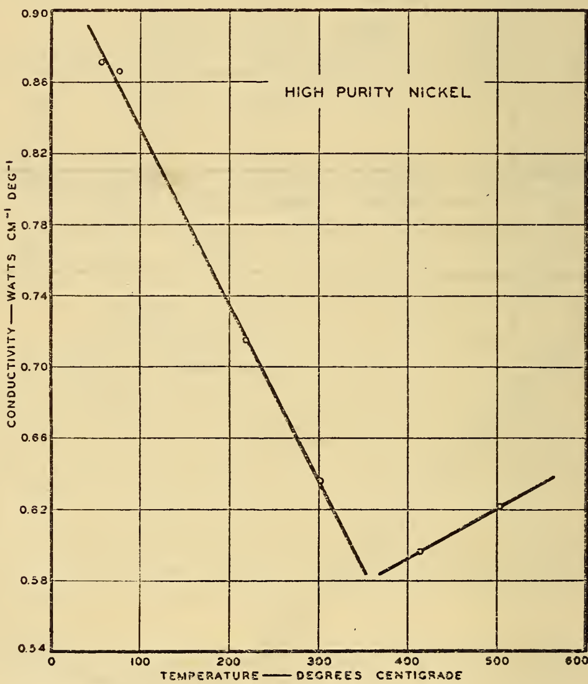

FIGURE 5.—*Results of measurements on high purity nickel.*

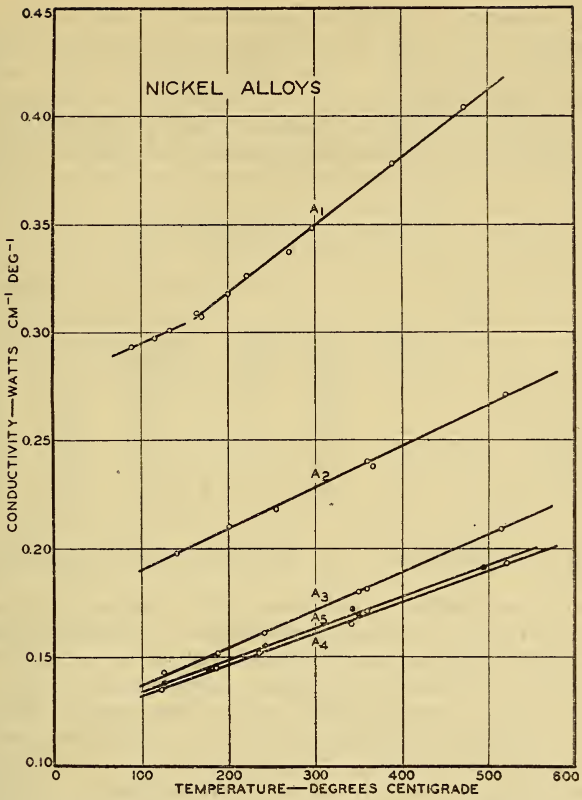

FIGURE 6.—*Results of measurements on alloys.*

TABLE 4.—*Description of test specimens*

Designation	Material	Chemical composition	Remarks
(N_1)	"High purity nickel."	Ni 99.94, C .005, S .004, Co .016, Si .006, Fe .03, Cu. 006.	Melted in vacuum furnace and furnace cooled. Large crystals. Bureau analysis.
(A_1)	"Alumel"	Ni 95%, Al 2%, Mn 2%, Si 1%.	Pouring temperature 1,540 C. Cast in 5-in. diam. ingot. Hot rolled to 1⅝-in. sq bar. Tested in "as rolled" condition. Mfgrs. analysis.
(A_2)	"Chromel P"	Ni 90%, Cr 10%	Pouring temperature 1,540 C. Cast in 5-in. diam. ingot. Hot rolled to 1⅝-in. sq bar. Tested in "as rolled" condition. Mfgrs. analysis.
(A_3)	"Chromel A"	Ni 80%, Cr 20%	Pouring temperature 1,540 C. Cast in 5-in. diam. ingot. Hot rolled to ¾-in. round bar. Tested in "as rolled" condition. Mfgrs. analysis.
(A_4)	"Chromel C"	Ni 61%, Cr 16%, Fe 23%	Pouring temperature 1,540 C. Cast in 5-in. diam. ingot. Hot rolled to ¾-in. round bar. Tested in "as rolled" condition. Mfgrs. analysis.
(A_5)	"Chromel 502"	Ni 34%, Cr 10%, Fe 56%	Pouring temperature 1,540 C. Cast in 5-in. diam. ingot. Hot rolled to ¾-in. round bar. Tested in "as rolled" condition. Mfgrs. analysis.

The conductivity of the high purity nickel was found to be considerably greater than that of commercial nickel, but the percentage variation with temperature was approximately the same. The alloys all show fairly large positive temperature coefficients of conductivity, and there is some indication of a slight change in the slope of the curve for alumel in the neighborhood of 150 C. Since alumel is 95 percent nickel, an effect of this nature is quite possible.

Table 5.—*Interpolated results*

Designation	Metal	Thermal conductivity in watts cm^{-1} deg^{-1}						
		0 C	*100 C*	*200 C*	*300 C*	*400 C*	*500 C*	*600 C*
(L.S.)	Lead	0.352	0.332	0.312	0.292			
(Z.S.)	Zinc		1.123	1.085	1.048			
(N.S.)	Nickel		.649	.599	.549	0.524	0.546	0.569
(N_1)	High purity nickel		.828	.732	.638	.593	.621	
(A_1)	"Alumel"		.296	.318	.350	.381	.412	
(A_2)	"Chromel P"		.190	.209	.228	.247	.266	
(A_3)	"Chromel A"		.136	.154	.172	.189	.206	
(A_4)	"Chromel C"		.132	.147	.161	.175	.190	
(A_5)	"Chromel 502"		.134	.149	.163	.177	.192	

VI. DISCUSSION OF SOURCES OF ERROR

It was not practicable to calibrate each thermocouple separately, since it was necessary to attach new couples to each specimen tested, and the program contemplated measurements on a large number of specimens. Several lots of wire from a single source were annealed, calibrated, and used during the course of the work. Each lot was wound on a nichrome reel about 8 inches in diameter and annealed in a furnace at about 750 C. Calibrations made from time to time on specimens from a given lot showed insignificant variations. The duration and temperature of the several annealings were not exactly the same, and the calibrations of the various lots differed from each other by a few degrees at the higher temperatures. Even if a single calibration had been used for all the lots of wire which were separately annealed, the error in temperature differences would have been almost negligible, since the differences in emf at any given temperature are nearly proportional to temperature. An error of a few degrees in mean temperature would hardly be noticed, since the change in conductivity of metals with temperature is small. The magnitude of errors due to inhomogeneity of wires can only be judged by the precision of the results obtained. Such errors would not be systematic, and some of the scatter in the results can no doubt be attributed to this cause.

The effect of heat conduction to or from the junctions along the thermocouple wires was minimized by using fine wires of low conductivity metals, by attaching the junctions in good thermal contact with the metallic specimen, and by locating a portion (about 2 cm length) of the wires adjacent to the junction in a region having nearly the same temperature as the junction. The cuts into which the thermocouple wires were peened were made as small as possible, the continuity of the bar being but very slightly impaired by this process. Distances between junctions could be measured to 1 percent or better by means of a pair of dividers and a steel scale.

As a guide in the design of apparatus, a mathematical analysis of the effect of heat transfer between specimen and guard tube was made at the outset of the work. This calculation indicated that if a nickel specimen were maintained at temperatures 1° C different from those at corresponding points on the guard tube, the maximum error due to heat leakage would be only about 0.2 percent. With any given adjustment of temperatures, the error due to heat leakage is inversely proportional to the conductivity of the specimen, so that better temperature control is required in measuring relatively poor heat conductors than is the case with relatively good conductors. Experimental tests were made with a zinc specimen, and calculated results approximately verified. No great difficulty was experienced in regulating temperatures within a few degrees, and it is therefore believed that errors greater than 1 percent were rarely introduced by heat transfer between specimen and guard tube.

On account of the large amount of insulation surrounding the apparatus, a long time was required to approximate a steady state. It was therefore desirable to ascertain how great a drift in temperature could be tolerated without causing significant error. Calculation showed that for a nickel specimen a drift of about 0.1° C per minute at the hot end, when this was at 500 C, would introduce an error of only about 0.6 percent. A steady state within this limit can be attained in a few hours.

It was found experimentally that appreciable errors were introduced by imperfect soldered joints, the resulting distortion of the lines of heat flow affecting the temperature at the thermocouple junctions nearest the joints. The possible presence of an effect of this kind is shown up by a large drop in temperature across the soldered joint. When this is found, the joint requires resoldering. A few metals on which measurements have been made, e.g., chromium steels, are rather difficult to tin, but it has always been found possible to make joints having low thermal resistance.

VII. COMPARISON WITH PREVIOUS RESULTS

The values of thermal conductivity given in this paper depend upon the value assigned to lead. A critical review of the previous work on lead indicates that the value assumed is probably correct within about 3 percent. Most of the previous results are in fairly good agreement in the region of room temperature, but depart widely at both high and low temperatures. Temperature coefficients of conductivity range from practically zero to a 0.12 percent decrease per degree C, excluding some apparently unreliable data. The value obtained in the present work (which is independent of the absolute value assumed for the conductivity of lead) is 0.057 percent decrease per degree C. Of the more recent measurements extending above 0 C, Meissner [2] observed practically no change in conductivity between 0 and 100 C, King [3] found a decrease of 0.017 percent per degree between 90 and 210 C, Konno [4] a decrease of 0.037 percent per degree from 0 to 300 C, and finally, Bidwell and Lewis [5] observed

[2] Meissner, Ann. der Phys., vol. 47, p. 1001, 1915.
[3] King, Phys. Rev., vol. 11, p. 149, 1918.
[4] Konno, Sci. Rep. Tohoku Imp. Univ. ser. 1, vol. 8, p. 169, 1919.
[5] Bidwell and Lewis, Phys. Rev. vol. 33, p. 249, 1929.

a decrease of 0.12 percent per degree between −50 and 100 C. In the neighborhood of 0 C, all these experimenters report practically the same value for the conductivity itself.

Comparison of the present results on zinc with those obtained previously does not have much significance, since Bidwell and Lewis [6] have shown that the conductivity of high purity zinc is considerably affected by the method of preparation of the specimen. So far as the change in conductivity with temperature is concerned, the results reported here are in good agreement with those of Konno,[7] and in fair agreement with those of Schofield.[8] The conductivity values obtained by Konno are consistently 2.5 percent lower than those reported here, and those of Schofield average about 6 percent lower.

Previous results on nickel are widely divergent, probably on account of the relatively large effect of small amounts of impurities on the conductivity of this metal, as well as the difficulty in the past of securing material in a high state of purity. The only published results on the thermal conductivity of nickel comparable in purity with the N_1 (high purity) nickel measured in the course of the present work are those of Sager.[9] His values are in general higher than ours, although in the region of 100 C the agreement is good. At about 175 and 300 C, Sager's results are some 5 percent higher than ours, and at 400 and 550 C approximately 20 percent higher. Other results reported in the literature are much lower, roughly comparable with our results on the (N.S.) commercial nickel, although all show a less pronounced minimum.

The only thermal conductivity data on nickel-chromium alloys which have come to our attention are those of Smith,[10] who worked with a large number of binary alloys, including 90 Ni—10 Cr and 70 Ni—30 Cr. Measurements were made at one mean temperature, approximately 50 C. The value of 0.197 watt cm^{-1} deg^{-1} was obtained for the conductivity of 90 Ni—10 Cr alloy at 56 C, which can be compared with 0.18 watt cm^{-1} deg^{-1}, obtained in the present work for chromel P, nominally of the same composition. This agreement can be considered good, in view of the fact that the curve representing conductivity as a function of composition for this series of alloys is very steep at the 90 Ni—10 Cr composition. No measurements were made by Smith on 80 Ni—20 Cr alloy (essentially chromel A), but interpolation indicates that the agreement of his results with ours is probably considerably better at this point. Smith also made measurements at about 50 C on zinc and lead of high purity, obtaining results on both in agreement with ours within about 2 percent. He apparently made no measurements on nickel, although the nickel used in the preparation of the various alloys contained only about 0.2 percent of impurities excluding cobalt.

Acknowledgment is made to the Hoskins Co., who kindly furnished the nickel alloys used in the measurements described in this paper.

WASHINGTON, February 24, 1934.

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[6] Loc. Cit. p. 439.
[7] Loc. Cit. p. 439.
[8] Schofield, Proc. Roy. Soc., London, series A, vol. 107, p. 206, 1925.
[9] Sager, Rensselaer Poly. Inst., Eng. and Science, series no. 27, 1930.
[10] Smith, Ohio State Univ. Eng. Exp. Stat. Bull. no. 31, 1925.