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Accuracy of Small Diameter Sheathed Thermocouples for the Core Flow Test Loop

R. L. Anderson
T. G. Kollie

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Contract No. W-7405-eng-26

INSTRUMENTATION AND CONTROLS DIVISION

ACCURACY OF SMALL DIAMETER SHEATHED THERMOCOUPLES FOR THE CORE FLOW TEST LOOP

R. L. Anderson and T. G. Kollie*

*Present address is Union Carbide Corporation, Y-12 Plant, P.O. Box Y, Oak Ridge, TN 37830

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Date Published: April 1979

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ACCURACY OF SMALL DIAMETER SHEATHED THERMOCOUPLES FOR THE CORE FLOW TEST LOOP

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HIGHLIGHTS

The potential errors in temperature measurements employing small diameter (0.5 mm), metal sheathed thermocouples were analyzed for the Core Flow Test Loop experiments. The error sources considered were extension lead wires, reference junctions, the data acquisition system, calibration of the sensors, decalibration of the sensors, thermal shunting, electrical shunting, electrical leakage, and electrical noise.

Because of the relatively small wire sizes (<0.1 mm diameter), the behavior of the small diameter thermocouples could not be extrapolated from the known behavior of larger diameter thermocouples, particularly above about 900°C.

Electrical shunting and leakage at these higher temperatures can cause large errors, their magnitude depending on the temperature profile through which the thermocouple passes. An analytical model was developed to calculate these errors, and the results are in excellent agreement with experimental results.

Size effects also play a large role in the decalibration of small diameter sheathed thermocouples. The sheath material is an important factor that causes decalibration above 900°C. Nicrosil vs Nisil thermocouples -- new alloys developed to replace type K alloys -- decalibrated more extensively than the type K materials in a sheath of either type 304 stainless steel or Inconel 600.

Small diameter thermocouples with platinum based thermoelements were even more sensitive to the choice of sheath material than the base metal alloys. Platinum based thermocouples with platinum alloy sheaths were superior to base metal thermocouples, particularly at higher temperatures (above 900°C).

Finally, the predictable temperature measurement errors were combined to yield an estimate of the maximum overall errors as a function of temperature.

1. INTRODUCTION

This report summarizes the research and development on 0.5-mm-diameter, compacted, metal sheathed thermocouples carried out in the Metrology Research and Development Laboratory (MRDL) of the Instrumentation and Controls Division at the Oak Ridge National Laboratory over a period of about two years. The objectives of this research effort have been: to identify and analyze the sources of temperature measurement errors in the use of 0.5-mm-diameter sheathed thermocouples to measure the surface temperature of the cladding of fuel-rod simulators in the Core Flow Test Loop (CFTL) at ORNL; to devise methods for reducing or correcting for these temperature measurement errors; to estimate the overall temperature measurement uncertainties; and to recommend modifications in the manufacture, installation, or materials used to minimize temperature measurement uncertainties in the CFTL experiments.

Accurate temperature measurements are essential to meet the objectives of the CFTL experiments. Both heat transfer calculations and structural integrity evaluations require reliable temperature measurements for interpretation and for comparison of the CFTL results with analyses and measurements from other laboratories. Temperature measurement sensors are the most numerous type of sensor in the CFTL, and these include both thermocouples and resistance thermometers.

Research to determine the characteristics of small diameter thermocouples is necessary for several reasons. Because of space limitations inside the ∿7.2-mm-diameter fuel-rod simulators of the CFTL, the diameter of the thermocouples for measurement of the temperatures of the cladding on the simulators cannot be larger than 0.5 mm. Since such small diameter, sheathed thermocouple materials have become available only recently, little performance data has been accumulated. Furthermore, the CFTL experiments require measurements to higher temperatures than previously required -- sometimes to 1370°C, the melting point of the stainless steel cladding on the simulators. (The single-rod and the multirod burst tests, for example, reach similar temperatures;[1] however, the duration of a test is only about 20 sec, which is not sufficient time for significant thermocouple decalibration to take place.)

In addition, the requirements for temperature measurement accuracy for the CFTL are more stringent than previously required in large scale engineering experiments.[2] The limits of temperature measurement uncertainties for the thermocouples on the fuel-rod simulators have been specified[3] as ±8°C to 800°C and ±15°C above 800°C.

Because the diameters of the thermoelement wires, as well as the separation between the wires or between the wires and the sheaths in the 0.5-mm-diameter thermocouple assemblies are so small, the mechanical, electrical, and chemical behaviors of these thermocouples are different from those of larger diameter thermocouples. Above 600°C, the thermoelectric properties of these thermoelements may be altered by a variety of causes. For example, chemical reactions may occur at the interface between the thermoelements and their surrounding materials, or the thermoelements may be contaminated by diffusion of impurities from the insulation or by diffusion of constituents of the sheath material into the wires. The effects of such reactions or contamination on the thermoelectric properties of the thermoelements will increase as the products of the reactions and the impurities penetrate to the core of the wires, the penetration time being a function of the wire thickness and temperature. In a 3-mm-diameter sheathed thermocouple, for example, the thermocouple wire diameters are ∿0.5 mm, but in a 0.5-mm-diameter sheathed thermocouple, the wire diameters are 0.1 mm or less. Thus, the decalibration of a 0.5-mm-diameter thermocouple is more rapid and severe at high temperatures than in a larger diameter thermocouple.

Nine major sources of temperature measurement errors relevant to thermocouple thermometry were evaluated for the CFTL fuel-rod simulator, small diameter thermocouples. These sources of error are listed in Table 1.

2. DEFINITIONS OF ERROR TERMINOLOGY

Since the central theme of this paper is the identification and analysis of temperature measurement errors, it will be worthwhile to digress, momentarily, to define the terms and the specific way in which they are used. These include error (random and systematic), uncertainty,

Table 1. Error sources in CFTL thermocouple thermometry

Source	Discussed in Section
1. Extension lead wires	4.1
2. Reference junctions	4.2
3. Data acquisition system	4.3
4. Calibrations	4.4
5. Decalibration	5
6. Thermal shunting	4.5
7. Electrical shunting	4.6
8. Electrical leakage	4.7
9. Electrical noise	4.9

deviation, tolerance, precision, and terms derived from these. This discussion relies heavily on the work of Eisenhart[4] and Ku.[5,6]

Error is the difference between a measured value and the "true" value of a physical property. There is an uncertainty associated with both a measured value and the true value. The *uncertainty* of a reported value is estimated by "stating credible limits to its likely inaccuracy."[4]

The true value of a quantity is unknowable in an absolute sense, since there is always some uncertainty associated with even the most accurate determinations, due to noise or ultimately the uncertainty principle. "Standard" values of physical quantities are adopted, along with an estimate of their uncertainty, by international agreement after careful examination of the available experimental work. An adopted value is usually the average of several independent determinations, and is subject to revision as more accurate determinations become available. Thus, it is of utmost importance that the standard values used in critical measurements be adequately referenced so that when more accurate standard values become available, the experimental results can be corrected and thus retain their validity.

Just as a standard value would be relatively useless without an estimate of its associated uncertainty, an "experimental" value reported

without a statement of estimated uncertainty is also worthless. A statement of uncertainty must include the estimated uncertainties contributed by the measurement system as well as those determined from examination of the experimental data. A propagation of error treatment[5] is frequently used to make an estimate of the contribution of several error sources to the uncertainties of experimentally determined values.

The ways in which estimates of errors can be combined depend on the types of errors involved. There are two general classifications of errors: random and systematic. Any experimental measurement will have associated with it both random and systematic errors, although in some cases one or the other may be so small that it may be neglected with respect to the other. Eisenhart recommends that the estimates of the random and systematic errors be reported separately in an explicit statement.[4]

The estimate of the standard deviation is frequently given as the estimate of the random error. At the same time, the confidence limits of the estimate are also given, so that the probability that an individual measurement will not deviate from the group average is 63% for 1 sigma, 95% for 2 sigma, and 99% for 3 sigma, where sigma is the estimate of the standard deviation of the mean of a large number of experimental measurements. If an experimental quantity is dependent on the combination of several measured values, and if the random errors associated with each value are *independent* and normally distributed, then the estimate for the random error of the final quantity is given by the RMS value of the sum of the squares of the individual independent random errors.

2.1 Systematic Errors

The estimate of systematic errors is more subjective and depends to some extent on the perspicacity and honesty of the observer. Systematic error can be both constant and changing. Constant systematic errors can often be corrected for if their effect is known. In a hypothetical example, suppose that a steel measuring tape was used to determine a length on two different days. The measurements made on the first day

had a random error of 0.1%. On the second day, a second set of determinations had a random error which was also 0.1%, but the average lengths of the same object measured on the two different days differed by 1%. An investigation into the cause of this disagreement disclosed that the ambient temperature on the two days differed by 5°C, and the effect on the steel tape due to the thermal expansion of the tape could account for the discrepancy. Thus, by measuring the temperature along with the length, the systematic error due to temperature could be corrected for and the overall uncertainty of the length measurements reduced. Of course, there might still be a systematic error due to a stretching of the tape, which could affect both sets of measurements and might not be detected. If the tape were stretched inelastically, then this could be detected by a recalibration of the measuring tape. If the stretching of the tape were elastic, however, this effect might not be detected by a recalibration unless the tape were suspended in the same way as in the original experiments. Thus, simple reproducibility, or *precision*, while necessary for accurate measurements, is not sufficient to guarantee accuracy. Even careful consideration of all known sources of systematic errors may not reveal all of the factors which contribute. In particular, as advancing technology makes possible more sensitive and accurate measuring instruments, factors which were formerly insignificant (and thus ignored) can become the limiting factors with the introduction of improved instrumentation. A danger lies in that these factors may not be recognized.

Selection of an appropriate method to combine estimates of systematic errors from several sources is fraught with uncertainty. Eisenhart[7] mentions five different methods ranging from "much too daring" to "a wee bit conservative." Ku[6] recommends that since there are no formally accepted ways to estimate systematic errors or to combine them, one should discuss these errors in sufficient detail to enable others to make their own judgments. In this paper we attempt to do this for the temperature in CFTL fuel-rod simulators made with 0.5-mm-diameter, internally attached, sheathed thermocouples. In combining the errors from the several sources, we have used a conservative approach of simple algebraic addition to yield overall error bounds. This approach gives an overall basis for comparison

of the relative magnitudes of the uncertainties from the different sources of temperature measurement errors. Actual experience with operating systems will make more accurate estimates of the probable errors possible.

Deviation is the difference between the measured value and a standard value.

Tolerance is a permissible uncertainty, and limit of tolerance is the maximum permissible uncertainty in a measured quantity.

2.2 Reporting the Estimated Errors of Experimental Results

Experimental results should be reported with an explicit statement of the estimates for both the random and systematic errors. Eisenhart[4] recognizes four different cases:

1. Systematic and random errors both negligible,
2. Systematic error not negligible, random errors negligible,
3. Neither systematic nor random errors negligible,
4. Systematic errors negligible, random errors not negligible.

In the discussion of thermocouple errors which follows, we will discuss the types of errors involved and how they should be treated.

3. REVIEW OF THERMOCOUPLE FUNDAMENTALS

Before the errors in CFTL thermocouple thermometry are discussed in detail, some fundamentals of thermocouple thermometry will be reviewed briefly, starting first with the basic equation for the thermoelectric effect. The emf output of a thermocouple, E, is given by the line integral along the path ℓ, as follows:

$$E = -\int^{\ell} S_i \, \vec{\nabla} T \cdot \vec{dx} \, , \tag{1}$$

where S_i is the absolute thermoelectric power (dE/dT of the thermoelement i along the path ℓ, the Seebeck coefficient), and $\vec{\nabla} T$ is the temperature gradient at x along the path ℓ. This is illustrated in Fig. 1a.

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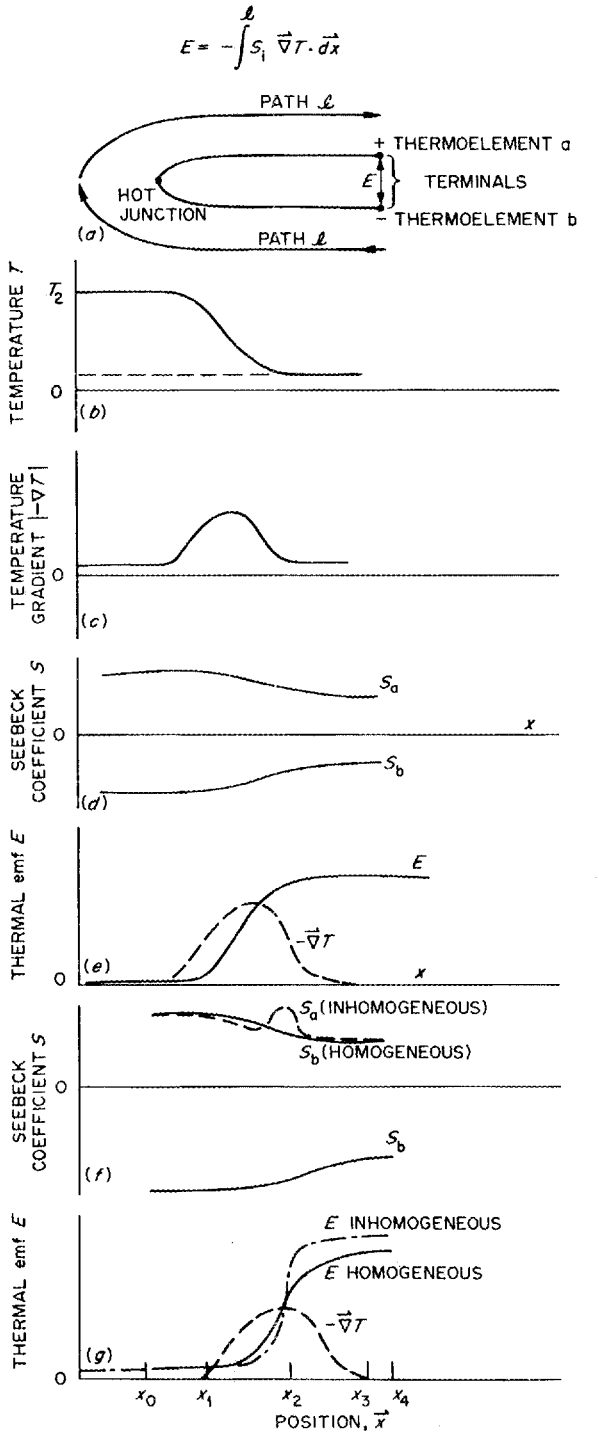

Fig. 1. Effects of thermal gradient and inhomogeneities on the emf output of a thermocouple.

If both thermoelements are homogeneous, that is, if S_i is not a function of position x (but may be a function of temperature), Eq. (1) is often simplified to

$$E = \int_{T_1}^{T_2} (S_a - S_b)\, dt \ . \tag{2}$$

This equation is an oversimplification, however, because it obscures the fact that the source of the thermocouple emf is in the temperature gradient, not the junction. The junction serves only to complete the electrical circuit. Furthermore, this equation is based on an assumption that the thermoelements are homogeneous. This is a poor assumption because chemical and physical changes can produce inhomogeneities after a thermocouple is put in service or even during its calibration, particularly in 0.5-mm-diameter thermocouples. For instance, with a simple temperature profile (Fig. 1b), suppose there is a corresponding temperature gradient (Fig. 1c). If the thermoelements in Fig. 1a are homogeneous, the corresponding Seebeck coefficients can be illustrated by Fig. 1d, and their gradual change with temperature represents their temperature dependence. Finally, the emf output of the homogeneous thermocouple [Eq. (1)] is shown in Fig. 1e.

If, however, one of the thermoelements is inhomogeneous in a portion of the thermocouple located in the temperature gradient, the emf output will not be as shown in Fig. 1e. For example, with the Seebeck coefficient of the inhomogeneous thermoelement represented by the dashed line in Fig. 1f, the resultant emf of the inhomogeneous thermocouple is shown in Fig. 1g, where the emf of the homogeneous thermocouple of Fig. 1e is shown for comparison. In this case, the postulated inhomogeneity has produced an increase, ΔE, in the emf output, with an attendant increase in the temperature, as indicated by the thermocouple ΔT, and this increase is a temperature measurement error. Depending on the cause, however, the development of inhomogeneities can either increase or decrease the Seebeck coefficient locally, resulting in either positive or negative temperature measurement errors. These errors are systematic and can be of the order of hundreds of degrees in extreme cases.

If thermoelement "a" were to develop an inhomogeneity in the portion of the thermocouple located around x_o, as shown in Fig. 2, with the temperature profile identical to that of Fig. 2a and the temperature gradient the same as that of Fig. 2b, then, $\vec{\nabla}T$ in the region of the inhomogeneity would equal zero. Thus, ΔE would also equal zero, since from Eq. (1) an inhomogeneity in a region of uniform temperature would have no effect on the emf output of the thermocouple, and, consequently, there would be no temperature measurement error.

Examples of both cases presented in Figs. 1 and 2 will be illustrated by experimental data in Sect. 5.

4. TEMPERATURE MEASUREMENT UNCERTAINTIES

4.1 Extension Lead Wires

In precision thermometry, the thermocouple wires are connected directly to a reference junction whose temperature is precisely known and/or controlled. That is, the thermocouple wires extend from the measuring junction to the reference junction in continuous, unbroken lengths without the intervention of extension lead wires or connectors.

Such practice is impractical in large scale engineering experiments such as the CFTL, because more than 200 thermocouple connections to the data system must be made and broken when the test apparatus is exchanged. At least six such exchanges will be made during the course of the CFTL experiments. In addition, the lengths of the small diameter thermocouples must be minimized to reduce the total electrical resistance of the thermocouple circuits. For these reasons, the thermocouples will be connected using extension lead wires.

Extension lead wires and the contacts in the thermocouple connectors are made from pairs of alloys which approximately match the thermoelectric properties of a specific thermocouple type (K, S, etc.) over a limited temperature range, typically 0 to 200°C. Type K extension wire, for example, is often made from type K alloys which for some reason or another do not meet the requirements for type K thermocouples over the entire range from 0° to 1260°C, but do provide a reasonable match of emf vs temperature from 0° to 200°C. The most widely used standard for the

allowable error in the match between the thermoelectric properties of the extension lead wire materials and the corresponding thermocouple types is given in the American National Standard for Temperature Measurement Thermocouples, ANSI MC96.1.[8] The tolerances are expressed as maximum allowable errors which would result in a temperature measurement due to a mismatch in thermoelectric properties between the extension lead wires and the thermocouple. For example, the limit of error is ±2.2°C (0 to 200°C) for type K (Chromel-P versus Alumel) and ±6.7°C (0 to 200°C) for type S (90% platinum-10% rhodium versus platinum).

Within a particular lot of extension wire pairs, these should be relatively constant, and therefore, systematic errors.

We have measured the deviations in a temperature range from 0 to 140°C of a random sampling of type S extension wire obtained from several sources at ORNL. Thus, the samples were from several manufacturers and came from lots of materials purchased over a period of about ten years. The range of deviations was within the ANSI tolerance stated in the preceding paragraph and agreed with the results of Starr and Wang.[9] The results (Fig. 3) show that calibration can reduce the uncertainty due to the systematic errors in the extension wires to ±0.1°C or less. This is also true for type K extension wires: calibration can reduce their measurement uncertainties to a few tenths of a degree Celsius.[10] In other words, calibration of each lot of extension lead wires used can reduce the systematic errors to the level of the random errors in the thermocouple measurement system.

4.2 Reference Junctions

The output of a homogeneous thermocouple unaffected by the other sources of error listed in Table 1 -- a condition almost never achieved -- is determined by the difference in temperature between the measuring junction at temperature T and the reference junction at temperature T_o. Therefore, an uncertainty in T_o will directly cause an uncertainty in the measured temperature. For a large group of thermocouples, such as in the CFTL, it is practice to install a zone box heated to constant temperature to establish T_o. Usually a zone box is thermostatically controlled at

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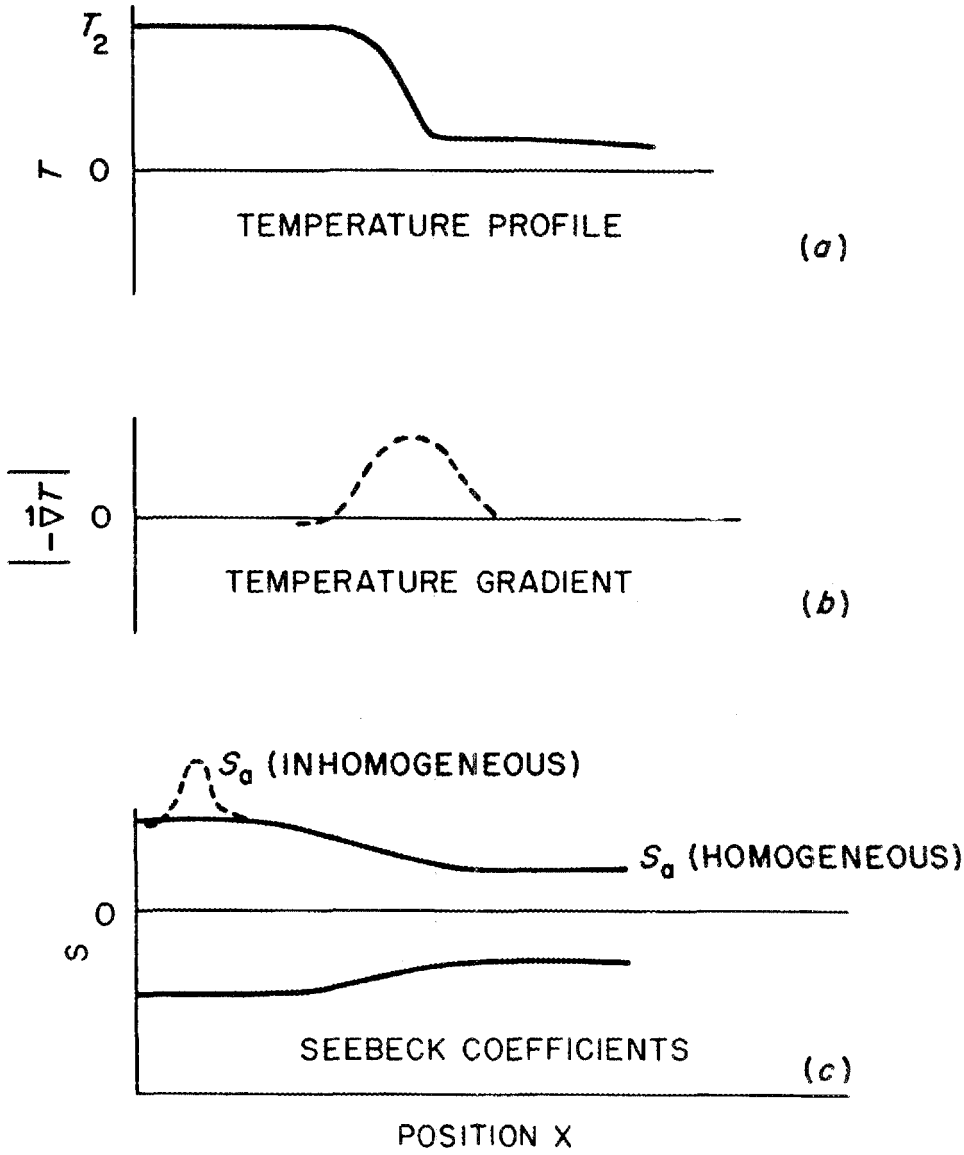

Fig. 2. Illustration of how the location of a temperature gradient can mask the decalibration of a thermocouple. This is a situation which is likely to arise if the thermocouple has decalibrated in use and is removed and inserted in a calibration furnace in an attempt to recalibrate it. The decalibrated section near X_o is not detected because $\vec{\nabla}T = 0$ in that region.

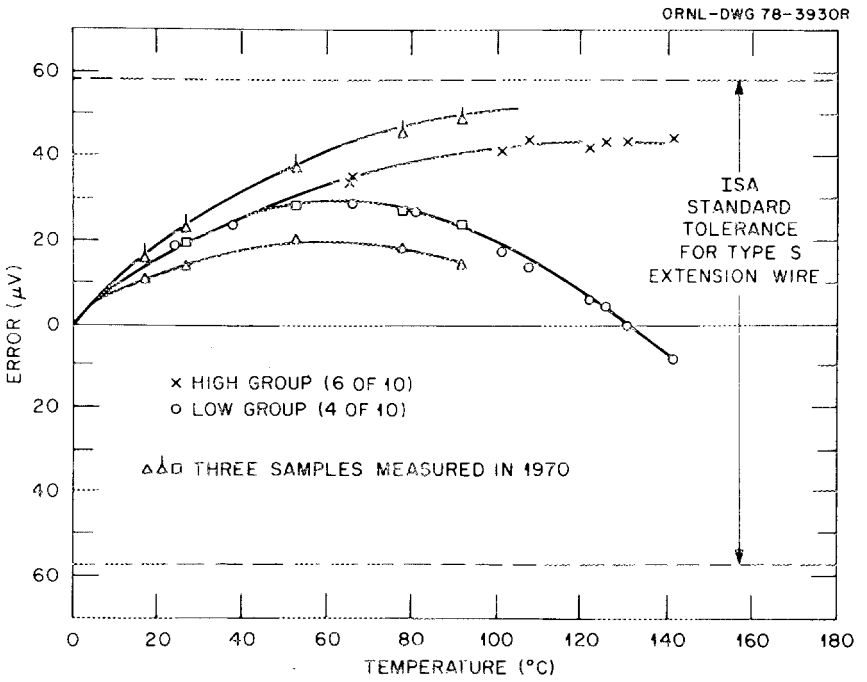

Fig. 3. Deviations of ten samples of type SX extension wire obtained from various sources at ORNL from 0 to 140°C.

65°C, and this temperature is monitored during an experiment by a thermometer, such as a resistance thermometer, that does not require a reference junction. The uncertainty attributable to a lack of uniformity of temperature within a zone box is about ±0.2°C. This will be a constant systematic error if the temperature gradients are small and constant within the zone box and the box remains in thermostatic control.

4.3 Data Acquisition System

The errors due to the measuring system in measuring the thermal emf of a thermocouple can vary from the equivalent of a few millidegrees with high quality laboratory potentiometers to tens of degrees with high speed data loggers. Measurement systems designed for steady state emf measurements can be, in general, more accurate than systems designed for transient measurements of emf. To paraphrase the Heisenberg uncertainty principle, the product of the *speed* of data acquisition and the *accuracy* of data acquisition is approximately constant.

Because the measurement of transient temperatures is of great importance in the CFTL, the data acquisition system proposed for the CFTL will have a 20-kHz analog-to-digital (A/D) converter with 10-, 20-, 40-, and 80-mV ranges. The uncertainty of this converter is ±0.25% of the full-scale range for each range and is a random error. The CFTL data acquisition system will be provided with self-calibration checks, so the systematic errors in the measuring system can be reduced to the level of the random errors. These uncertainties will result in the temperature measurement errors listed in Table 2. Because of the ±0.25% uncertainty for each range of the converter, the greater emf output of the type K thermocouple has no particular advantage over type S in some of the most important temperature ranges to be used in the CFTL, namely, 485 to 1035°C.

4.4 Calibrations

Temperature is hotness, and values of temperature are measures of hotness. To compare values of temperature, one must refer to a scale of temperature, and many different scales are in use: Fahrenheit, Rankine,

Table 2. Errors in data acquisition due to analog-to-digital converter[a]

Temperature Range (°C)	emf Range for Type K (mV)	Error for Type K[b] (°C)	emf Range for Type S[c] (mV)	Error for Type S (°C)
0 - 250	10	±0.6	10	±3.2
250 - 485	20	±1.2	10	±2.7
485 - 965	40	±2.4	10	±2.4
965 - 1035	80	±5.0	10	±2.2
1035 - 1372	80	±5.0	20	±4.2
1372 - 1768	---	---	20	±4.2

[a]Data by T. G. Kollie.[5]

[b]Type K is a Chromel-P versus Alumel thermocouple.

[c]Type S is a 90% platinum-10% rhodium versus platinum thermocouple.

Centigrade, Celsius, Kelvin, etc. The scale of temperature which has widest usage in scientific and engineering work is the International Practical Temperature Scale of 1968 (IPTS-68)[11] adopted by the International Conference on Weights and Measures. This scale not only defines values of temperature for selected, reproducible fixed points, but also prescribes standard instruments and methods for realizing the scale.

All measurements of temperature in the CFTL should ultimately be referable to the IPTS-68. This will be accomplished by calibration of the temperature sensors. The calibration uncertainty of temperature sensors installed in the CFTL is, therefore, the uncertainty of the calibration of these sensors with respect to IPTS-68. The uncertainties in the measuring system and the reproducibility of the standards used in the Metrology Research and Development Laboratory of the Instrumentation and Controls Division do not contribute more than 0.2°C uncertainty for calibrations to 1400°C. The standard thermocouples in the metrology laboratory were calibrated by the U. S. National Bureau of Standards (NBS) with an uncertainty of ±1°C with respect to the IPTS-68. Because of the greater variability of the working sensors installed in the CFTL, the *initial* calibration uncertainty is not more than ±1°C to 1000°C, increasing to ±1.5°C at 1400°C. Additional uncertainties may be introduced during the fabrication of the fuel-rod simulators due to additional handling, cold-working, and annealing of the thermocouples.

Calibration errors are systematic errors, and since decalibration errors are treated separately, they are constant systematic errors.

4.5 Thermal Shunting

Thermal shunting is a broad category which includes all errors that result from the thermocouple not actually attaining the temperature of the location it is intended to measure.

A thermocouple, just as any other contacting temperature sensor, disturbs the temperature distribution of any object to which it is attached because the thermocouple has a finite size and conducts heat from (or to) the object. The thermocouple itself loses heat to (gains heat from) its surroundings by conduction. This heat transfer can cause

the thermocouple hot junction to be at a different temperature, higher or lower, than that of the object. This temperature difference plus any temperature change of the object due to the presence of the thermocouple, is a temperature measurement error, which is called a "thermal shunting" error.

Thermal shunting errors frequently result from the way in which the sensor is attached. Without good thermal contact, the junction of the sensor will not attain the temperature of the object. It is frequently necessary to electrically isolate the thermocouple from the object by inserting a thin sheet of insulating material between the junction and the object (Fig. 4). In this case, because of the various heat transfer processes, the temperature of the measuring junction will always be more or less than the temperature of the object, depending on the temperature of the surrounding media.

Thermal shunting errors in the CFTL will result from uncertainties in (a) the sensor location in the fuel-rod simulator; (b) the thermal contact of the sensor with the sheath of the simulator; (c) changes in the temperature distribution in the simulator because of the presence of the sensor; and (d) temperature variations along the length of the heater due to heater inhomogeneities, insulator density variations, and differences in thermal contact at the various interfaces.

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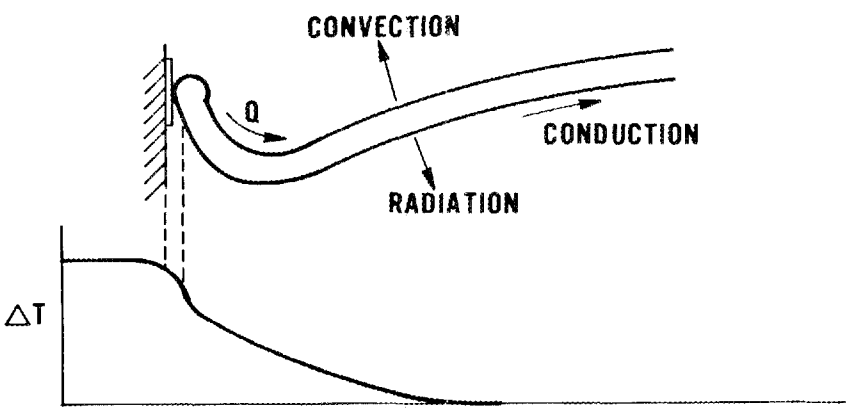

Fig. 4. Thermal shunting is affected by the wire size of the thermocouple, the relative thermal conductivities of the object and the thermocouple, the temperature of the surroundings, and the heat transfer coefficient of the surrounding medium.

In transient-temperature measurements, the effects of thermal shunting are intensified and can often be the dominant error. For very fast transients, the estimated probable error for the CFTL is 30°C.[12] Thermal shunting errors are nonconstant systematic errors, and their evaluation will require a combination of experimental tests and analytical modeling.

4.6 Electrical Shunting

The electrical resistance of all insulating materials used in sheathed thermocouple assemblies decreases exponentially with increasing temperature. The sorption water by compacted ceramic powder insulation in metal sheathed thermocouples can alter the electrical insulation resistance of such assemblies by several orders of magnitude. As an example, during repeated thermal cycling of a 0.5-mm-diameter thermocouple assembly between 25 and 1000°C, the insulation resistance changed as shown in Fig. 5.[13] Data recorded during the initial heating of the as-received

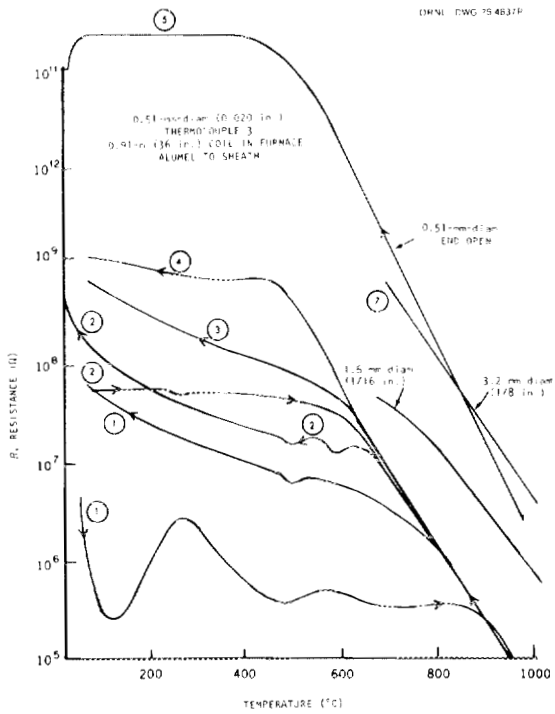

Fig. 5. Illustration of the changes in the insulation resistance measured on repeated thermal cycling. The insulation resistances of 1.6- and 3.3-mm-diameter assemblies are shown for comparison.

thermocouple are plotted as curve 1, which we believe to be the result of water adsorbed by the hygroscopic magnesium oxide (MgO) insulation. We believe that the sharp decrease in the insulation resistance at 100°C is due to the release of loosely bound water which had been physically adsorbed on the large surface area of the MgO.[14] The complete removal of water, which includes that chemisorbed on the MgO, requires temperatures of 900°C or more.[15] The improvement of the insulation resistance with thermal cycling shown by the curves marked 2, 3, 4 is probably due to "pumping" of the water vapor to cooler portions of the assembly or to removal of the water through chemical reaction with the sheath and thermoelements, or both.[16] To confirm this mechanism, the end of a thermocouple was cut open, and the open end was located within the hot zone. Curve 5 shows the insulation resistance measured during cooling from 1000°C. The open end of the thermocouple allowed the desorbed water vapor to escape from the heated length, resulting in the improved insulation resistance shown. As the thermocouple cooled below about 50°C, the insulation resistance began to decrease because moisture was absorbed from the air into the open end of the thermocouple. For comparison, measurements of the insulation resistance of 1.6-mm and 3.2-mm-diameter thermocouple materials are shown for temperatures between 700 and 1000°C.

Figures 6a, 6b, and 6c illustrate three different ways in which the lower insulation resistance at temperatures above ∿1000°C can cause errors in thermocouple thermometry.

In 0.5-mm-diameter thermocouples with ∿0.1-mm-diameter wires, the loop resistance of the thermocouple is large (∿140 Ω/m). The electrical resistance of the insulation decreases with increasing T, and at a sufficiently high temperature where the insulation resistance approaches the loop resistance, a significant loss of the thermocouple emf can occur because of electrical leakage through the thin insulation layer between the thermoelements, resulting in erroneous indications of temperature above about 1000°C.[13]

Figure 6a illustrates the most common way electrical shunting errors occur. If a thermocouple is inserted into a simple temperature profile

as illustrated, electrical shunting will occur in the portion of the thermocouple above ∿1000°C.

A second related effect is the creation of a "virtual junction" at temperatures above ∿1000°C. If a portion of the length of a thermocouple is located in a temperature profile such as shown in Fig. 6b, where a part of the thermocouple is hotter than the measuring junction, the thermocouple will indicate a temperature that is more nearly the temperature of the hottest part of the thermocouple — *not that of the measuring junction*. This phenomenon is shown schematically in Fig. 6b, and some experimental results are shown in Fig. 7. In the length of the thermocouple located in the part of the temperature profile with temperatures greater than 1000°C, the electrical insulation resistance is lowered, allowing a conduction path to be established between the thermoelements, thereby creating a "virtual" junction at this point. This effect has been observed in 0.5-mm-diameter thermocouples with no junctions. For example, an emf was measured on an open-circuited "thermocouple" which extended through a peak temperature of ∿1000°C that was 90% of the emf expected from a close-circuited thermocouple with its measuring junction at that temperature. The effect is not so great with larger diameters.

Electrical shunting errors are temperature dependent, systematic errors.

4.7 Electrical Leakage

A third effect observed during a decrease in insulation resistance is caused by leakage of small dc currents from the heating element of the fuel rod simulator into the thermocouple circuit (Fig. 6c). This effect was measured by passing a small dc current long the sheath of one m of a 0.5 mm diameter thermocouple assembly heated to 1000°C or greater, resulting in temperature measurement errors of hundreds of degrees Celsius for a few milliamperes of dc current on the thermocouple. However, the errors in a CFTL fuel-rod simulator would not be nearly so large as this. The driving force for the leakage current is the voltage drop along the sheath of the thermocouple, the error introduced is strongly dependent on this sheath resistance. In the CFTL fuel-rod

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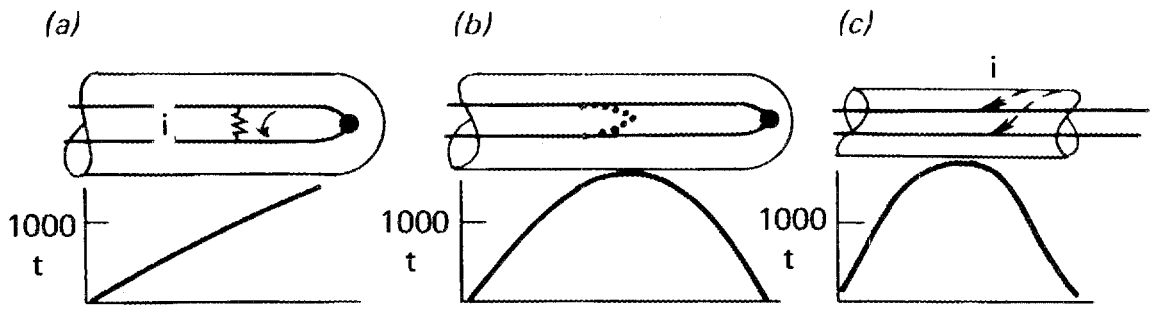

Fig. 6. Three types of temperature measurement errors which can occur because of the reduced electrical insulation resistance of the insulant in a sheathed thermocouple assembly: (a) shunting of the electrical signal; (b) creation of a virtual junction which tends to make the thermocouple indicate the hottest temperature through which it passes; and (c) leakage of currents on the sheath onto the thermocouple wires. (Since the thermoelements are normally of different resistances, this results in a net emf at the output of the thermocouple, which can be positive or negative depending on the direction of the leakage current.)

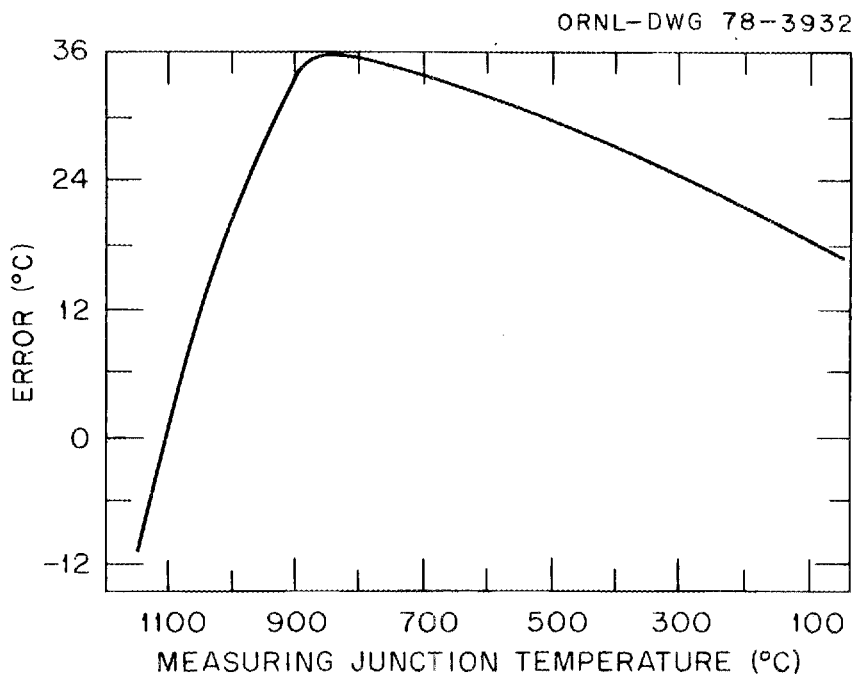

Fig. 7. Temperature measurement error measured when the thermocouple passes through a hot zone held at 1200°C and the temperature of the measuring junction is varied.

simulator, the sheath resistance of the thermocouple is shunted by the clad resistance of the rod, which results in an "effective" thermocouple sheath resistance of a few hundredths of an ohm per meter, rather than several ohms per meter. Thus, the effect of leakage current in the clad of the rod on the thermocouple output is reduced proportionally.

The net effect on a thermocouple which is inserted *through* a temperature profile is shown in Fig. 8 is a combination of both electrical shunting and the creation of a virtual junction. The temperature measurement errors in this case depend on the location of the thermocouple with respect to the temperature profile. If the measuring junction is located to the left of the temperature maximum in Fig. 8, the indicated temperature is less than the true temperature (− error). As the measuring junction is moved through the temperature maximum, the error first decreases to zero as the measuring junction approaches a point "a" on the temperature profile; then it increases, but of the opposite sign (+ error), as the measuring junction is moved beyond "a", that is, the temperature indicated by the thermocouple is higher than the true temperature of its measuring junction.

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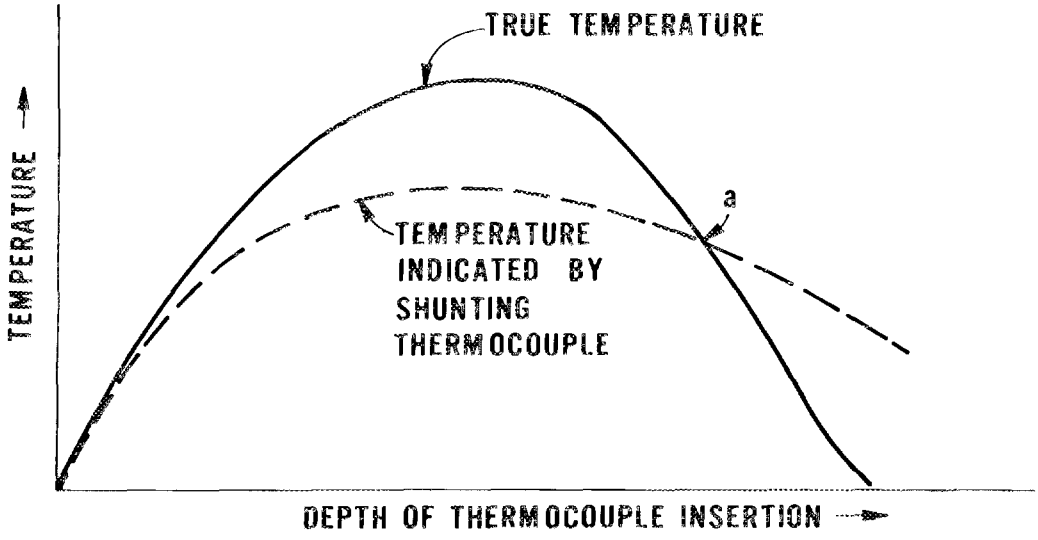

Fig. 8. Temperature indicated by a shunting thermocouple as it is inserted through a symmetric temperature distribution.

Electrical leakage errors are also systematic and depend on temperature and the direction and magnitude of the current on the sheath.

4.8 Analytical Model for Electrical Shunting and Electrical Leakage

Earlier experiments showed that errors due to electrical leakage and shunting in the fuel-rod simulator thermocouples can be large. Thus, an analytical model based on electrical transmission line theory for three parallel conductors with distributed voltage sources was developed for small diameter sheathed thermocouples.[17] In this model, the thermocouple is divided into small sections of length Δx (Fig. 9). The selections for individual sections are combined, along with a known temperature profile, and a solution is obtained that gives a good approximation to the errors caused by either electrical leakage, electrical shunting, or both.

The necessary parameters for the model (electrical resistance per unit length, Seebeck coefficients, and conductances per unit length) were determined experimentally at several temperatures. The maximum deviations between the calculated and measured errors resulting from electrical leakage are shown in Fig. 10. The agreement is excellent, even though the parameters measured for input to the model changed with time at higher temperatures during the experiment.

The model was used to calculate the effects of shunting and of a related phenomenon, the virtual junction. This is illustrated in Fig. 11 for a 0.5-mm-diameter, type K thermocouple sheathed in stainless steel.

The simple temperature profile used in the calculation is shown in Fig. 11a. In Fig. 11b the temperature measurement errors resulting from locating the measuring junction at various positions, L, in the temperature profile are shown. As the location of the measuring junction is changed from L = 0 to 1 m (500 to 1000°C), the effect of electrical shunting becomes barely discernible at about 900°C. As the measuring junction is moved from the 1 m to the 2 m position (1000 to 1200°C), the electrical shunting effect results in a negative (∿2°) temperature measurement error. Moving the measuring junction from the 2 m position to the 3 m position (1200 to 1000°C) results in the large (∿16°C) error

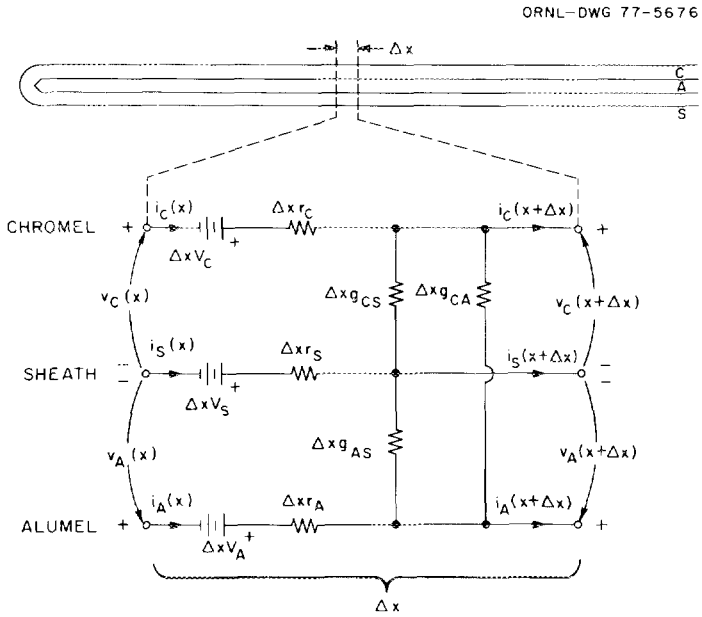

Fig. 9. Approximate electrical model of a short section of a thermocouple of length Δx. V_c, V_a, and V_s represent the voltages per unit length generated by the Seebeck effect; r_c, r_a, and r_s represent the series resistances per unit length of the Chromel, Alumel, and sheath, respectively; and g_{cs}, g_{as}, and g_{ca} represent the shunt conductances per unit length for Chromel to sheath, Alumel to sheath, and Chromel to Alumel; v_c and v_a represent the voltages on the thermocouple, Chromel to sheath, and Alumel to sheath; and i_c, i_a, and i_s represent the currents in the Chromel, Alumel, and sheath elements, respectively.

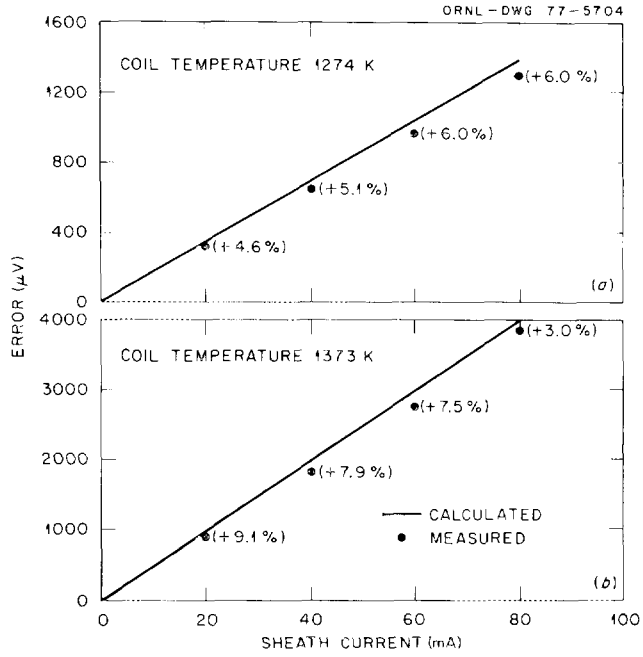

Fig. 10. Calculated and measured electrical leakage errors versus sheath current (error of 40 μV ≅ 1 K).

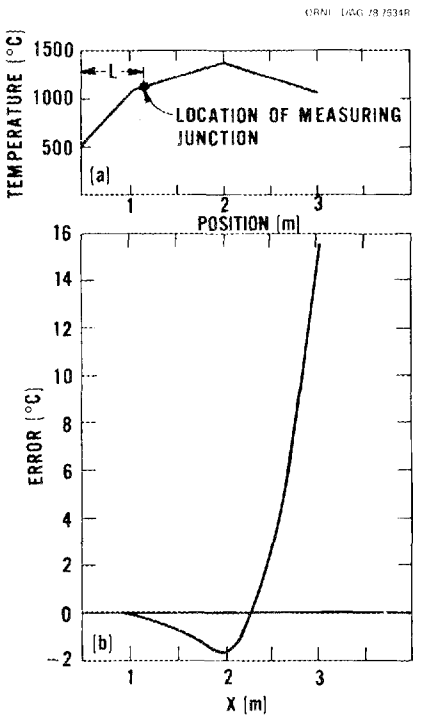

Fig. 11. Shunting errors calculated using model. (a) Temperature profile with measuring junction at X = L and reference junction at x = 0. (b) Temperature measurement error versus L.

due to the development of a virtual junction at the location along the thermocouple at the maximum 1200°C temperature. These results have been reported in detail elsewhere.[17]

4.9 Electrical Noise

In the CFTL, as in any system in which large amounts of power are handled, electrical noise usually will be introduced by induction and leakage into the low-voltage-level thermocouple circuits. The relative effects of noise pick-up on intrinsic, grounded and ungrounded junction thermocouples have been determined in a room temperature simulation of CFTL fuel-rod simulators to be in a ratio of about 10:2:1.[18] Passive filtering could reduce the effect of this induced noise, but, because this method would impair the ability of the thermocouples to make high-speed measurements during the fast CFTL transients, it would not be acceptable. Active filters can essentially solve this problem. Active filters with low-drift operational amplifiers in a thermostatically

controlled enclosure, would add less than 1°C to the overall errors.[19] This error introduced into the measurement system by the drift of the filter amplifiers will be relatively constant and systematic.

5. DECALIBRATION

5.1 Introduction

The calibration of a thermocouple establishes an initial functional relationship between the emf output of the thermocouple and the temperature of the measuring junction. This is referred to as the "temperature-emf relationship." DEcalibration is the result of changes of the temperature-emf relationship with time. Decalibration destroys the homogeneity of the thermocouple, and the temperature-emf relationship becomes dependent on the location of the decalibrated portion of the thermocouple with respect to temperature gradients. As a result, REcalibration usually is not possible.

Decalibration can be caused by changes in (1) the metallurgical state of the thermocouple materials or (2) the composition of the thermoelements. The rate and extent of these changes depend on factors such as temperature, composition of the thermoelements, composition of the surrounding materials (insulators, protective sheaths, gases), and size of the thermocouple wires.

To elucidate the effect of decalibration on thermocouple thermometry, the decalibration errors that result from any the above factors are classified as decalibration errors of the first kind or of the second kind. This classification does not refer to the type or origin of the decalibration errors, but to the ways in which the errors are manifested by experimental procedures. They may differ enormously in magnitude; the magnitude of an error of the second kind usually is several times greater than that of the first kind.

Decalibration errors of the first kind are observed as a drift of the temperature-emf relationship with the location of the temperature gradient *fixed* with respect to the thermocouple. Most cases of drift reported in thermocouple calibrations or applications are in this category.

Decalibration errors of the second kind occur when a decalibrated thermocouple is moved with respect to the temperature gradient. That is,

the effect of decalibration is to make the temperature-emf relationship of the thermocouple position dependent. A change in the location or shape of the temperature gradient with respect to the decalibrated portion of a thermocouple will cause a change in the output, even if the measuring junction should remain at the same temperature. Decalibration errors of the second kind are important for experiments such as the CFTL because changes in the power input and helium flow will change the temperature gradients imposed on the fuel-rod simulator thermocouples, which, as discussed in succeeding sections, can result in large nonconstant systematic temperature measurement errors.

The 0.5-mm-diameter sheathed thermocouples for the CFTL heater rods were studied to determine the causes of decalibration, to develop means to minimize decalibration errors, and to estimate the temperature measurement errors which result from decalibration. The results of this study are discussed below to explain why temperature measurement errors cannot be reduced by in situ, steady-state calibrations and why efforts to reduce temperature measurement errors due to decalibration must be limited to the selection of the optimum combination of thermocouple and sheath materials and manufacturing procedures which minimize decalibration. One would expect, as well, that the CFTL experiments would be conducted in such a manner as to minimize the errors in the thermocouple thermometry within the experimental goals of the CFTL.

5.2 Experimental Measurements and Results

5.2.1 Measurement Systems

Two different furnaces were used in this work. Both are used regularly for calibration work in the ORNL Metrology Research and Development Laboratory (MRDL). Both are electrically heated tube furnaces of about 6.5 cm ID. One (No. I) contains an Inconel-clad, copper equalization isothermal zone block which provides an immersion depth of ∿23 cm and a temperature range from 20 to 1000°C. The second furnace (No. II) has a platinum-rhodium alloy heating element which permits operation to ∿1500°C. In furnace I, the test thermocouples were sealed into an Inconel-600 protection tube that was first evacuated and then filled with

argon before insertion into the furnace. In furnace II, a sealed, crystallized alumina tube was used with flowing argon to protect the test thermocouples from oxidation.

Several systems were used to measure the thermocouple emf's, including a Guildline 9930 DCC potentiometer with a resolution better than 0.1 μV; and a Leeds and Northrup precision digital voltmeter, which has a resolu tion of 0.1 μV, together with a low-thermal, 30-channel scanner. The digital voltmeter and scanner were controlled by a minicomputer which also calculated and logged the data. The leads between the measurement systems and the test thermocouples were untinned copper telephone cable.

The overall uncertainty of the measuring systems which includes the measuring instrument, the switching, and lead wires is estimated to be ±1 μV.

The inhomogeneity test rig (Fig. 12) consists of a stirred salt bath maintained at ∿150°C, a motorized carriage to lower the test thermocouples into the bath at a uniform rate, a voltage suppression unit, and an X-Y recorder. To test a thermocouple for homogeneity, the thermocouple is attached to the motorized carriage and lowered into the bath about 2 cm. The thermocouple emf is suppressed using the volt suppression unit, and this low-level differential emf is amplified and recorded on the Y-axis of the recorder. The amplifier on the X-axis of the recorder is adjusted to give a 1:1 correspondence between the distance traveled by the motorized carriage and the recorder pen. As the thermocouple is lowered into the bath at a uniform rate, the change in the thermocouple output is recorded by the X-Y recorder which plots the presence of inhomogeneities in the thermocouples as a function of position. This apparatus was used extensively to observe, semiquantitatively, the decalibration of thermocouples during the drift tests.

The changes in the thermoelectric power along the length of a thermocouple measured in the inhomogeneity test rig at 150°C cannot, in most cases, be used to predict temperature measurement errors at other temperatures. The usefulness of these tests lies mainly in the ability to detect the presence or absence of inhomogeneities resulting from decalibration. The magnitude of temperature measurement errors due to such decalibration depends uniquely on the location of the inhomogeneities

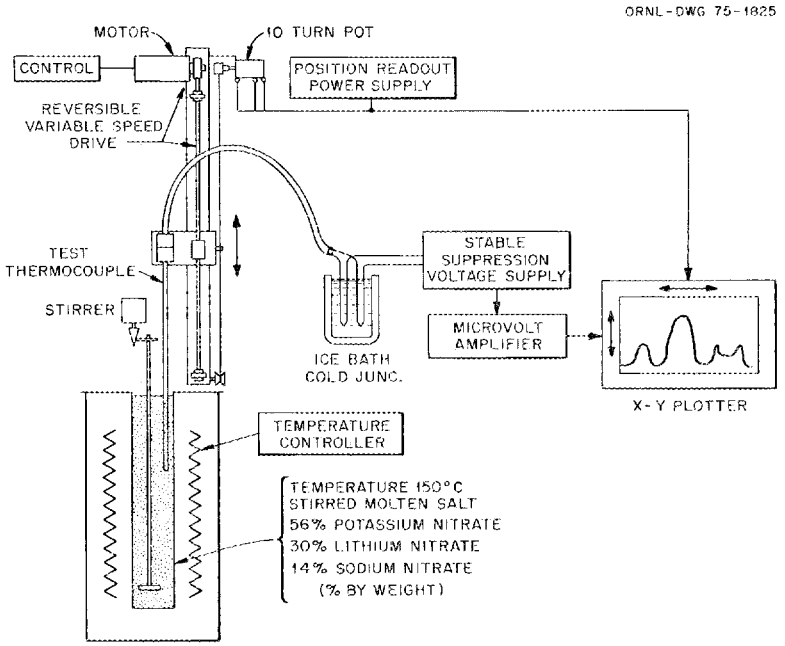

Fig. 12. Diagram of the inhomogeneity test rig.

with respect to the temperature gradients in any particular application, as discussed in Sect. 3, and on the cause of the decalibration. If the decalibration is the result of compositional changes in the thermocouple, the change in the thermoelectric power measured at 150°C in the inhomogeneity test rig is not simply related to a corresponding change in thermoelectric power at any other temperature. This is discussed more extensively in Sect. 5.3.3.

5.2.2 Drift Tests

A variety of materials were tested to determine the rate and extent of decalibration as a function of temperature (Table 3).

The test thermocouples were fabricated in the MRDL from bulk 0.5-mm-diameter sheath thermocouple materials, most as grounded junction thermocouples. The measuring junction and its end seal were welded using a tungsten-inert-gas welder. At the reference end of the thermocouple, a 3- to 5-mm length of the sheath was stripped off, and the end of the

Table 3. Summary of drift tests conducted on CFTL type thermocouples

Thermocouple Type	Sheath Material	Drift Tests Conducted Time (hr)	Drift Tests Conducted Temp (°C)
K	304 stainless steel	962	600
		531	800
		303	900
K	Inconel 600	100	1000
		50	1100
		50	1150
Nicrosil vs Nisil	304 stainless steel	962	600
		531	800
		303	900
		100	1000
S	90%Pt-10%Rh	50	1100
S	90%Pt-10%Rh	Calibrated to 1370°C	
S	80%Pt-20%Rh		
S	304 stainless steel		
S	Inconel-600		
B	80%Pt-20%Rh		
B	304 stainless steel		
B	Inconel-600		

thermocouple was sealed with a fast-setting (5 min) epoxy resin. The thermocouple wires were spot-welded to copper lead wires, and the connections were insulated electrically with additional epoxy, which also provided mechanical support and protection. The reference junctions were immersed in an ice bath which provided the reference temperature.

Drift tests on several types of thermocouples and thermocouple-sheath material combinations were run at the same time to ensure identical heat treatments for the thermocouple types being compared. They were sealed into a protection tube and inserted into a hot furnace, which was preheated to the temperature of the particular drift test being run, along with a calibrated, type S, standard thermocouple. The emf's of the test thermocouples and the standard were recorded periodically while the thermocouples were in the furnace. At selected intervals, the test thermocouples were withdrawn from the furnace, quenched in an ice bath, and removed from the protection tube. After the test thermocouples were scanned to determine where inhomogeneities had formed, they were returned

to the furnace in accordance with the procedure described in the preceding discussion for further heat treatment.

5.2.3 Results of Drift Tests

At 600°C. Three sets of thermocouples (two per set) were tested for a total of 962 hr at 600°C: (1) type K in type 304 stainless steel sheaths, (2) type K in Inconel-600 sheaths, and (3) Nicrosil versus Nisil in type 304 stainless steel sheaths. The thermocouples were removed from the furnace at 1, 6, 28, 96, 249, and 962 hr and scanned.

For the first set (type K in stainless steel) the maximum emf recorded in each scan (Fig. 13) occurred at a position 25 cm from the measuring junction (left-hand-side of Fig. 13). The thermocouples were immersed ∿23 cm into the heat treatment furnace, and these maxima are at a point ∿2 cm outside the furnace. In another test with the furnace at 600°C, the temperature profile inside the protection tube was scanned; the temperature inside the protection tube at the location of the maxima of the emf outputs was ∿350°C. The magnitude of the maxima in the emf's represents a change of ∿1% in the Seebeck coefficient. This correlates well with the measured short-range-order errors in type K thermocouples, which produce a maximum error of ∿1.1% at 350°C.[20] The temperatures measured during the drift tests changed a total of +2.6°C in 962 hr, or ∿0.4%; this is a decalibration error of the first kind.

For the second set of thermocouples with Inconel sheaths the scans for one of these thermocouples (Fig. 14) show a positive maximum in the emf output at ∿25 cm, which would result from a change of ∿1.1% in the Seebeck coefficient. This is an excellent agreement with the value from short-range-order measurements. The change in indicated temperatures after 962 hr at 600°C was 1.3°C, or ∿0.2% — again a decalibration error of the first kind.

The Nicrosil versus Nisil thermocouples (third set) are improved thermocouple materials which were formulated to minimize decalibration due to short-range order and high-temperature oxidation. The overall change in the two thermocouples was about 0.2°C in the 962 hr period. The scan shows a minimum in the emf output at the same location corresponding to the short-range-ordering maximum in the emf of the companion

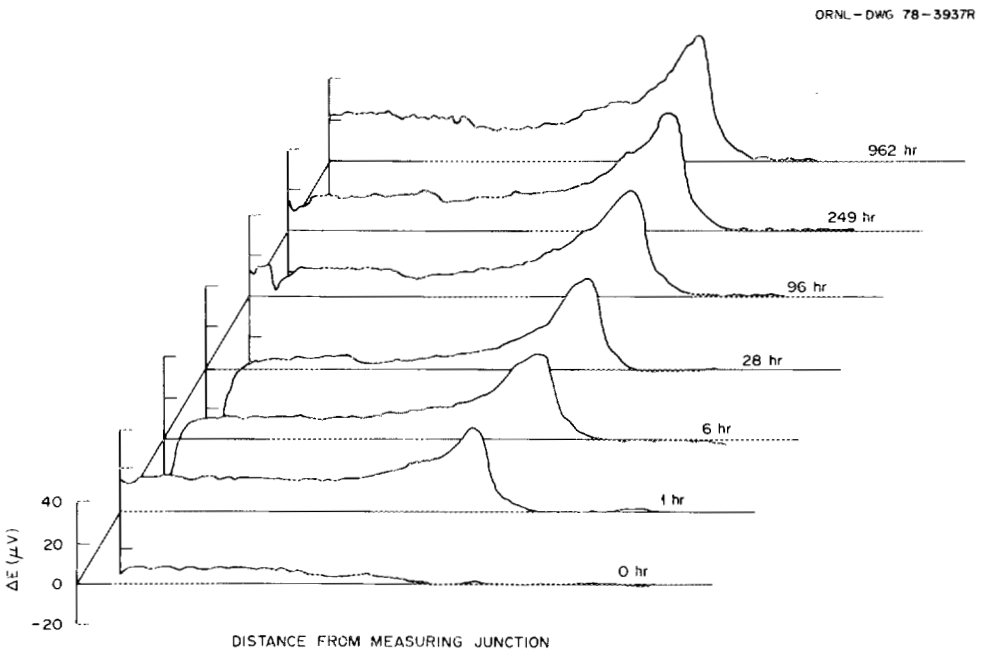

Fig. 13. Inhomogeneity scans of a type K thermocouple in a type 304 stainless steel sheath after exposure at 600°C for the indicated times.

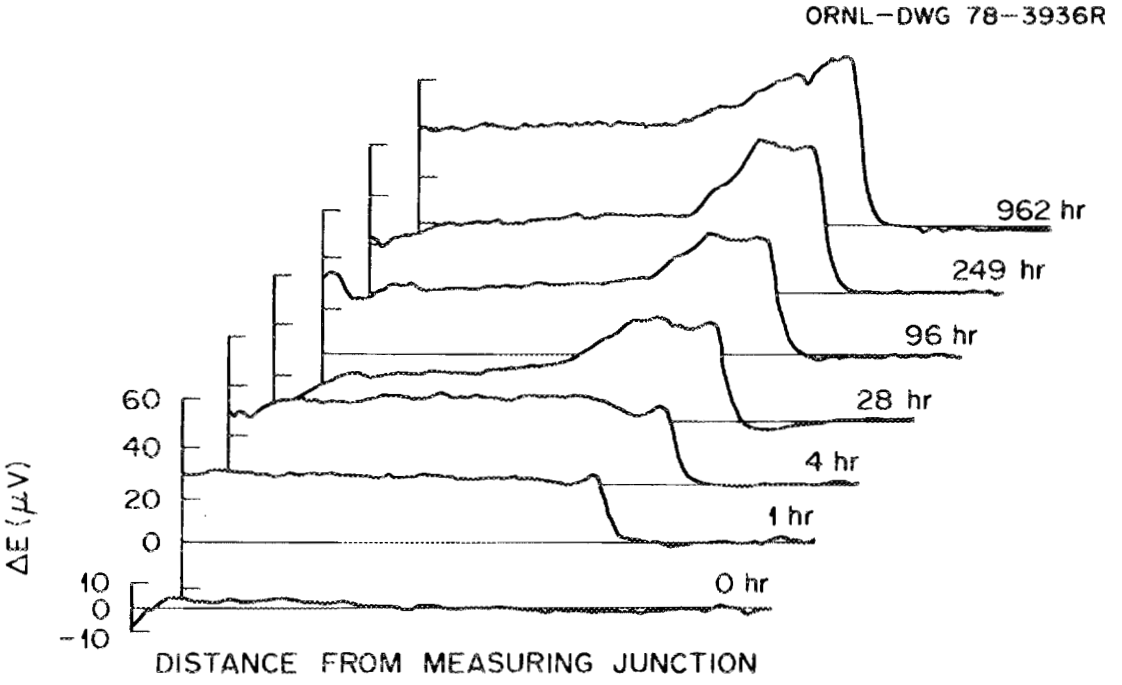

Fig. 14. Inhomogeneity scans of a type K thermocouple sheathed in Inconel-600 after exposure at 600°C for the indicated times.

type K thermocouples. The chromium content of the Nicrosil in these thermocouples was ∿1% greater than the 14.2 wt.% Cr which has been found to minimize the ordering effect on the thermoelectric properties of Ni-Cr alloys. Because experiments at 900 and 1000°C showed that these thermocouples in a sheathed assembly decalibrate to a greater extent than type K thermocouples of a similar size, they will not be discussed extensively.

At 800°C. Two type K thermocouples in type 304 stainless steel, two type K in Inconel-600, but only one Nicrosil versus Nisil in type 304 stainless steel were tested at 800°C for a total of 531 hr. These thermocouples were removed from the furnace and scanned after 1, 4, 7, 43, 184, 250, 321, 485, and 531 hr at 800°C.

The scans for the first set (Fig. 15) show that the average indicated temperature for the two thermocouples had increased 1.4°C after about 500 hr of exposure at 800°C. Also, a peak due to short-range ordering had formed in the steep temperature gradient region of the furnace. It is evident that some other mechanism caused decalibration of the portion inside the furnace.

A scan of one of the two type K thermocouples sheathed in Inconel-600 (Fig. 16) shows that a decalibration other than ordering occurred in the temperature gradient region of the furnace because the temperature indicated by these two thermocouples decreased 2.7°C after 500 hr at 800°C, but the temperature indicated by the single Nicrosil versus Nisil thermocouple decreased 1.4°C.

Because the inhomogeneity tests at 150°C can only indicate the presence of inhomogeneities, two additional tests were conducted with this set of thermocouples after completion of the drift test to obtain an estimate of the errors in temperature measurement caused by the decalibration at 800°C at other temperatures. With the furnace controlling at either 200 or 800°C, the thermocouple assembly was withdrawn from the furnace in measured increments to a total of 12.5 cm, and the thermocouple emf was read after sufficient time had been allowed for thermal equilibrium to be established. The system was judged to be in thermal equilibrium when successive readings of the thermocouple emf's taken at 5-min intervals repeated within ±2 μV. At 200°C, the Nicrosil versus Nisil in stainless

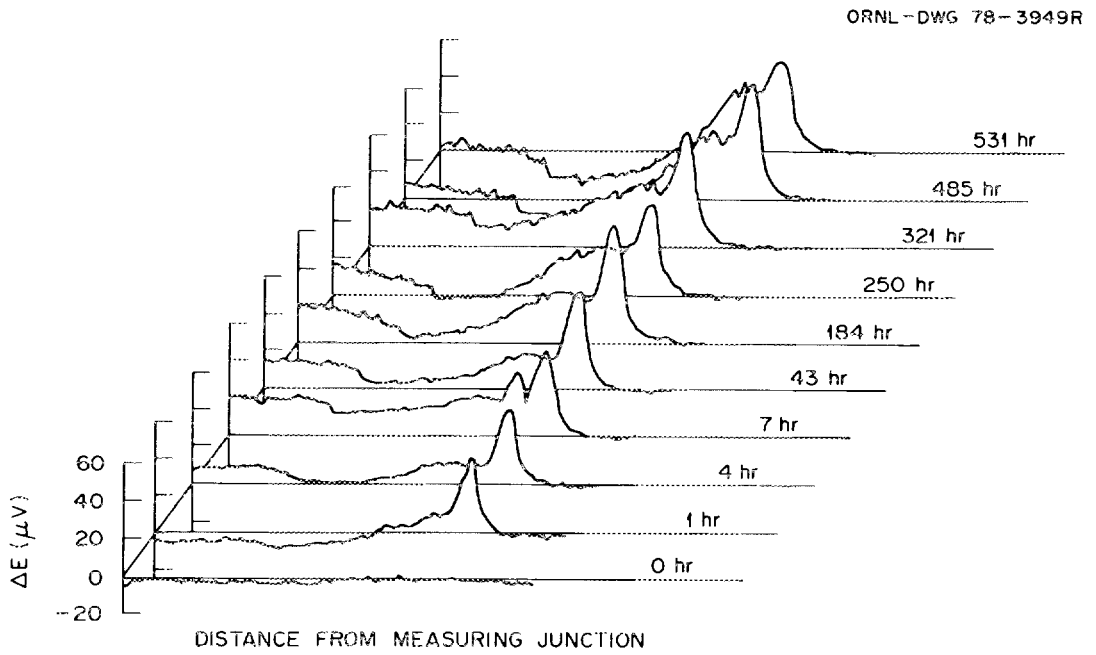

Fig. 15. Inhomogeneity scans of a type K thermocouple in a type 304 stainless steel sheath after exposure at 800°C for the indicated times.

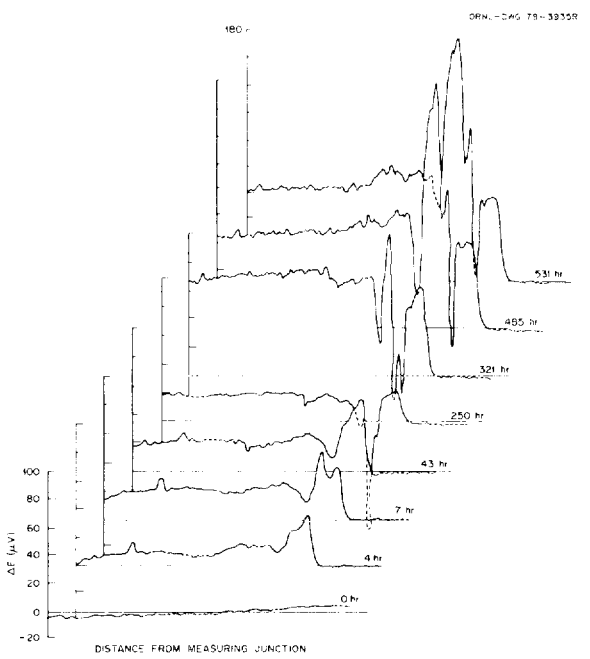

Fig. 16. Inhomogeneity scans of a type K thermocouple sheathed in Inconel-600 after exposure at 800°C for the indicated times.

steel, the type K in stainless steel, and the type K in Inconel sheaths, respectively, changed by +0.3, -2, and -1°C, respectively. At 800°C the changes observed were -2.8, -8.9, and +3.9°C, respectively. These are decalibration errors of the second kind.

At 900°C. Figure 17 is a plot of homogeneity scans of a type K thermocouple in a stainless steel sheath after 0, 4, 23, 47, 95, and 189 hr at 900°C. (During another scan at 303 hr, the decalibration had become so severe that the scale factors had to be changed on the recorder, and this scan could not be plotted on Fig. 17.) The change in the Seebeck coefficient, represented in Fig. 17 by the deep minimum in the emf about halfway between the measuring junction and the end of the furnace, was 1.3% at 189 hr and 2% at 303 hr. The height of the maximum in the emf due to short-range ordering remained about the same as before. The indicated temperature decreased by ∿1.8°C in 300 hr.

A different pattern is observed in the scans of a type K thermocouple in an Inconel-600 sheath (Fig. 18). Clearly, the mechanism of decalibration is dependent on the sheath materials above 800°C. The indicated temperature increased 1.5°C in 300 hr, and this increase is a decalibration error of the first kind.

At 1000°C. Figure 19 is a plot of scans of a (1) type K thermocouple in a type 310 stainless steel sheath, (2) a type K thermocouple in an Inconel-600 sheath, and (3) a Nicrosil versus Nisil thermocouple in a type 304 stainless steel sheath after 100 hr at 1000°C. The Seebeck coefficient for the first of these three thermocouples changed -2.5%, whereas the coefficient for the second thermocouple changed only +0.7%. In the Nicrosil versus Nisil thermocouple a -6.6% change in the Seebeck coefficient was observed at the large emf minimum. The decalibration errors of the first kind observed after 100 hr at 1000°C were (in the same order as in the preceding list): (1) -1.6°C, (2) +2.6°C, and (3) -6°C.

The CFTL program plan[2] calls for about 50 hr of total exposure at 1150°C, which represents the most severe long-term tests. (The subsequent approach to 1370°C is expected to be relatively short term.) The following tests were, therefore, performed to simulate the expected high temperature exposure.

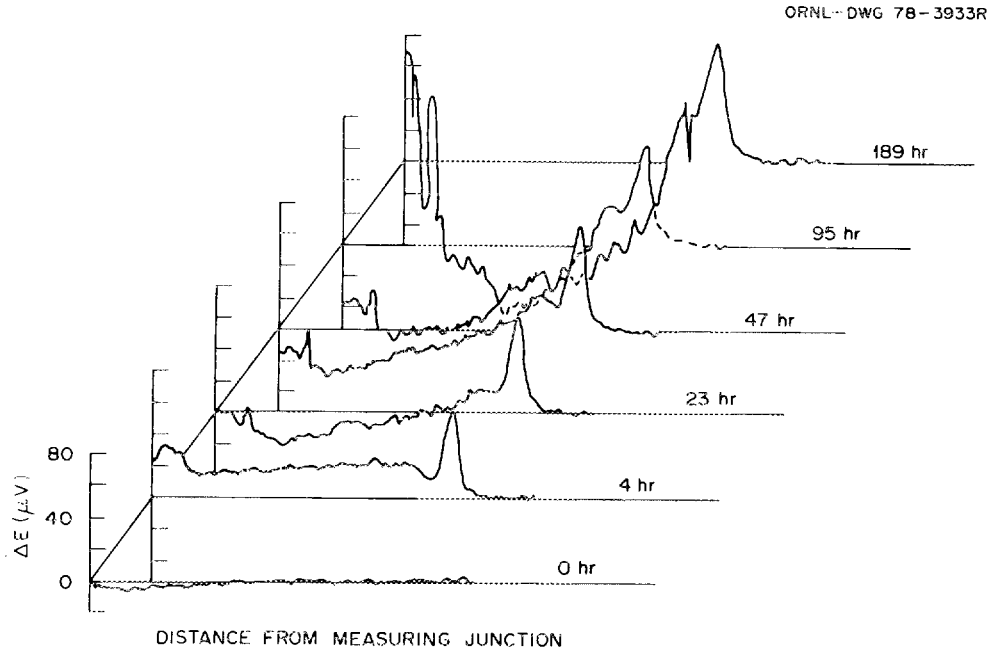

Fig. 17. Inhomogeneity scans of a type K thermocouple in a type 304 stainless steel sheath after exposure at 900°C for the indicated times.

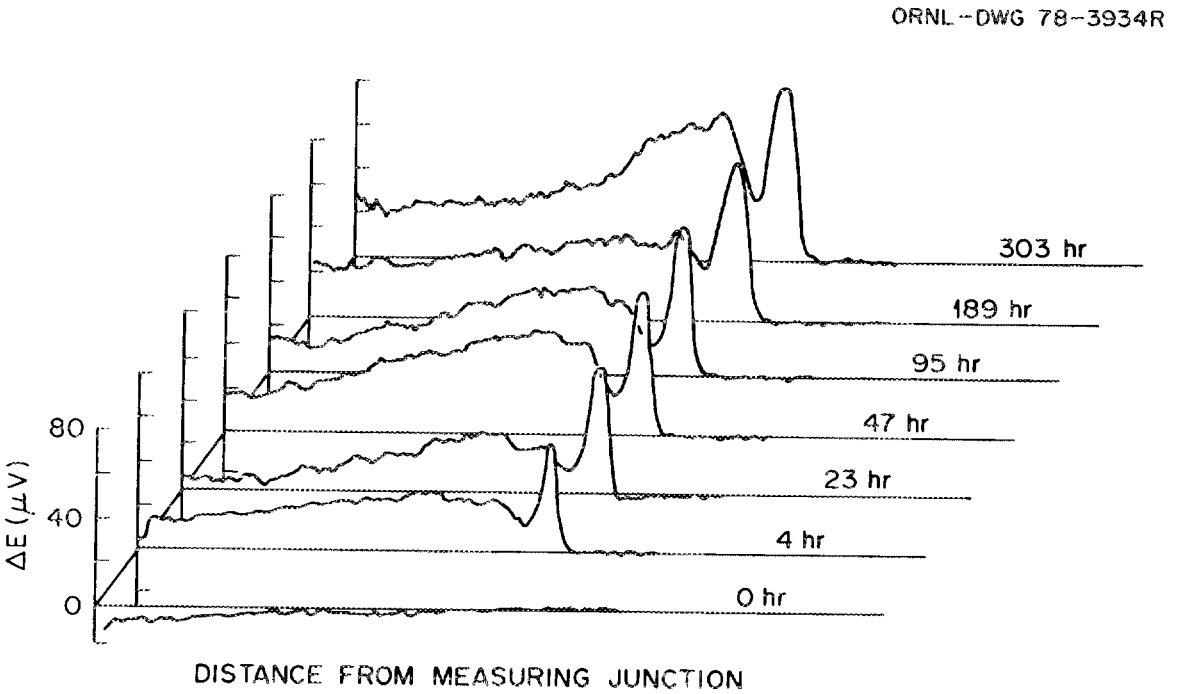

Fig. 18. Inhomogeneity scans of a type K thermocouple in an Inconel-600 sheath after exposure at 900°C for the indicated times.

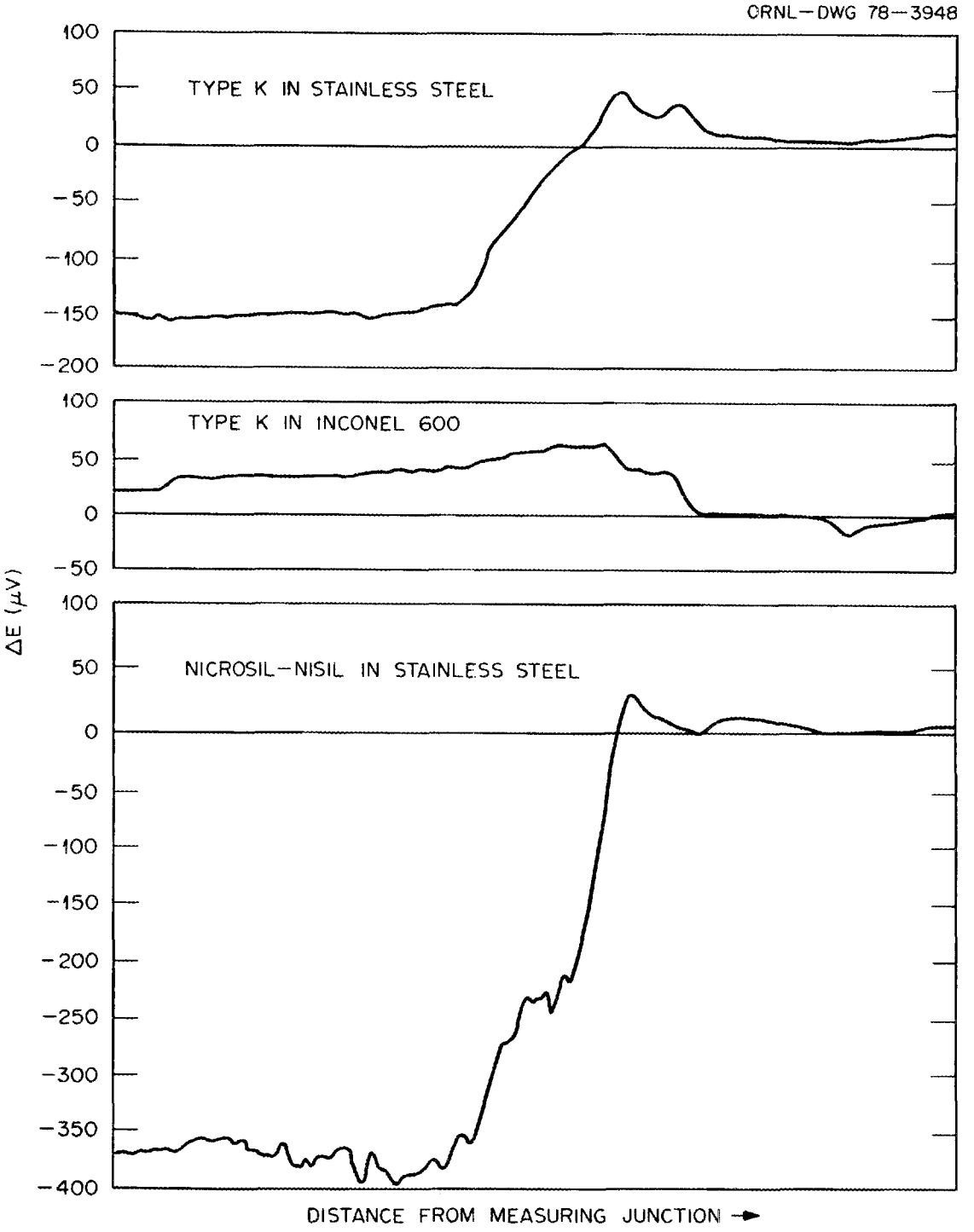

Fig. 19. Inhomogeneity scans of a type K thermocouple sheathed in a type 304 stainless steel sheath, a type K thermocouple in an Inconel-600 sheath, and a Nicrosil vs Nisil thermocouple in a type 304 stainless steel sheath after exposure at 1000°C for 100 hr.

At 1100°C. Two type K thermocouples were tested at 1100°C for 50 hr. One was sheathed in type 310 stainless steel, and the other in Inconel-600. An initial intuitive evaluation of the test data for the stainless steel sheathed thermocouple (Fig. 20) was that the thermocouple was defective and the results should be discarded. Later, however, after stainless steel sheathed, type K thermocouples installed in a prototypic fuel-rod simulator showed a similar behavior during a dc leakage test (Fig. 21), the data were reconsidered. During the same test, the type K thermocouple in Inconel indicated a decalibration error of the first kind of less than 1°C.

In these tests, in addition to the outputs of the thermocouples, the outputs of the individual thermoelements referenced to platinum were recorded. Figure 22 shows an anomalous peak at ∿30 hr due to a change in the Alumel element. Some change was also observed in the Chromel element (Fig. 23). In addition, calibration data were recorded as the thermocouples were heated to 1100°C. At the conclusion of the 50-hr test, additional calibration data were recorded from 1100 to 1300°C, and from 1300 to 600°C (Fig. 24). At the conclusion of the test, the decalibration error of the first kind was ∿30°C at 600°C.

To demonstrate the errors of the second kind, the thermocouple test assembly was moved ±2.5 cm with respect to the furnace. The change in the indication of the standard thermocouple (a part of the test assembly) was less than ±0.5°C; the temperatures of the measuring junctions of all thermocouples in the assembly remained almost constant. This test was conducted in four steps: (1) from the original position of the thermocouple assembly maintained during the 50-hr drift test at 1100°C, the assembly was moved 2.5 cm farther into the furnace; (2) after the assembly became equilibrated, it was moved back to its original position; (3) after equilibrium was established in step 2, the assembly was moved farther out of the furnace by 2.5 cm; and (4) after equilibrium was established in step 3, the assembly was returned to its original position of step 1. The thermocouple emf was recorded approximately every 3.5 min. The results for the type K thermocouple in the stainless steel sheath (Fig. 25) were as follows: after the shift into the furnace (step 1), the indicated temperature increased ∿13.7°C; after the return to the original position (step 2), the indicated temperature returned to the value observed prior

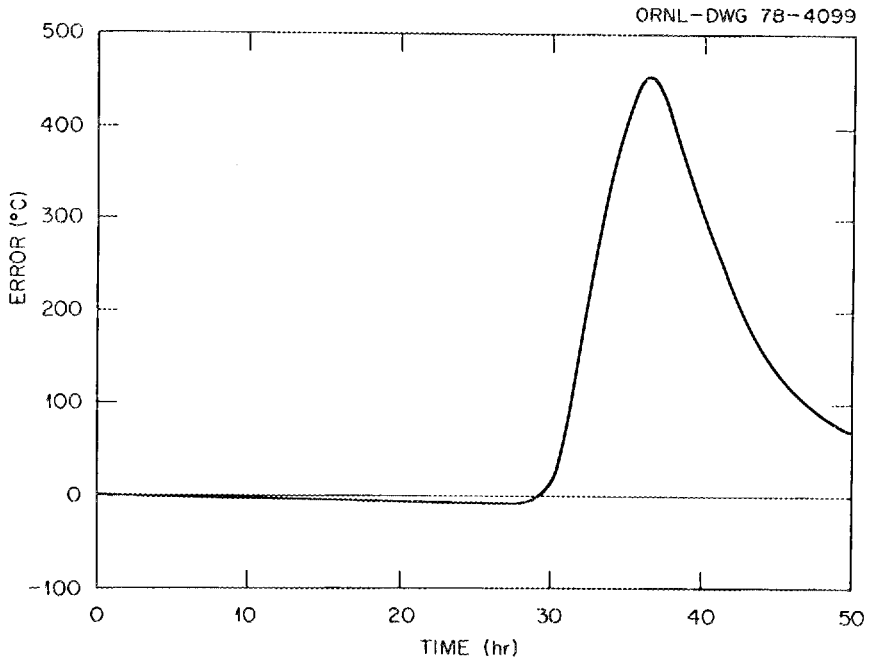

Fig. 20. Results of a 50 hr exposure at 1100°C on a type K thermocouple.

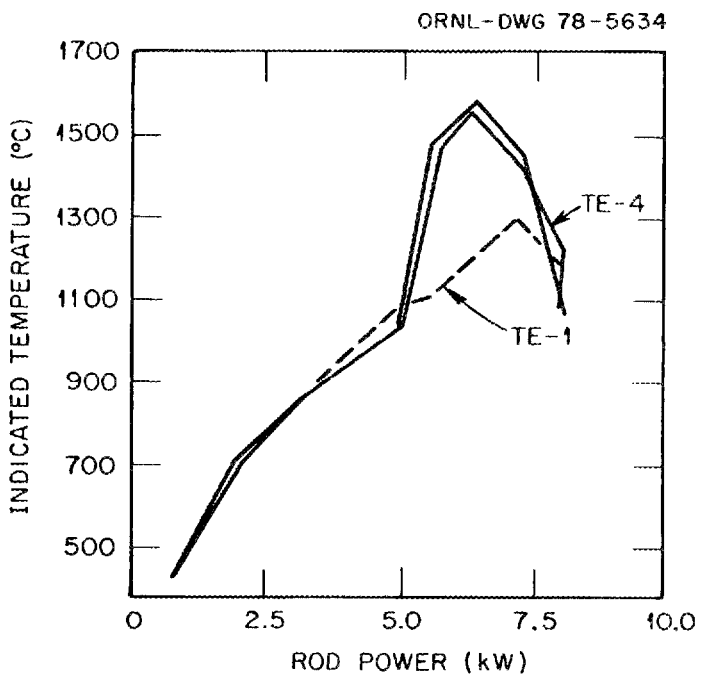

Fig. 21. Anomalous behavior of type K thermocouples sheathed in stainless steel during a dc leakage test.

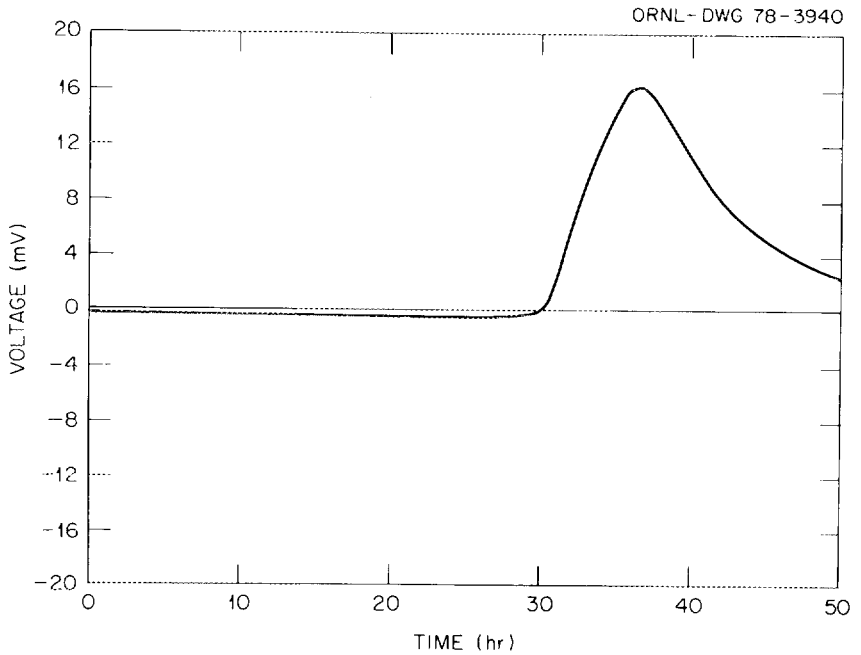

Fig. 22. Changes observed in the Alumel element of a type K thermocouple sheathed in stainless steel during a 50 hr exposure at 1100°C.

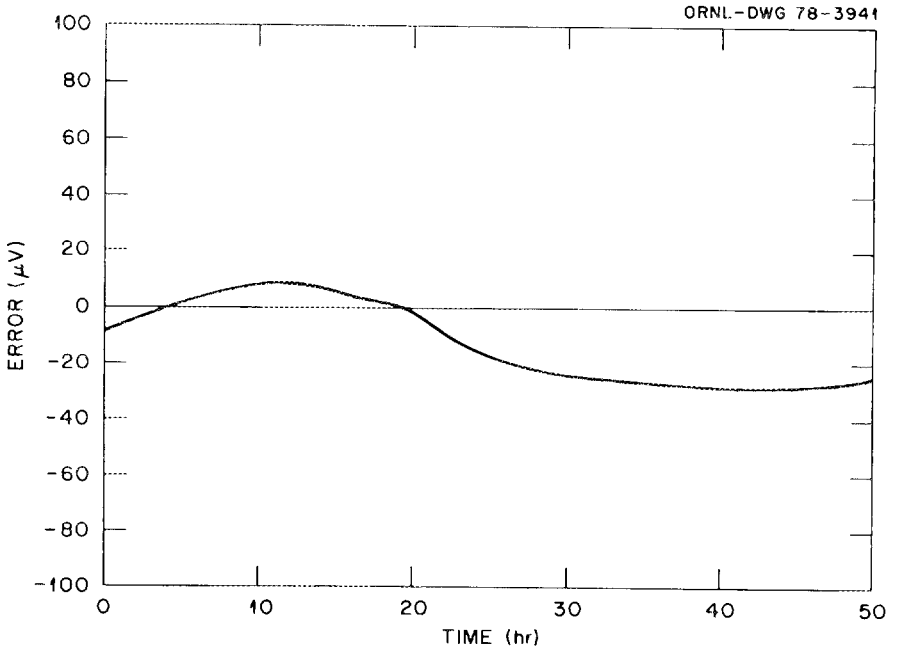

Fig. 23. Changes observed in the Chromel element of a type K thermocouple during a 50 hr exposure at 1100°C.

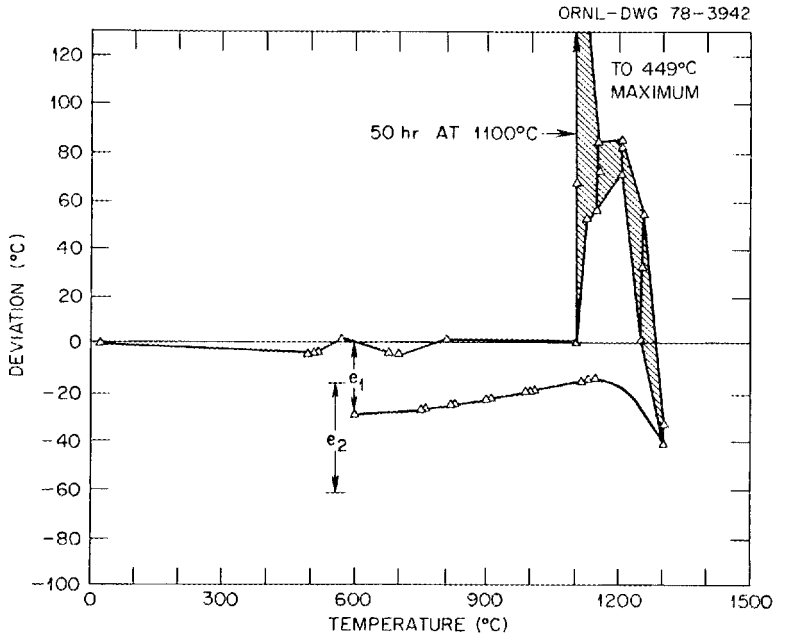

Fig. 24. Calibration of the stainless steel sheathed type K thermocouple before and after the 50 hr exposure at 1100°C. The decalibration error of the first kind at 600°C is indicated by e_1. The decalibration errors of the second kind produced by moving the thermocouple ±2.5 cm in the furnace gradient is indicated by e_2.

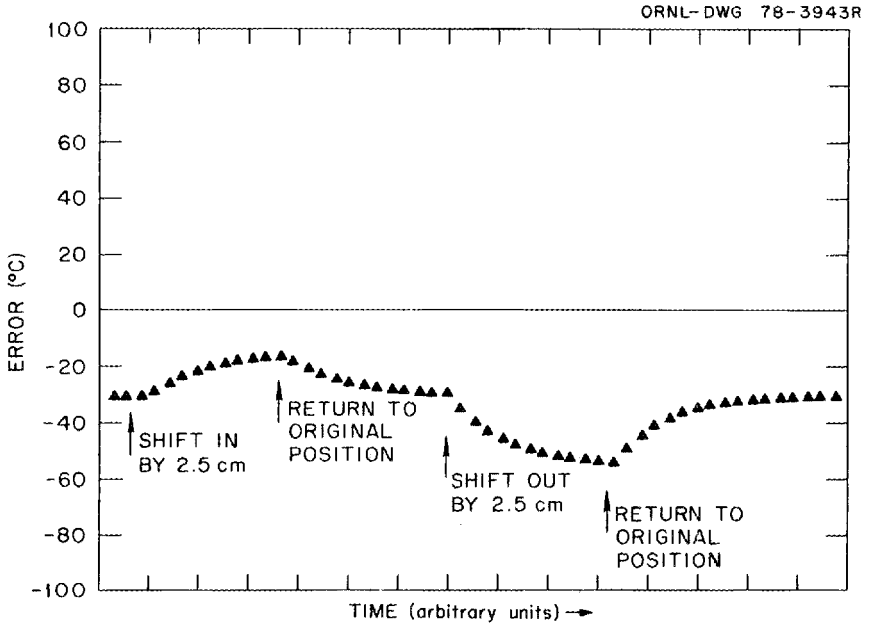

Fig. 25. Changes in indicated temperatures resulting from moving the thermocouple ±2.5 cm in the calibration furnace held at 600°C.

to step 1 within ≈1°C; after the shift out of the furnace (step 3), the indicated temperature decreased ∿25°C; and after the return to the original position (step 4), the indicated temperature again agreed with that observed prior to step 1.

The corresponding changes in the indicated temperature observed for the type K thermocouple in the Inconel-600 sheath were: step 1, +2°C; step 2, returned to the original indicated temperature within 0.15°C; step 3, -4.1°C; and step 4, returned to the original indicated temperature within 0.1°C.

This test will, perhaps, clarify the distinction between decalibration errors of the first and second kinds. At the end of the 50-hr drift test and a short calibration run to 1300°C, the indicated temperature at 600°C had decreased ∿25°C (indicated by e_1 in Fig. 24). This is a decalibration error of the first kind, that is, a change in the indicated temperature with the thermocouple in a fixed location.

Changing the position of the thermocouple only ±2 cm with respect to the furnace and the steep temperature gradient at the entrance resulted in changes in the indicated temperature denoted by the error bar e_2 in Fig. 24. Thus, a decalibration error of the second kind is, in effect, a measure of the uncertainty of decalibration error of the first kind. The magnitude of an error of the second kind depends on several factors: the relative displacement of the inhomogeneities induced by the decalibration with respect to the temperature gradient, the extent of the decalibration, that is, the change in the Seebeck coefficient, in the length of the thermocouple in the temperature gradient; the shape and magnitude of the temperature gradient; and the temperature. An increase of the displacement to ±4 cm might increase the magnitude of the errors of the second kind by 100% or more. The extent of compositional decalibration depends on the thermal history of the thermocouple, since the rate of chemical reactions and the rate of diffusion of impurities increase with temperature. Thus, these errors are, in practice, indeterminate in most cases. If the thermocouple can be calibrated in place by comparison with a standard thermometer under actual operating conditions, the overall effect of errors of the first and second kind may be determined. In the case of the CFTL, it is not possible to install a standard for a calibration

under operating conditions, and this will probably be true in many other cases. Where a standard can be installed, it is necessary to show that the presence of the standard does not disturb the temperature gradients with respect to the thermocouple being calibrated. To reduce temperature measurement uncertainties, therefore, combinations of the thermocouple, sheath, and insulator materials should be selected which minimize decalibration.

At 1150°C. - Two thermocouples, a type K in a stainless steel sheath and a type K in an Inconel-600 sheath were heated at 1150°C for 50 hr. The behavior of the type K in stainless steel at 1150°C, was markedly different from the test at 1100°C (Fig. 26). In this test at 1150°C, the outputs of the individual thermoelements versus platinum as well as the outputs of the thermocouples were recorded and are shown in Fig. 26. The change of the Alumel can explain the largest fraction of the total change in the output of the thermocouple. Decalibration of the Chromel resulted in the anomalous behavior in the temperature vs time curve beginning at ∿30 hr. The total drift after 50 hr at 1150°C was -13.5°C, or 1.1%. This is a decalibration error of the first kind.

Figure 27 shows a similar plot for the type K thermocouple in Inconel. The measured drift during the 50 hr was less than +2°C.

After their removal from the drift tests, these thermocouples were sectioned for metallographic studies and the ion microprobe mass analyzer (IMMA). The results of the IMMA analysis are discussed in Sect. 5.3.3.

Additional tests at 1100°C. - Because of the encouraging results at high temperature with type K thermocouples in Inconel-600 sheaths, a second set of drift tests was conducted for 50 hr at 1100°C. Before and after these tests, the thermocouples were calibrated to allow an estimate of the decalibration errors of the second kind as well as those of the first kind, which were measured directly. Figure 28 shows the results of the calibration of two thermocouples from room temperature to 1100°C. Curves K1 and K2 are least-squares fits of the calibration data for the two thermocouples taken both before and after the 50-hr drift test. The spread of the data points around the fitted lines was about ±1 μV and gives estimates of the size of the decalibration errors of the first kind.

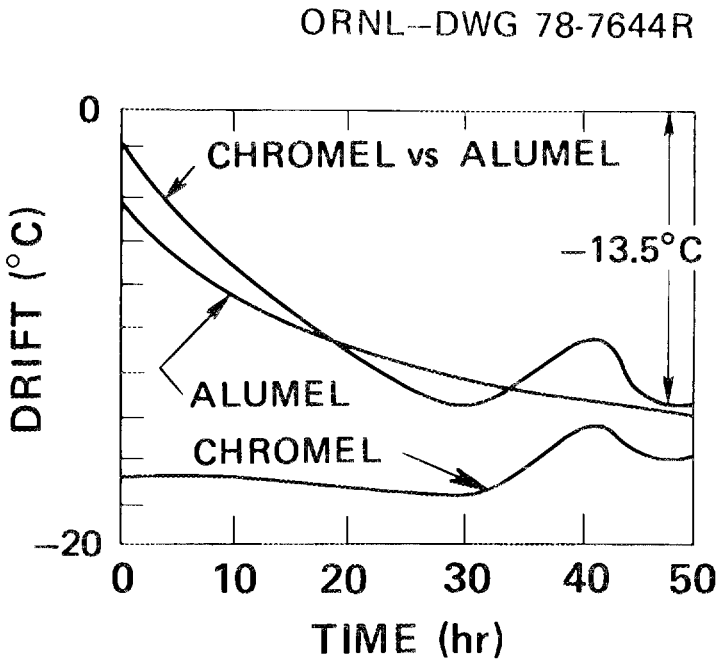

Fig. 26. Changes observed in a type K thermocouple sheathed in type 304 stainless steel during a 50 hr exposure at 1150°C.

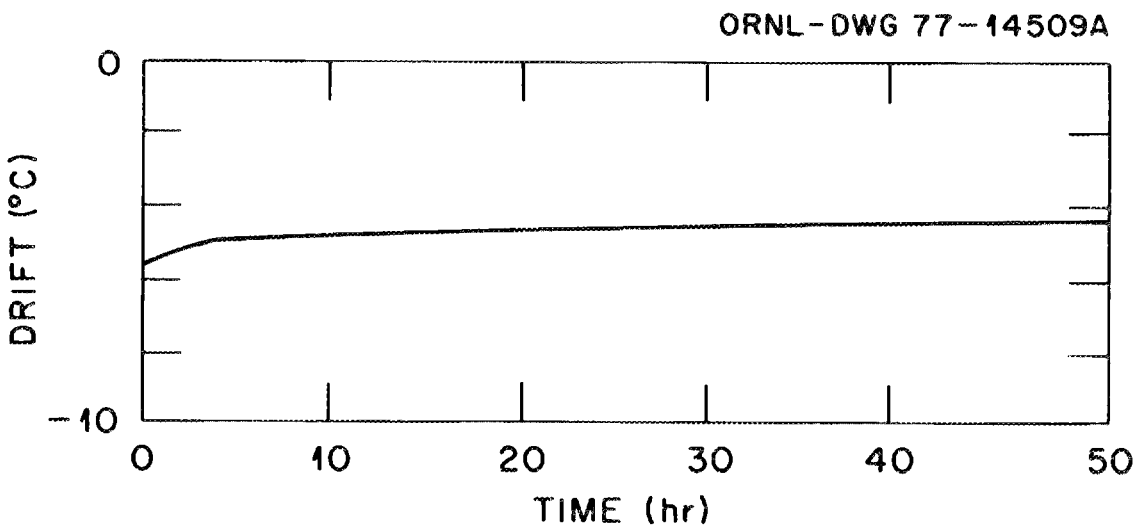

Fig. 27. Changes observed in a type K thermocouple sheathed in Inconel-600 during a 50 hr exposure at 1150°C.

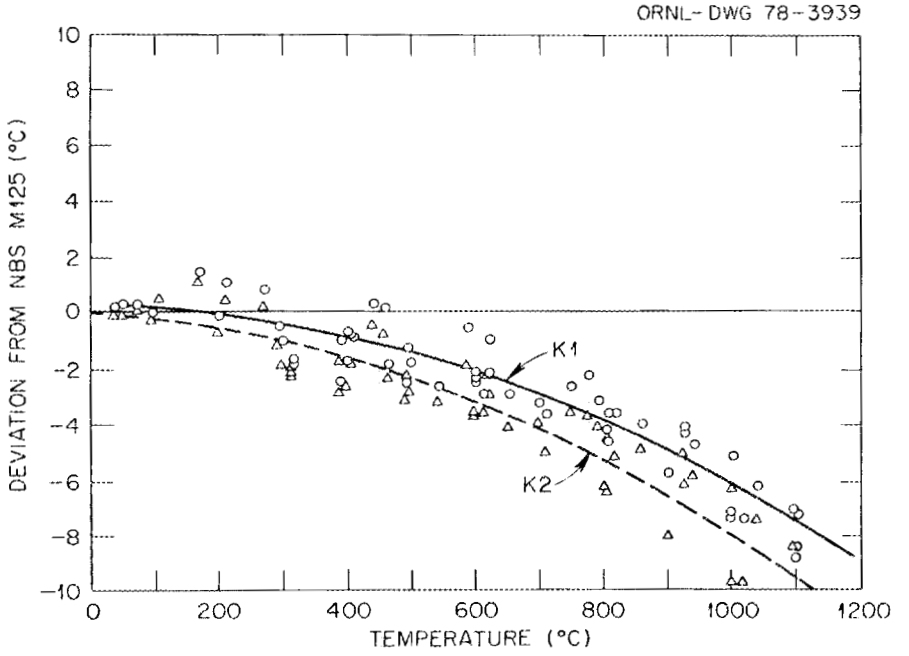

Fig. 28. Results of a calibration of two type K thermocouples sheathed in Inconel-600. After the calibration data were taken up to 1100°C, the thermocouples were held at 1100°C for 50 hr and calibration data were also taken in cooling to ∿300°C. The curves marked K1 and K2 are the result of a least squares fit of the calibration data.

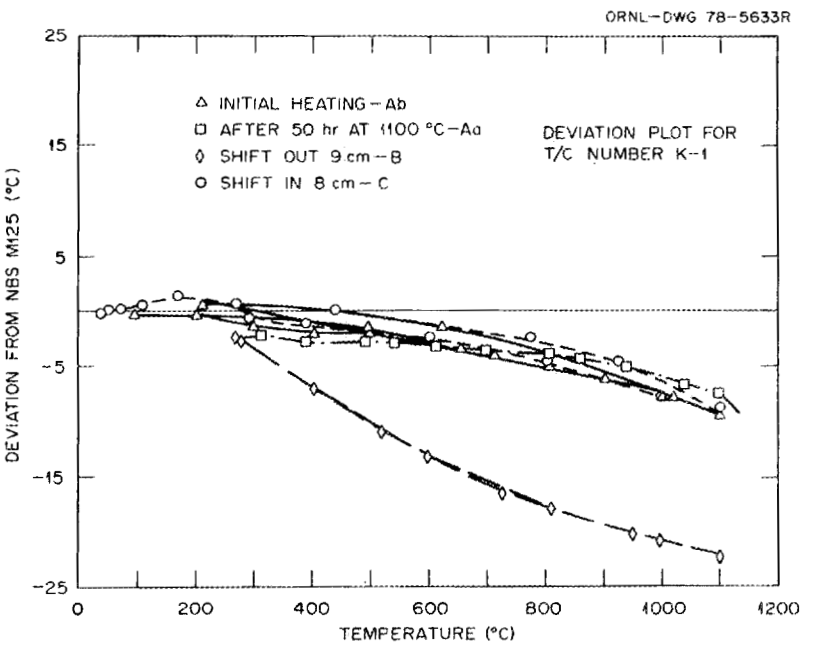

Fig. 29. Decalibration errors of the second kind revealed by shifting the thermocouple in the decalibrating furnace after a 50 hr exposure at 1100°C.

Another study was made during this test to obtain information about the effects of decalibration errors of the second kind. After the two thermocouples had been heated at 1100°C for 50 hr, they were cooled in the furnace to ∿300°C. Calibration data were taken at discrete steps during the cooling process; and these data, along with the calibration data taken before the 50-hr test, are plotted in Fig. 29 (curve marked Aa). The assembly was shifted out of the furnace 9 cm, placing the temperature gradient of the furnace in a region of the thermocouples which had been isothermal at 1100°C for 50 hr. A new calibration was performed from 300 to 1100°C and back (curve B in Fig. 29). The thermocouple assembly was then shifted into the furnace 8 cm beyond the original position so that virgin material would be in the temperature gradient of the calibration furnace. A third calibration was performed, and these results are plotted as curve C in Fig. 29. This last calibration essentially reproduced the original calibration. At 600°C, the difference between the average of curves A and C and curve B is ∿10°C, or 1.7%. This difference is a decalibration error of the second kind.

Decalibration of sheathed noble-metal thermocouples. – Samples of type S and type B materials listed in Table 3 were calibrated to 1370°C. To determine the reproducibility of the calibration of these thermocouples, the calibration was performed in cycles of increasing temperatures; that is, calibration data were recorded at selected intervals from room temperature to ∿500°C. The temperature was then stepped down to ∿300°C and a new cycle was begun. In the second cycle, the maximum temperature reached was about 700°C. Again, the temperature was stepped down to 300 to 400°C before a new cycle was begun. The results of this procedure also indicated the maximum temperature at which each type of thermocouple could be expected to perform well. An example of the results of the reproducibility test is shown in Fig. 30 for the calibration of a type S thermocouple sheathed in type 304 stainless steel to 1300°C. The overall decalibration was –88°C after exposure to temperatures above 1200°C.

Calibration to 1200°C of a type S thermocouple sheathed in type 304 stainless steel (Fig. 31) shows that up to ∿900°C the reproducibility was within about ±1°C (error of the first kind). At temperatures above 900°C, the reproducibility became worse. Table 4 lists the change in the

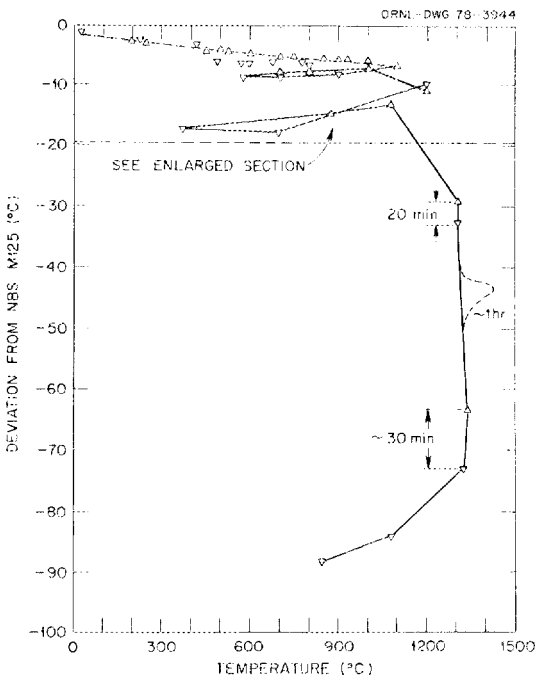

Fig. 30. Decalibration of a type S thermocouple sheathed in type 304 stainless steel.

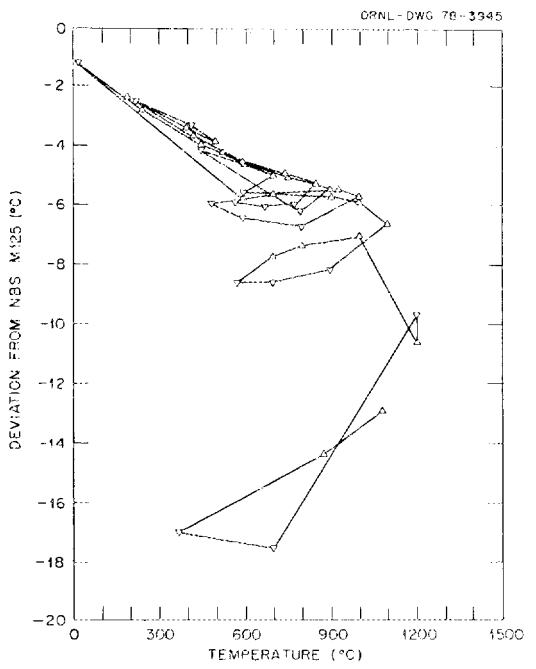

Fig. 31. Expansion of the calibration data shown in Fig. 30 from 0 to 1200°C. At temperatures above 900°C, the reproducibility is poor.

Table 4. Change in indicated temperature at 600°C for a type S thermocouple in a type 304 stainless steel sheath as a function of the maximum temperature during the cycle

Maximum Temperature of Cycle (°C)	Change in Indicated Temperature at ∿600°C (°C)
900	-1
1000	-2
1100	-4
1200	-13

indicated temperature of the thermocouple at 600°C as a function of the maximum temperature of the cycle.

Finally, while the temperature of the furnace was held at 1305°C, the outputs of the thermocouples were recorded for a period of 20 min. The drift rates for all thermocouples in the test (plotted in Fig. 32) show that the stability of the noble-metal thermocouples is dependent on the sheath material. The type S thermocouple in a 90%Pt-10%Rh sheath performed best. The drifts of thermocouples in stainless steel sheaths were greater than those in Inconel-600 sheaths. The drifts and drift rates for type S thermocouples were higher than those for type B thermocouples. This is to be expected since the type S thermocouple with a pure platinum thermoelement is more sensitive to contamination than the type B thermocouple in which both thermoelements are alloys.

At the conclusion of the test, these thermocouples were scanned for inhomogeneities. The scans for the type S thermocouples in the various sheaths are shown in Figs. 33-37. (Note: In comparing the base-metal sheathed thermocouple scans with those of the noble-metal sheathed thermocouples allow for differences in the scales.) For the type S thermocouples in 90%PT-10%Rh, 80%Pt-20%Rh, type 304 stainless steel, and Inconel-600 sheaths, the decreases in the Seebeck coefficients ∿2 cm from the measuring junction were 0.3%, 5%, 64%, and 55%, respectively.

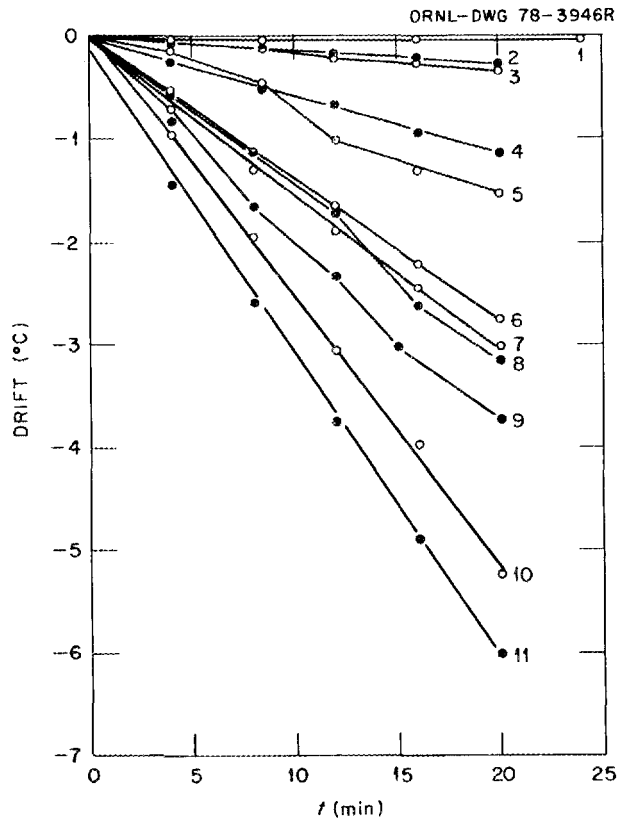

Fig. 32. The effect of various sheath materials on the drift rate of noble-metal thermocouples at 1300°C. The various thermocouple types and sheath materials, along with the drift rates, are given below.

Curve	Thermocouple Type	Sheath Material	Drift Rate at 1300°C
1	S	90%Pt-10%Rh	1 mK/min
2	S	80%Pt-20%Rh	10 mK/min
3	S	80%Pt-20%Rh	70 mK/min
4	B	Inconel-600	70 mK/min
5	B	Inconel-600	70 mK/min
6	B	Type 304 stainless steel	140 mK/min
7	B	Type 304 stainless steel	140 mK/min
8	S	Inconel-600	170 mK/min
9	S	Inconel-600	170 mK/min
10	S	Type 304 stainless steel	300 mK/min
11	S	Type 304 stainless steel	300 mK/min

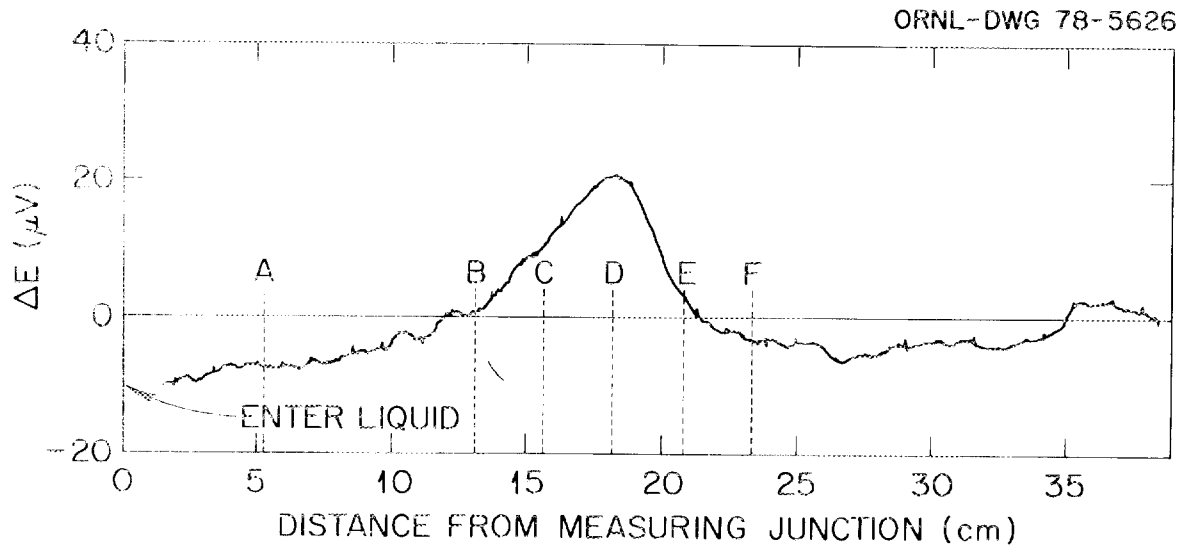

Fig. 33. Inhomogeneity scan of a type S thermocouple sheathed in 90%Pt-10%Rh after calibration to 1350°C.

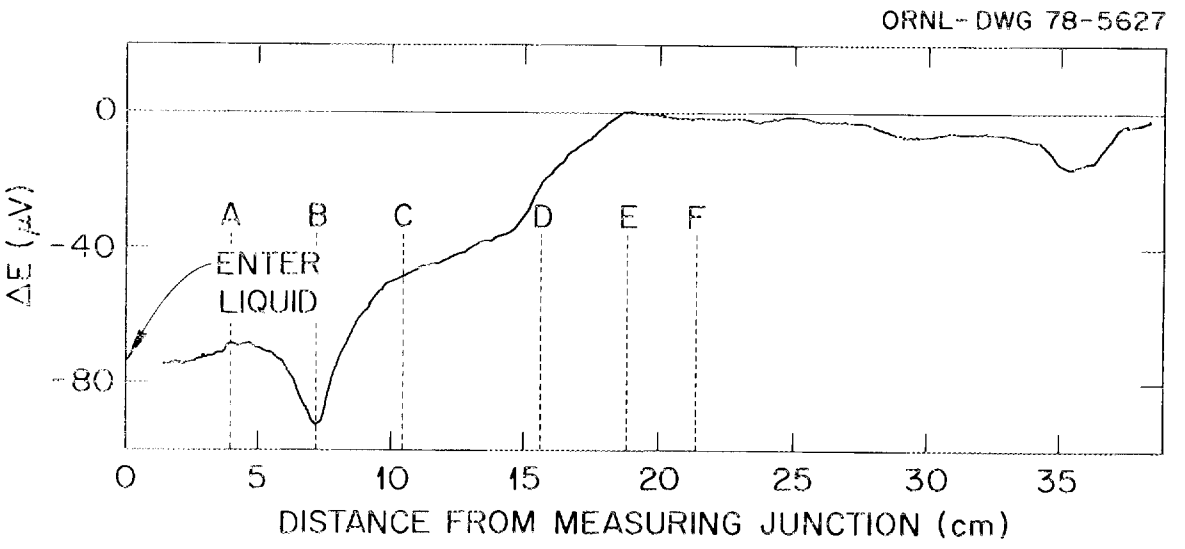

Fig. 34. Inhomogeneity scan of a type S thermocouple sheathed in 80%Pt-20%Rh after calibration to 1350°C.

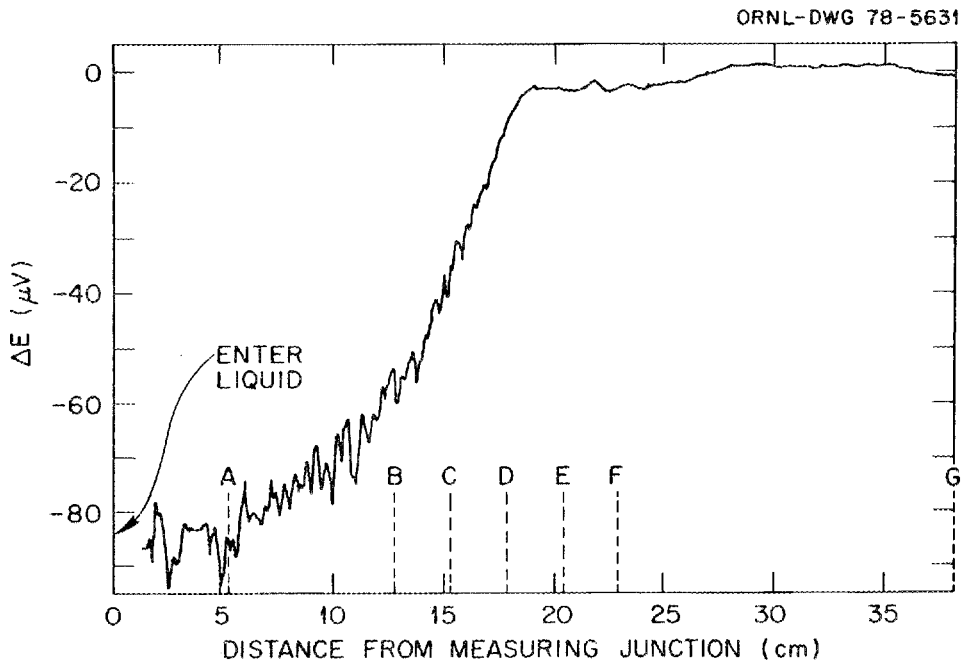

Fig. 35. Inhomogeneity scan of a type B thermocouple sheathed in Inconel-600 after calibration to 1350°C.

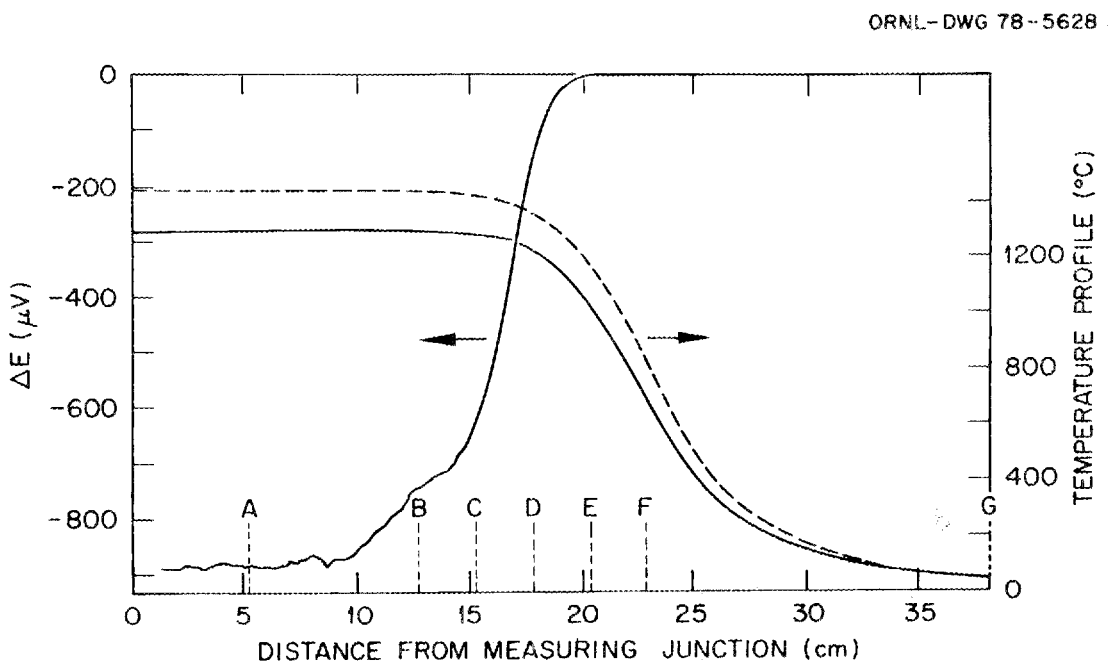

Fig. 36. Inhomogeneity scan of a type S thermocouple sheathed in Inconel-600 after calibration to 1350°C.

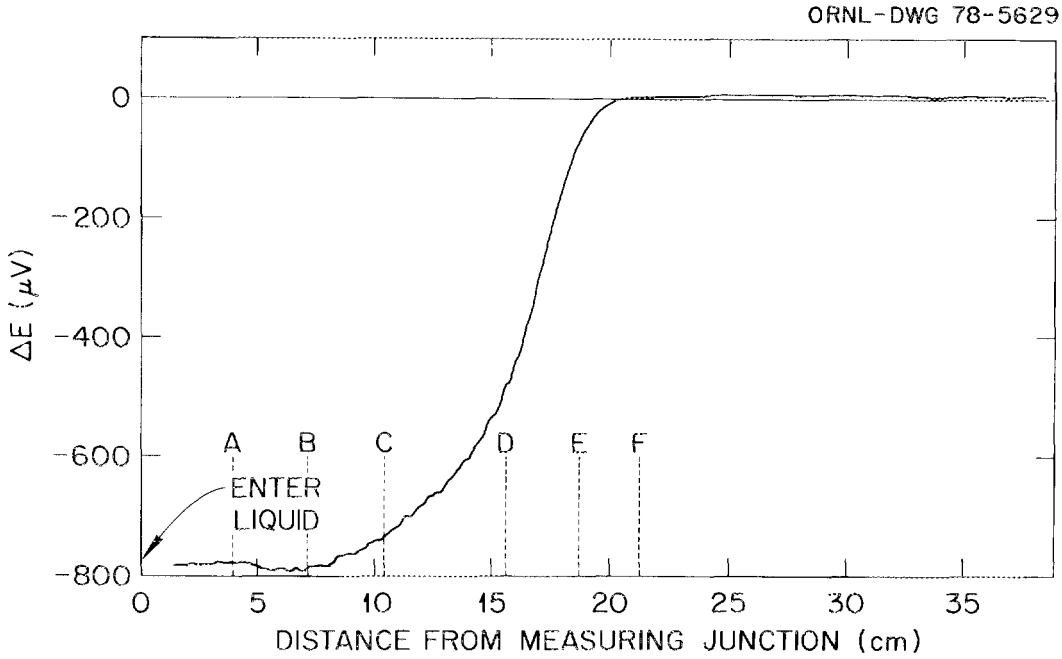

Fig. 37. Inhomogeneity scan of a type S thermocouple sheathed in type 304 stainless steel after calibration to 1350°C.

Drift test of type S thermocouple in Pt-10%Rh Sheath at 1100°C. - Three type S thermocouples in 90%Pt-10%Rh sheaths were included in the tests discussed in Sect. 5.2.3 under "Additional tests at 1100°C." The results of this test on this type of thermocouple are shown in Fig. 38. (The designation of the curves is the same as in Fig. 29.) The difference between the average of curves A and C and curve B is ∿4°C at 600°C, or 0.7%. As in Fig. 29, this is an estimate of the decalibration errors of the second kind. The scan (Fig. 39) of this thermocouple indicates little change after 50 hr at 1100°C. The maximum change in the Seebeck coefficient near the measuring junction is ∿2.5%.

5.3 Causes of Decalibration

The causes of decalibration are many and include changes in the metallurgical state, as well as in composition.[21] Metallurgical changes can be caused by mechanical deformations of the thermoelements, the annealing procedure, or solid-state phase transformations such as short-range ordering in Chromel. Compositional changes may be caused by

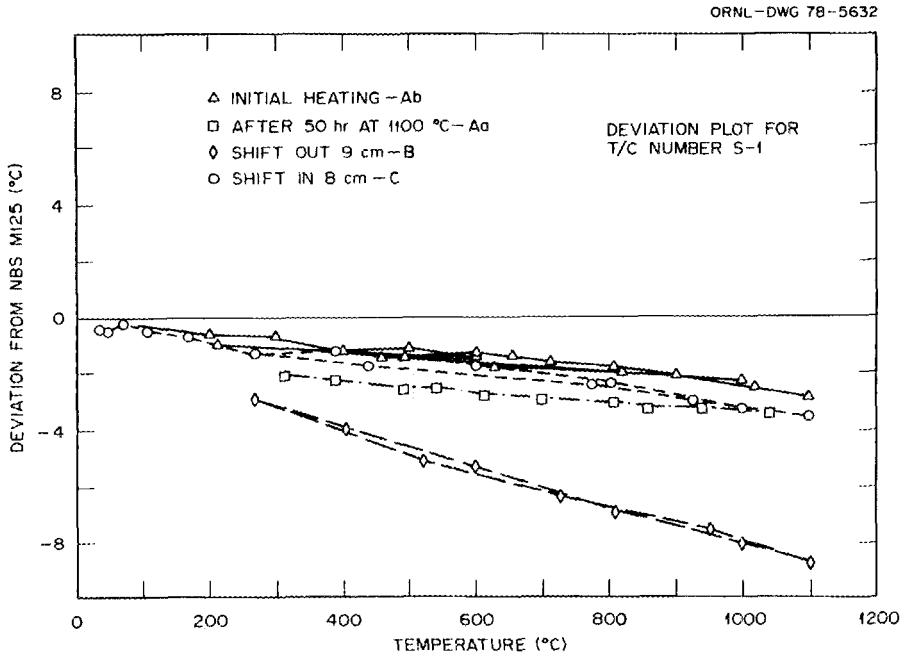

Fig. 38. Calibration curves and decalibration errors of the second kind revealed on a type S thermocouple sheathed in 90%Pt-10%Rh after 50 hr at 1100°C.

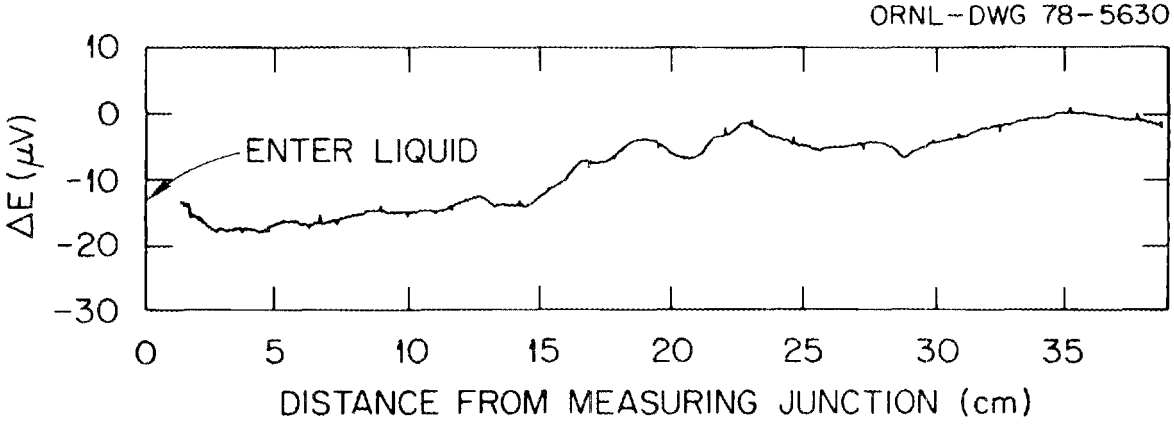

Fig. 39. Inhomogeneity scan of type S thermocouple after 50 hr exposure at 1100°C.

reactions between the thermocouple wires and the insulation; by impurities in the insulation or gases surrounding the wires; by preferential evaporation of one or more components of the thermocouple alloys; or by diffusion of impurities through the insulation from the sheath (Fig. 40). In the following sections, the discussions are limited to decalibration of types K and S thermocouples.

5.3.1 Effect of Wire Size

In 0.5-mm-diameter sheathed thermocouples, the average diameter of the thermocouple wires is 0.1 mm or less, about the diameter of a human hair (Fig. 41). Because of the large surface-to-volume ratio of such small-diameter wires, chemical effects that normally would be apparent after an extended period of time in larger-diameter wires are discernible after a much shorter time in the smaller-diameter wires. In addition, since chemical reaction rates increase exponentially with temperature (a 10°C temperature increase will double the reaction rate), decalibrations become rapid at higher temperatures (cf. Fig. 32).

5.3.2 Order-Disorder Effect in Type K

Kollie et al.[21] have reviewed the order-disorder phenomenon in Chromel and its effect on the emf output of type K thermocouples. Some of the pertinent points are as follows: between 200 and 600°C, the nickel and chromium atoms in Chromel tend to occupy specific sites in the crystal lattice (the ordered state); above ∿600°C, the atoms are distributed randomly among the lattice sites (the disordered state); a change from the ordered to the disordered state or vice versa is reversible; the rate and extent at which the ordered state is formed are time and temperature dependent (Fig. 42); and between 0 and 600°C, the temperature measurement errors caused by the order-disorder transformation can approach 1.1% of the measured temperature.

The results of the order-disorder transformation on temperature measurements made with type K thermocouples are illustrated by Fig. 43.[21] The initial calibration curve of an annealed thermocouple, curve A, lies within the ±3/8% ISA allowable error for special grade Chromel-Alumel,

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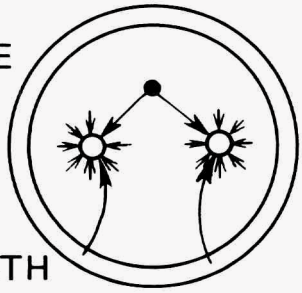
CHEMICAL REACTIONS CAN AFFECT BOTH ELEMENTS OF THE THERMOCOUPLE

REACTIONS MAY TAKE PLACE BETWEEN THE THERMOCOUPLE WIRES AND:



1. THE INSULATION
2. IMPURITIES IN THE INSULATION
3. THE ATMOSPHERE INSIDE THE SHEATH
4. THE SHEATH

RATES OF CHEMICAL REACTIONS, AS A GENERAL RULE, DOUBLE FOR EACH 10°C RISE IN TEMPERATURE.

Fig. 40. Illustration of several sources of decalibration at high temperature in sheathed thermocouple assemblies.

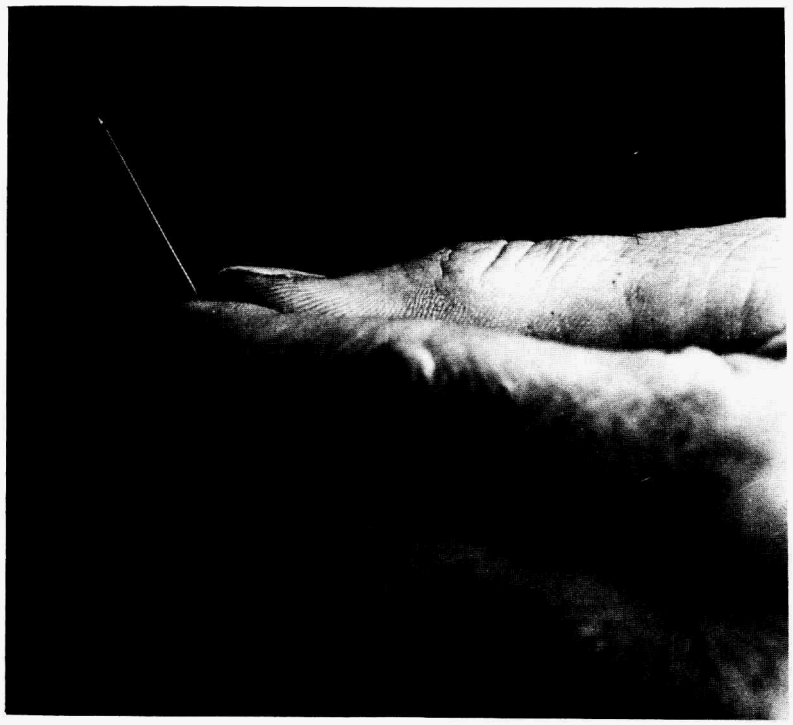


Fig. 41. Photograph of the end of a 0.5-mm-diameter sheathed thermocouple. The sheath has been stripped back to show the relative wire sizes, which are ∿0.01 mm diameter.

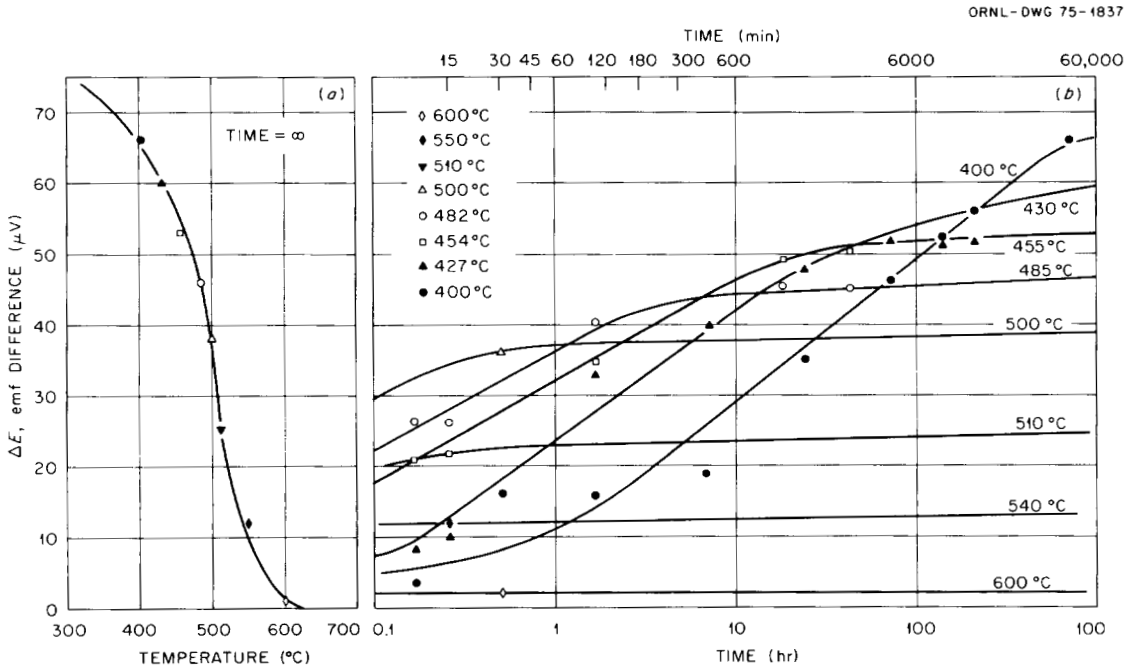


Fig. 42. Short-range order (ΔE) in Chromel as a function of temperature and time: (a) short-range order vs temperature at infinite time; (b) short-range order vs time at temperatures of 400, 430, 455, 485, 500, 510, 540, and 600°C.

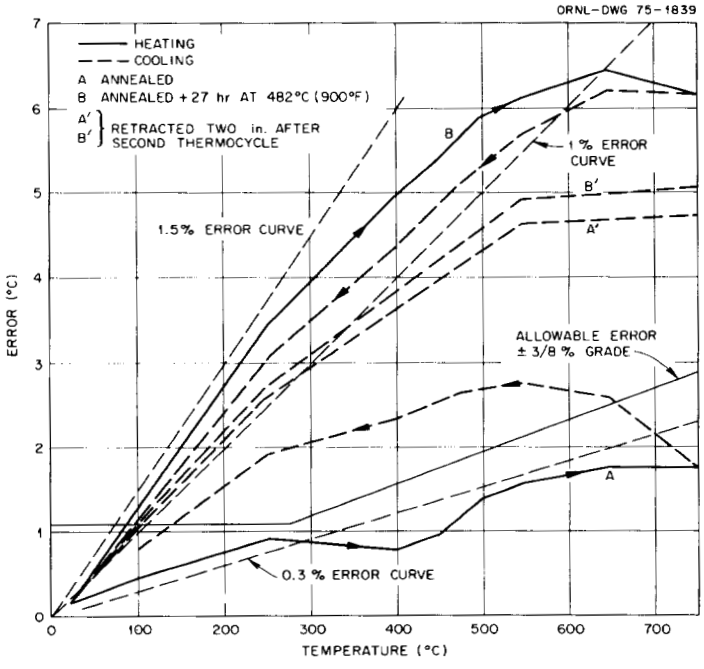


Fig. 43. Curves from calibration data taken on heating and cooling an annealed and a heat-treated type K thermocouple.

but data taken during cooling lie well outside this limit. The calibration curve for a thermocouple which was "preordered" at 482°C is labeled curve B, and the hysteresis observed during cooling of this thermocouple was much less than that for the annealed thermocouple. These are errors of the first kind. After the thermocouples had been shifted in the furnace and recalibrated, different calibration curves for the two thermocouples were obtained, which are indicated by curves A' and B', respectively. The differences A-A' and B-B' are estimates of decalibration errors of the second kind for thermocouples A and B.

In the drift tests conducted at 600°C, the maxima in the scans were formed in the temperature gradient of the furnace where the temperature was less than 600°C. These decalibrations were dominant and were due to the effects of ordering (cf. Figs. 15 and 16). As discussed in Sect. 6, errors due to order-disorder would degrade the fuel-rod simulator temperature measurements if type K thermocouples were used. More particularly, these errors would occur in the temperature region of greatest interest in the CFTL experiments, namely, between ∿400 and 600°C.

5.3.3 Decalibration by Compositional Changes in Type K Thermocouples

Numerous investigators have studied the decalibration of type K thermocouples in air. Burley,[22] in one of the more recent studies, investigated the decalibration of 3.3-mm-diameter, bare-wire, type K thermocouples in air at temperatures to 1000°C for up to 3000 hr. Figure 44 summarizes some of his results. For Chromel-Alumel pairs from four different sources, the changes in the emf output of these thermocouples (Fig. 44a) are caused by changes in both the Chromel element (Fig. 44b) and the Alumel element (Fig. 44c). At 600 and 800°C the changes in the thermocouple output are caused predominately by changes in the Chromel element, but at 1000°C the changes in the thermocouple output result mainly from changes in the Alumel element.

Our investigation of small diameter sheathed thermocouples shows much more rapid and extensive decalibrations. We attribute this to differences in the sizes of the thermoelements and to our use of sheathed thermocouples. The presence of a sheath has been shown[23] to contribute

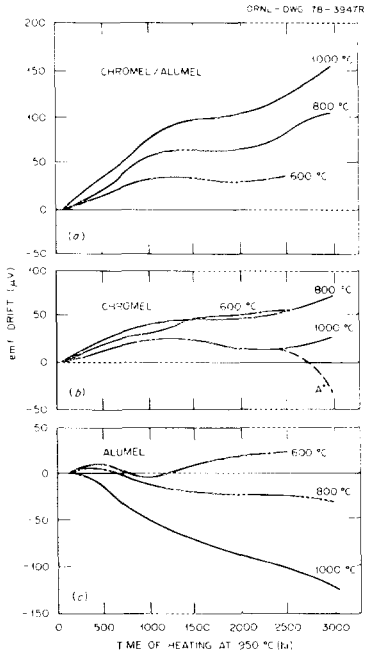


Fig. 44. Thermocouple emf drift vs time curves for conventional type Chromel and Alumel in terms of averages of all four. (Adapted from Burley.[22])

Table 5. Nominal composition of typical Alumel and Chromel alloys

	Composition (units)						
Alloy	Cr	Fe	Mn	Co	Al	Si	Ni
Alumel[a]	trace	trace	2.7	1	1.8	1	Remainder
Chromel[a]	9.5	trace	trace	0.8	trace	1.2	Remainder

[a]Since the compositions of these alloys are adjusted during manufacture so that their temperature-emf relationship matches the reference tables, the composition will vary not only from manufacturer to manufacturer but from batch to batch. This is particularly true of trace elements.

to the decalibration for two reasons: a sheath is a source of impurities which contaminate the thermoelements, and it limits the supply of oxygen needed to passivate the surface of the thermoelement wires.

One feature of a Chromel-Alumel thermocouple which has lead to its wide use is its excellent resistance to oxidation at high temperatures in air. (Table 5 lists the nominal composition of Alumel and Chromel.) The resistance of Chromel to oxidation is due to the formation of an impervious, passive layer of chromic oxide on the surface of the wire. Burley has postulated[24] that the oxidation resistance of Alumel is due to the formation of a protective (passivating) layer of silica (SiO_2) or a silicate at the metal-oxide interface. In a sheathed, small diameter thermocouple (0.5 mm diameter) with a compaction density of the insulation of 75% (25% of the insulation volume is air), an estimated 0.3% of the chromium content of the Chromel wire per unit length could be oxidized to Cr_2O_3 by the oxygen contained within the insulation. This estimate assumes that there are no competing reactions. This assumption is not valid, because the inside surface of the sheath and the Alumel wire both compete for the oxygen. As a result, the partial pressure of oxygen inside the sealed sheath is reduced substantially when the wires and sheath are oxidized at high temperatures. Consequently, a protective film of oxides cannot form on the surfaces of the thermocouple wires, and decalibration proceeds rapidly, particularly for small diameter wires.

The hygroscopic property of MgO has been discussed previously. Lowell[25] has shown that the presence of water vapor accelerates the oxidation of Ni-Cr alloys by activating the vapor phase transfer of Cr_2O_3, which destroys the protective oxide layer on the alloy.

Samples were cut from four locations on the type K thermocouples that had been subjected to different maximum temperature exposures in the 50-hr drift test at 1150°C. The compositional changes in these samples were studied with the ion microprobe mass analyzer (IMMA) to determine the relative changes in major constituents and impurities in the thermoelements resulting from high temperature exposure. The changes indicated in the preliminary results listed in Table 6 are relative to IMMA samples of bare-wire Alumel and Chromel taken from Metrology

Table 6. Results of ion microprobe mass analysis of type K thermocouple samples after 50 hr at 1150°C

Sample	Label[b]	Distance from hot end (cm)	Change in Content of					
			Cr	Fe	Mn	Co	Al	Si
Alumel in Inconel sheath	A	1	+	+	–	0	0	0
	D	14	0	–	(–)	0	0	(–)
Alumel in stainless steel sheath	A	1.5	+	+	0	0	–	+
	E	13	+	(–)	0	0	–	–
Chromel in Inconel sheath	A	1	0	0	+	0	+	0
	D	14	0	0	(+)	(+)	–	0
Chromel in stainless steel sheath	A	1.5	0	0	+	–	–	0
	E	13	0	0	+	0	(+)	+

[a] A + or – symbol in parentheses indicates a slight change.

[b] A, sampled near measuring junction; D and E, sampled from part of the thermocouple outside the furnace.

Laboratory stock. A sample designated "A" was taken near the measuring junction of the thermocouple. Samples marked "D" or "E" taken from a part of the thermocouple located outside the furnace, represent the state of the as-received materials and were included for comparison with the heat treated samples.

In general, the composition changes in samples sheathed in stainless steel were greater than those sheathed in Inconel. In particular, the Alumel element in the stainless steel sheathed thermocouple showed an increase of Cr, but a loss of Al. In the parts of the thermocouple between the A and D sections that were heated, differences in compositions of the Chromel and Alumel wires in the Inconel and stainless steel sheathed thermocouples were measured. These samples showed an increase in Cr and Fe, but the Alumel in stainless steel showed appreciably more Cr pickup than did the Alumel in Inconel. The former also showed a substantial loss of Al, but the Si content increased. The emf data taken during the drift test showed clearly that changes in the Alumel in the stainless steel sheathed thermocouple contributed the major fraction of the change in the thermocouple emf. An estimate of the Cr content of the Alumel based on the IMMA measurements is about 1.3% Cr.

During the manufacture of these sheathed materials, the materials are annealed for a few minutes at ∿1000°C, followed by rapid cooling to room temperature, as the diameter is reduced in stages to 0.5 mm. Production of 0.5-mm-diameter thermocouple materials requires many reduction and annealing steps. Data taken during the initial heating of these thermocouples in both the 1100 and 1150°C, 50-hr tests showed that at 600°C there was a difference of ∿2.5°C in the calibrations between the Inconel sheathed and the stainless steel sheathed thermocouples because the E section of the Alumel in stainless steel showed more Cr pickup than did the D section of the Alumel in Inconel-600.

The effect at 260°C of the concentration of various elements on the thermoelectric power (Seebeck coefficient) of Ni is shown in Fig. 45, which was adapted from Wang, Starr, and Brown.[26] Figures 46 and 47 show the effects on the emf of Ni at several temperatures versus the concentrations of Cr and Fe, respectively. Adding Cr to Ni decreases the Seebeck coefficient more or less regularly with increasing temperature.

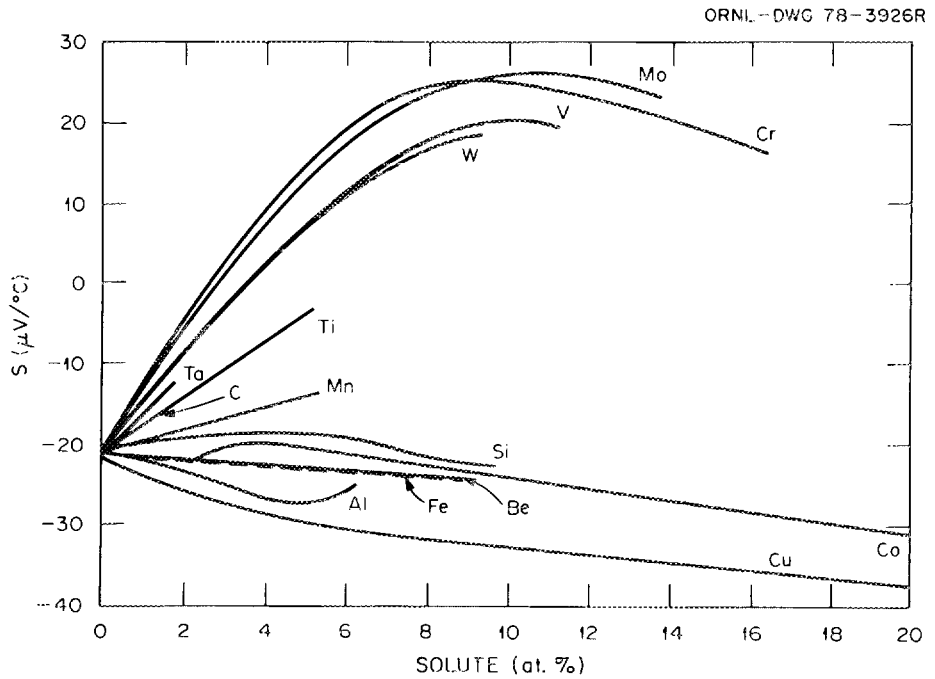


Fig. 45. Absolute thermoelectric power of binary alloys of nickel at 260°C. (Adapted from Wang et al.[26])

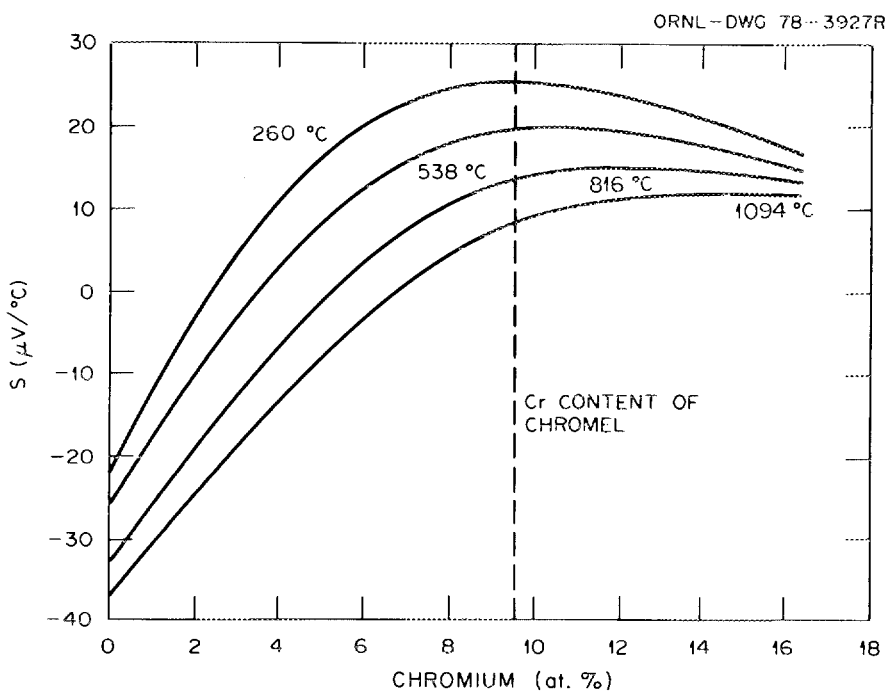


Fig. 46. Absolute thermoelectric powers of binary nickel-chromium alloys as a function of composition and temperature. (Adapted from Wang et al.[26])

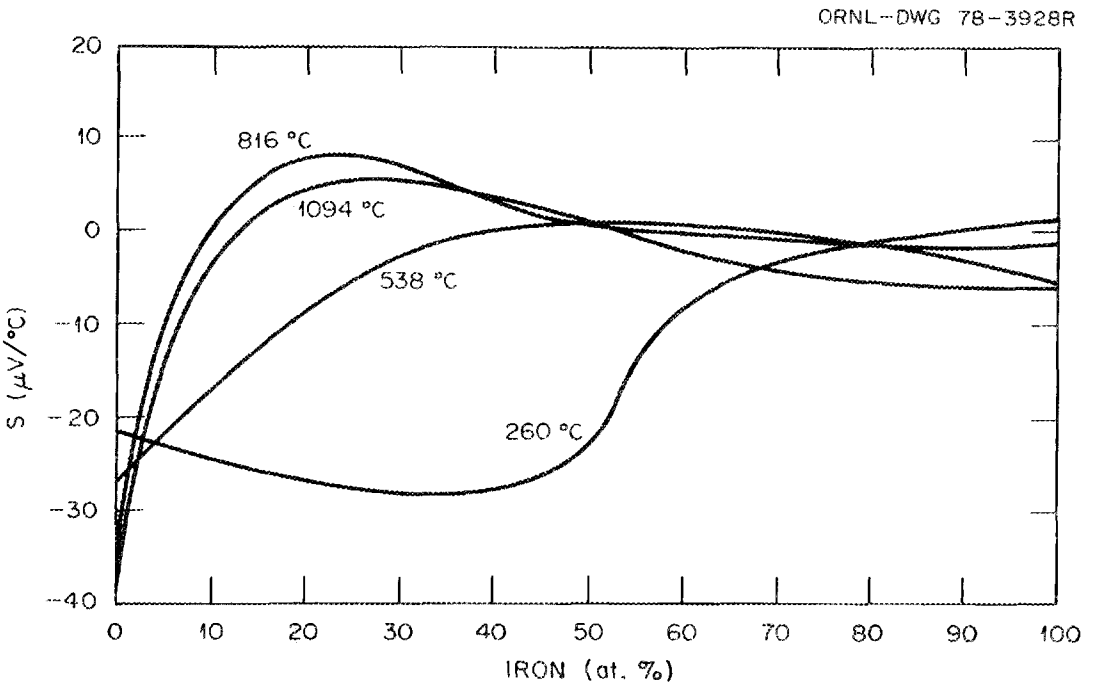


Fig. 47. Absolute thermoelectric power of binary nickel-iron alloys as a function of composition and temperature. (Adapted from Wang et al.[26])

The effect on the Seebeck coefficient of increasing the concentration of Fe, on the other hand, changes sign with increasing temperature. Since Alumel is ∿94% Ni, it might be expected that the effects of impurities in Alumel would be similar to the effects of impurities in pure Ni.

The anomalous effects of impurities such as Fe (or Co) demonstrate that decalibrations measured at one temperature cannot be used to estimate the effects of decalibrations at other temperatures.

5.3.4 Decalibration of Noble-Metal Thermocouples

Two of each of the noble-metal thermocouple-sheath combinations listed in Table 3 were calibrated to 1370°C. The results were presented in Sect. 5.2 under "Decalibration of Sheathed Noble-Metal Thermocouples." One thermocouple of each type was cut at positions selected to yield samples that had received different, maximum temperature exposures. The samples were mounted and prepared for IMMA by the Metallography Section at ORNL.

Although IMMA yielded a host of data which have not been analyzed completely, several facts are obvious. For example, noble-metal thermocouples and base-metal sheaths are incompatible. As with the type K materials, the as-received materials showed contamination from the sheath resulting from the manufacturing process. The elements Al, Mg, Cr, Ni, Mn, and Fe were found in small quantities in the section that had not been heated in the calibration experiments. The results were, in general, similar to those which have been reported for a larger-diameter, Inconel-sheathed, type S thermocouple.[23]

As seen in Fig. 32, the decalibration of 90%Pt-10%Rh sheathed, type S thermocouples was substantially less than that of any other thermocouple-sheath combination, and the drift rate at 1305°C was ∿1 mK/min. In addition, during the initial calibration of these thermocouples, the deviations from the NBS reference tables for type S thermocouples were essentially of the same magnitude as those of high quality, "nominal," bare-wire, type S standards.

5.4 Why Recalibration Will Not Improve Temperature Measurement Accuracy

It has been suggested that the fuel-rod simulator couples could be recalibrated in situ when the loop is held at steady state, thereby enabling correction of the original calibrations. In the preceding sections we have shown that decalibration will affect type K thermoelectric materials to a greater or lesser extent at all temperatures at which they will be used in the CFTL. In the following discussion we will explain why such recalibration could not appreciably reduce temperature measure ment errors; instead such recalibration would obscure real and serious changes in the temperature-emf relationship of the fuel-rod simulator thermocouples.

The reason for this inability to reduce temperature measurement errors through in situ calibration is that the temperature gradients imposed on the fuel-rod simulator thermocouples under steady state conditions will be different from those imposed during an experiment. In

fact, different temperature gradients will occur under different conditions of power and flow, even at steady state. In addition, decalibration effects are temperature dependent.

One could surmise that if these thermocouples were initially homogeneous and remained so in use, or if a decalibration occurred uniformly over the entire length of the thermocouple, the thermocouple would remain homogeneous and it could be recalibrated. This is not so. The Chromel in a Chromel versus Alumel thermocouple becomes appreciably inhomogeneous within a few minutes in sections of the thermocouple heated between ∿350 and 600°C. More inhomogeneities will form due to compositional changes in both the Chromel and Alumel at temperatures above 600°C. The effect on the measured temperature of changes in the temperature gradients imposed on a decalibrated thermocouple were shown in Fig. 43 for the order-disorder decalibration of a type K thermocouple and in Figs. 29 and 38 for the compositional decalibration of types K and S thermocouples, respectively.

5.4.1 Application to CFTL Thermocouples

The locations of the fuel-rod simulator thermocouples in a particular test will be determined after evaluation of two considerations: (1) a sufficient number of thermocouples must be installed to evaluate the heat transfer and thermohydraulic performance of the various bundle configurations, and (2) the temperatures at certain critical locations must be measured so that damage of the bundle will be avoided. Saunders[27] has proposed such a distribution, and we will use this tentative scheme to evaluate the probable errors which can arise because of decalibration of the thermocouples in these locations. A diagram for the placement of these thermocouples is shown at the top in Fig. 48. Calculations of the clad temperature profiles for a 37-rod bundle in a transient test have been made by the General Atomics Co.[28] Figure 48 shows two temperature profiles calculated for an average of 23.7 kW (curve A, state II) and 2.37 kW per rod (curve B, state III), which are for the steady state conditions before the power transient and ∿2 min after the power transient, respectively. Curves A′ and B′ directly below represent the

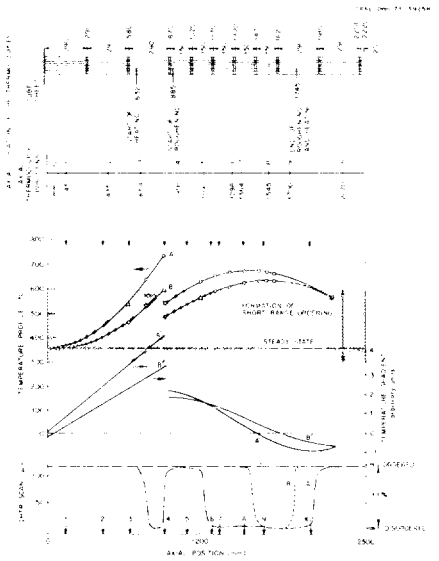


Fig. 48. The effects of the formation and disruption of short range ordering for type K thermocouples as installed in suggested locations in the CFTL bundles. The temperature profiles corresponding to three states are shown in the middle. Curve I is at steady state at 350°C with no power applied to the heaters. Curve A is with 23.7 kW of power applied to each rod, and curve B is with 2.37 kW applied to each rod. Below the temperature profiles are the calculated temperature gradients A′ and B′ dT/dx plotted in arbitrary units. At the bottom are three hypothetical inhomogeneity scans which might result in each of the three states I, II, and III.

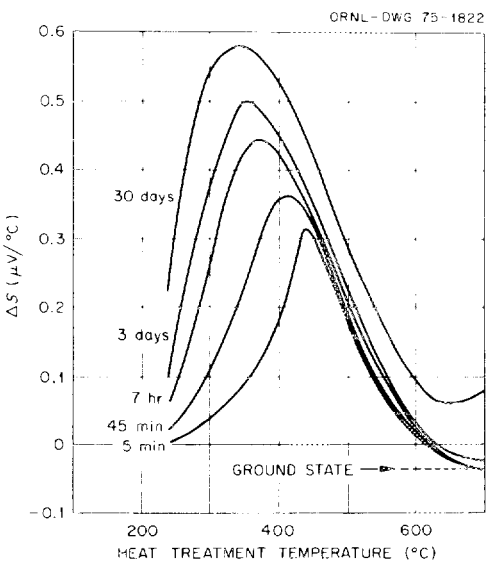


Fig. 49. Change in Seebeck coefficient of Chromel as a function of time at temperature. (Adapted from ref. 20.)

corresponding temperature gradients dT/dx. (No scale is given for the Y axis, as these curves are plotted in arbitrary units.) The band between 300 and 600°C represents the range of temperatures in which the formation of short-range ordering in the Chromel introduces inhomogeneities in type K thermocouples. Plotted at the lower part of the figure and indicated by I, A´´, and B´´, are hypothetical inhomogeneity scans which would be obtained if the fuel-rod simulator thermocouples could be scanned in any of these three states. State I is that of the unpowered bundle.

State I, the unheated bundle. - The program plan for the CFTL specifies a series of studies of unheated bundles in which various tests will be performed with helium gas circulated at ∿350°C. Some of these tests will be extensive, requiring that the bundle temperature be maintained at ∿350°C for hours or days. Under these conditions, based on the data in Fig. 49, the Seebeck coefficient will have increased by ∿1% after 7 hr. This will cause short-range ordering uniformly along the entire length of the thermocouples in the test section. The hypothetical inhomogeneity scan for this condition is indicated by the dashed line I of Fig. 48.

State II, bundle heated, 23.7 kW/rod. - With 23.7 kW/rod, the temperature of two sections of a typical heater rod will be greater than 600°C (Fig. 48). Above 600°C, the short-range ordering that had formed when the thermocouples were at a lower temperature will become disordered, and the portions of the thermocouples in these regions will return to their original "annealed" state. Thermocouples in locations 1, 2, and 3 will not be heated above 600°C and will remain in an ordered state. Portions of thermocouples in locations 6, 7, 8, and 9 will be annealed, and the short-range ordering will be destroyed. The degree of ordering in portions of the lengths of thermocouples in locations 4, 5, and 10 will increase and decrease. Thus, after application of power to the heater rods, thermocouples in locations 4 through 10 will no longer be homogeneous over their lengths that are within the test bundle, and the Seebeck coefficient will become a function of the location, x.

Curve A´´ represents an inhomogeneity scan for this situation. The temperature gradients, dT/dx, given by curve A´, are also a function of

x. The emf output given by Eq. (1) is thus a function of the relative locations of the inhomogeneities and the temperature gradient. As shown by Kollie et al.[20] this can cause an uncertainty in the temperature measurement of as much as 1.1%.

State III, bundle heated, 2.37 kW/rod. – When the power to the bundle is reduced to 2.37 kW/rod, a new temperature profile B and a new temperature gradient B′ will be produced. Thermocouples in positions 4 through 8 will be returned to the region of temperatures in which ordering occurs. Thus, these thermocouples will tend to reorder at a rate which is both temperature- and time-dependent. Thermocouples in locations 9 and 10 will retain their disordered state in those portions above 600°C; however, the portions below 600°C will reorder. Because the location of the disordered portions of thermocouples 9 and 10 will change with respect to the location of the new temperature gradient, their temperature-emf relationship will also be uncertain. Curve B′′ illustrates the inhomogeneities in this situation.

5.4.2 Effect of Order-Disorder on Recalibration

As explained in the preceding discussion, thermocouples in different locations will behave differently, depending on the state of the loop. At this point, a question to be considered is: What will be the effect on temperature measurement accuracy if any one of these states is chosen for "steady state" recalibration?

Recalibration in state I, the unheated bundle. – If the fuel-rod simulator thermocouples are to be recalibrated at a steady state with the bundle unheated after a series of tests has been completed in which the bundle was heated, what is to be expected? First, the loop would probably have to remain at this condition for several hours — perhaps overnight — before it would become equilibrated. Consequently, a uniform temperature would be established across the bundle; that is, dT/dx, the temperature gradient, would be zero within the test section. The state of order or disorder of portions of the thermocouples inside the bundle would be irrelevant. If this were the case, the emf of the thermocouples would be determined entirely by the portion of the thermocouple

in the temperature gradient at the entrance to the test bundle. During powered tests, however, it would be precisely these decalibrated portions of the thermocouples inside the test bundle that will cause temperature measurement errors which would not be revealed by steady state recalibrations. Under steady state conditions, the precision of measurement of temperature with type K thermocouples has been shown to be $\sim$0.2°C.[20]

It would be wrong to assume, however, that the measurement accuracy of a standardized procedure which reproduces the original steady state calibration would also represent temperature measurement accuracy. Reproducibility, or precision, is a necessary condition for measurement accuracy, but it is not sufficient. Temperature measurement accuracy must refer to the agreement between experimental thermocouples and an accepted scale of temperature, IPTS-68.

Recalibration in state II, 23.7 kW/rod. - Heating the bundle will tend to destroy the short-range ordering in portions of thermocouples in certain locations, as discussed previously. Doubt as to the extent of the remaining order, the rate at which ordering occurs, and the location of the order-disorder induced inhomogeneity with respect to the temperature gradient will result in temperature measurement uncertainty. However, there is another consideration. All thermocouples with their measuring junctions located at a given level will tend to agree since they will all pass through similar temperature gradients. Again, the precision of the indicated temperatures at a particular level does not represent temperature measurement accuracy.

Recalibration in state III, 2.37 kW/rod. - The temperature profile B in Fig. 48 is that predicted less than 2 min after the power transient. Establishment of a steady state presumably would take considerably longer so that the predicted temperature gradients in Fig. 48 would be considerably different at steady state. If the recalibration data were to be recorded after the maximum temperature in the bundle at location 9 had reached, say, 500°C, the entire length of the thermocouples would be in a region of temperature conducive to the formation of short-range order. This recalibration would tend to agree with the steady state calibration in state I, but it would not reflect the true uncertainty in temperature measurement immediately after the power transient.

Other factors. - A single case was taken for simplicity in the preceding examples. In the CFTL experiments, there will be changes not only in power levels, but the helium flow rate will be changed and the power applied to the bundle will be skewed across the bundle. All of these changes will interact to rearrange the temperature gradients in the bundle.

5.4.3 Recalibration

All of the preceding arguments against recalibration were directed toward the order-disorder decalibration effect in type K thermocouples. We believe that this effect will be a major cause of uncertainties in the fuel-rod simulator temperature measurements, and, furthermore, these uncertainties will occur in a temperature region in which most of the experimental work will be concentrated. The use of type S thermocouples, sheathed with 90%Pt-10%Rh, will avoid the errors due to short-range ordering in type K thermocouples. Type S thermocouples, however, will be subject to decalibration caused by compositional changes at high temperatures; but, since the high temperature experiments on each bundle will be conducted at the end of the test series, the compositional decalibration of type S thermocouples will not affect the balance of the measurements taken prior to the high temperature runs. Under these conditions, the temperature measurement uncertainties using type S thermocouples will remain within the CFTL limits of error over most of the temperature range.

6. OVERALL TEMPERATURE MEASUREMENT UNCERTAINTIES

After the sources of temperature measurement errors listed in Table 1 are considered, the cumulative effects of these errors on thermocouple thermometry in the CFTL fuel rod simulators can be estimated. In the CFTL program plan[2] the desired temperature measurement uncertainties for the CFTL experiments are specified as ±8°C below and ±15°C for temperatures above 800°C. Because the magnitudes of many of the errors are temperature dependent, they cannot be presented easily in tabular form, but are best visualized graphically.

The cumulative uncertainty due to the extension lead wires, the reference zone box, and the calibration of a single, type K, Inconel-sheathed thermocouple is shown as ±2°C in the middle of Fig. 50. To this uncertainty is added the uncertainties attributed to the emf measuring system indicated by the curves marked +DAS and -DAS: the thermostated active filters of the measuring system introduce an estimated uncertainty of ±1°C; and the full-scale uncertainty of 0.25% in the analog-to-digital converter contributes the step changes in the uncertainty because of range changes at 20 (270°C), 40 (485°C), and 80 mV (967°C).

Shown in Fig. 51 is a similar plot of cumulative uncertainties for a type S thermocouple. The uncertainties due to the extension lead wires (calibrated), reference box, and calibration total ±1.6°C to 1000°C; this total increases to ±2°C between 1000 and 1400°C owing to an increased uncertainty of the calibration data. Because of the lower output of a type S thermocouple, the analog-to-digital converter will continue to operate its most sensitive range to ∿1035°C; consequently, the resultant uncertainty is ±2.4°C between 350 and 1035°C. We believe that even with the lower output of the type S thermocouple the additional uncertainty in the data acquisition system due to drift in the active filter amplifiers need not exceed ±1°C. The greater uncertainty below ∿300°C in Fig. 51 is caused by the decrease in sensitivity of the type S thermocouple from 300 to 0°C. (Since this temperature region is below the range of temperatures contemplated in the CFTL experiments, discussion of errors within the region is irrelevant to this report.)

We reemphasize the following important point: one cannot conclude that because the output of a type K thermocouple is higher than that of a type S thermocouple, the temperature measurement accuracy in a region of major importance to the CFTL, namely 485 to 800°C, will be improved. Instead, one can expect the higher output of the type K thermocouple in this region to be offset by the greater uncertainty in the analog-to-digital converter when it is switched automatically from the 20-mV range to the 40-mV range.

Uncertainties due to decalibration will add to the uncertainties due to the measuring system. With type K thermocouples there is an even

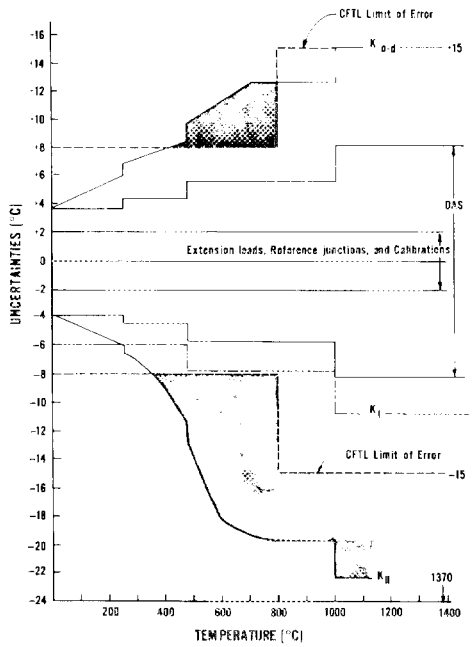


Fig. 50. Cumulative uncertainties for a type K thermocouple sheathed in Inconel-600 as a function of temperature.

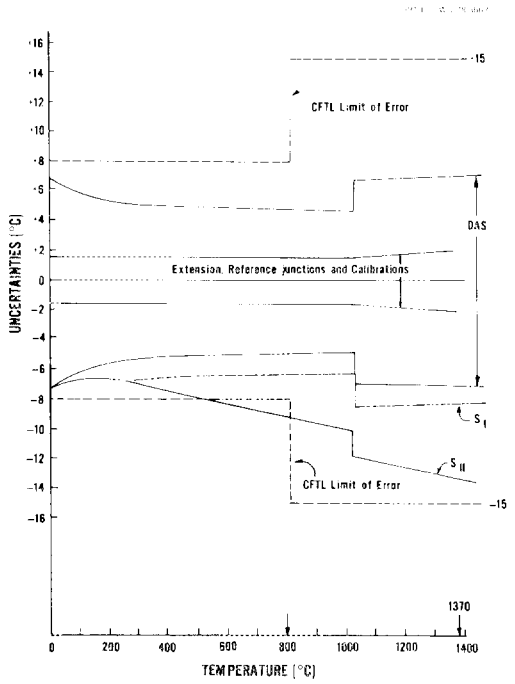


Fig. 51. Cumulative uncertainties for a type S thermocouple sheathed in 90%Pt-10%Rh as a function of temperature.

greater uncertainty due to the order-disorder transformation (line K_{o-d} in Fig. 50). With the inclusion of uncertainties due to the order-disorder transformation, the overall uncertainty of a fuel-rod simulator temperature measurement would exceed the tolerance limits in the region of major importance to the CFTL experiments, namely 400 to 800°C. Since the error due to order-disorder transformation is always positive, the K_{o-d} line occurs only in the upper half of the plot of cumulative uncertainties. The compositional decalibration errors of the first and second kinds are indicated by K_I and K_{II}, respectively. Since the compositional changes will affect the thermocouples only after exposures to temperatures above ∿800°C, uncertainties to the extent indicated by K_{II} will occur only after ∿50 hr at 1100°C. Also, since the thermocouples will be exposed to high temperatures only at the end of each test, uncertainties of this magnitude will not occur during the major portion of the tests. In this case, the error limit in the lower half of the plot is bounded by the "-DAS" curve.

For type S thermocouples, there is no appreciable order-disorder transformation in either Pt or 90%Pt-10%Rh alloys; thus there is no additional uncertainty over that of the "+DAS" curve in the upper half of Fig. 51. In the lower half of Fig. 51, decalibration errors of the second kind indicated by curve S_{II} exceed the CFTL tolerance limits between ∿400 and 800°C. For the same reasons given before, errors of this magnitude probably will not occur unless the test assembly is subjected to additional testing in this temperature region after testing at ∿1100°C has been completed.

Uncertainties due to thermal shunting, electrical leakage, and electrical shunting are not included in Figs. 50 and 51. Accurate evaluation of these errors will have to await the start up of the loop, since this will require a knowledge of the actual temperature profiles.

Figures 50 and 51 illustrate temperature measurement uncertainties with a single calibrated thermocouple. In practice, it may be impractical for the CFTL to calibrate each thermocouple installed in a fuel-rod simulator. Specifications written for the purchase of type K thermocouple assemblies include a specification of either "standard" grade (3/4%) or "special" grade (3/8%). More specifically, the ISA tolerances for these

grades are as follows:[8] (1) for the 3/4% grade, ±2.2°C from 0 to 277°C, and 0.75% of T from 277 to 1250°C; and (2) for the 3/8% grade, ±2.2°C from 0 to 277°C, and 0.4% of T from 277 to 1250°C. Figure 52 shows the result of adding the ISA tolerances for special grade (3/8%) type K thermocouple materials to the cumulative error plot indicated by the curves ±3/8%.

The ISA tolerance is considered a "batch" tolerance; that is, during their first heating cycle, the deviation of a batch of thermocouple materials should be within this tolerance with reference to the NBS thermocouple reference tables. The variation among thermocouples made from one batch of materials might reasonably be expected to be considerably smaller. From experience in the Metrology Research and Development Laboratory and in the field shops of the Instrumentation and Controls Division with calibrations of larger-diameter, type K thermocouples (1 to 3 mm diameter), we have observed that the variation within a batch of type K thermocouples is usually ±5°C or less at 1000°C. For reasons previously cited concerning the greater variability of 0.5-mm-diameter thermocouple materials, the variation observed within single batches of 0.5-mm-diameter thermocouple assemblies is much larger. Twelve 0.5-mm-diameter thermocouples selected randomly from a batch of 240 standard grade (0.75%), type K thermocouples assemblies sheathed in Inconel-600 procured for fuel-rod simulators being developed for the CFTL were calibrated in our laboratory. One-third of the sampled thermocouple assemblies were out of tolerance at 1000°C, and there was a spread of 14°C at this point between the highest and lowest of the 12.

Random samples from two large batches of type K thermocouples in stainless steel sheaths ordered from the same manufacturer showed a marked difference in the initial deviations in acceptance calibrations. Six assemblies were selected randomly from an order of 1064 assemblies and calibrated at 500°C. The average deviation was 0.12%, well within the ISA 3/8% tolerance limit. Calibration of a second random sampling of 20 assemblies from an order of 200 assemblies showed that 25% exceeded the 3/4% tolerance. Another lot was purchased from a second manufacturer to meet the 3/8% specification, but 56% of the assemblies were outside this

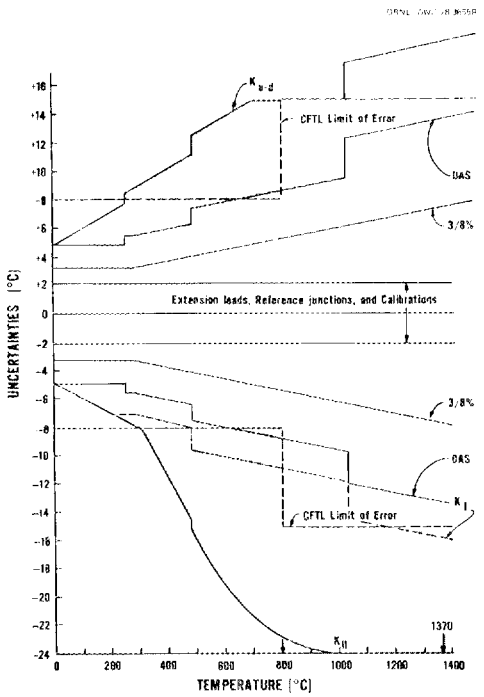


Fig. 52. Cumulative uncertainties for Inconel-600 sheathed, type K thermocouples after adding the ISA 3/8% tolerance.

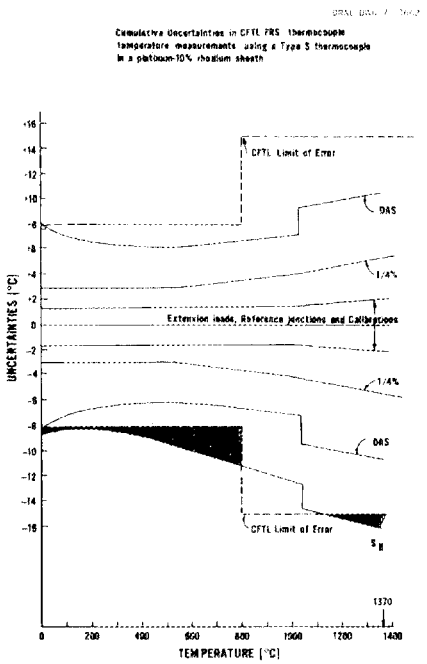


Fig. 53. Cumulative uncertainties for type S thermocouples, including the ISA 1/4% tolerance.

limit. Thus, the 3/8% tolerance added to the cumulative error plot in Fig. 52 may be too optimistic.

The cumulative error plot in Fig. 53 shows the results of adding the ISA tolerance for standard grade, type S thermocouples (±1.5°C or ±0.25%, whichever is greater). As mentioned in the preceding discussion of Fig. 51, the errors that exceed the CFTL tolerance limits in the region below 800°C, in practice, would not be manifested until after completion of the planned test schedule. Above ∿1150°C, the tolerance limits would also be exceeded; however, the temperature measurement accuracy requirements could probably be relaxed by ∿1°C for the single tests with each assembly that will approach the melting point of stainless steel (1370°C).

7. SUMMARY

Fuel-rod simulator cladding temperatures in the CFTL experiments will be determined using 0.5 mm diameter sheathed thermocouples attached to the inner walls of the simulators. A detailed analysis of the potential sources of errors in these temperature measurements determined that insufficient information existed about the characteristics of these small diameter thermocouple materials, especially above 800°C. It was found that performance data available on larger diameter thermocouples cannot be extrapolated to predict the behavior of small diameter sheathed thermocouples, nor can the data available from lower temperatures be extrapolated to higher temperatures. Many of the processes which degrade the performance of the thermocouples depend exponentially on temperature. The small wire size of the thermoelements in these sensors results in more rapid and extensive decalibration than in larger diameter thermocouples. The performance of a variety of materials and material combinations was measured to provide enough information to choose a combination of thermocouple type and sheath material for optimum behavior. In addition, more reliable estimates can now be made of the overall temperature measurement uncertainties resulting from the use of 0.5 mm diameter sheathed thermocouples.

An analysis of nine sources of temperature errors showed that additional information was required about electrical insulation resistance -- and the related effects such as electrical shunting and electrical leakage -- and decalibration effects, particularly above 900°C (but also between 350 and 600°C).

Measurements of the electrical insulation resistance and its temperature dependence have been reported elsewhere.[13] Additional data were obtained on the effects of electrical leakage and shunting on temperature measurements, particularly above 900°C. These measurements were used to develop a mathematical model to correct the errors arising from these effects. A report of this work has also been published.[17]

Studies on the decalibration of 0.5-mm-diameter sheathed thermocouples showed that the stability of these materials was strongly influenced by the composition of the sheath. Substitution of Inconel-600 for type 304 stainless steel for the sheath material of 0.5-mm-diameter type K thermocouples, for instance, resulted in markedly improved stability at temperatures above ∿900°C. Some small diameter sheathed Nicrosil vs Nilsil thermocouples were tested along with the type K materials from 600 to 1000°C. They displayed superior performance to ∿800°C, but at 1000°C the rate of decalibration was 2 to 3 times that of type K. It was also found that noble-metal thermocouples (type S or type B) decalibrated severely at 1000°C and above when base metals were used for the sheath material. With noble-metal sheaths, however, the stability of the 0.5-mm-diameter sheathed thermocouples approached that of standard grade, bare-wire noble-metal thermocouples.

Ion microprobe investigations of decalibrated, type K and type S, small diameter sheathed thermocouples disclosed that the thermoelements in the bulk 0.5-mm-diameter thermocouple materials as received from the manufacturers were contaminated by impurities from the sheath and the insulation. Few samples of 0.5-mm-diameter sheathed type K materials were found which had temperature errors within the ISA 3/8% tolerance limits over the range of CFTL usage. Variations greater than 3/4% were observed in a random sample of 12 assemblies from a single batch of 240 thermocouple assemblies.

The effects of short-range ordering errors on the temperature measurement uncertainties were analyzed, and it was demonstrated that recalibration of decalibrated thermocouples could, and probably would, lead to erroneous and misleading results.

Finally, the temperature measurement uncertainties were combined to give an overall estimate of the error bounds for the range of temperatures to be encountered in the CFTL experiments. It was shown that the uncertainties with type K thermocouples would exceed the accuracy requirements of the CFTL program plan, but that type S thermocouples would be satisfactory.

From the results of the research and testing on small diameter thermocouples for CFTL, we can state the following:

1. No small diameter thermocouples have met the ISA 3/8% tolerance over the range of CFTL usage; therefore, each thermocouple assembly procured should be calibrated to provide corrections.

2. The decalibration of type K thermocouples is due to effects in both the Chromel and Alumel elements. These effects tend to cancel at some temperatures but to add at other temperatures in a fixed temperature gradient. A change of the temperature gradients, which can be expected to happen during CFTL transient experiments and to a lesser extent during changes in power or flow, will produce errors that will exceed CFTL uncertainty limits in the region of greatest interest, namely, from 400 to 800°C.

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