# NATIONAL BUREAU OF STANDARDS REPORT

8389

## THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY OF A SPECIMEN OF ARMCO IRON

by

T. W. Watson and H. E. Robinson

Report to

Headquarters U. S. Army Missile Support Command U. S. Army Missile Command Redstone Arsenal, Alabama



U. S. DEPARTMENT OF COMMERCE NATIONAL BUREAU OF STANDARDS

### THE NATIONAL BUREAU OF STANDARDS

The National Bureau of Standards is a principal focal point in the Federal Government for assuring maximum application of the physical and engineering sciences to the advancement of technology in industry and commerce. Its responsibilities include development and maintenance of the national standards of measurement, and the provisions of means for making measurements consistent with those standards; determination of physical constants and properties of materials; development of methods for testing materials, mechanisms, and structures, and making such tests as may be necessary, particularly for government agencies; cooperation in the establishment of standard practices for incorporation in codes and specifications; advisory service to government agencies on scientific and technical problems; invention and development of devices to serve special needs of the Government; assistance to industry, business, and consumers in the development and acceptance of commercial standards and simplified trade practice recommendations; administration of programs in cooperation with United States business groups and standards organizations for the development of international standards of practice; and maintenance of a clearinghouse for the collection and dissemination of scientific, technical, and engineering information. The scope of the Bureau's activities is suggested in the following listing of its four Institutes and their organizational units.

Institute for Basic Standards. Electricity. Metrology. Heat. Radiation Physics. Mechanics. Applied Mathematics. Atomic Physics. Physical Chemistry. Laboratory Astrophysics.\* Radio Standards Laboratory: Radio Standards Physics; Radio Standards Engineering.\*\* Office of Standard Reference Data.

Institute for Materials Research. Analytical Chemistry. Polymers. Metallurgy. Inorganic Materials. Reactor Radiations. Cryogenics.\*\* Office of Standard Reference Materials.

Central Radio Propagation Laboratory.\*\* Ionosphere Research and Propagation. Troposphere and Space Telecommunications. Radio Systems. Upper Atmosphere and Space Physics.

Institute for Applied Technology. Textiles and Apparel Technology Center. Building Research. Industrial Equipment. Information Technology. Performance Test Development. Instrumentation. Transport Systems. Office of Technical Services. Office of Weights and Measures. Office of Engineering Standards. Office of Industrial Services.

<sup>\*</sup> NBS Group, Joint Institute for Laboratory Astrophysics at the University of Colorado.

<sup>\*\*</sup> Located at Boulder, Colorado.

# NATIONAL BUREAU OF STANDARDS REPORT

### **NBS PROJECT**

**NBS REPORT** 

421,06-30-4210642

### 1 July 1964

8389

THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY OF A SPECIMEN OF ARMCO IRON

by

T. W. Watson and H. E. Robinson Heat Transfer Section Building Research Division Institute for Applied Technology

#### Report to

Headquarters U. S. Army Missile Support Command U. S. Army Missile Command Redstone Arsenal, Alabama

#### IMPORTANT NOTICE

NATIONAL BUREAU OF STAN for use within the Government. Be and review. For this reason, the p whole or in part, is not authorize Bureau of Standards, Washington the Report has been specifically pr

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

accounting documents intended bjected to additional evaluation sting of this Report, either in Office of the Director, National e Government agency for which es for its own use.

U. S. DEPARTMENT OF COMMERCE National Bureau of Standards

#### Thermal Conductivity and Electrical Resistivity of a Specimen of Armco Iron

by

Thomas W. Watson and Henry E. Robinson

#### 1. INTRODUCTION

This report presents results of thermal conductivity and electrical resistivity measurements in the temperature range -162° to 200° C of a sample of Armco Iron submitted by U. S. Army Missile Support Command, U. S. Army Missile Command, Redstone Arsenal, Alabama.

Also presented, as a matter of pertinent interest, are data on the thermal conductivity of another sample of Armco iron, measured here in 1961, which is designated herein as "Armco B" to distinguish it from the Redstone Arsenal specimen.

#### 2. SAMPLE

The sample submitted was a bar about one inch in diameter, which was machined to yield a test specimen having a uniform diameter of 2.54 cm and a length of 36.9 cm. The overall length of the test specimen was increased to 37.0 cm by soldering a piece of iron 0.1 cm in length to the top or cold end of the specimen.

The chemical composition of the specimen is given in Table 1, as determined from a spectrochemical analysis made by the National Bureau of Standards Spectrochemistry Section.

#### 3. TEST APPARATUS AND METHOD

The thermal conductivity of the sample was determined by means of a steady-state flow of heat longitudinally in the bar specimen, with measurements of the temperatures existing at the ends of six consecutive, approximately 3.51-cm, spans along the central length of the bar. Each determination required a pair of tests at moderately different temperature conditions, and yielded values of thermal conductivity at six different mean temperatures [1]<sup>a</sup>.

a See references in 6. REFERENCES

The test apparatus is shown schematically in figure 1.

The specimen, a bar approximately 37 cm long and of uniform external dimensions over the metering length, was supported at the top (coolant) end concentrically within a stainless steel guard tube of 0.8-cm wall thickness, which in turn was held concentrically within a cylindrical outer container. The specimen was drilled at each end with a 1.35-cm hole 5.5 cm deep. An electrical heater was inserted and secured in the hole at the bottom (hot) end by a completely-enclosing metal cap (in lieu of the strap shown in figure 1), and the supporting fixture at the top end provided a liquid-tight connection for circulating a coolant through the top drill-hole.

Temperatures along the specimen were indicated by seven thermocouples located symmetrically about the longitudinal center of the specimen, spaced approximately 3.51 cm apart, with one additional thermocouple near the bottom end of the specimen. Thermocouples were similarly located in almost exactly corresponding longitudinal positions on the guard tube.

The guard tube was equipped near its lower end with an external circumferential electric heater, as shown. The guard tube was cooled at the top by means of a copper-tube coil soldered circumferentially at a position corresponding in effect to that of the specimen coolant well. Coolant (liquid nitrogen at -196° C) was pumped through the guard coil and specimen well in series connection, as shown.

The electrical heater for the specimen consisted of 26-gage nichrome heater wire threaded back and forth through longitudinal holes in a porcelain cylinder 1.25 cm in diameter and 5.2 cm long. Its resistance at 25° C was approximately 21 chms. Current was brought to the heater through relatively large heater leads, to which separate potential leads were connected at the point where they entered the porcelain core. The heater was energized by an adjustable constant-voltage d-c source. Heater current and voltage drop measurements were made using standard resistors and the high-precision manual potentiometer used for thermobouple observations. The guard was heated with alternating current governed by a sensitive temperature controller actuated by the guard remperature at a selected position.

The thermocouples were made from calibrated chromel and alumel 20 gage wires, welded by gas-oxygen flame to form a butt joint about 0.042 cm in diameter.

The thermocouple junctions were pressed into transverse grooves 0.04 cm wide by 0.05 cm deep and 0.6 cm in length in the convex surface of the bar and tightly secured by peening the metal around the groove. The bare thermocouple leads were individually insulated electrically with high-temperature flexible sleeving, and led out into the powder insulation in the same transverse plane as the junction (one wire in each direction around the bar), forming a 2.6-cm circle. The wires were brought out through the powder insulation near the guard tube. The thermocouples in the guard tube were electrically welded to form a spherical junction about 0.10 cm in diameter. The junctions in the guard were inserted into radially-drilled holes 0.11 cm in diameter and 0.17 cm deep, and tightly secured by punch-pricking the metal around the hole. The wires were similarly brought out through the powder insulation. The longitudinal positions of the thermocouple junctions were taken as those of the centers of the grooves, or of the drilled holes, measured to the nearest 0.01 cm with a laboratory cathetometer.

Current leads (0.1 cm Pt) were attached to the two ends of the bar specimen for passing a direct current of about 8 amperes along the bar for making electrical resistivity measurements. The lead at the hot end was led in a flat spiral in the powder insulation, in a plane transverse to the bar axis, to near the inner radius of the guard tube, from which point it was electrically insulated with broken ceramic tubing and brought upwards through the powder insulation near the guard tube.

After installation of the specimen, the space between it and the guard tube was filled with diatomaceous earth powder insulation, which also was used to insulate the space surrounding the guard tube. The tests were conducted with the insulation exposed to atmospheric air.

In principle, if there were no heat exchange between the specimen and its surroundings, the conductivity could be determined from the measured power input to the specimen and the average temperature gradient for each of the six spans along the specimen, all of uniform known cross-sectional area. In practice, a perfect balance of temperatures between the bar and guard all along their lengths is not possible because of differences in their temperature coefficients of conductivity, and the effect of the outward heat losses of the guard. In addition to heat exchanges between the bar and guard from this cause, a relatively smaller longitudinal flow of heat occurs in the powder insulation surrounding the specimen, and the contribution of the specimen to this heat flow must depend somewhat on the bar-to-guard temperature unbalance.

In order to evaluate the heat flow in the bar at the center points of each of the six spans, a partly empirical procedure was used. Two steady-state test-runs were made with slightly different bar and guard temperatures and power inputs. In the two tests, the heat flow and the observed temperature drop from end to end of a given span differed, as did also the approximate integral with respect to length of the observed temperature differences between bar and guard, summed from the hot end of the bar to the span center point. It is thus possible to write for each span two equations (one for each test-run) of the form

 $\frac{Ak\Delta t}{\Delta x} + fS = Q$ 

where A is the cross-sectional area of the specimen,

- k is the specimen conductivity at the mean temperature of the span,
- At is the temperature drop from end to end of the span,
- $\Delta x$  is the length of the span,
- fS represents the total net heat loss from the bar from its bottom end at the heater to the midpoint, x, of the given span, expressed as the product of S, which is the integral
  - $\int_{0}^{x} (t_{bar} t_{guard}) dx$ , and an average heat transfer coeffi-
  - cient f for the thermal path from bar to guard,
- Q is the measured power input to the specimen heater.

The two equations written for each of the six spans of the bar can be solved simultaneously to determine k and f. For this to be strictly valid, k and f must have equal values in the two equations. Since the mean temperatures of the span in the two tests will in general differ slightly, and the conductivity of the bar may vary with temperature, a slight adjustment is made to the observed values of  $\Delta t$  so that k corresponds to the mean of the span mean temperatures in the two tests. The equality of f in the two tests is not so readily assured, but because the magnitude of fS in these tests was generally on the order of one percent of Q, a moderate difference in the values of f in the two equations would affect the solved value of Ak/ $\Delta x$  only slightly.

Electrical resistivity measurements for each span were made at the end of, but at the temperature conditions existing at, each pair of runs for determining the thermal conductivity, by passing a d-c current of about 7.7 A along the bar, and observing the potentials of the chromel and/or alumel leads of the span thermocouples, with the current direction forward and reversed. The average of the two potential drops between two adjacent alumel or chromel leads indicated the net potential drop due to the current flowing in the span, and thus enabled calculation of its resistivity. Due to a slight warming of the bar during the period of current flow, the resistivity was assigned to correspond to the time-average of the span mean temperature over this period. The same electrical and temperature measurements also enabled calculation of the thermoelectric power of the iron relative to the chromel P thermocouple wire used.

 $T \cdot e$ 

The computation of results directly from the observed data was effected by an IBM 7094 digital computer suitably programmed to compute the thermal conductivity, the electrical resistivity and the thermoelectric power, and the corresponding mean temperatures, for each of the six spans.

#### 4. RESULTS

The results of the thermal conductivity and electrical resistivity determinations are shown in figure 2 and table 2. The 12 individual values of thermal conductivity plotted represent two sets of tests each with values for the six spans. The 42 values of electrical resistivity plotted represent 18 measurements made concurrently with the thermal conductivity determinations and, in addition, 24 determinations made by taking 12 points with the specimen in ice (0° C) and 12 points with the specimen at room temperature (26° C) using either the chromel or the alumel leads. The averages of each set of 12 points are shown by the two solid circles. The solid lines of figure 2 represent the trend of the data from which the values tabulated in table 2 were taken.

The calculated values of the thermoelectric power of the Armco iron relative to the chromel P thermocouple wire used are plotted in the upper part of figure 3. Values of the Lorenz function,  $k\rho/T$ , were calculated from the values of thermal conductivity, electrical resistivity, and temperature tabulated in table 2. They are given in table 2 and represented by the smooth curve in the lower part of figure 3.

#### 5. DISCUSSION OF RESULTS

The individual values of thermal conductivity and electrical resistivity plotted in figure 2 show moderate scattering from the smooth curve. The extreme departure of an individual value of thermal conductivity from the smooth curve is less than two percent. The uncertainty in the smoothed conductivity values is believed to be not more than one percent.

As shown in figure 2, the thermal conductivity decreased with increasing temperature over the range from -160 to 200° C. The dashed line in figure 2 represents the values obtained previously at NBS on another sample of Armco iron ("Armco B"), the spectrochemical analysis of which is also given in table 1.

The obtained values of electrical resistivity are shown in the upper part of figure 2. The resistivity increased with temperature over the temperature range -160 to 200° C. The points plotted as triangles near the resistivity curve show values of resistivity reported by Roeser 1941 [2] for iron of 99.99 percent purity; the squares show values of White and Woods [3] for iron of "ideal purity."

The obtained values of thermoelectric power of the Armco iron, relative to the chromel P thermocouple wire used, are shown in figure 3, with symbols consistent with those of figure 2. The plotted triangles represent values of thermoelectric power of iron relative to chromel P derived from data on thermal emf of iron, and of chromel P, relative to platinum [4].

The lower curve of figure 3 represents the values of the Lorenz function  $(k\rho/T)$  given in table 2, derived from the thermal conductivity and electrical resistivity results. The theoretical (Sommerfeld) value 2.443 x  $10^{-8}$  V<sup>2</sup>/deg<sup>2</sup> is shown by the horizontal dashed line.

Photomicrographs of the two Armco iron specimens, as viewed at 100X, are shown in figure 4. The Redstone Arsenal specimen exhibits an oriented microstructure typical of a cold-worked material. The other specimen (Armco B), which had been annealed at 850° C for 1/2 hour prior to testing, exhibits large grains with no particular orientation. The Rockwell B hardness of the Redstone Arsenal specimen was determined as 72, while that of the Armco B specimen varied between 30 and 50.

In general, cold-working raises the electrical resistivity and lowers the thermal conductivity of a given material, the effect on thermal conductivity increasing at lower temperatures (see, for example, the work of White [5] on gold, silver, and copper). Thus, on the basis of the cold-worked state versus the annealed state only, assuming identical chemical composition, the thermal conductivity of the Redstone Arsenal specimen would be expected to be lower than that of the Armco B specimen rather than higher, as was found. The electrical resistivity of the Redstone Arsenal specimen was significantly lower than that of the other specimen (which had an ice point resistivity of 9.88  $\mu\Omega$  cm as compared to 9.36  $\mu\Omega$  cm for the Redstone Arsenal specimen), implying greater purity of the Redstone Arsenal specimen, and confirming the finding of a higher thermal conductivity for it than for the Armco B specimen.

In a recent paper, Godfrey <u>et al</u>. [6] report (pages 26-29) a quantitative chemical analysis showed the presence of 0.086 percent  $O_2$ , 0.023 percent S, and 0.013 percent C (all weight percentages) in an Armco iron sample which they investigated. They report the presence, in this sample, of about 0.9 volume percent of a second phase, presumed to consist of oxides, sulfides, and phosphides. On a sample of Armco iron from the same stock as that from which the Armco B specimen tested at NBS was obtained, they found 1.3 volume percent of a second phase. The amount of non-metallic impurities in a sample of Armco iron and, as pointed out by Godfrey <u>et al</u>. (page 40), also the physical state of the impurities, may be of quite significant importance as regards the thermal and electrical conductivities. Impurities in solution would be expected to have a much greater effect on thermal and electrical conductivity than would impurities present as a dispersed second phase.

Significant differences in purity between the Redstone Arsenal specimen and the Armco B specimen could only be determined by a more complete chemical analysis of both specimens, including quantitative analysis for non-metallic impurities. A detailed microstructural analysis would also be required to determine the physical state of the impurities present. It is interesting whether the orientation of the microstructure in the cold-worked specimen would result in an anisotropicity in the electrical and thermal conductivities of the metal in its present state.

#### 6. REFERENCES

- T. W. Watson and H. E. Robinson, Thermal conductivity of some commercial iron-nickel alloys, Trans. ASME Jour. of Heat Transfer, 83C, 403 (1961).
- [2] W. F. Roeser, Temperature, Its Measurement and Control in Science and Industry, p. 1312 (Reinhold Publishing Corp., New York, N. Y., 1941).
- [3] G. K. White and S. B. Woods, Electrical and thermal resistivity of the transition elements at low temperature, Phil. Trans. Roy. Soc. <u>251A</u>, 273 (1959).
- [4] American Institute of Physics Handbook, p. 4-8, 9 (McGraw-Hill Book Co., Inc., 1963, Second Edition).
- [5] G. K. White, The thermal conductivity of gold at low temperatures, Proc. Phys. Soc. (London) A66, 559 (1953).

G. K. White, The thermal conductivity of silver at low temperatures, Proc. Phys. Soc. (London) <u>A66</u>, 844 (1953).

G. K. White, The thermal and electrical conductivity of copper at low temperatures, Aust. J. Physics <u>6</u>, 397 (1953).

[6] T. G. Godfrey, W. Fulkerson, T. G. Kollie, J. P. Moore, and D. L. McElroy, Thermal conductivity of uranium dioxide and Armco iron by an improved radial heat flow technique, ORNL-3556 (June 1964).

## TABLE 1

					Present Sam	p1	e					Armco B
Mn	•	٠			< 0.1	٥		e			ø	< 0.1
Si		0	9	e	< 0.015		٠	٠	e	÷	÷	< 0.015
Cu		•		0	0.03		÷	•		0		0.06
Ni	٠	•	•	ę	0.04			ø		•	•	0.06
Cr	÷	0		Ģ	0.01		٠		٠		0	0.01
V	٠		٥	0	< 0.01	æ	0	9	٠		٠	< 0.01
Мо	٠	ø		9	0.01	•	•	•	•	•		0.01
W	•	•		0	< 0.02	٠	٠	٠	•	٠		< 0.02
Со		٠	٠		< 0.01	•	٠	٠	0	٠	۰	< 0.01
Ti		٠	ø	٠	0.006	•	0	٠	0		٠	0.006
Sr	٠	٠	٠		0.02	•	•	٠	٠	٠		0.02
Nb	٠	6	٠	٠	< 0.01						۰	< 0.01
Zr	٥	ø	0	٠	< 0.003		٠		٥	0	e	< 0.003
Fe*	6	ø		٠	99.80 ± 0.08	34			0			99.75 ± 0.084

# Chemical Composition -- Percent

\*By difference

## TABLE 2

# Thermal Conductivity and Electrical Resistivity

## of a

## Sample of Armco Iron

Temperature, t					Thermal nductivity		Re	Electrical sistivity	L , ,		Lorenz $\begin{pmatrix} k\rho \\ T \end{pmatrix}$			
°C		W/cm deg					anaigeo	μΩ cm		V <sup>2</sup> /deg <sup>2</sup>				
- 162	٠	٠	٠	٠	0.920	٠	٠	٠	٠	2.48	•	•	•	2.06 x 10 <sup>-8</sup>
- 150		ø	٠	٠	. 903	•	٠	•	٠	2.87	•	•	٠	2.11
···· 100	٠	•	•	٠	.844	٠	٠	٠	0	4.71	٠	٠	0	2.30
50	ø	ø		٠	.795	٠	•	٠		6.87	٠	•		2.45
0	9	٠	•	٠	.754	٠	•	•	٠	9.36	٠	٠	٠	2.59
50	٠		v	٠	.720	٠	•	•		12.17	•	•	•	2.71
100	Ð	•	e	÷	.687		•	•		15,30	•	٠	•	2.82
150	ø	•	•	0	.655	ø		•	٠	18.76	٠		٠	2.90
200		ø			.620	0		•		22.54	•	٠		2.95



APPARATUS FOR MEASURING THE THERMAL CONDUCTIVITY OF METALS



FIGURE 2. THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY OF ARMCO IRON SPECIMEN VERSUS TEMPERATURE





<b>A</b> 0		



SAMPLE A

SAMPLE B

FIGURE 4. MICROSTRUCTURES OF TWO DIFFERENT SAMPLES OF ARMCO IRON.

- A. REDSTONE ARSENAL SAMPLE, x 100.
- B. ARMCO B SAMPLE, x 100.





- - 4