NBS SPECIAL PUBLICATION 260-52

U.S. DEPARTMENT OF COMMERCE / National Bureau of Standards

Standard Reference Materials:

THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY STANDARD REFERENCE MATERIALS: TUNGSTEN SRM'S 730 AND 799, FROM 4 TO 3000 K

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Standard Reference Materials:

Thermal Conductivity and Electrical Resistivity Standard Reference Materials: Tungsten SRM's 730 and 799, from 4 to 3000K [SP no. 260-52]

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PREFACE

Standard Reference Materials (SRM's) as defined by the National Bureau of Standards are "well-characterized materials, produced in quantity, that calibrate a measurement system to assure compatibility of measurement in the nation." SRM's are widely used as primary standards in many diverse fields in science, industry, and technology, both within the United States and throughout the world. In many industries traceability of their quality control process to the national measurement system is carried out through the mechanism and use of SRM's. For many of the nation's scientists and technologists it is therefore of more than passing interest to know the details of the measurements made at NBS in arriving at the certified values of the SRM's produced. An NBS series of papers, of which this publication is a member, called the NBS Special Publication - 260 Series is reserved for this purpose.

This 260 Series is dedicated to the dissemination of information on all phases of the preparation, measurement, and certification of NBS-SRM's. In general, much more detail will be found in these papers than is generally allowed, or desirable, in scientific journal articles. This enables the user to assess the validity and accuracy of the measurement processes employed, to judge the statistical analysis, and to learn details of techniques and methods utilized for work entailing the greatest care and accuracy. It is also hoped that these papers will provide sufficient additional information not found on the certificate so that new applications in diverse fields not foreseen at the time the SRM was originally issued will be sought and found.

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THERMAL CONDUCTIVITY AND ELECTRICAL RESISTIVITY STANDARD REFERENCE MATERIALS: TUNGSTEN

SRM's 730 and 799, from 4 to 300 K

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Abstract

A historical review of the development of thermophysical Standard Reference Materials, SRM's, is given and selection criteria of SRM's are listed. Thermal conductivity and electrical resistivity data for arc cast and sintered tungsten are compiled, analyzed, and correlated. Recommended values of thermal conductivity (SRM 730) and electrical resistivity (SRM 799) for these lots of tungsten are presented for the range 4 to 3000 K. These values are based on low temperature NBS measurements and higher temperature measurements by participants of the AFML-AGARD project. The uncertainty of the thermal conductivity values below ambient is 2% and rises to 5% between ambient and 2000 K. Above 2000 K the uncertainty rises to a maximum of about 8%. The uncertainty of the electrical resistivity values is 2% over the entire temperature range.

Key words: Electrical resistivity; high temperature; Lorenz ratio; low temperature; Standard Reference Material; thermal conductivity; thermopower; tungsten.

1. Introduction

Design and development engineers continually demand thermal and electrical property data of technically important materials. Often these data are not in the published literature and immediate measurements must be performed. Since only a handful of laboratories have the proven expertise to make such measurements, they are often performed by inexperienced personnel using unproven apparatus. The results, as can be seen from the literature, exhibit excessive scatter; 50% differences are commonplace. In such situations, Standard Reference Materials, SRM's, are invaluable to predetermine the accuracy of the engineering measurements. Currently, an inaccuracy of 10% is allowable for most engineering thermal property data, and therefore, SRM's for engineering applications need to be established with an uncertainty no larger than about 5%.

A few research laboratories performing thermal and electrical measurements are obtaining data with uncertainties at the state-of-the-art level, 1% for thermal conductivity and lower for electrical resistivity. SRM's for use at such laboratories must be correspondingly more accurate, and though they may indeed be possible, they have not yet been established.

Considerable effort has been directed toward the development of suitable thermophysical SRM's*, over a period of many years, with limited success. This lack of success may be due, in part, to the tacit assumption that SRM data must be accurate to state-of-the-measurement-art to be useful. There are several reasons why the achievement of thermal and electrical property SRM's with certified inaccuracies of less than 1% is extremely difficult. The principal reason is that material variability, generally, causes property variations of greater than 1%, even with the most up-to-date production control techniques. The effects of material variability lead to the consideration of three categories of calibration materials and three concomitant certification inaccuracies: (1) A characterized type of material, e.g., copper, gold, iron, etc. Based on past experience it appears

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* The term SRM is used here in a broad sense to denote any material or specimen that is to serve as a calibration standard. The term, as coined by the Office of Standard Reference Materials, generally implies a specific lot of material prepared under strict control and subsequently characterized for chemical composition and homogeneity.

that inaccuracies of 5-10% can be expected. (2) A characterized specific lot of a given type of material, e.g., austenitic stainless steel, SRM 735, or electrolytic iron, SRM 734. Data uncertainties of one percent appear to be near the lower limit of current production control techniques. (3) Characterized specimens of material. At first glance, it may be thought that the latter SRM's would be invariant; but it is known that the thermal and electrical properties of specimens are dependent on their thermal and mechanical histories, and some specimens change spontaneously with time at ambient temperature. These effects are most important at low temperatures, especially for highly purified materials. Appropriately chosen well-characterized specimens, handled with care to avoid physical and chemical changes, and frequently reexamined to detect changes, presently represent the only means to achieve accuracies in the state-of-the-measurement-art range. This is the basis of round-robin measurements used by standardizing laboratories for state-of-the-art apparatus intercomparisons (see, for example, Laubitz and McElroy [1]). Category (2) is considered to be the most cost-effective to satisfy engineering needs and, to a lesser extent, the needs of standards laboratories. It is also the philosophical basis of the Office of Standard Reference Materials, National Bureau of Standards.

This report is a result of a program to establish several thermal and electrical conductivity metal SRM's with conductivities ranging from pure metals (high conductivity) to structural materials (low conductivity). Plans are being formulated to extend this program to insulating materials and dielectric solids. The current effort has resulted in two additional reports: one on iron (medium-to-high conductivity, 4 to 1000 K) and another on austenitic stainless steel (low conductivity, 4 to 1200 K). The material reported on here, tungsten, is in the high conductivity range.

This paper reviews the historical development of thermal conductivity SRM's. A listing is given of selection criteria for SRM's and a justification is presented for the establishment of both engineering and standards laboratory SRM's. Data are compiled and best values are selected to establish two lots of tungsten as electrical resistivity and thermal conductivity SRM's, 799 and 730, respectively. As discussed later, thermal conductivity and electrical resistivity data have been obtained to certify these SRM's over the range 4 to 3000 K to within engineering accuracy. This material appears to have the qualities of a useful SRM. An adequate supply of this material exists to insure measurement compatibility among laboratories for about ten years.

The following historical review of SRM efforts is presented to indicate the relatively large amount of research that has been conducted, compared to the few thermophysical SRM's that have been established. It is this divergence between expended efforts and concrete results that has prompted us to establish potentially useful SRM's, at what may seem to some as a premature phase of the work. Based on past experience, it appears that if this is not done, a vast amount of research is essentially wasted because the stock of material, on which the research was performed, is generally no longer available. This consideration also points out the significance of continuity in SRM projects.

2. Historical Review

2.1 Early Efforts

Thermophysical property reference material investigations began, for all practical purposes, in the 1930's with the work of R. W. Powell at the National Physical Laboratory (NPL), Teddington, England [2] on iron and Van Dusen and Shelton at NBS [3] on lead. These efforts were successful in that they resulted in frequently used reference materials of thermal conductivity. Powell's work resulted in the establishment of ingot iron* (category 1) as a standard which is still being used today. Lucks [4] recently reviewed the massive amount of work which has been done on this material and recommends the continued use of ingot iron as a reference material. Van Dusen and Shelton's work resulted in an unofficial lead standard based on a well-characterized lot of pure lead (category 2) distributed by NBS as a freezing point standard.

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* The ingot iron used for this purpose is Armco iron produced by Armco Steel Corporation. The use of trade names of specific products is essential to the proper understanding of the work presented. Their use in no way implies any approval, endorsement, or recommendations by NBS.

2.2 Iron

Since the 1930's, reference material investigations have been sporadic with notable efforts by researchers from the NBS (National Bureau of Standards, U.S.), NPL (National Physical Laboratory, England), ORNL (Oak Ridge National Laboratory, Tennessee), BMI (Battelle Memorial Institute, Ohio), and AFML (Air Force Materials Laboratory, Ohio). The material that has been the subject of the most extensive investigations is ingot iron.

Renewed interest in this material was spurred by the round-robin† experiments initiated by C. F. Lucks of Battelle Memorial Institute during 1959. Twenty-four laboratories requested and received the round-robin material for measurements. Data from eight laboratories were ultimately reported and compiled by Lucks [4]. These data are on specimens obtained from a single lot of ingot iron. The literature, however, contains data on a total of eleven distinct lots of ingot iron. Lucks [4] has shown that ingot iron is an acceptable reference material at temperatures from about 100 K to 1000 K. In this range, material variability affects thermal conductivity and electrical resistivity by about 5%. At higher temperatures, reported variations increase. At lower temperatures, especially at liquid helium temperatures, variations of 10% have been reported on a single 30 cm long rod by Hust et al. [5,6]. Electrolytic iron, SRM 734, was established as a low-temperature standard by Hust and Sparks [7] because it exhibits relatively small low-temperature variability. Based on their high temperature study of ingot iron and a high purity iron, Fulkerson et al. [8] also concluded that high purity iron is a more homogeneous and stable SRM than ingot iron. The thermal conductivity and electrical resistivity SRM's of electrolytic iron have been extended to 1000 K by Hust and Giarratano (NBS Special Publication 260-50).

2.3 NBS, Washington Efforts

D. R. Flynn of NBS, Washington began a study of potential thermal conductivity SRM's during the early 1960's. He examined several ceramics* and metals††. None of these materials has achieved the status of an SRM. Descriptions of these efforts appear in the unpublished proceedings of the early thermal conductivity conferences. Laubitz and Cotnam [9] reported that Inconel 702 exhibits transformation effects of several percent in thermal conductivity and recommended against its use as a reference material.

At the 1963 Thermal Conductivity Conference, Robinson and Flynn [10] presented the results of a survey of thermal conductivity SRM needs. SRM's with a data uncertainty of 3-5% were in greatest demand. The intended use of SRM's, most often stated, was to check and calibrate apparatus. Needs were indicated for SRM's of conductivities from 0.01 W/mK to 500 W/mK at temperatures from 4 to 3300 K.

2.4 NBS, Boulder Efforts

R. L. Powell of NBS, Boulder initiated a low-temperature SRM project during the early 1960's. This project has been continued by the first author since that time. Materials studied include ingot iron, electrolytic iron, gold, tungsten, graphite, and stainless steel. As a result of these studies, electrolytic iron and stainless steel have been established as SRM's of electrical resistivity and thermal conductivity. Current efforts are directed toward establishing graphite as SRM's at temperatures up to near 3000 K. It is anticipated that this project will continue until a sufficiently wide range of conductivities and temperatures are included to satisfy existing demands for thermophysical SRM's.

2.5 AFML-AGARD Project

Minges [5th Thermal Conductivity Conference, 1965] reported on the initiation of an AFML sponsored high-temperature reference materials program. This program was divided into two phases. Phase I included the preliminary selection and characterization of materials as potential reference materials. Selection criteria were established, dozens

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† The use of the term "round-robin" is different here from that used earlier where the use of a single specimen was implied; however, this double meaning is allowed to be consistent with the literature on ingot iron.

* Pyroceram 9606 and Pyrex 7740 (trade names of Corning Glass Works).

†† Inconel 702 (trade name of International Nickel Company, Inc.), lead, and 60% platinum - 40% rhodium alloy.

of materials screened, and about 15 were chosen for experimental evaluation. Phase II included further measurements on those materials selected from Phase I studies. Arthur D. Little Corp. contracted with AFML to perform this study. The results were reported in reference [11]. The materials of particular interest in Phase II of this program were aluminum oxide, thorium oxide, tungsten, and graphite.

After partial completion of the AFML program, an international program, principally high-temperature, was initiated under the auspices of the Advisory Group for Aerospace Research and Development, NATO (AGARD). E. Fitzer of Karlsruhe University, Germany, directed this program in close cooperation with the AFML program. The establishment, progress, and results of this program are described in a series of reports by Fitzer [12]. Minges has also summarized some of the results on AFML-AGARD programs [13]. The materials, internationally distributed and measured by numerous laboratories, are: platinum, gold, copper, austenitic steel alloy, tungsten (both sintered and arc-cast), tantalum - 10% tungsten alloy, alumina, and graphite.

3. SRM Selection Criteria

The criteria for screening and selecting potentially useful materials for thermophysical property SRM's are generally well-understood and accepted. These criteria are not met absolutely by any material, but serve as a guide to determine which materials are most suitable. Some of the more significant factors are:

1. The material should be homogeneous* and isotropic throughout a lot. The lot should be large enough to be adequate for at least a decade and renewable with a minimum of effort.

2. Thermophysical properties should not vary with time and should be relatively unaffected by the environment of the measurement apparatus. The material should have chemical stability, thermal shock resistance, low vapor pressure, and insensitivity to stress.

3. The material should be readily available, machinable, relatively inexpensive, and have sufficient strength to be handled without causing damage.

4. The material should have characteristics similar to the material to be measured.

5. The material should be useful over a wide temperature range.

The two lots of tungsten described in this report satisfy these criteria reasonably well above 90 K. Below 90 K, individual specimen characterization is required.

4. Material Characterization

Extensive reliance is placed on electrical resistivity variability as an indicator of thermal conductivity variability for pure metals. The justification for this is presented below.

The electrical resistivity, ρ, and thermal conductivity, λ, are intimately related for pure metals and 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:

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* The term homogeneous refers here to the uniformity of the thermophysical property in question. Homogeneity of a thermophysical SRM implies not only chemical homogeneity, as in chemical composition SRM's, but also homogeneity of physical characteristics of the material. The parameters affecting physical property homogeneity are so numerous that detailed characterization of each is prohibitive. Instead, one often reverts to aggregate characterization methods, such as by electrical resistivity as discussed later.

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

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. Generally the ice-point is a sufficiently high temperature and liquid helium is a sufficiently low temperature to approximately satisfy the WFL law.

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.

$$\rho(T) = \rho_o + \rho_i(T). \tag{3}$$

This separation of the total resistivity into a constant term, ρ_o, and temperature dependent term, $\rho_i(T)$, is known as Matthiessen's rule. Although Matthiessen's rule is not strictly valid, it is a sufficient approximation to be useful for our purposes.

At ambient temperatures the residual resistivity is a negligibly small fraction of the total resistivity; consequently, the total resistivity, $\rho(T)$, is nearly equal to the intrinsic resistivity, $\rho_i(T)$, and therefore a characteristic of the metal itself. As the temperature approaches absolute zero, however, the intrinsic resistivity becomes very small and the total resistivity is essentially the value of ρ_o. The temperature at which $\rho(T)$ becomes constant depends upon the purity of the sample, but for most materials available at the present time, the intrinsic resistivity will be negligible at 4 K (the boiling point of helium). For the tungsten studied here the total resistivity is temperature independent within the accuracy of our measurements, below 15 K.

The residual resistivity, which is caused primarily by impurities and imperfections, provides a good indication of a specimen's purity and freedom from strain. Rather than using the residual resistivity itself for this purpose, a common procedure is to determine a specimen's resistance at the ice-point, R_{273}, and at 4 K, R_4, and calculate the ratio between these two, R_{273}/R_4. This is nearly equal to the ratio of the resistivities at the same temperatures, as the geometric form factor nearly cancels in the ratio. The geometric form factors are not quite the same because of thermal expansion, which is about 0.5% between 273 K and 4 K. This ratio is called the residual resistivity ratio, RRR, and its magnitude is an indication of the purity and physical perfection of the specimen. Since the specimens measured here were generally in the annealed condition, the RRR value should indicate the effective chemical purity (electrical purity).

To support the validity of this statement, we computed the residual resistivity based on the measured chemical composition of these tungsten lots. The specific resistivity of impurities in tungsten was estimated to average 3 μΩ·cm/at.% on the basis of Blatt's [14] table of specific resistivities for many other metals. (This is a crude estimate since no data are listed for tungsten.) We obtained a value of 0.6 nΩ·m assuming that all impurities are in solution for the two lots of tungsten studied. The measured residual resistivities range from 0.5 to 0.8 nΩ·m. This indicates reasonable agreement between directly measured chemical purity and electrically measured purity.

Electrical resistivity variations are accompanied by thermal conductivity variations of nearly equal and opposite sign as indicated by the WFL law. Therefore, the determination of relative residual resistivity variability will directly indicate thermal conductivity variability. The measurement of electrical resistivity is much easier than the determination of thermal conductivity.

Other characterization data are useful to establish the nature of these particular tungsten lots. Of these, composition is the most significant, but hardness, grain size, density, and other physical properties are valuable aids in data analyses. These data are presented in the following sections for AFML arc cast and NBS sintered tungsten.

4.1 AFML Arc Cast Tungsten

In an earlier section, the AFML-AGARD project was briefly described and it was indicated that both sintered and arc cast tungsten were distributed for measurements. The arc cast tungsten discussed in this paper is from the lot originating with that study and therefore is referred to as AFML arc cast tungsten. The raw material for the starting billet was high purity tungsten powder. A billet was formed from the powder by vacuum arc melting to a bar approximately 25 cm in diameter and 46 cm in length. The billet was machined to 20 cm diameter and then extruded to a rod of 10.5 cm diameter. After an acid etch the rod was swaged to 4 cm diameter. Portions of this rod were swaged to form smaller diameter rods. These rods were dye penetrant inspected for defects and heat treated at 1700 K for 1/2 hour. The composition of these rods as determined by the supplier is listed in table 1. A considerable portion of this arc cast tungsten was used in the AFML-AGARD program. Part of it was sent to the European Transuranium Institute, Karlsruhe, Germany, in care of H. E. Schmidt. The remaining rods were supplied to NBS, OSRM for future distribution.

The available diameters in cm are 0.51, 0.64, 0.83, 1.02, 1.27, 2.54, 3.18, and 4.06. The measured density of these rods is 19.20 ± 0.05 g/cm^3. Each of the rod sizes of this heat of arc cast tungsten was characterized with electrical resistivity measurements at the ice point and at liquid helium temperature. The variability of electrical resistivity at the ice-point indicates primarily the homogeneity of macro-physical defects, such as cracks and voids. The variability at liquid helium temperature indicates homogeneity of the lot from the standpoint of both chemical impurities and physical defects as discussed earlier.

Table 2 lists the observed resistivities at 4 K and 273.15 K and calculated values of RRR and intrinsic resistivities for AFML arc cast tungsten specimens of various diameters. The range of resistivities observed at the ice point is ± 0.7% from the mean indicating that the macro-homogeneity and the high temperature electrical resistivity and thermal conductivity variability are quite acceptable for SRM's. The values observed at 4 K range from 0.5 to 0.8 nΩ·m, indicating a relatively large percentage variation in chemical purity. This is not surprising since for pure materials, a small absolute variation in chemical impurity can amount to a large percentage variation. The variation observed is diameter dependent, indicating that impurity pickup is greater for the small diameter specimens, probably because of the increased surface-to-volume ratio. The large variation in ρ, and therefore λ, at 4 K detracts from the apparent usefulness of this lot of material at low temperatures. However, by a simple measurement of ρ_o for each specimen, the entire range of ρ and λ can be predicted to within engineering accuracy, as is shown later. Above 90 K the effect of ρ_o variations is strongly diminished and can often be neglected.

Measurements on specimens of arc cast tungsten from different heats resulted in RRR values similar to those in table 2 when annealed at 1700 K. However, the RRR values increased to above 100 with less variability when annealed at 2300 K and above. This indicates that an anneal at 1700 K is too low to remove most of the physical defects and therefore, much of the variability may be the result of defect variation rather than composition variation. An anneal of 2300 K for one hour is recommended.

The intrinsic resistivity is characteristic of the pure, defect-free base metal and its constancy in table 2 serves as a check on the accuracy and precision of the measurements. The range of values is ± 0.5% of the mean, which is consistent with our estimated measurement uncertainty.

4.2 Sintered Tungsten

The AFML-AGARD project initially included sintered tungsten as candidate reference material. This material was later rejected for use as a reference material because of its inhomogeneity and instability at high temperatures. This instability was observed primarily with the larger diameter specimens. The larger diameter specimen densities were lower than the small diameter specimens by more than 5%. The instability is, therefore, believed to be caused by incomplete sintering and heat treatment. For this reason, a lot

Table 1. Composition of AFML Arc Cast and NBS Sintered Tungsten

Element	Composition in Weight PPM	
	AFML Arc Cast	NBS Sintered
Al	< 10	-
Ca	< 5	< 10
Si	< 20	10-100
Mo	140	10-100
Fe	16	< 10
Cr	< 10	< 10
Ni	< 55	-
Cu	< 1	< 10
Mn	< 10	-
Mg	< 1	< 10
Sn	< 20	-
Pb	< 20	-
Ti	< 5	-
V	< 10	-
Zr	< 10	-
H_2	14	-
N_2	28	-
C	26	-
O_2	36	-

Table 2. Electrical Resistivity Characterization of AFML Arc Cast Tungsten (Anneal = 1700 K: 1/2 hour)

Bar Diameter (cm)	Electrical Resistivity (n Ω•m) 273.15 K	4 K	273.15 K (Intrinsic)	RRR* $\rho_{273\ K}/\rho_{4\ K}$
0.51				
left end	49.4	0.821	48.6	60.2 ± 0.2
right end	49.3	0.759	48.5	64.9 ± 0.2
0.64				
left end	49.3	0.788	48.5	62.5 ± 0.2
right end	49.3	0.786	48.5	62.7 ± 0.2
0.83				
left end	49.0	0.64	48.4	77 ± 1
right end	48.9	0.63	48.3	78 ± 1
1.02				
left end	48.9	0.50	48.4	97 ± 1
right end	48.8	0.49	48.3	100 ± 1
1.27				
left end	48.7	0.53	48.2	92 ± 2
right end	48.7	0.53	48.2	92 ±2

*RRR = Residual resistivity ratio

of sintered tungsten stocked by NBS-OSRM was investigated for use as thermal and electrical SRM's. Its density is 19.23 ± 0.05 g/cm^3, comparable to the small diameter specimens previously studied and the same as the arc cast material. The composition of this sintered tungsten, referred to as NBS sintered tungsten, is listed in table 1. No difference was observed in the composition of the two available diameters, 0.64 cm and 0.32 cm. The overall purity is determined as 99.98% tungsten. Hardness has been measured as 405 DPH for a specimen annealed at 2020°C and 514 for an unannealed specimen.

As with the arc cast tungsten, electrical resistivity measurements have been performed to characterize the homogeneity of NBS sintered tungsten. Specimens were selected at random from the entire lot in both the 0.64 and 0.32 cm diameter rods. Electrical resistivity measurements were performed on specimens annealed for 60 hours at 1200°C and on specimens annealed for one hour at 2020°C. Similar to the arc cast tungsten, increasing the anneal temperature increased the residual resistivity ratio and the apparent homogeneity. A specimen diameter dependence was again observed, indicating that the smaller diameter specimens were slightly less pure. In this case, however, the mean electrical resistivities of the 0.64 cm and 0.32 cm diameter were within 2σ (σ being the standard deviation). Thus, statistically it is difficult to make a clear and unambiguous distinction between them.

The effect of high temperature cycling (2800 K) was also studied on three 0.32 cm specimens. These specimens were temperature cycled by R. E. Taylor of Purdue University using electrical self heating. The RRR measurements were performed at NBS. The results show no further increase in RRR over the specimens annealed at 2020°C and no physical degradation. The above described RRR results are listed in table 3. The intrinsic ice point resistivity as determined for three of the 0.32 cm diameter specimens is 48.5 ± 0.2 nΩ·m, which is the same as that determined for arc cast tungsten as listed in table 2.

The conclusions drawn from these and earlier AFML characterization data are as follows. Both AFML arc cast tungsten and NBS sintered tungsten are useful as SRM's of thermal conductivity and electrical resistivity at temperatures up to at least 2800 K if annealed before use at 2300 K for one hour. Below ambient the effects of material inhomogeneity gradually became significant. At liquid helium temperature, the thermal and electrical conductivities of NBS sintered tungsten vary from specimen-to-specimen by about ± 5%. The corresponding variation for the arc cast tungsten may be larger primarily because of the dependence on rod diameter. The effect of these variations can be determined and corrections applied by a relatively simple residual electrical resistivity measurement on each specimen. The effect of inhomogeneity on electrical resistivity and thermal conductivity decreases rapidly with increasing temperature. These variations are relatively small above 90 K and essentially insignificant above 300 K. This is demonstrated in later sections.

5. Apparatus and Measurements

Numerous thermal conductivity and electrical resistivity measurements have been performed on sintered and arc cast tungsten. However, neither of these materials has been measured at both cryogenic and elevated temperatures. The AFML arc cast tungsten has been measured at temperatures above ambient by several participants of the AFML-AGARD project. The NBS sintered tungsten has been measured at temperatures below ambient at NBS, Boulder. A sintered tungsten was also measured at elevated temperatures as part of the AFML-AGARD project. These data are discussed below according to whether they are principally below or above ambient temperature.

5.1 Low-Temperature (Below Ambient) Measurements

The electrical resistivity, thermal conductivity, and thermopower of NBS sintered tungsten were measured from 6 to 280 K by Hust [15]. These measurements were performed simultaneously on a single specimen using a longitudinal heat-flow, multi-property apparatus. The specimen was 3.1 mm in diameter and 23 cm long. Eight thermocouples were mounted at equally spaced positions along the length of the specimen to determine temperature gradients in the range 4 to 300 K. An unannealed specimen was measured from 6 to 280 K and a specimen annealed at 2020°C for one hour was measured from 7 to 90 K. These data were represented with the following equations for smoothing and analysis.

$$\ell n\lambda = \sum_{i=1}^{n} a_i \, [\ell nT]^{i+1} \qquad (4)$$

Table 3. Residual Resistivity Ratio ($\rho_{273\ K}/\rho_{4\ K}$) of NBS Sintered Tungsten

Specimen	RRR	Specimen	RRR	Specimen	RRR
Unannealed		2020°C, 1 hr; 0.64 cm dia.		2020°C, 1 hr; 0.32 cm dia.	
14-7	39.8	1-1	78.0	10-2	73.6
1200°C, 60 hr; 0.64 cm dia.		2-2	76.1	11-2	79.0
		3-1	83.2	12-6	80.1
1-1	66.2	4-2	93.5	13-1	75.5
1-2	48.8	5-1	82.8	14-7	74.9
3-1	69.5	6-1	79.0	15-7	81.3
2-2	63.9	6-2(1)	79.9	16-4	79.4
6-1	44.2	6-2(2)	83.1	17-5(1)	69.8
3-2	65.4	6-2(3)	80.8	17-5(2)	71.2
mean =	59.6	6-2(4)	82.3	17-5(3)	69.8
std. dev. =	9.2	6-2(5)	83.1	17-5(4)	70.4
		mean =	82.0	17-5(5)	70.8
1200°C, 60 hr; 0.32 cm dia.		std. dev. =	4.5	17-8	71.5
		2525°C; 0.32 cm dia.		18-6(1)	76.8
10-2	56.6			18-6(2)	75.1
10-7	46.6	10-2		mean =	74.6
14-2	43.5	30 min.	71.0	std. dev. =	4.0
14-7	45.2	12-6			
18-2	61.9	10 min, 5 times	78.2		
18-7	62.5				
mean =	52.7	11-2			
std. dev. =	8.6	10 min, 10 times	78.4		

$$\rho = \sum_{i=1}^{m} b_i \, [\ell n T]^{i-1} \qquad (5)$$

$$S = \sum_{i=1}^{\ell} c_i \, [\ell n T']^{i}/T' \; ; T' = \frac{T}{10} + 1 \qquad (6)$$

where λ = thermal conductivity, ρ = electrical resistivity, S = thermopower, and T = temperature. The deviations of the experimental data from values calculated with these equations are illustrated in figures 1-6. The horizontal bars in figures 3-6 indicate the temperature span of each run. Smoothed low-temperature values of thermal conductivity, electrical resistivity, Lorenz ratio, and thermopower as calculated from equations 4-6 are illustrated in figures 7-10. The RRR values for the annealed and unannealed specimens are 76 and 40, respectively. The estimated uncertainties of the smoothed values are:

thermal conductivity - 2.5% at 300 K, decreasing to 0.7% at 200 K, 0.7% from 200 K to 50 K, and increasing inversely with temperature to 1.5% at 4 K

electrical resistivity - 0.5%

thermopower - 0.2 μv/K at 4 K, decreasing to 0.03 μv/K at 80 K and above.

Further details of the NBS measurement procedure and data analysis are given by Hust et al. [6].

5.2 High-Temperature (Above Ambient) Measurements

Several participants of the AFML-AGARD project have measured thermal conductivity, electrical resistivity, thermal diffusivity, specific heat and other thermophysical properties of tungsten at temperatures above ambient. These data have been presented by Fitzer [12] and reviewed by Minges [13]. Various apparatus were used to obtain these data. The general description of these apparatus and methods can be obtained from the original papers. Our interest is concerned primarily with the accuracy of the resultant data. The stated uncertainties of the thermal conductivity data are generally in the 5 to 10% range.

Six participants reported thermal diffusivity data for AGARD sintered tungsten. The scatter between these data sets is about ± 10%. The scatter within each of the data sets is about ± 5%. The effect of high temperature exposure (2846 K) as reported by one participant was to increase the thermal diffusivity by approximately 10%. Four participants reported thermal diffusivity data for AFML-AGARD arc cast tungsten. These data agreed to within about ± 5%. The effect of high temperature exposure was not reported. Two participants reported specific heat data for sintered tungsten. Although they do not overlap in temperature range, the agreement at the common temperature is within about 1%. These data agree with existing literature data for other tungsten to within about 2%.

Two participants reported thermal conductivity data for sintered tungsten. These are widely separated in temperature (one set ranges from 400 to 900 K and the other from 2300 to 2800 K) thus, no comparisons between them are possible. The high temperature data are appreciably below other tungsten data reported in the literature.

Only one participant reported thermal conductivity data for arc cast tungsten (from 1600 to 2800 K). These data agree with literature data for other tungsten to within 10%.

One participant reported the electrical resistivity of sintered tungsten and another reported the same for arc cast tungsten. These two sets of data overlap from 2000 K to 2700 K and agree with each other to within 1%. Minges [13] compared resistivity measurements on various forms of tungsten, including single crystal, arc cast, sintered, and ultra high purity. All of these data agree to within about ± 2%. These and other intercomparisons are presented in the following section.

Moore et al. [16,17] measured electrical resistivity, thermopower, and thermal conductivity of two specimens of tungsten from 100 to 1700 K. One of them was a sintered specimen very similar in composition to the NBS sintered tungsten, although its density ($19.1\ g/cm^3$) was slightly lower. Its RRR was 31. The other was a higher purity electron beam melted specimen with an RRR of 400.

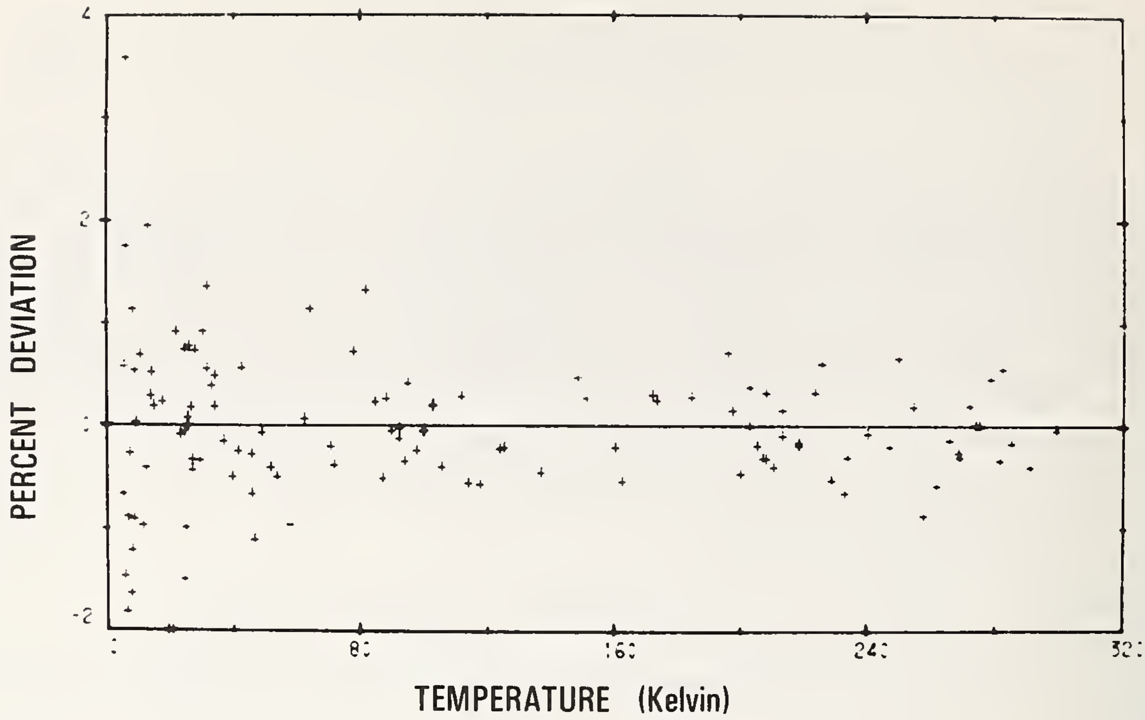

Figure 1. Thermal conductivity deviations for NBS sintered tungsten (unannealed).

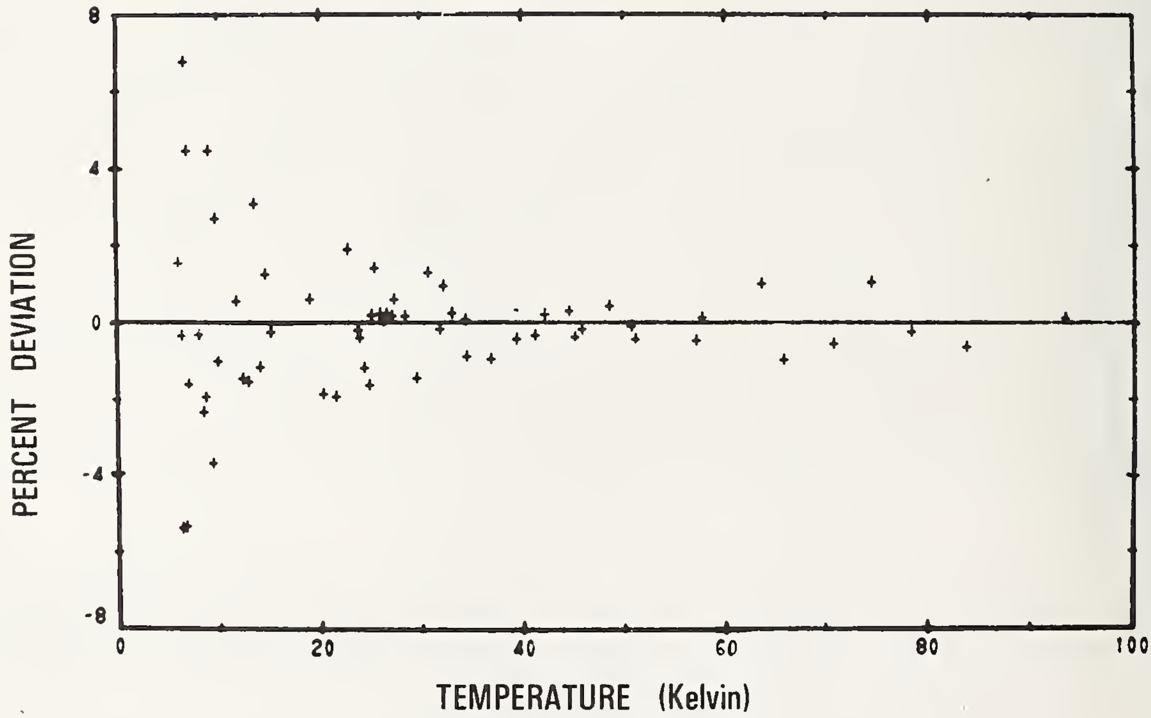

Figure 2. Thermal conductivity deviations for NBS sintered tungsten (annealed).

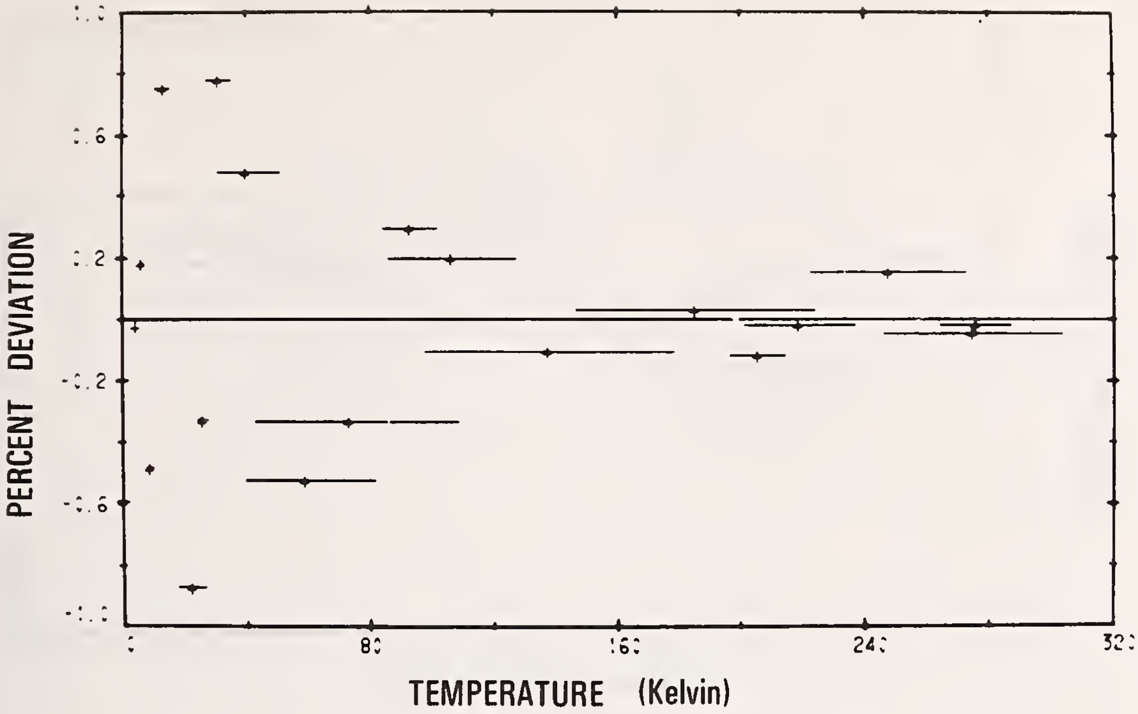

Figure 3. Electrical resistivity deviations for NBS sintered tungsten (unannealed).

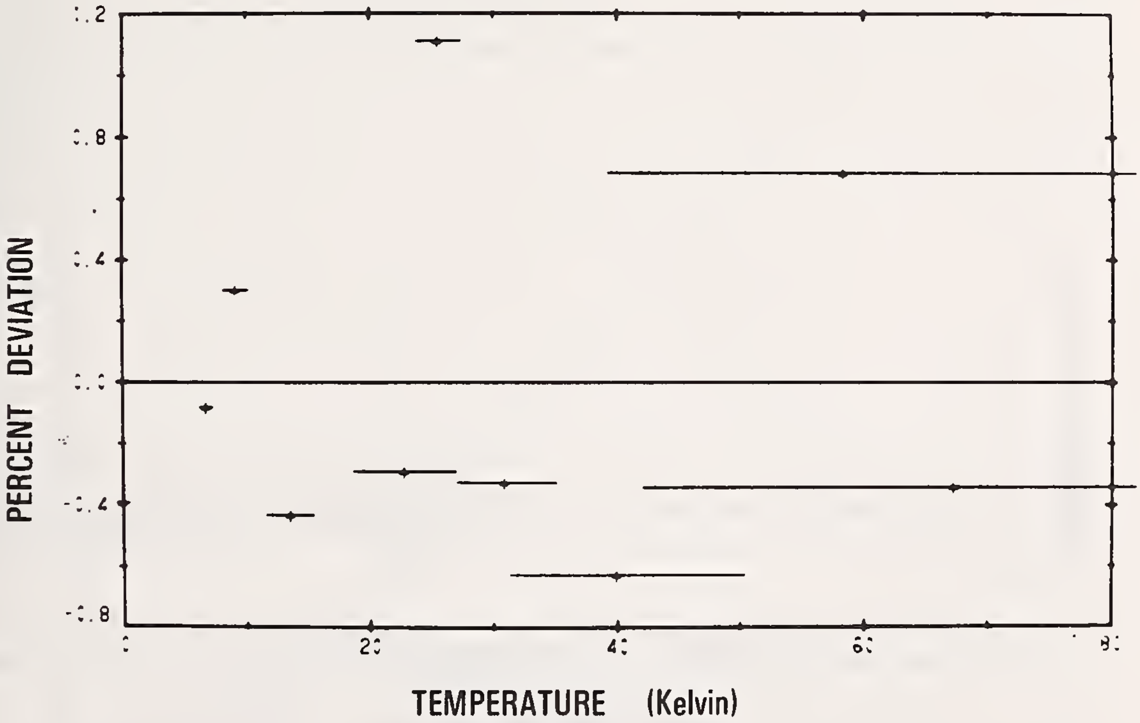

Figure 4. Electrical resistivity deviations for NBS sintered tungsten (annealed).

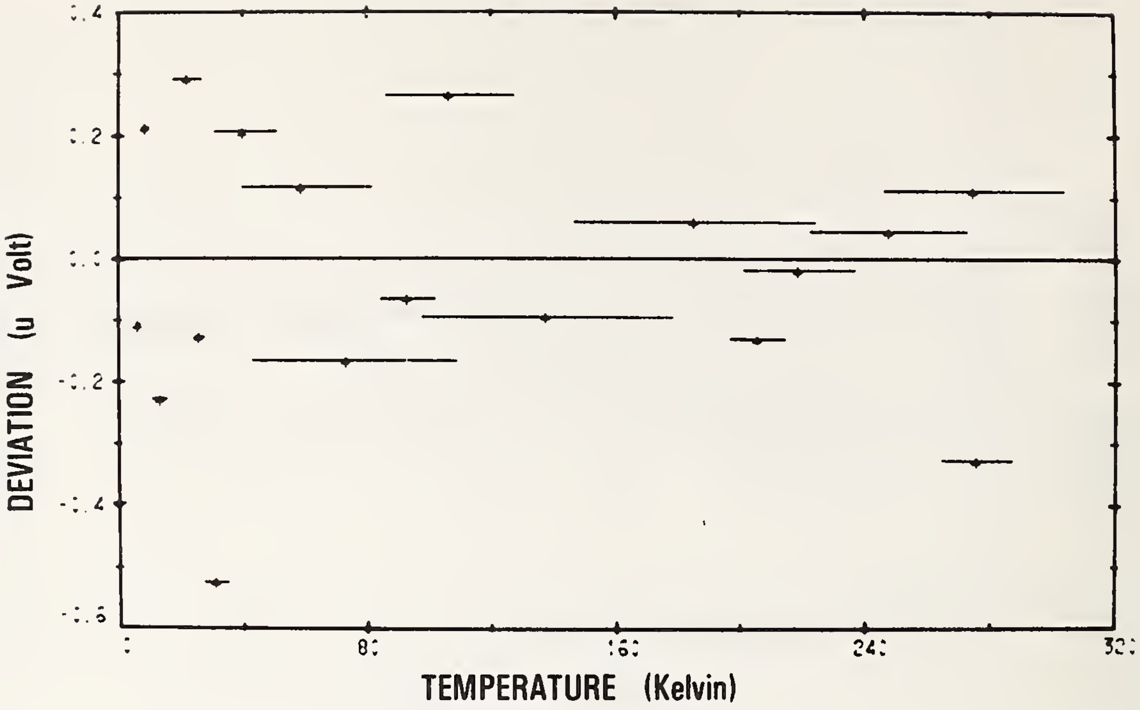

Figure 5. Thermovoltage deviations for NBS sintered tungsten (unannealed).

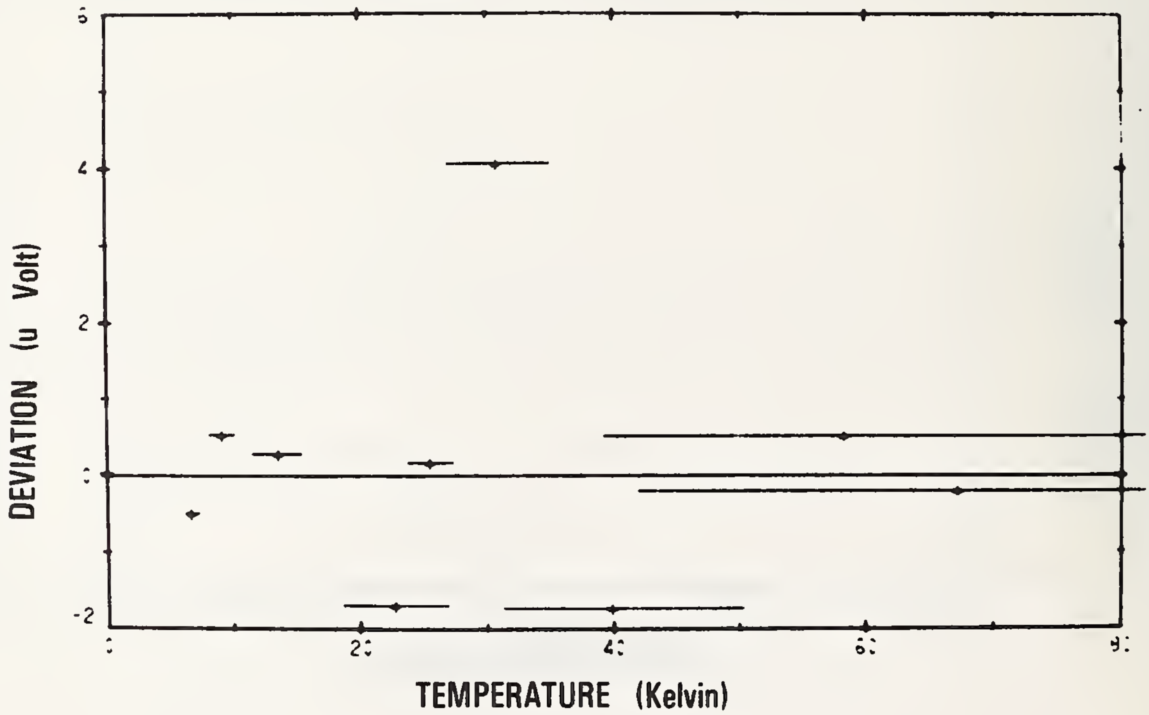

Figure 6. Thermovoltage deviations for NBS sintered tungsten (annealed).

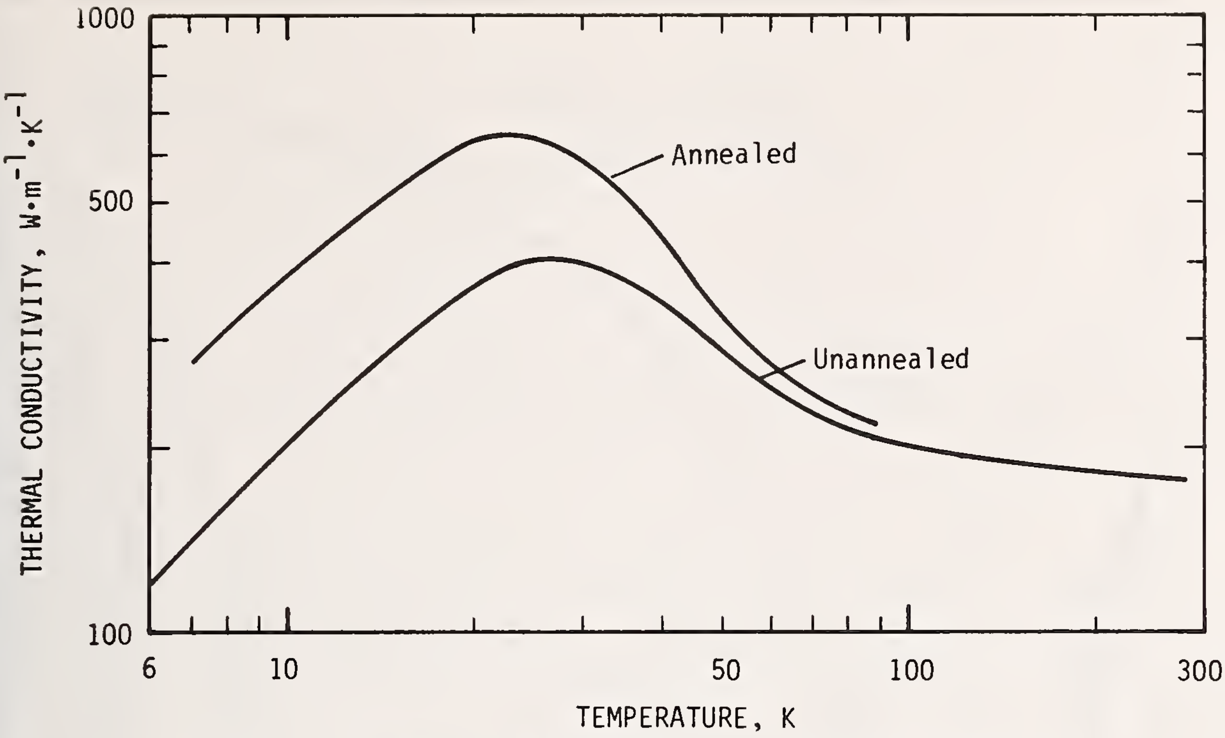

Figure 7. Thermal conductivity of NBS sintered tungsten.

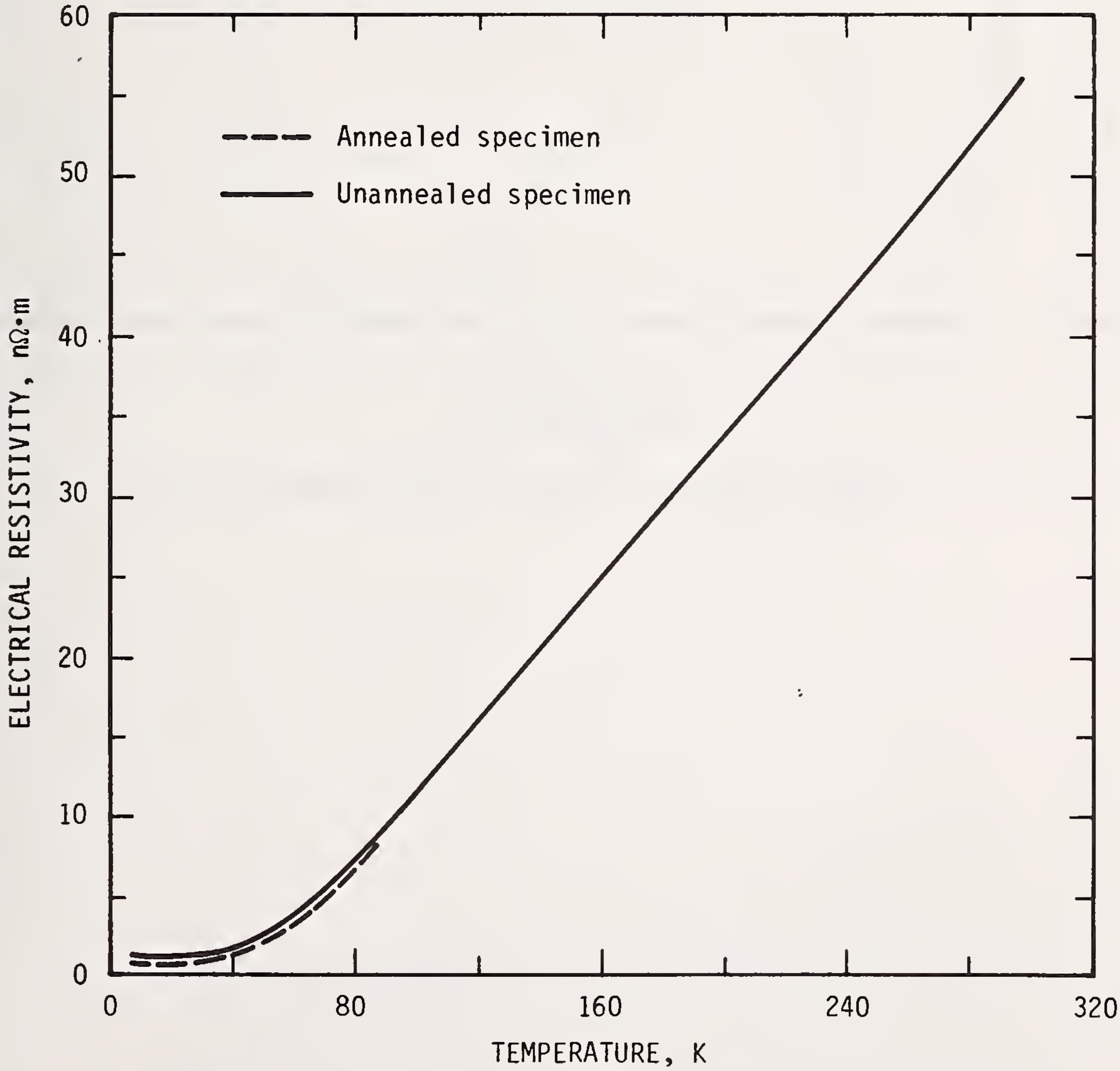

Figure 8. Electrical resistivity of NBS sintered tungsten.

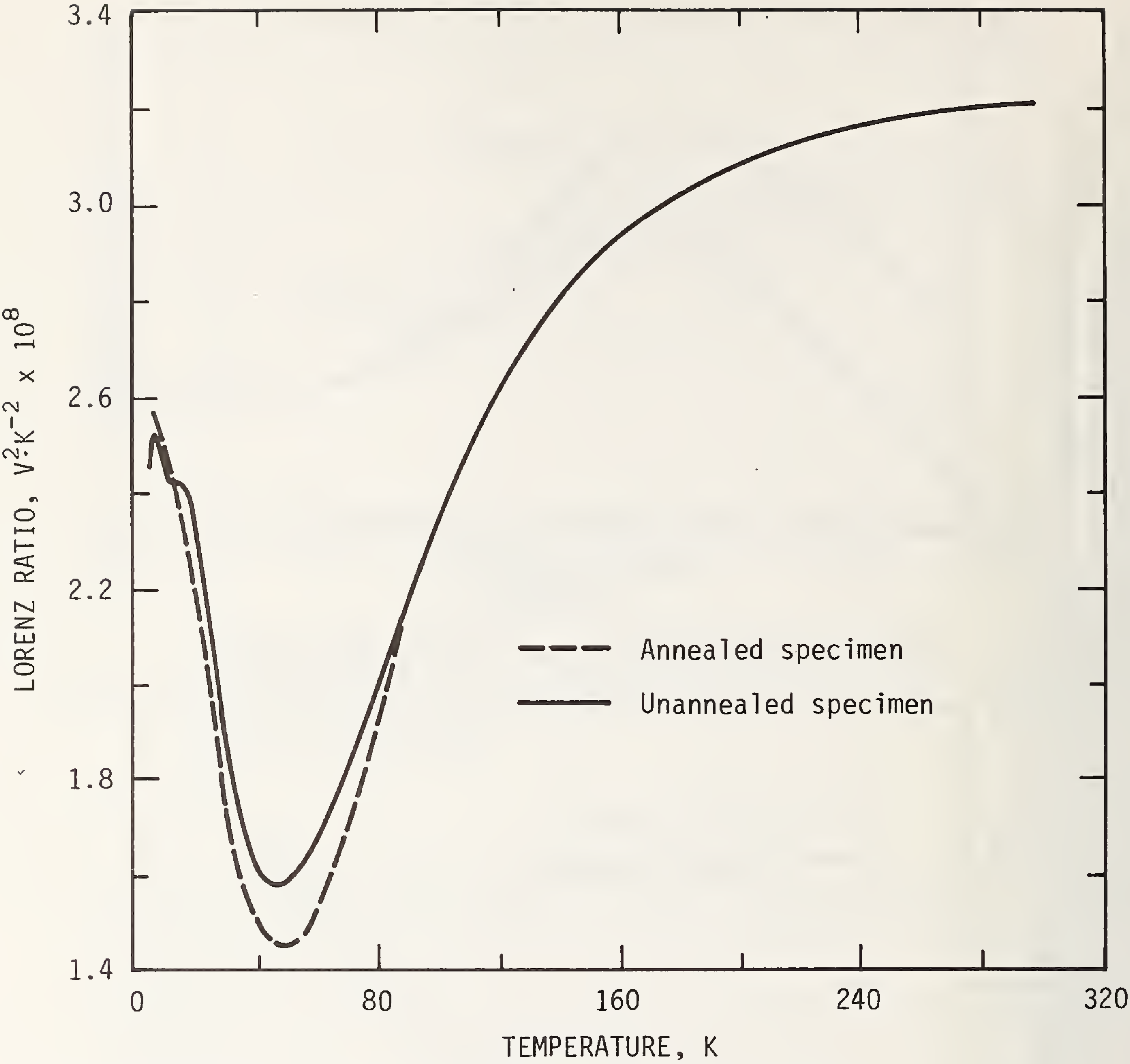

Figure 9. Lorenz ratio of NBS sintered tungsten.

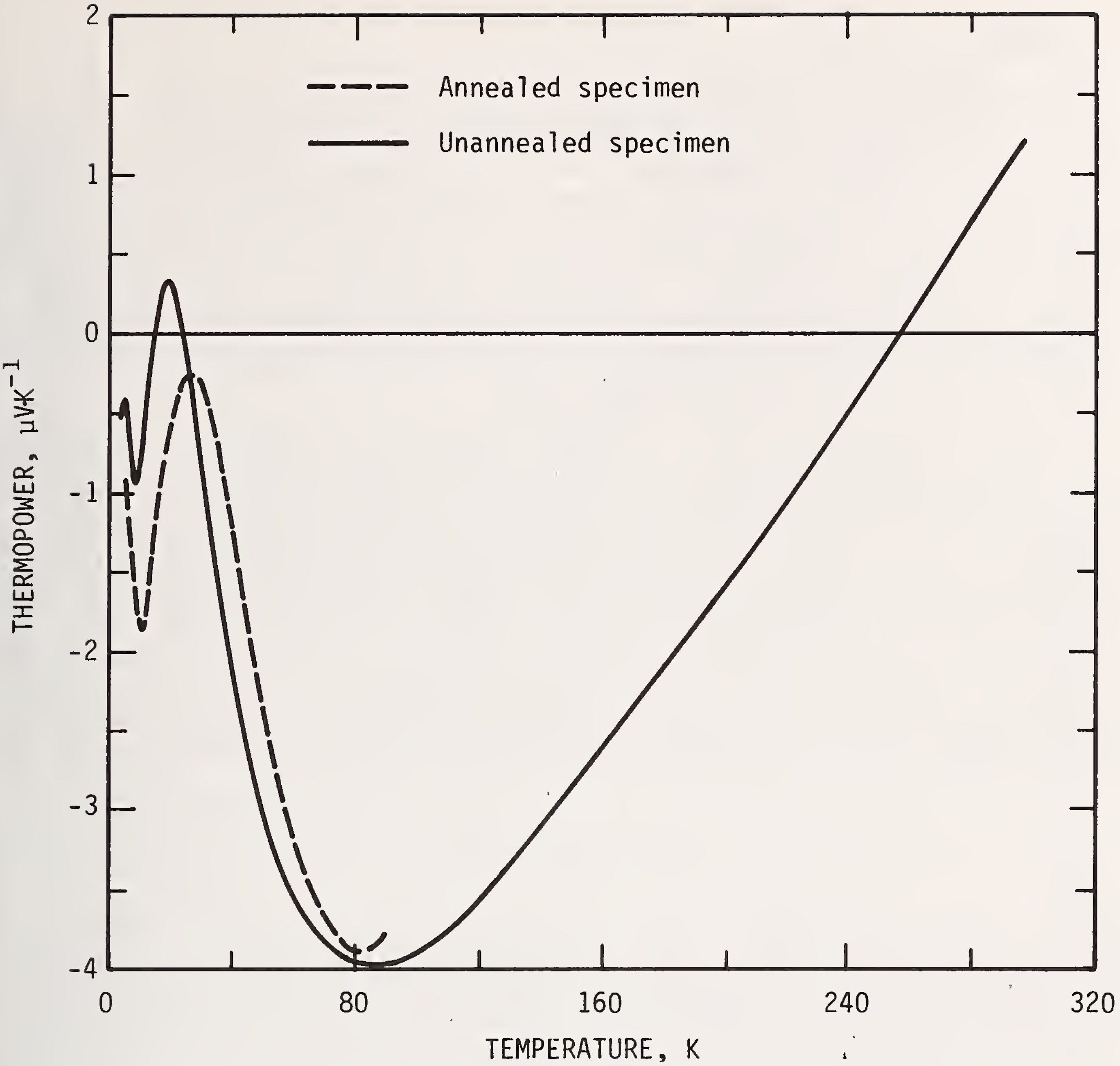

Figure 10. Thermopower of NBS sintered tungsten.

6. Data Analysis (Selection of Best Values)

6.1 Electrical Resistivity

Because of the many data sources and the existing comparisons, it is difficult to present a simple comparison of all of these data. Thus, it has been decided to present several comparison graphs using a reference equation to facilitate comparison between graphs. First, the previously described experimental electrical resistivity data sets are illustrated in figure 11. This figure includes 153 data points from nine sources. The points are so closely spaced and self consistent on this graph that it is impractical to delineate the various sources. The main purpose of this graph is to illustrate the primary data sets [12,15-19] and the range of data involved in the analysis. It is clear from figure 11 that only 2 data points are excessively discordant with the data set. These data sets will be unambiguously defined and compared in the following deviation plots. Our objective is to obtain and present best values of resistivity for two particular lots of tungsten, arc cast and sintered; therefore, only the data on tungstens similar in composition to these are considered primary data.

Figure 11 illustrates the effect of impurity and state of anneal on the low temperature resistivity of these specimens. Below about 100 K these variations are pronounced. At 4 K the resistivity is determined almost entirely by these imperfections. Because of these low temperature variations, the representation problem becomes essentially three dimensional, i.e., resistivity as a function of temperature and residual resistivity. For the range of residual resistivity to be studied, it was assumed that Matthiessen's rule would be valid, i.e., the total resistivity function is separable into additive residual and intrinsic resistivities, $\rho(T) = \rho_i(T) + \rho_o$. To confirm this hypotheisis, we expressed $\rho_o(T)$ as a power series in ℓnT (equation 7) and fitted it to the data illustrated in figure 11. The deviations of this fit are shown in figure 12. Since the deviations shown in figure 12 do not differ from each other systematically with RRR, we conclude that, for this range of residual resistivities, Matthiessen's rule is valid within the scatter of the data.

$$\rho_i = \sum_{i=1}^{n} a_i (\ell nT)^i, \qquad (7)$$

The power series representation of electrical resistivity data over a wide temperature range has several disadvantages. First, a relatively high order, eleven, is required to obtain a good fit. Second, the number of significant digits that must be carried in the calculations is too large to be asthetically satisfactory. Last, but not least, power series representations have a tendency to introduce unreal oscillations within the range of the fitted data and can generally not be extrapolated beyond the data set. For these reasons, a less complex function was sought to represent these data. By analyzing the low and high temperature data sets separately, the following rational function was formulated.

$$\rho = (aT^n + bT^3)/(1 + C/T^m) + \rho_o. \qquad (8)$$

Using an iterative non-linear least squares fitting technique, the best values of the parameters were determined to be

$$a = .04535$$

$$b = -2.90 \times 10^{-9}$$

$$c = 3.442 \times 10^5$$

$$n = 1.2472$$

$$m = 2.98$$

with T in kelvin and ρ in $n\Omega\cdot m$. The deviations of the experimental data, uncorrected for thermal expansion, from this equation are illustrated in figure 13. Also shown are deviations of equation 7 and the oscillatory nature of a power series is clearly evident. Although figure 13 suggests that small systematic deviations exist between equation 8 and the real resistivity behavior of tungsten, the simplicity of this function and the elimination of the disadvantages of the power series representation make this equation more

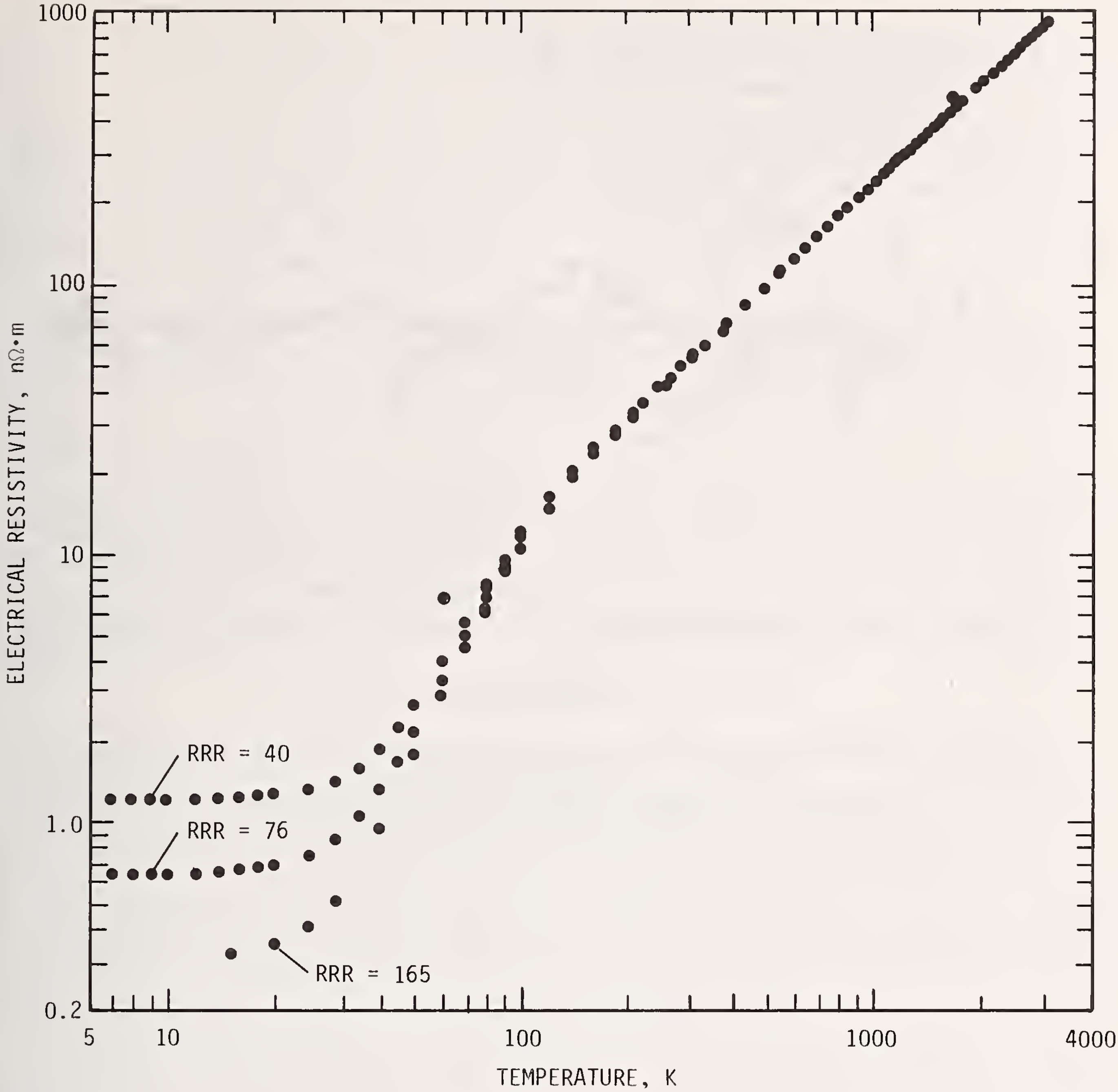

Figure 11. Primary electrical resistivity data [12,15-19] for arc cast and sintered tungsten.

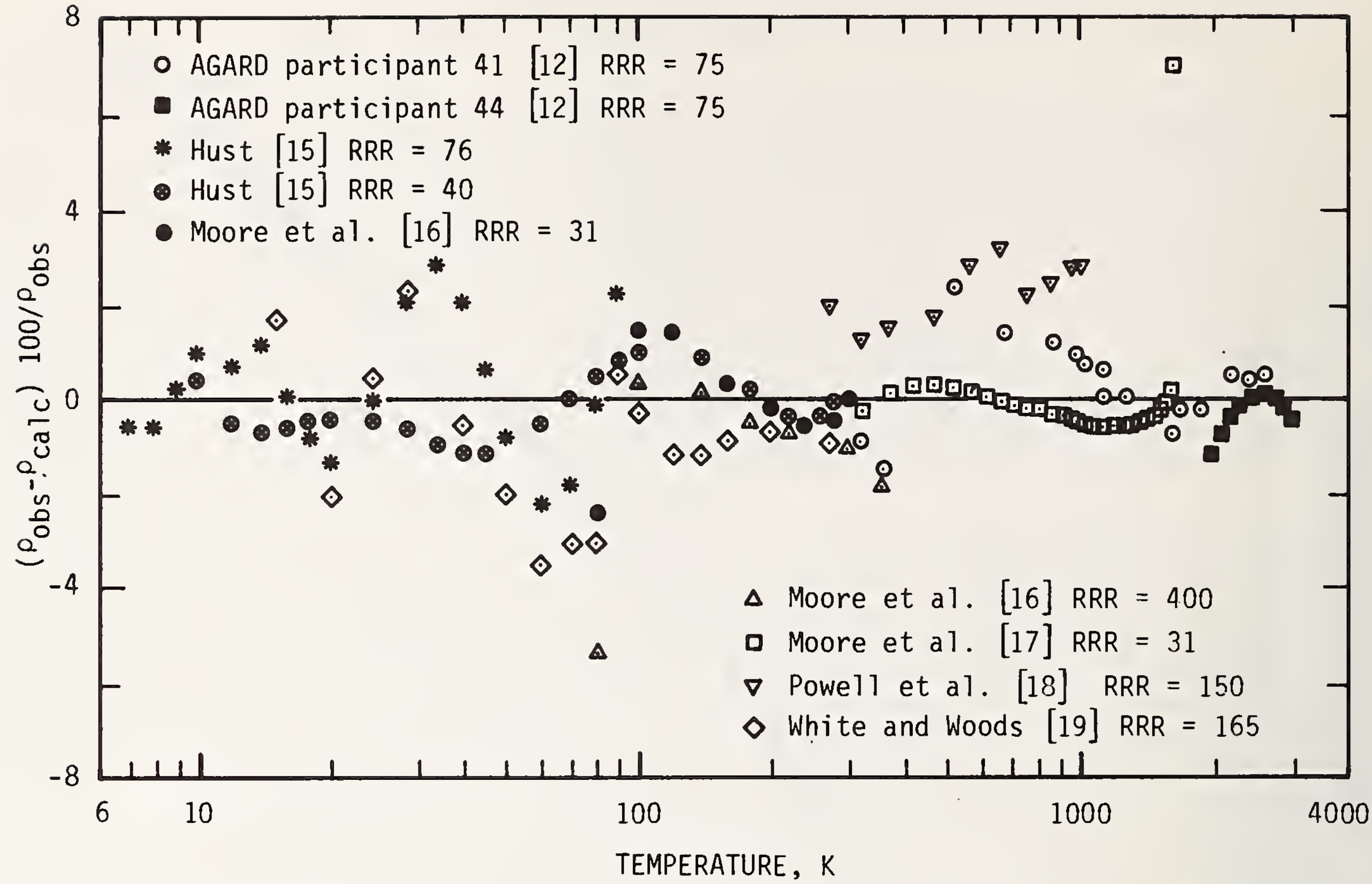

Figure 12. Deviations of primary intrinsic electrical resistivity data for arc cast and sintered tungsten from the eleven term power series representation (equation 7).

desirable as the baseline equation. The numerator of this rational fraction predicts the high temperature behavior while the denominator compensates for low temperature deviation from the higher temperature behavior. Omitting the term C/T^m from the denominator produces a change of less than 1% above 330 K. The bT^3 term represents only a small correction to the numerator, reaching a maximum of 8% at the upper temperature of 3000 K. Thus, the high temperature resistivity of tungsten can be reasonably well represented with simply aT^n. The low temperature resistivity is reasonably represented by $\frac{a}{c}\ T^{n+m} + \rho_o$. The transition region between low and high temperature behavior occurs in the vicinity of 100 K.

Comparisons to other than the primary data set and other smoothing equations is facilitated through the use of equation 8. Figure 14 is a plot of arc cast and sintered tungsten data presented by Minges [13] (reprinted by permission). Superimposed on this plot is a line as computed from equation 8 showing excellent agreement. Figure 15 is a deviation plot presented by Minges [13] and again calculated values from equation 8 have been added for comparison.

The data presented by Minges are uncorrected for residual resistivity variation since the residual resistivity was not known for each of the specimens. Since it is likely that all of these specimens have RRR values above 30, such correction could be as large as 3% at 0°C and 0.5% at 800°C. The omission of these corrections may account for the 2-3% deviation between the high purity (RRR - 4900) and lower purity (RRR = 31) tungsten at ambient temperature as shown in figure 15. Minges [13] compared data to the following baseline equation:

$$\rho = 4.64 + 2.533 \times 10^{2}T + 3.162 \times 10^{6}T^{2}$$

with ρ in μΩ•cm and T in °C. This equation was for the range 20 to 1800°C. It is corrected linearly for thermal expansion. The correction is zero at 20°C and 0.7% at 1800°C. The large deviation between equation 8 and Minges' equation in the vicinity of 300 K is apparently caused by the fact that his equation extrapolates to a resistivity of 4.64 μΩ•cm at the ice point, 0°C. Note that this is outside the range of validity of his equation by 20°C, but our results indicate an ice point resistivity of about 4.94 μΩ•cm for both of these tungsten lots. Also note that the Minges equation should not be extrapolated above the upper temperature, 1800°C, specified by him, as indicated by the strong divergence between his equation and equation 8. This is an example of the danger of extrapolating even a very simple power series, as mentioned earlier. Figure 16 is a deviation plot presented by Moore et al. [17] with the addition of equation 8.

The above comparisons show that good agreement exists between the intrinsic resistivity of various forms of tungsten, including sintered, arc cast, single crystal, and very high purity tungsten. Typical deviations among various data sources are ± 2% for temperatures from 4 to 3000 K.

Examination of the various comparisons illustrated in figures 12 through 16 has led the authors to accept equation 8 for the prediction of best values of electrical resistivity for these lots of arc cast and sintered tungsten. It could be argued that equation 7 is a better least squares representation of the entire data set. However, consideration of the existing material variability, the data uncertainties, and the previously discussed disadvantages of power series, has strongly influenced the decision to adopt an inherently smooth equation rather than one which follows the intricacies of each data set more exactly. The difference between the two equations of course is small as illustrated in figure 13; and the power series representation contains what appear to be unreal oscillations as a function of temperature. Values of resistivity calculated from equation 8, for values of ρ_o spanning the range of these tungsten lots, are listed in table 4. For values of ρ_o other than those listed, it is a simple matter to form a new column by adding ρ_o to the ρ_o = 0 resistivities. The uncertainty in the tabulated values of electrical resistivity is estimated at ± 2% for the entire temperature range. A paper published by Williams [23], after the above work was completed, contains a thorough analysis of the high temperature resistivity of zone-refined tungsten. He presents smoothed values of resistivity for the range 1050 to 2500 K. These data agree with the values listed in table 4 for ρ_o = 0 to within 1% below 2000 K (Williams values are larger); above 2000 K the differences increase to 2% at 2500 K.

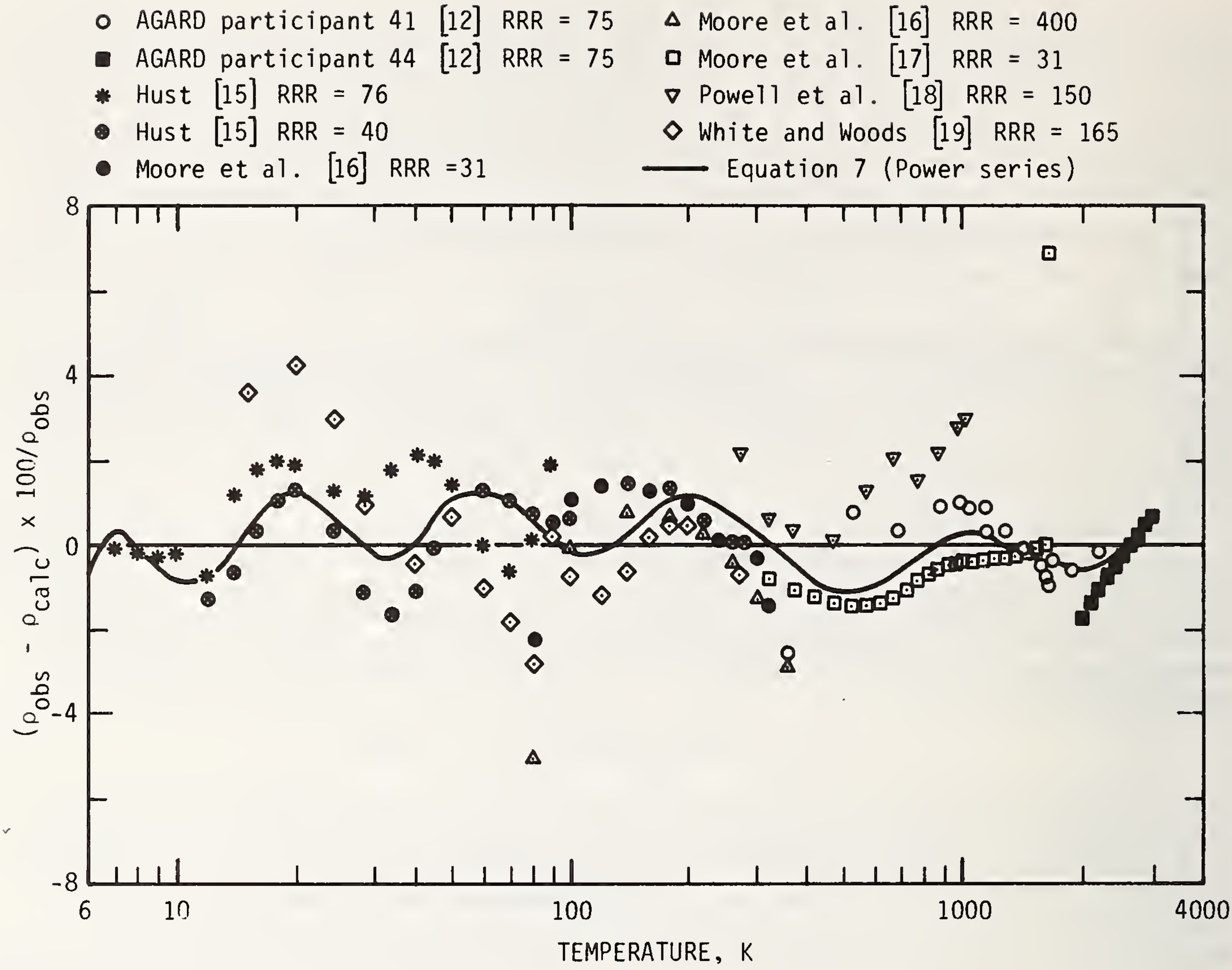

Figure 13. Deviations of primary intrinsic electrical resistivity data for arc cast and sintered tungsten from rational fraction representation. (Equation 8).

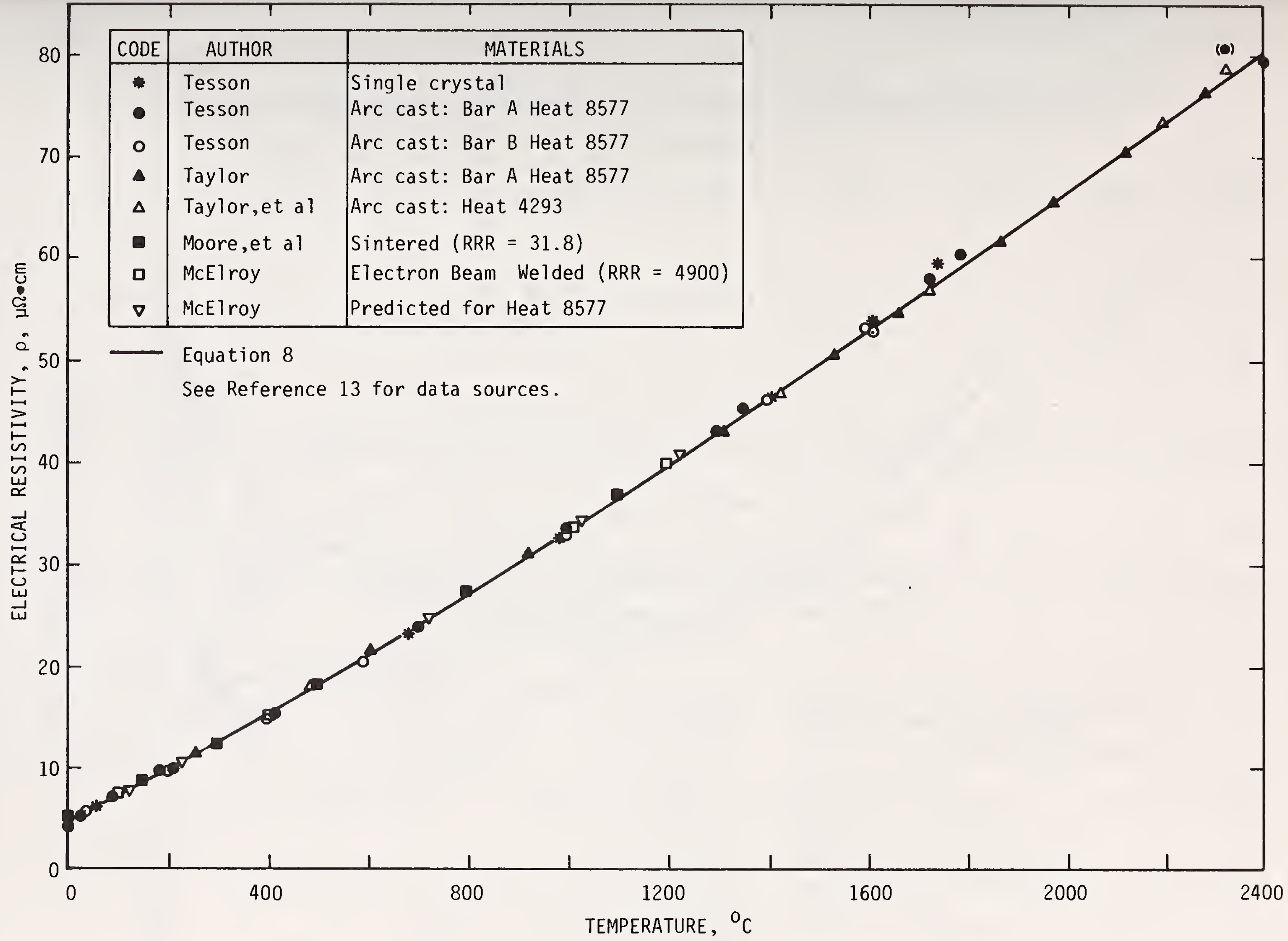

Figure 14. High-temperature electrical resistivity of tungsten. Literature and unpublished data. (Reprinted from Minges [13]).

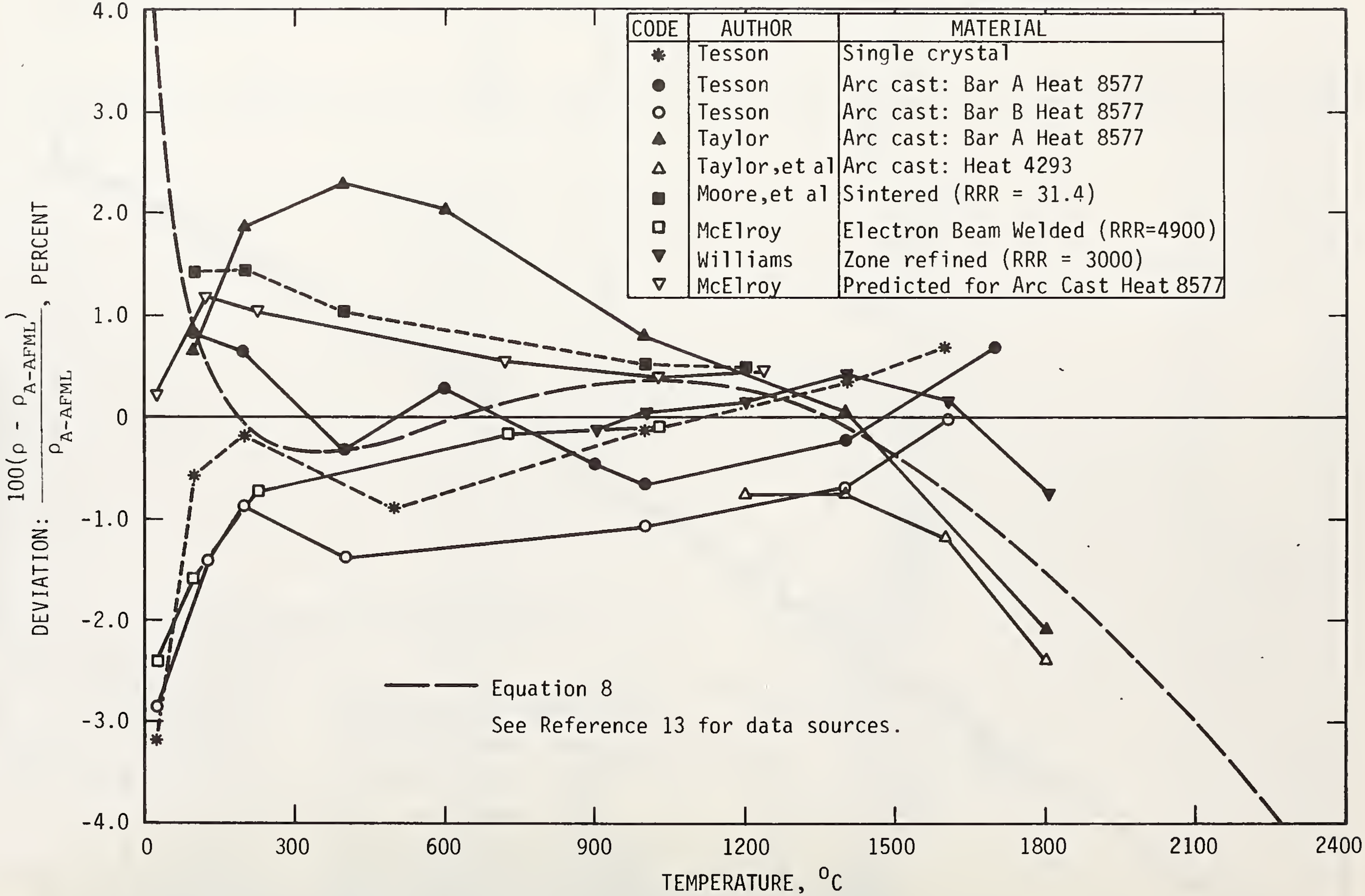

Figure 15. Electrical resistivity deviation of Tungsten. (Reprinted from Minges [13]).

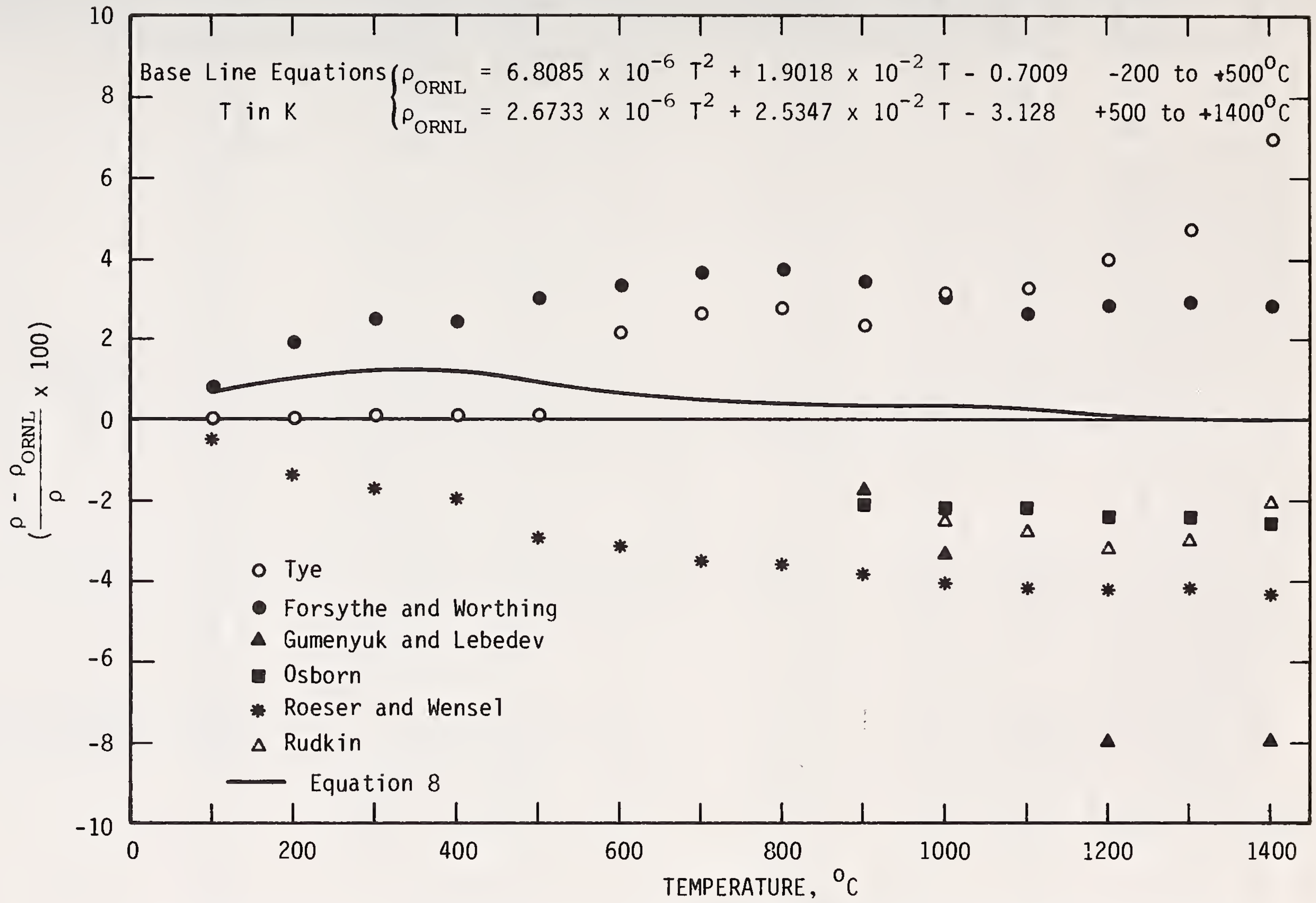

Figure 16. Electrical resistivity deviations from ORNL results. (Reprinted in part from Moore et al. [17])

6.2 Thermal Conductivity

Again, because of the multiplicity of data sets, thermal conductivity intercomparisons are difficult to present without the aid of a reference, or baseline equation. Considerable effort was expended to find such an equation for the range 4 to 3000 K. This equation would need to not only represent a complex temperature dependence, but also, describe the low temperature impurity variation as evidenced by variations in residual electrical resistivity. Figure 17 shows the primary data [12, 15–18] to be represented. Delineation of the separate sources will be given in later deviation plots.

Consideration was given to using a power series fit of these data, as in the case of the electrical resistivity data, and using the thermal equivalent of Matthiessen's rule to obtain the variation with ρ_o. However, it was found that the thermal equivalent of Matthiessen's rule was not adequate to obtain a representation accurate to within the uncertainty of the data. A three dimensional power series equation, $\lambda = \lambda(\rho_o, T)$, was not seriously considered because undoubtedly it would need to be unnecessarily complex to represent the data.

We thus resorted to the formulation of an equation based in part on theory but primarily on empirical grounds. Cezairliyan and Touloukian [20] has shown that for pure metals the low temperature thermal conductivity behavior can be reasonably well described by

$$\lambda = \frac{1}{\alpha T^n + \beta / T} \qquad (9)$$

where β is taken as a linear function of ρ_o, i.e., $\beta = \beta' \rho_o$, α may be a weak function of ρ_o, and n is near 2. This theoretically based equation is generally valid up to about 0.1 of the Debye temperature, or in this case, approximately 40 K. For the range of residual resistivities of interest here, we felt it would be possible to neglect the dependence of α on ρ_o. Thus, we performed an iterative nonlinear least squares fit of the data below 40 K to determine best values of α, n, and β'. It was found that this fit represented the data below 35 K to within their uncertainty. The deviations above 35 K increased very rapidly since this function falls rapidly to zero, and the experimental data fall off much more slowly. By examining the nature of the λ deviation and the character of the Lorenz ratio curve for tungsten, we empirically found that the additive term

$$A \frac{T}{\rho} \text{EXP} \left(- (\Theta_1/T)^2\right) \qquad (10)$$

allowed us to represent the experimental data to 300 K within their uncertainty. Extrapolating this equation to higher temperatures resulted in relatively small deviations with respect to the data which could be fitted with yet another exponential

$$A' \frac{T}{\rho} \text{EXP} \left(-(\Theta_2/T)^2\right). \qquad (11)$$

Thus our final equation for the entire range 4 to 3000 K is

$$\lambda = \frac{1}{\alpha T^n + \beta' \rho_o / T} + A \left[\text{EXP}\left(-(\Theta_1/T)^2\right) + B\ \text{EXP}\left(-(\Theta_2/T)^2\right)\right] \frac{T}{\rho}. \qquad (12)$$

An iterative nonlinear least squares fit of the parameters in this equation to the primary data set resulted in the following best values:

Table 4. Recommended Electrical Resistivity Values for AFML Arc Cast and NBS Sintered Tungsten for Several Values of RRR

Temperature (K)	Electrical Resistivity (nΩ·m) RRR = 50 $\rho_o = 0.97$	RRR = 75 $\rho_o = 0.65$	RRR = 100 $\rho_o = 0.49$	RRR = ∞ $\rho_o = 0$
4	0.970	0.650	0.490	0.000
6	0.970	0.650	0.490	0.000
8	0.971	0.651	0.491	0.001
10	0.972	0.652	0.492	0.002
12	0.975	0.655	0.495	0.005
14	0.979	0.659	0.499	0.009
16	0.986	0.666	0.506	0.016
18	0.996	0.676	0.516	0.026
20	1.011	0.691	0.531	0.041
30	1.185	0.865	0.705	0.215
40	1.635	1.315	1.155	0.664
50	2.469	2.149	1.989	1.498
60	3.713	3.393	3.233	2.740
70	5.31	4.986	4.826	4.333
80	7.15	6.83	6.67	6.18
90	9.15	8.83	8.67	8.18
100	11.25	10.93	10.77	10.27
120	15.54	15.22	15.06	14.57
140	19.88	19.56	19.40	18.91
160	24.24	23.92	23.76	23.26
180	28.61	28.29	28.13	27.64
200	33.02	32.70	32.54	32.05
250	44.25	43.93	43.77	43.28
300	55.8	55.5	55.4	54.8
350	67.8	67.5	67.3	66.8
400	80.1	79.8	79.6	79.1
450	92.7	92.4	92.2	91.7
500	105.7	105.3	105.2	104.7
600	132.4	132.1	131.9	131.4
700	160.1	159.8	159.6	159.2
800	188.7	188.4	188.2	187.8
900	218.1	217.8	217.6	217.1
1000	248.1	247.8	247.6	247.2
1200	309.9	309.6	309.4	309.0
1400	373.5	373.2	373.0	372.6
1600	438.6	438.3	438.1	437.6
1800	505	504	504	504
2000	572	571	571	571
2200	639	638	638	638
2400	706	706	706	705
2600	774	773	773	773
2800	841	840	840	840
3000	907	907	907	906

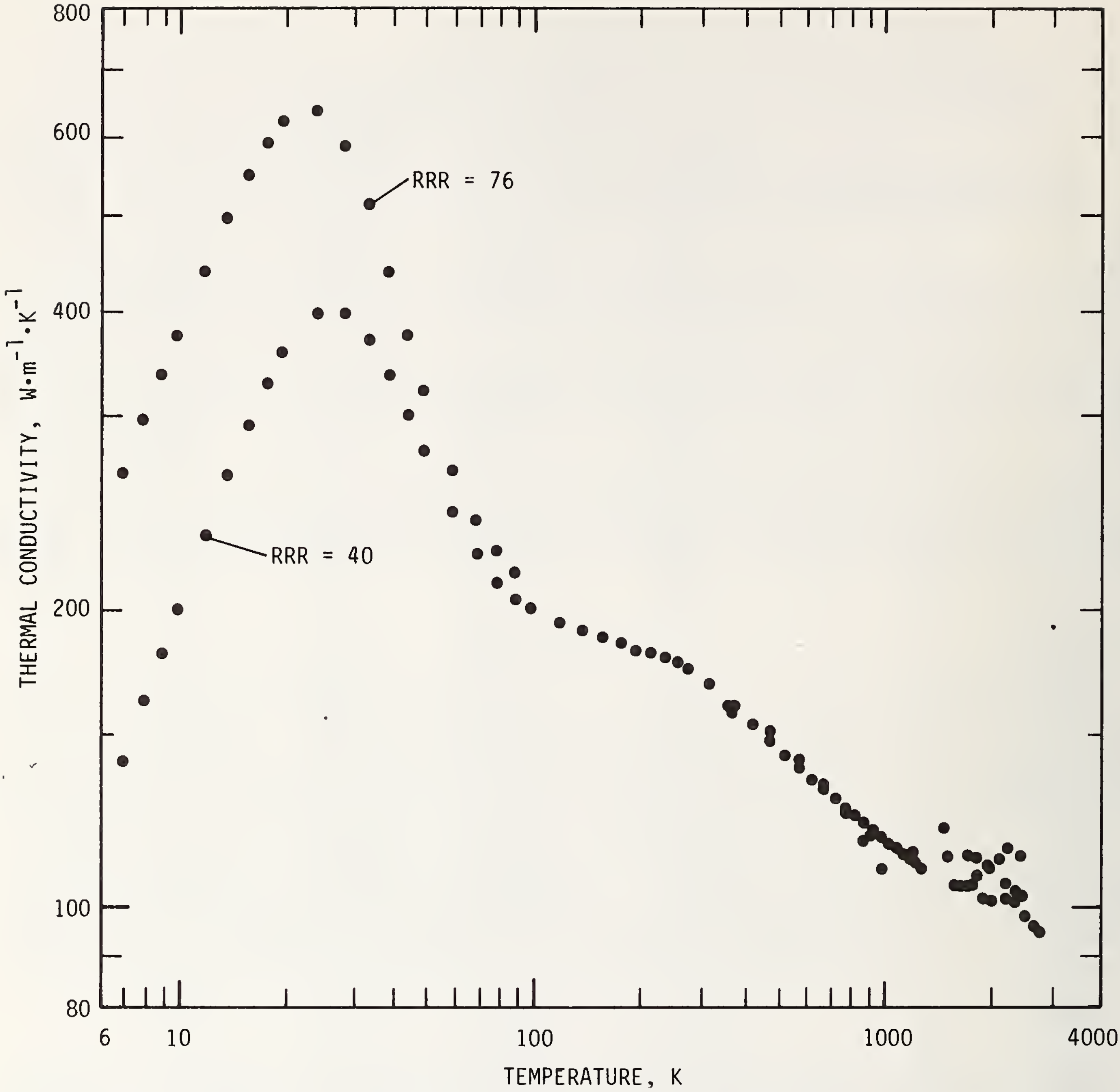

Figure 17. Primary thermal conductivity data for AFML arc cast and NBS sintered tungsten.

$$\alpha = 2.705 \times 10^{-7}$$
$$\beta' = 0.03982$$
$$n = 2.367$$
$$A = 35.06$$
$$B = -0.1689$$
$$\Theta_1 = 89.62$$
$$\Theta_2 = 370.4$$

with λ in $W \cdot m^{-1} \cdot K^{-1}$, T in helium, and ρ and ρ_o in $n\Omega \cdot m$. The deviations of the primary data [12,15,16,17] from this equation are illustrated in figure 18. Deviations with respect to the TPRC recommended curve [22] are included in figure 18. The differences are within the combined uncertainties of both sets of recommended values. However, above 2000 K these two curves are divergent. Further measurements are desirable to reduce the uncertainty in this range. Minges [13] presented two graphs illustrating high temperature thermal conductivity data as obtained from various sources and several prediction methods. We have added values as calculated from equation 12 to these graphs, figures 20 and 21, as a convenient method of illustrating further intercomparisons.

No attempt has been made in this formulation to separate the conductivity of tungsten into electronic and lattice conductivity components, and therefore, any effort to attribute the exponential terms to specific interaction mechanisms is not recommended. In the formulation of this and other potentially useful equations, we examined the possibility of simplifying the representation problem by subtracting the lattice conductivity as predicted by Moore et al. [16,21] and others. This potentially heuristic approach, although esoterically more satisfying, resulted in a more complicated rather than simplified function, and therefore this approach was abandoned.

We decided that equation 12 is the best representation of the arc cast and sintered tungsten. Values of thermal conductivity based on this equation and several values of ρ_o are listed in table 5. Values of Lorenz ratio as computed from the recommended values of ρ and λ are illustrated in figure 19. The uncertainties of the thermal conductivity data are estimated as 2% from 4 to 300 K, increasing to 5% at 2000 K, and rising to a maximum of 8% above 2000 K. Above 2000 K the deviations (figure 18) are larger than 8%, but it is noted that these data are converted diffusivity results. The direct thermal conductivity values are well within 8% of equation 12.

7. Discussion

The principal factors determining the validity of SRM data are measurement uncertainty and material variability. Measurement uncertainty is a highly speculative quantity, as evidenced by the fact that most experimentalists present optimistically low uncertainties for their own work. The best way to obtain realistic uncertainties is through round-robin type measurements using apparatus as basically different as possible. Such programs are expensive, and therefore not often performed. It is essential for standardizing laboratories to be involved in such programs because this forms the basis of essentially all other measurements. SRM's resulting from measurements by these standards laboratories make it possible for all other laboratories to perform measurements on a common basis.

Material variability is determined by the degree of control exercised during material production and the sensitivity of property values to physical and chemical variations in the material. As pointed out earlier, however, transport properties at low temperatures are strongly dependent on the detailed nature of the microscopic material structure. Because of this, it is necessary to make measurements to determine the property variability of a lot of material produced even under the best of conditions. The only truly foolproof method of determining material variability effects is to measure the property of interest on a random sampling of specimens from the entire lot of material. For a thermal conductivity SRM, this is costly and one must resort to less expensive characterization measurements and careful production record keeping to insure maximum benefit from a minimum number of measurements.

Fixed-point electrical resistivity, density, grain size, and hardness data have been compared earlier in the text. These comparisons for AFML arc cast and NBS sintered tungsten

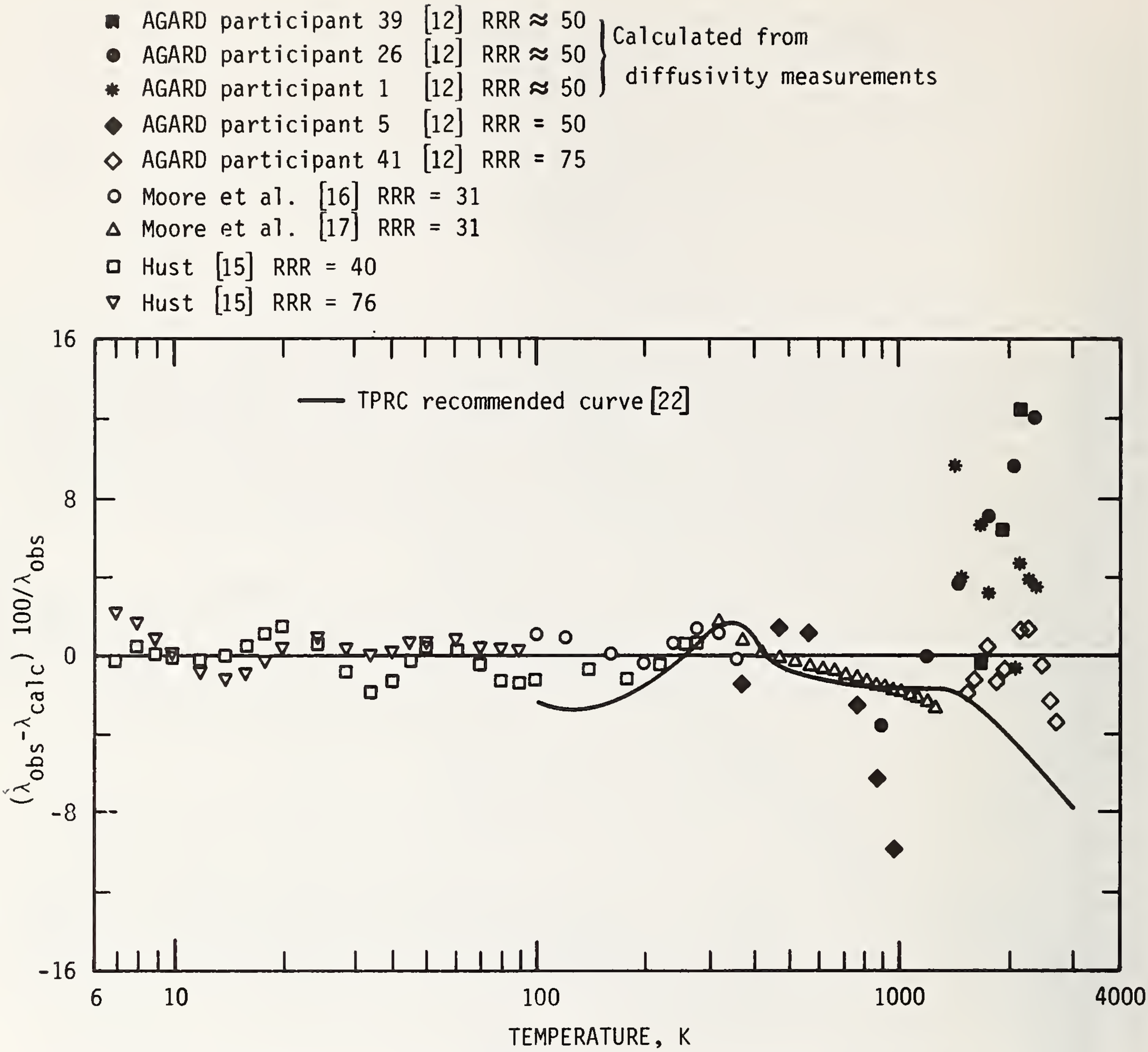

Figure 18. Thermal conductivity deviations of primary data from equation 12.

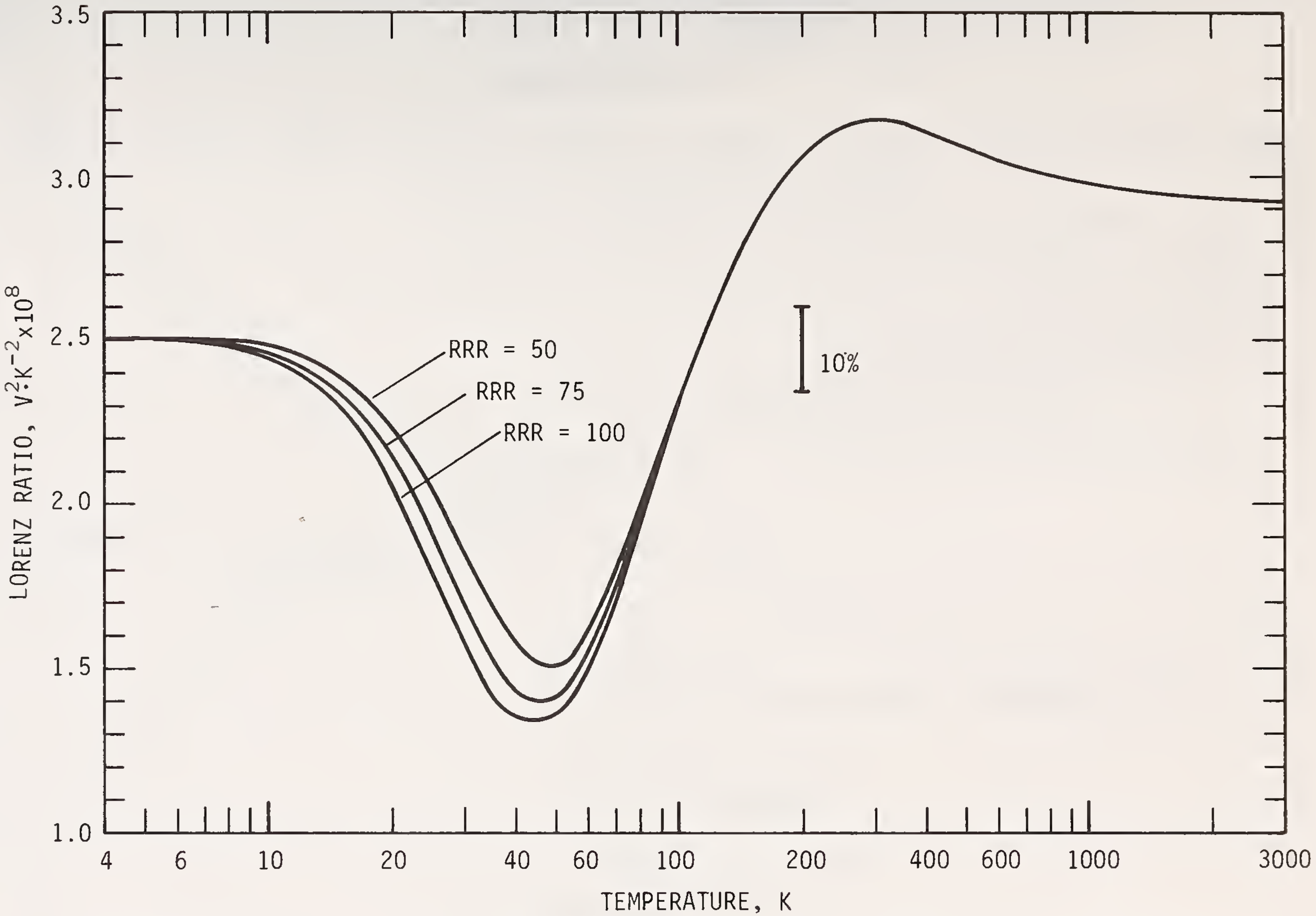

Figure 19. Lorenz ratio of tungsten as computed from recommended values of thermal conductivity (equation 12) and electrical resistivity (equation 8).

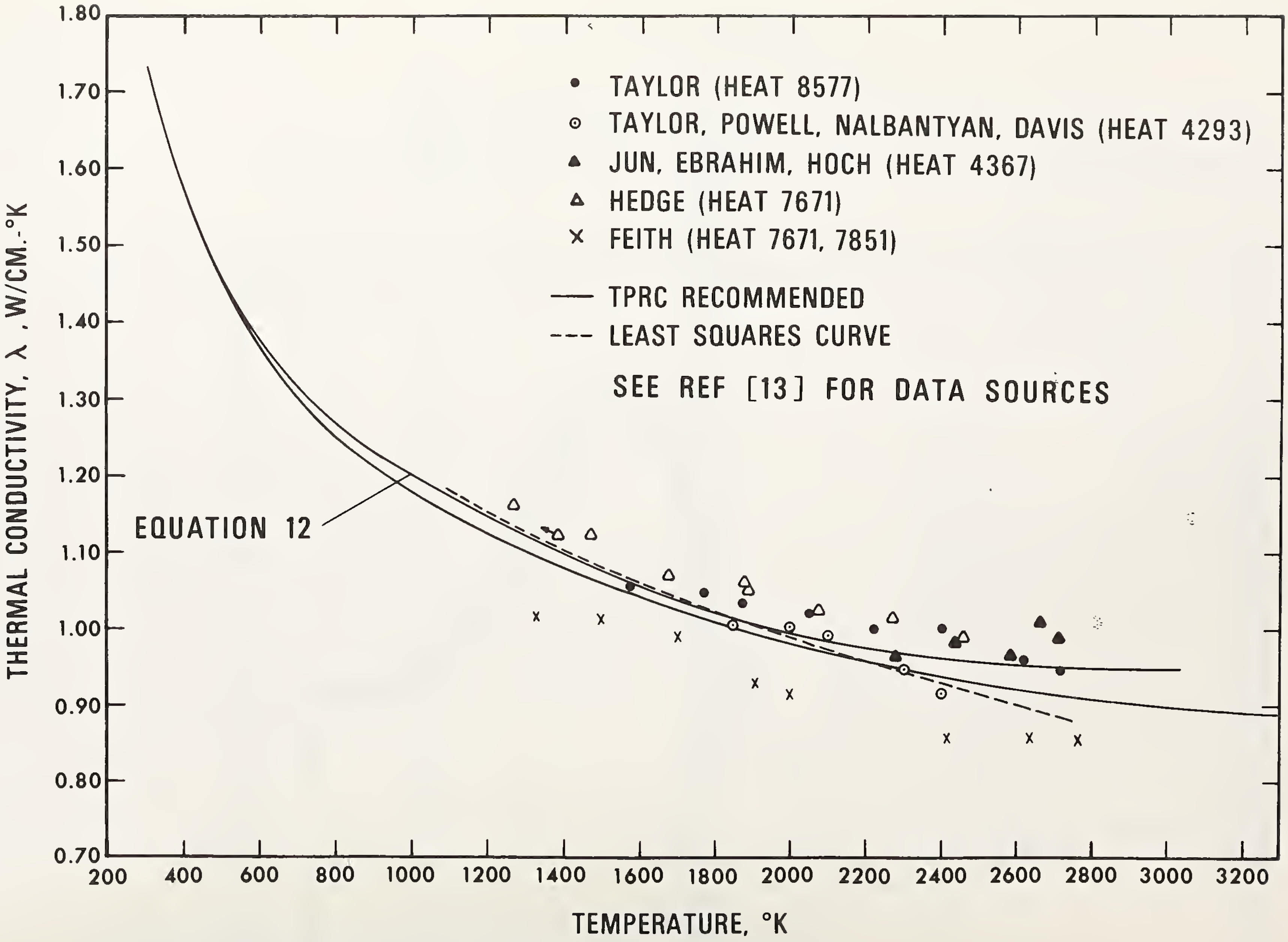

Figure 20. High temperature thermal conductivity data as presented by Minges [13] compared to values calculated from equation 12.

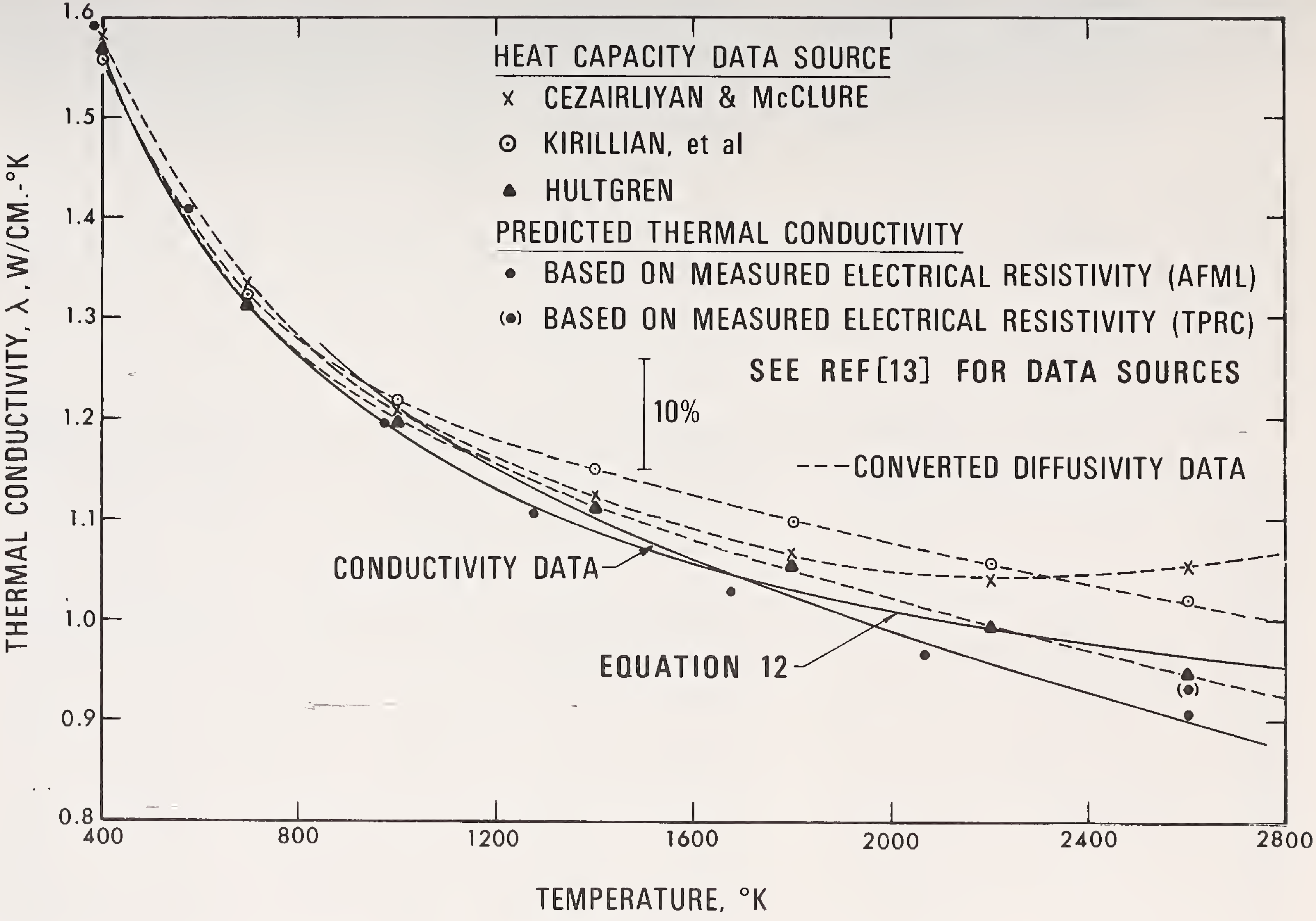

Figure 21. High temperature thermal conductivity values obtained from thermal diffusivity and electrical resistivity data as presented by Minges [13] compared to values calculated from equation 12.

Table 5. Recommended Thermal Conductivity Values for AFML Arc Cast and NBS Sintered Tungsten for Several Values of RRR

	Thermal Conductivity ($Wm^{-1}K^{-1}$)		
Temperature (K)	RRR = 50	RRR = 75	RRR = 100
4	103	154	205
6	155	231	306
8	206	306	404
10	255	377	496
12	302	444	580
14	345	503	652
16	384	553	709
18	417	591	748
20	443	618	769
30	468	585	667
40	384	438	470
50	305	330	345
60	260	275	283
70	236	245	251
80	221	229	232
90	212	218	221
100	206	211	213
120	199	202	204
140	194	197	199
160	191	194	195
180	189	190	191
200	186	187	188
250	178	180	180
300	171	172	172
350	163	164	164
400	157	157	158
450	151	151	152
500	146	146	147
600	138	138	139
700	132	132	132
800	127	127	127
900	123	123	123
1000	120	120	120
1200	114	114	114
1400	110	110	110
1600	107	107	107
1800	105	105	105
2000	102	102	102
2200	101	101	101
2400	99	99	99
2600	98	98	98
2800	97	97	97
3000	97	97	97

show that significant material variability effects exist at low temperatures (below 90 K). At higher temperatures, the effects of material variability are less well defined because of the larger-than-desirable measurement uncertainty, especially above 2000 K. The present analysis demonstrates that the effects of material variability can be accounted for by residual resistivity characterization.

Minges [13] and Fitzer [12] have indicated that irreversible effects may occur due to high temperature use of these SRM's (above 2000°C). This conclusion is based on measurements using large diameter sintered tungsten specimens produced early in the AFML-AGARD program. The evidence suggests that this occurred primarily due to the incomplete sintering of that lot of sintered tungsten. This is not to say that irreversible effects are totally absent in these lots of tungsten. It would be surprising if such changes did not occur; however, we believe that they will be within the previously mentioned uncertainties.

Although the SRM's described in this paper are considered quite adequate for engineering use, improvement in the accuracy and credibility of the values presented could be accomplished with additional measurements. Through its use as an SRM, this material will be measured by other laboratories. The resulting data will be compiled and, when sufficient reduction in uncertainty is achieveable, the recommended values will be updated. Anyone measuring this material with an absolute method is urged to make the data available to the author.

8. Summary

Recommended values of electrical resistivity and thermal conductivity for two specific lots of tungsten, AFML arc cast and NBS sintered tungsten, have been presented for the range 4 to 3000 K. These recommended values are based on an analysis and correlation of various characterization data, primarily those of NBS and AFML-AGARD. The semi-empirical functions chosen as the basis for the recommended values are inherently smooth, and therefore may be extrapolated somewhat without fear of strong divergence from true physical behavior. Although, it is believed that these functions are applicable to a wide range of commercially available tungsten, the intent is to represent only the two specific lots of interest here. The data presented are for ambient dimensions, uncorrected for thermal expansion.

With an anneal of 2000°C for one hour in vacuum and a residual resistivity measurement, the uncertainties of the recommended values are estimated as follows:

	Thermal Conductivity	Electrical Resistivity
4-300 K	2%	2%
300-2000 K	2%-5%	2%
Above 2000 K	5%-8%	2%

The above anneal and residual resisitivity measurement must be done after the specimen has been machined to size and completely readied for use.

If the specimens are to be used only at high temperatures (above ambient), the residual resistivity measurement is unnecessary, at the expense of a slight increase in uncertainty. An anneal of 2000°C for one hour will produce specimens of nominal RRR = 75 and the corresponding columns of tables 4 and 5 are used. These specimens are not recommended for use below 90 K without a residual resistivity determination.

These SRM's are available in the form of rods from the Office of Standard Reference Materials, National Bureau of Standards, Washington, D.C. 20234. Available sizes are as follows:

Sintered tungsten:

SRM 730-S1S, 799-S1S	(0.32 cm dia, 5 cm long)
SRM 730-S2S, 799-S2S	(0.32 cm dia, 10 cm long)
SRM 730-S3S, 799-S3S	(0.32 cm dia, 20 cm long)
SRM 730-M1S, 799-M1S	(0.64 cm dia, 5 cm long)
SRM 730-M2S, 799-M2S	(0.64 cm dia, 10 cm long)
SRM 730-M3S, 799-M3S	(0.64 cm dia, 20 cm long)

Arc cast tungsten:

SRM 730-S1A, 799-S1A	(0.64 cm dia, 5 cm long)
SRM 730-S2A, 799-S2A	(0.64 cm dia, 10 cm long)
SRM 730-S3A, 799-S3A	(0.64 cm dia, 20 cm long)
SRM 730-MA	(0.83 cm dia, 5 cm long)
SRM 730-LA	(1.02 cm dia, 5 cm long)
SRM 730-LXA	(1.27 cm dia, 5 cm long)

Multiple continuous lengths may be obtained by special order. The larger diameter arc cast specimens are in relatively limited supply.

9. Acknowledgements

This work has been in progress for several years, and over this period of time many people have assisted in various ways. R. L. Powell was instrumental in suggesting the importance of the work and in formulating the preliminary program. We wish to thank R. E. Michaelis of NBS, OSRM and M. Minges of AFML for supporting this work and for many helpful discussions. The cooperation of R. E. Taylor of Purdue University, in performing the high temperature cycling of the NBS sintered tungsten, is also acknowledged.

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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.)*

A historical review of the development of thermophysical Standard Reference Materials, SRM's, is given and selection criteria of SRM's are listed. Thermal conductivity and electrical resistivity data for arc cast and sintered tungsten are compiled, analyzed, and correlated. Recommended values of thermal conductivity (SRM 730) and electrical resistivity (SRM 799) for these lots of tungsten are presented for the range 4 to 3000 K. These values are based on low temperature NBS measurements and higher temperature measurements by participants of the AFML-AGARD project. The uncertainty of the thermal conductivity values below ambient is 2% and rises to 5% between ambient and 2000 K. Above 2000 K the uncertainty rises to a maximum of about 8%. The uncertainty of the electrical resistivity values is 2% over the entire temperature range.

17. KEY WORDS *(six to twelve entries; alphabetical order; capitalize only the first letter of the first key word unless a proper name; separated by semicolons)* Electrical resistivity; high temperature; Lorenz ratio; low temperature; Standard Reference Materials; thermal conductivity; thermopower; tungsten.

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