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Standard Reference Materials:

THERMAL CONDUCTIVITY OF AUSTENITIC STAINLESS STEEL, SRM 735, FROM 5 to 280 K

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Standard Reference Materials:

Thermal Conductivity of Austenitic Stainless Steel, SRM 735, from 5 to 280 K

J. G. Hust and L. L. Sparks

Institute for Basic Standards
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Boulder, Colorado 80302

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THERMAL CONDUCTIVITY OF AUSTENITIC STAINLESS STEEL, SRM 735, FROM 5 TO 280 K*

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Thermal conductivity data are presented for a well-characterized austenitic stainless steel. Thermal conductivity and electrical resistivity measurements were conducted on two lots of this steel. Electrical resistivity measurements were performed on the second lot both before and after the material was hot-swaged and reannealed to a size 1/10 the original diameter. These measurements indicate that this steel can be swaged and reannealed without an appreciable change in thermal conductivity. Electrical resistivity measurements as well as direct thermal conductivity measurements on several specimens from both lots indicate a material variability in these lots of less than 1% in thermal conductivity.

Key words: Cryogenics; electrical resistivity; stainless steel; Standard Reference Material; thermal conductivity.

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*This work was carried out at the National Bureau of Standards, Boulder, Colorado, under the sponsorship of NASA-Space Nuclear Systems Office, Cleveland, Ohio, and the National Bureau of Standards Office of Standard Reference Materials (NBS-OSRM), Washington, D. C.

1. INTRODUCTION

This report is a result of a program to establish several thermal conductivity Standard Reference Materials (SRM's). Measurements are planned for SRM's of high, medium and low conductivity. An earlier report [1] describes the establishment of SRM 734 material as a medium-high thermal conductivity SRM. The material reported on here, stainless steel SRM 735* [2], is in the low conductivity range.

Design and development engineers in the aerospace industry continue to have urgent need for thermal and mechanical property data for new materials. For most materials, especially new or uncommon alloys, measured values of thermal conductivity are not available and predictions cannot be made with adequate confidence. To help satisfy these needs, we have constructed an apparatus for the simultaneous measurement of thermal conductivity, electrical resistivity, and thermopower. We intend to measure several specimens of materials that appear to be useful as standards. SRM data are useful for intercomparison of existing thermal conductivity apparatus, for debugging new apparatus, and for calibration of comparative apparatus. The apparent large differences (50% is not uncommon) among the results of various investigators for a given material is evidence of the need for reliable thermal conductivity SRM's.

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* This steel was originally prepared and distributed for use as a Standard Reference Material for high-temperature thermophysical properties by the Advisory Group for Aerospace Research and Development (AGARD) under the direction of NATO. It was measured at this laboratory to establish a standard at low temperatures as well. Temperatures from 5 to 280 K are included in this work. Later, as data from other laboratorys are reported, it will be possible to extend the range to higher temperatures.

The availability of SRM's will result in more accurate and more permanent transport property data for technically important solids.

The basic characteristics of a thermal conductivity SRM are that it be: (a) stable and reproducible under the conditions of use, (b) uniform throughout a single specimen and from specimen-to-specimen, (c) similar in thermal conductivity to that of the materials to which it will be applied, (d) readily machined and fabricated to appropriate size and shape, (e) chemically inert with its environment, and (f) usable over a wide temperature range. Stainless steel SRM 735 satisfies these criteria reasonably well.

2. APPARATUS AND DATA ANALYSIS

The apparatus is based on the axial one-dimensional heat flow method. The specimen is a cylindrical rod 11.3 mm in diameter and 23 cm long with an electric heater at one end and a temperature controlled heat sink at the other. The specimen is surrounded by glass fiber and a temperature controlled shield. Eight thermocouples are mounted at equally spaced points along the length of the specimen to determine temperature gradients in the range 4 to 300 K.

The experimental data are represented by arbitrary functions over the entire range and smooth tables are generated from these functions. The number of terms used to represent each of the data sets is optimized, through the use of orthonormal functions, so that none of the precision of the data is lost by underfitting, nor are any unnecessary oscillations introduced by overfitting. A detailed description of this apparatus and the methods of data anlaysis are given by Hust, et al. [3].

3. SPECIMEN CHARACTERIZATION

Initially, AGARD supplied four steel rods for characterization and measurement. Two rods were 6 mm diameter and the other two were 10 mm diameter. The smaller rods were used for electrical resistivity measurements while the larger two were machined for thermal conductivity measurements. These rods are referred to as lot 1 and measurements on them suggested that this steel would be a useful standard of thermal conductivity. A second lot of this material was purchased for further measurements and for stocking. The 12 rods from lot 2 were each 35 mm in diameter and 1 meter in length. Six five-cm-long pieces were cut from the ends of six of these rods for electrical resistivity characterization. These measurements showed that lot 2 is indistinguishable, within 1%, from lot 1.

Preparation of specimens by NBS-OSRM was then begun. Because of high specimen preparation costs and the excessive waste that would result from machining the 35-mm rods to smaller size, we decided to hot swage the rods to the desired diameters. However, this raised the question of whether the specimens would remain unchanged after hot swaging and reannealing. We performed additional characterization measurements on six specimens prepared by this method. The results, listed in table 1, show that specimens prepared by this procedure are within 1% in resistivity of the specimens from lots 1 and 2 as received. Based on a total of 21 elctrical resistivity determinations, it is concluded that the thermal conductivity variability of specimens from these lots is less than 1%. A discussion of the connection between electrical resistivity and thermal conductivity variability is presented in Appendix I.

The nominal composition of stainless steel SRM 735 is as follows: (The composition of lot 1 as determined by the supplier is given in parentheses)

Element	Percent, by weight	
Ni	20.0-20.5	(19.90)
Cr	16.0-16.5	(16.41)
Mn	1.0- 1.2	(1.20)
Si	0.2- 0.3	(0.27)
Nb	8-12 x C	(0.10)
Mo	<0.2	(0.01)
C	<0.02	(0.009)
N	<0.01	(0.009)
P	<0.015	(0.005)
S	<0.015	(0.006)
Fe	bal	(bal)

4. RESULTS

The thermal conductivity of three specimens, two from lot 1 and one from lot 2, was measured from 5 to 280 K. These data were functionally represented with the following equation:

$$\ell n\lambda = \sum_{i=1}^{n} a_i [\ell nT]^{i+1} \tag{4}$$

where λ = thermal conductivity in $Wm^{-1}K^{-1}$ and T = temperature in K. Temperatures are based on the IPTS-68 scale above 20 K and the NBS P2-20 (1965) scale below 20 K. The parameters, a_i, determined by least squares for the mean conductivity of these three specimens, are presented in table 2. Further details of the method of data analysis are given by Hust, et al. [3]. The deviations of the experimental data from this equation are given in figures 1a and 1b. Calculated values of thermal conductivity are presented in table 3 and figure 2.

A detailed error analysis for this system has been presented previously by Hust, et al. [3]. Based on this analysis of systematic and random errors, the uncertainty estimates (with 95% confidence) are as follows:

> 2.5% at 300 K, decreasing as T^4 to 0.70% at 200 K, 0.70% from 200 K to 50 K, increasing inversely with temperature to 1.5% at 4 K.

5. SUMMARY

We have established low temperature thermal conductivity standard reference data for stainless steel SRM 735. Thermal conductivity measurements have been made on this steel from 4 to 300 K. These data were fitted to an empirical equation that was used to generate tabular values. Material variability is estimated to be less than $\pm$ 1% in thermal conductivity, and measurement uncertainty is less than 2.5%.

We wish to thank R. E. Michaelis of NBS-OSRM, for assistance in specimens procurement and helpful discussions. This measurement program has been carried out under the helpful guidance of R. L. Powell.

6. FOOTNOTES AND REFERENCES

[1] Hust, J. G., Sparks, L. L., Thermal Conductivity of Electrolytic Iron, SRM 734, from 4 to 300 K, Nat. Bur. of Stand. Spec. Pub. 260-31, 19 p. (Nov. 1971).

[2] This SRM is available in the form of rods of three diameters and may be ordered from the Office of Standard Reference Materials, National Bureau of Standards, Washington, D. C. 20234. SRM 735-S is 0.65 cm in diameter and 30 cm long. SRM's 735-M1 and M2 are 1.25 cm in diameter and 15 and 30 cm long, respectively. SRM's 735-L1 and L2 are 3.5 cm in diameter and 5 and 10 cm long, respectively.

[3] Hust, J. G. Powell, R. L., and Weitzel, D. H., Thermal Conductivity Standard Reference Materials from 4 to 300 K. I. Armco Iron: Including Apparatus Description and Error Analysis, J. Res. Nat. Bur. Stand. (U.S.), <u>74A</u> (Phys. and Chem.) No. 5, 673-690 (1970).

Appendix I. Discussion of electrical resistivity and thermal conductivity variability.

The electrical resistivity, ρ, and thermal conductivity, λ, of metals are intimately related, especially for pure metals, but also for alloys to a lesser extent. This relationship exists because in a metal most of the heat is transported by the electrons. Some heat is also transported by the lattice vibrations. The total thermal conductivity is the sum of the electronic, λ_e, and the lattice, λ_g, (the German word for lattice is Gitter) components.

$$\lambda = \lambda_e + \lambda_g. \tag{1}$$

In most pure metals λ_g is small compared to λ_e, but in transition metals λ_g may be as large as 20% of λ_e, and in some alloys λ_g is much larger than λ_e. For pure metals and dilute alloys, the relationship between ρ and λ at both high and low temperatures is reasonably well described by the Wiedemann-Franz-Lorenz (WFL) law:

$$\frac{\rho\lambda}{T} = L_o = 2.443 \times 10^{-8}\ V^2K^{-2}, \tag{2}$$

where L_o is the Sommerfeld value of $\rho\lambda/T$ and T is the temperature. At intermediate temperatures, large deviations from the WFL law are observed. For our purposes the ice point is a sufficiently high temperature and liquid helium is a sufficiently low temperature to satisfy the WFL law. In complex alloys such as this steel eq (2) does not hold, but it has been observed that the value of the Lorenz function, $\rho\lambda/T$, is reasonably independent of material within a given class of alloys such as austenitic stainless steel (Fe-18Cr-8Ni alloys). This indicates a close but unexplained relationship between ρ and λ even λ_e is small compared to λ_g.

In metals there are two mechanisms that account for most of the scattering of electrons: the interaction of electrons with chemical impurities and physical imperfections, and the interaction of electrons with thermal vibrations of the atoms of the lattice. The former mechanism is usually taken to be independent of temperature while the latter is temperature dependent. If we assume that each of these mechanisms is independent of the other, we may assign a separate resistivity to each. The resistivity arising from impurity and imperfection scattering is usually referred to as the residual resistivity, ρ_o, while the resistivity due to thermal scattering is called the intrinsic resistivity, $\rho_i(T)$. The total resistivity, $\rho(T)$, may be written as the sum of these two terms.

$$\rho(T) = \rho_o + \rho_i(T). \qquad (3)$$

This separation of the total resistivity into a constant term (ρ_o) and a temperature dependent term ($\rho_i(T)$) is known as Matthiessen's rule. Although Matthiessen's rule is not strictly valid, it is a sufficiently good approximation for our purposes. For steels, the residual resistivity is a significant part of the total resistivity, even at room temperature; thus, values of either room temperature resistivity or residual resistivity can be used as indicators of material variability. This differs from pure metals in that ρ_o is much smaller than $\rho(293\ K)$ for pure metals; therefore, $\rho(293\ K)$ is not an indicator of purity. The variability in resistivity for various specimens in a given lot of material is an indication of the variability in chemical composition and physical imperfection concentration in the lot. These material variations also cause thermal conductivity variations as indicated by the Lorenz function for a given class of alloys. Therfore, a determination of resistivity variability will usually approximate the thermal conductivity variability in alloys. The determination of electrical resistivity is considerably easier than the determination of λ.

Table 1. Characterization data of SRM 735 stainless steel.

Specimen	Lot 1	Lot 2	Lot 2*
$\rho_{273\ K}$, (nΩm) ±2	788	786	788
$\rho_{4\ K}$, (nΩm) ±2	596	590	590
RRR = $\rho_{273\ K}/\rho_{4\ K}$ ±0.002	1.322	1.331	1.334
hardness, (Rockwell) ±2	B45	B48	B46
grain size, (mm) ±0.01	0.04	0.04	0.07
density, (g/cm^3) ±0.004	8.004	8.009	8.006

*after hot-swaging and reannealing.

Table 2. Parameters, a_i, of equation (4).

i	a_i
1	-4.85984600
2	6.59025067
3	-3.74701178
4	1.16265324
5	$-2.05457295 \times 10^{-1}$
6	$1.93981539 \times 10^{-2}$
7	$-7.59098428 \times 10^{-4}$

Table 3. Thermal conductivity of austenitic stainless steel (SRM 735).

Temperature (K)	Thermal Conductivity ($Wm^{-1}\ K^{-1}$)	Temperature (K)	Thermal Conductivity ($Wm^{-1}\ K^{-1}$)
5	0.466	75	7.97
6	0.565	80	8.27
7	0.676	85	8.55
8	0.796	90	8.80
9	0.921	95	9.04
10	1.05	100	9.25
12	1.32	110	9.65
14	1.58	120	9.99
16	1.86	130	10.3
18	2.13	140	10.6
20	2.40	150	10.9
25	3.07	160	11.1
30	3.72	170	11.4
35	4.34	180	11.6
40	4.92	190	11.9
45	5.47	200	12.1
50	5.98		
55	6.45	220	12.6
60	6.88	240	13.0
65	7.28	260	13.4
70	7.64	280	13.8

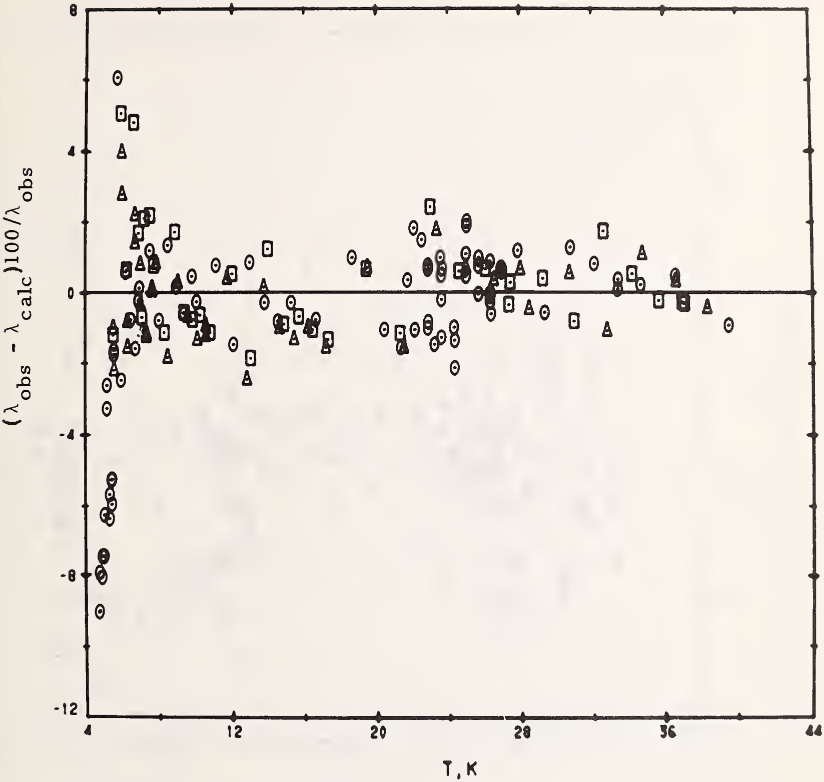

Figure 1a. Experimental thermal conductivity deviations from mean values of three specimens from 5 to 40 K.

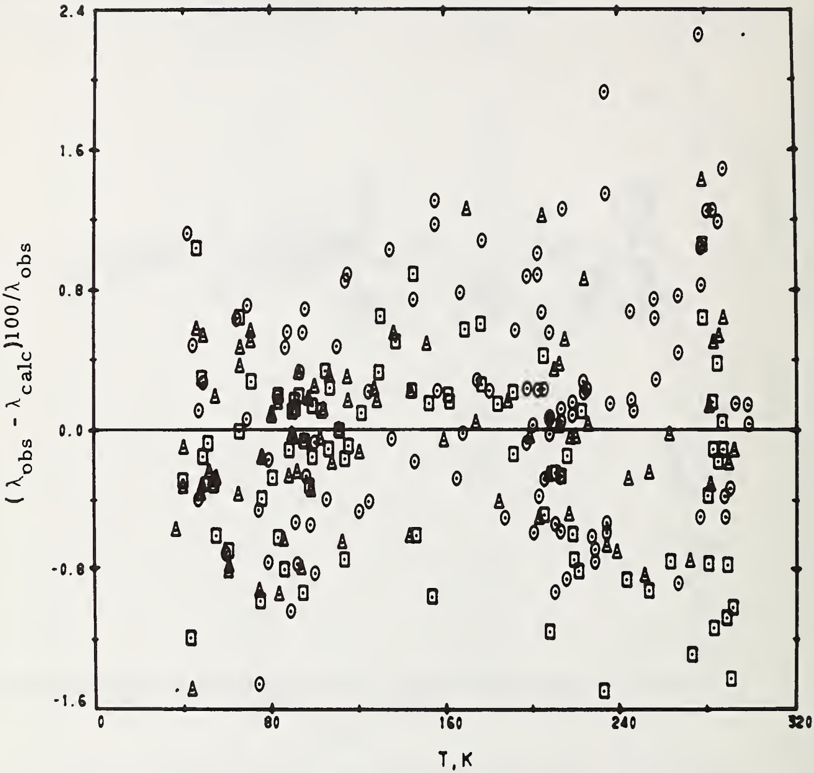

Figure 1b. Expermental thermal conductivity deviations from mean values of three specimens from 40 to 300 K.

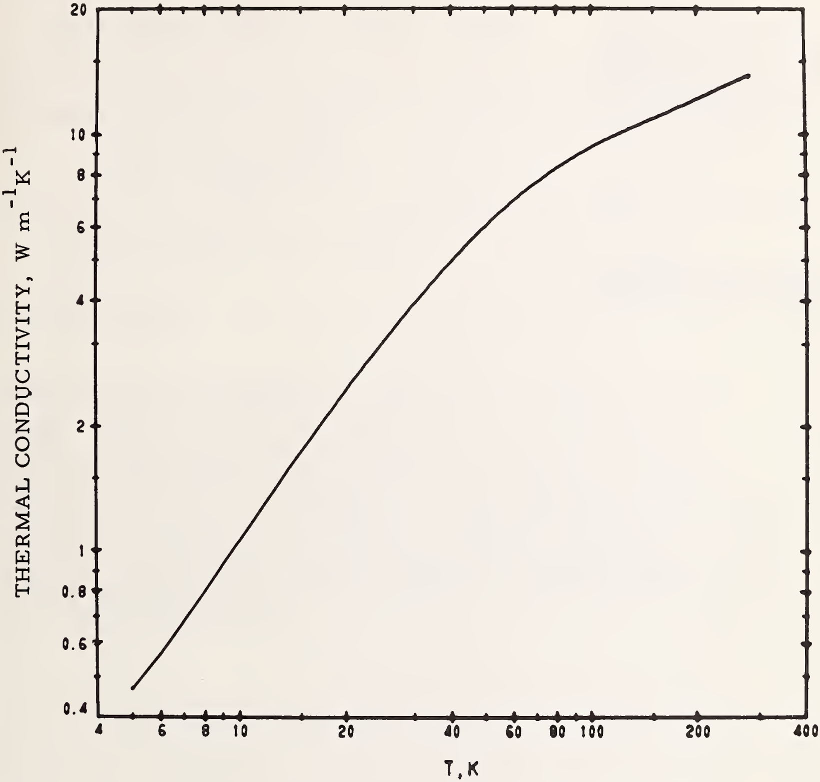

Figure 2. Thermal conductivity of SRM 735

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16. ABSTRACT (A 200-word or less factual summary of most significant information. If document includes a significant bibliography or literature survey, mention it here.)

Thermal conductivity data are presented for a well-characterized austenitic stainless steel. Thermal conductivity and electrical resistivity measurements were conducted on two lots of this steel. Electrical resistivity measurements were performed on the second lot both before and after the material was hot-swaged and reannealed to a size 1/10 the original diameter. These measurements indicate that this steel can be swaged and reannealed without an appreciable change in thermal conductivity. Electrical resistivity measurements as well as direct thermal conductivity measurements on several specimens from both lots indicate a material variability in these lots of less than 1% in thermal conductivity.

17. KEY WORDS (Alphabetical order, separated by semicolons) Cryogenics; electrical resistivity; stainless steel; Standard Reference Material; thermal conductivity.

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