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THERMOCOUPLE ERROR ANALYSIS IN THE DESIGN OF LARGE ENGINEERING EXPERIMENTS *

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CONTENTS

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 at ORNL. The sources of error considered were extension lead wires, reference junctions, the data acquisition system, calibration and decalibration of the sensors, thermal shunting, electrical shunting, electrical leakage, and electrical noise.

It was found that because of the relatively small dimension (wire sizes of 0.1 mm diameter) the behavior of 0.5 mm diameter thermocouples could not be extrapolated from the known behavior of larger diameter thermocouples, particularly above 900°C.

Electrical shunting and leakage at higher temperatures can result in large temperature measurement errors. The magnitude of these errors depends on the temperature profile through which the thermocouple passes.
Size effects are also important in the decalibration of small diameter sheathed thermocouples. The sheath material becomes a factor in decalibration above 900°C. Nicrosil vs Nisil thermocouples--new alloys develoned to replace type K alloys--decalibrated more extensively above 800°C than the type K materials in either type 304 stainless steel or Inconel 600.

Small diameter thermocouples with platinum based thermoelements are even more sensitive to the choice of sheath material than the base metal alloys. Platinum based thermocouples with platinum alloy sheaths are, however, superior to base metal thermocouples, particularly 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

The Core Flow Test Loop (CFTL) is a large scale engineering experiment at the Oak Ridge National Laboratory (ORNL) to provide structural and heat transfer data for the design of fast gas-cooled nuclear reactors. It is a simulation experiment, in that full-scale high density electrically heated "fuel-rod simulators" are substituted for nuclear fuel rods. Experiments on 0.5-mm-diameter, compacted metal sheathed thermocouples which will be used to measure cladding temperatures in the fuel-rod simulators were carried out in the Metrology Research and Development Laboratory (MRDL) of the Instrumentation and Controls Division at ORNL. The objectives were: identify and analyze the sources of temperature measurement errors in 0.5-mm-diameter sheathed thermocouples used to measure the surface temperature of the cladding of the fuel-rod simulators in the Core Flow Test Loop (CFTL); devise methods for reducing or correcting for these temperature measurement errors; estimate the overall temperature measurement uncertainties; and recommend modifications in the manufacture, installation, or materials used, to minimize temperature measurement uncertainties in the experiments.

Because the diameters of the thermoelement wires, as well as the separation between the wires or between the wires and the sheath in the 0.5-mm-diameter thermocouple assemblies are so small (on the order of 0.05 to 0.1 mm) the mechanical, electrical, and chemical behavior of these thermocouples are different from larger diameter thermocouples. Above 600°C, the thermoelectric properties of these thermoelements may be altered by chemical reactions 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 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 0.5-mm-diameter thermocouples is more rapid and severe at high temperatures than larger diameter thermocouples.

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

Table 1. Error sources in CFTL thermocouple thermometry

1. Extension lead wires
2. Reference junctions
3. Data acquisition system
4. Calibrations
5. Decalibration
6. Thermal shunting
7. Electrical shunting
8. Electrical leakage
9. Electrical noise

2. 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, precision, and terms derived from these. This discussion relies heavily on the work of Eisenhart[2] and Ku.[3,4]

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."[2]

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[4] 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.

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 should also be 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 <u>independent</u> and normally distributed, then the estimate for the random error of the final quantity is given by the square root 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 errors can be both constant and changing. Constant systematic errors can often be corrected for if their effect is known. Simple reproducibility, or <u>precision</u>, 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 instumentation. 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[5] mentions five different methods ranging from "much too daring" to "a wee bit conservative." Ku[3] recommends that since there are no formally accepted ways to estimate systematic errors or to combine them, these errors should be discussed in sufficient detail to enable others to make their own judgments. In this paper the error sources for the temperature measurements in CFTL fuel-rod simulators made with 0.5-mm-diameter, internally attached, sheathed thermocouples have been identified and classified as to type. To combine the errors from the sources for which it is possible to make a priori estimates, we have used a conservative approach of simple algebraic addition to yield overall error bounds. This approach gives a basis for comparison of the relative magnitudes of the uncertainties from the different sources of temperature measurement errors. Actual experience with operating systems will be required to make more accurate estimates of the probable errors.

3. TEMPERATURE MEASUREMENT UNCERTAINTIES

3.1 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 that 200 thermocouple connections to the data system must be broken and made when the test apparatus is exchanged. In addition, the total length of the small diameter thermocouples must be minimized to reduce the electrical resistance of the thermocouple circuits. For these reasons, the thermocouples will be connected using extension lead wires and connectors.

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. A 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.[6] 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 over a limited temperature range. 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 are relatively constant, and therefore, systematic errors.

The deviations were measured from 0 to 140°C of samples of type S extension wires from several manufacturers from lots of materials purchased over a period of about ten years. The range of deviations was within the ANSI tolerance stated in preceding paragraph. The results (Fig. 1) show that calibration can reduce the uncertainty due to the systematic errors from the extension wires to ±0.1°C or less. (This is also true for type K extension wires.) In other words, calibration of each lot of extension lead wires used can reduce the systematic errors from this source to the level of the random errors in the thermocouple measurement system.

3.2 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_0. Therefore, an uncertainty in T_0 will result in a comparable uncertainty in the measured temperature. For large numbers of thermocouples, such as in the CFTL, it is common practice to install a zone box to establish T_0. Usually a zone box is thermostatically controlled at 65°C, and this temperature 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 is a constant systematic error if the temperature gradients are small and constant within the zone box and the box remains in thermostatic control.

3.3 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 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 acquistion 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.

3.4 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, 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)[7] adopted by the Internaltional 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 will 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. For the working sensors installed in the CFTL, the <u>initial</u> 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 by 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.

3.5 Thermal Shunting

Thermal shunting is a broad category which includes errors that result when the thermocouple does not actually attain the temperature of the location it is intended to measure.

A thermocouple, just as any other contacting temperature sensor, disturbs the temperature distribution of an 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 and radiation. 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 is frequently the result of 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 isolate the thermocouple electrically from the object by inserting a thin sheet of insulating material between the junction and the object (Fig. 2). 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 distbriution 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.

In transient-temperature measurements, the effects of thermal shunting are intensified and can often be the dominant error. For fast transient of the order of 100°C/s, one estimate of the probable error in the CFTL fuel-rod simulators is 30°C.[8] Thermal shunting errors are nonconstant systematic errors, and their evaluation will require a combination of experimental tests and analytical modeling.

3.6 Electrical Shunting

The electrical resistance of all insulating materials used in sheathed thermocouple assemblies decreases exponentially with increasing temperature.

Figures 3a, 3b, and 3c 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, 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 the insulation resistance approaches the loop resistance. A significant loss of the thermocouple emf can occur at this temperature and above because of electrical leakage through the thin insulation layer between the thermoelements. This results in erroneous indications of temperature which are less than the actual temperature above about 1000°C[9] as illustrated in Fig. 3a.

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. 3b, 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 -- <u>not that of the measuring junction</u>. This effect has been observed in 0.5-mm-diameter thermocouples with no junction. For example, the emf measured on an open-circuited "thermocouple" which extended through a peak temperature of ∼1000°C with the open end at room temperature 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 diameter thermocouples.

Electrical shunting errors depend on the temperature and the temperature profile and are systematic errors.

3.7 Electrical Leakage

Leakage of small dc currents from the heating element of the fuel rod simulator into the thermocouple circuit (Fig. 3c) can be observed with decreased insulation resistance. 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. The resulting temperature measurement errors amounted to hundreds of degrees Celsius for a few milliamperes of dc current on the thermocouple. However, the errors in a CFTL fuel-rod simulator will not be nearly so large as this. Since 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 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.

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

3.9 Electrical Noise

In the CFTL, as in any system in which larger 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 intrisinc, 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.[10] Passive filtering would reduce the effect of this induced noise, but would impair the ability of the data system to follow high-speed measurements during the fast CFTL transients, and therefore, would not be acceptable. Active filters can essentially solve this problem. Active filters have been designed with low-drift operational amplifiers in a thermostatically controlled enclosure, which will add less than 1°C to the overall errors.[11] This error is introduced into the measurement system by the drift of the filter amplifiers and will be relatively constant and systematic.

4. DECALIBRATION

4.1 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 of (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.

Decalibration errors are most frequently observed as a drift of the temperature-emf relationship (and therefore the emf output) 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 can also 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 remains at the same temperature. Decalibration errors of this kind are important for experiments such as the CFTL because changes in the power input and helium flow change the temperature gradients imposed on the fuel-rod simulator thermocouples. This can result in large nonconstant systematic temperature measurement errors.

The magnitude of decalibration errors 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, (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. 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 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 type, sheath, and insulator material should be selected which minimize decalibration.

4.2 Some Experimental Results

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. 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. 4. 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%.

Figure 5 shows a similar plot for the type K thermocouple in Inconel. The measured drift at 1150°C 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 (INMA).

<u>Decalibration of sheathed noble-metal thermocouples</u>. - Samples of type S and type B materials listed in Table 3 were calibrated to 1370°C. During the calibration the temperature of the furnace was held at 1305°C for a period of 20 min, while the outputs of the thermocouples were recorded. The drift rates for the different types of thermocouples in the test, plotted in Fig. 6 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.

4.3 Causes of Decalibration

The causes of decalibration are many and include changes in the metallurgical state, as well as in composition.[12] 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 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. 7). In the following sections, the discussions are limited to decalibration of types K and S thermocouples.

4.3.1 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. 8). Because of the large surface-to-volume ratio of such small diameter wires, chemical effects that normally would be apparent only 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.

4.3.2 Order-Disorder Effect in Type K

Kollie et al.[13] 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; and between 0 and 600°C, the temperature measurement errors caused by the order-disorder transformation approach 1.1% of the measured temperature in this temperature range.

The results of the order-disorder transformation on temperature measurement made with type K thermocouples are illustrated by Fig. 9.[13] The initial calibration curve of an annealed thermocouple, curve A, lies within the ±3/8% ISA allowable error for special grade

Chromel-Alumel, 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. 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.

In the drift tests conducted at 600°C, the decalibrations were due to the effects of ordering. These errors, due to order-disorder will degrade the fuel-rod simulator temperature measurements where type K thermocouples are used. More particularly, these errors occur in the temperature region of greatest interest in the CFTL experiments, namely, between ~400 and 600°C.

4.3.3 Decalibration by Compositional Changes in Type K Thermocouples

Numerous investigators have studied the decalibration of type K thermocouples in air. Burley,[14] 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 300 hr. Figure 10 summarizes some of his results. For Chromel-Alumel pairs from four different sources, the changes in the emf output of these thermocouples, (Fig.10a) were caused by changes in both the Chromel element (Fig. 10b) and the Alumel element (Fig. 10c). At 600 and 800°C the changes in the thermocouple output were caused predominantly by changes in the Chromel element, but at 1000°C the changes in the thermocouple output resulted 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[15] to contribute 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. 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. Hughes and Burley have postulated[16] 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 with an insulation compaction density of 75% (15% of the insulation volume is air or gas), 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. The partial pressure of oxygen inside the sealed sheath is reduced substantially by oxidation of the wires and sheath at high temperatures. Consequently, the protective oxide films cannot form on the surfaces of the thermocouple wires, and decalibration proceeds rapidly, particularly with small diameter wires.

The hygroscopic property of MgO has been discussed previously.[17,18] Lowell[19] has shown that the presence of water vapor accelerates the oxidation of Ni-Cr alloys by activating the vapor phase transfer of $Cr2O_3$, 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 (INMA) to determine the relative changes in major constituents and impurities in the thermoelements resulting from high temperature exposure.

In general, the composition changes samples sheathed in stainless steel were greater than those sheathed in Inconel. Differences in compositions of the Chromel and Alumel wires in the Inconel and stainless steel sheathed thermocouples were measured in sections of the thermocouple that were heated. 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 in sections near the measuring junction 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 requirers many such 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. The section of the Alumel in stainless steel which was outside the furnace in this experiment (and therefore, representative of the as-received materials) showed more Cr pickup than did the corresponding section of the Alumel in Inconel-600.

4.4 Why Steady-State 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 in the unpowered steady state at 350°C, thereby enabling periodic correction of the original calibrations. In the preceding sections it was 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. The following discussion will explain why a steady-state recalibration could not appreciably reduce temperature measurement 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 both powered and unpowered 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.9 for the order-disorder decalibration of a type K thermocouple and Figs. 11 and 12 for the compositional decalibration of types K and S thermocouples, respectively.

4.5 An Acceptable Method for Recalibration

All of the preceding arguments against recalibration were directed toward the order-disorder decalibration effect in type K thermocouples. This effect will be a major cause of uncertainties in the fuel-rod simulator temperature measurements, and furthermore, these uncertainties will occur in a temperaure region in which most of the experimental work will be concentrated. Inclusion of type S thermocouples, sheathed with 90%Pt-10%Rh, would allow the errors due to short-range ordering in type K thermocouples to be evaluated. 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 a 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 would remain within the CFTL limits of error over most of the temperature range.

5. 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. The desired temperature measurement uncertainties for the CFTL experiments are ±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.13. 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 ±0.25% full-scale uncertainty of 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. 14 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 of the standard. 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. 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. 14 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 this 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 a greater uncertainty due to the order-disorder transformation (line K in Fig. 13). 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. Compositional decalibration errors indicated by K_{II}. 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 the 90%Pt-10%Rh alloy; thus there is no additional uncertainty over that of the "+DAS" curve in the upper half Fig. 14. In the lower half of Fig. 14, decalibration errors 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. 13 and 14. Accurate evaluation of these errors will have to await the start up of the loop, since this will require experimental determination of the actual temperature profiles.

Figures 13 and 14 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 normally 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: (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 15 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 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 at ORNL with calibrations of larger-diameter, type K thermocouples (1 to 3 mm diameter), the variation observed 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, larger differences are observed within a single batch of 0.5-mm-diameter thermocouple assemblies.

The cumulative error plot in Fig. 16 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. 14, 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).

6. 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.[9] Additional data were obtained on the effects of electrical leakage and shunting on temperature measurements, particularly above 900°C.[1]

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, type K thermocouples have met the ISA 3/8% tolerance over the range of CFTL usage; therefore, each thermocouple assembly procured may need to 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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Table 2. Errors in data acquistion due to analog-to-digital converter

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

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

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

FIGURE CAPTIONS

Fig. 1. Deviations of ten samples of type SX extension wire obtained from various sources at ORNL from 0 to 140°C.

Fig. 2. 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.

Fig. 3. 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. 4. Changes observed in a type K thermocouple sheathed in type 304 stainless steel during a 50 hr exposure at 1150°C.

Fig. 5. Changes observed in a type K thermocouple sheathed in Inconel-600 during a 50 hr exposure at 1150°C.

Fig. 6. 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. 7. Illustration of several sources of decalibration at high temperature in sheathed thermocouple assemblies.

Fig. 8. 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. 9. Curves from calibration data taken on heating and cooling an annealed and a he‹ .-treated type K thermocouple.

Fig. 10. Thermocouple emf drift vs time curves for four conventional type Chromels and Alumels in terms of averages of all four. (Adapted from Burley.[214])

Fig. 11. Decalibration errors revealed by shifting the thermocouple in the decalibrating furnace after a 50 hr exposure at 1100°C.

Fig. 12 Calibration curves and declibration errors for a type S thermocouple sheathed in 90%Pt-10%Rh after 50 hr at 1100°C.

Fig. 13. Cumulative uncertainties for a type K thermocouple sheathed in Inconel-600 as a function of temperature.

Fig. 14. Cumulative uncertainties for a type S thermocouple sheathed in 90%Pt-10%Rh as a function of temperature.

Fig. 15. Cumulative uncertainties for Inconel-600 sheathed, type K thermocouples after adding the ISA 3/8% tolerance.

Fig. 16. Cumulative uncertainties for type S thermocouples, including the ISA 1/4% tolerance.

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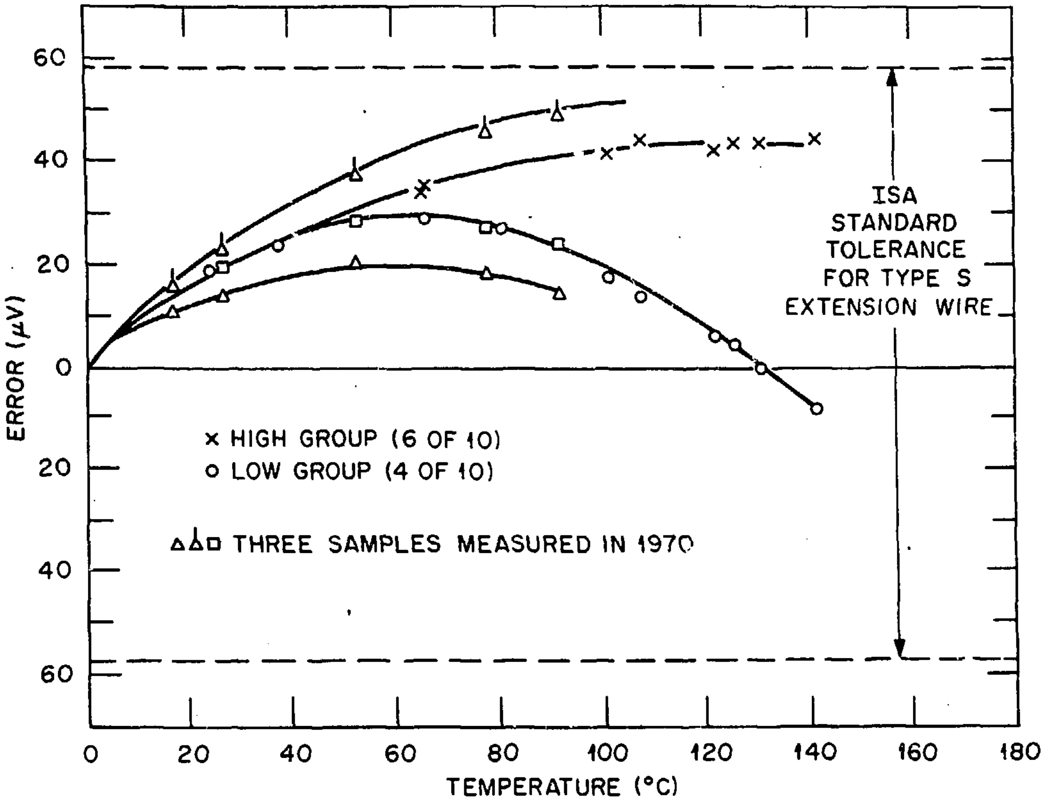

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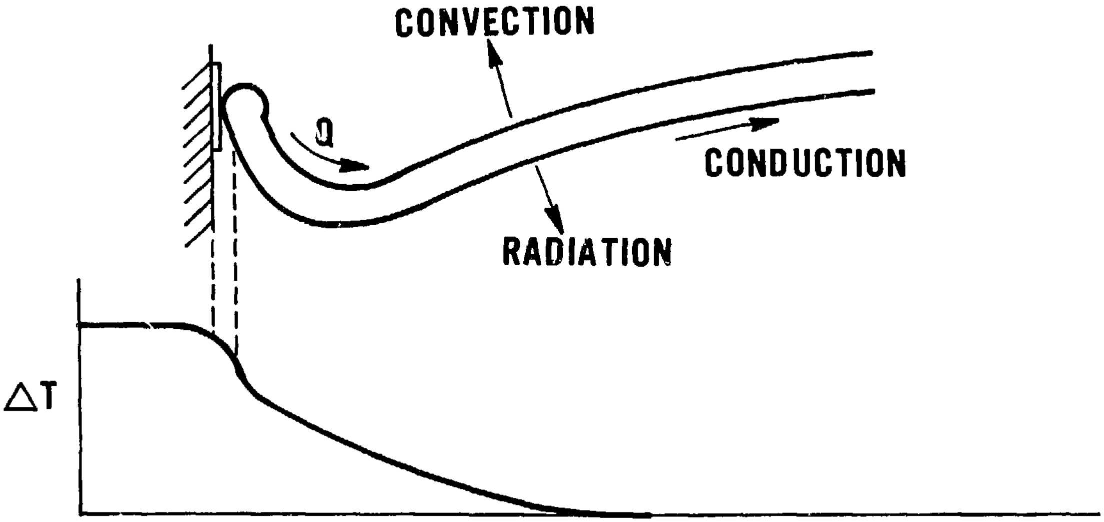

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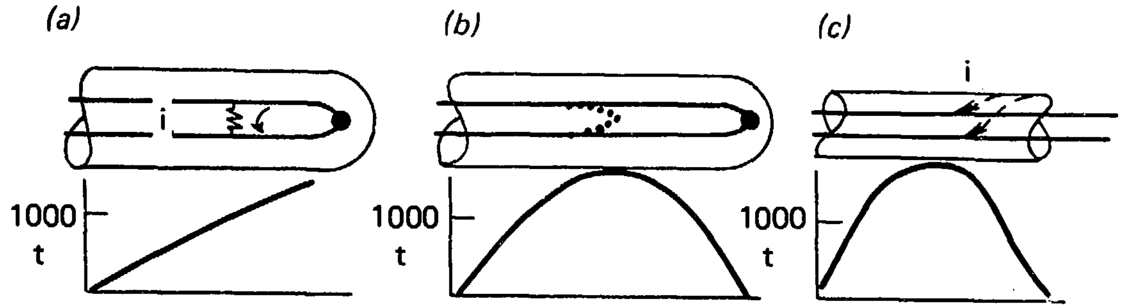

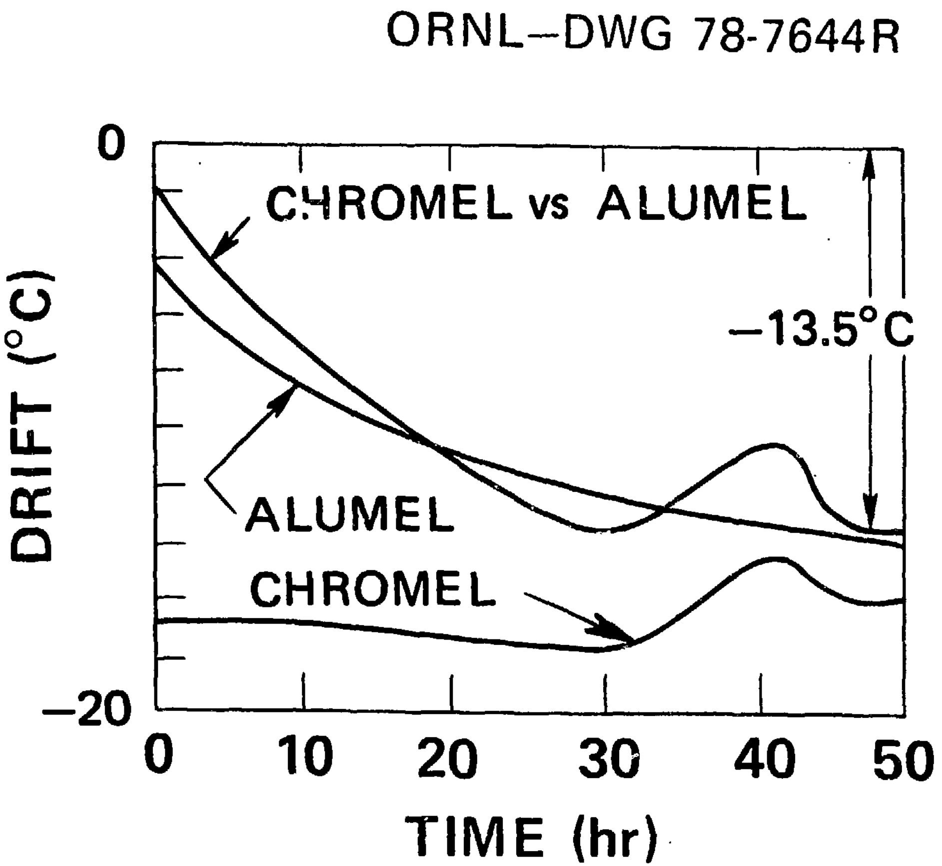
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0
–20
DRIFT (°C)
CHROMEL vs ALUMEL
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CHROMEL
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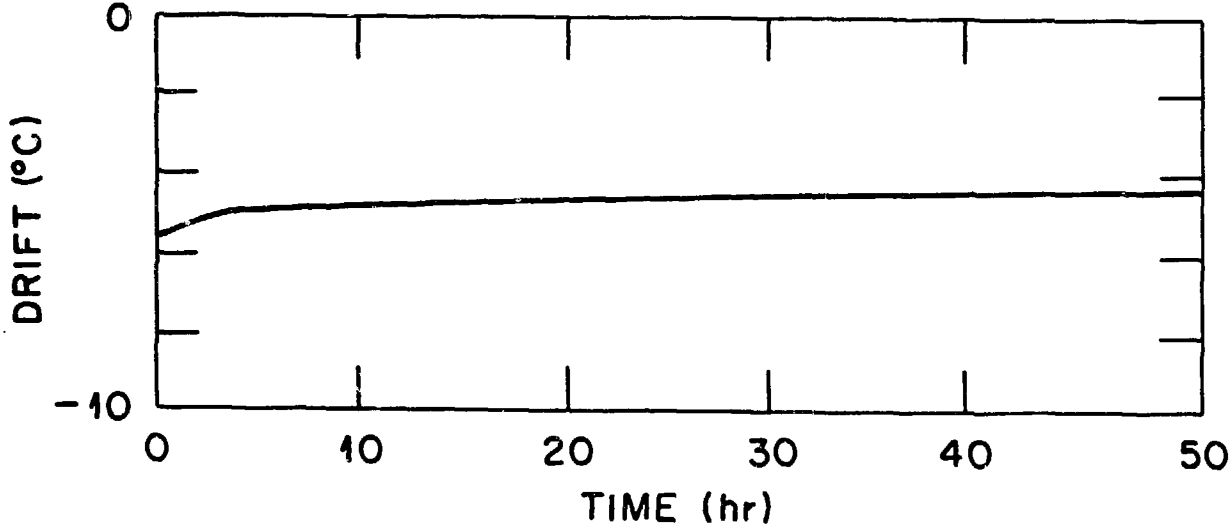
10
20
30
40
50
TIME (hr)

ORNL-DWG 77-14509A



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 at ORNL. The sources of error considered were extension lead wires, reference junctions, the data acquisition system, calibration and decalibration of the sensors, thermal shunting, electrical shunting, electrical leakage, and electrical noise.

It was found that because of the relatively small dimension (wire sizes of 0.1 mm diameter) the behavior of 0.5 mm diameter thermocouples could not be extrapolated from the known behavior of larger diameter thermocouples, particularly above 900°C.

Electrical shunting and leakage at higher temperatures can result in large temperature measurement errors. The magnitude of these errors depends on the temperature profile through which the thermocouple passes.
Size effects are also important in the decalibration of small diameter sheathed thermocouples. The sheath material becomes a factor in decalibration above 900°C. Nicrosil vs Nisil thermocouples--new alloys developed to replace type K alloys--decalibrated more extensively above 800°C than the type K materials in either type 304 stainless steel or Inconel 600.

Small diameter thermocouples with platinum based thermoelements are even more sensitive to the choice of sheath material than the base metal alloys. Platinum based thermocouples with platinum alloy sheaths are, however, superior to base metal thermocouples, particularly 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.

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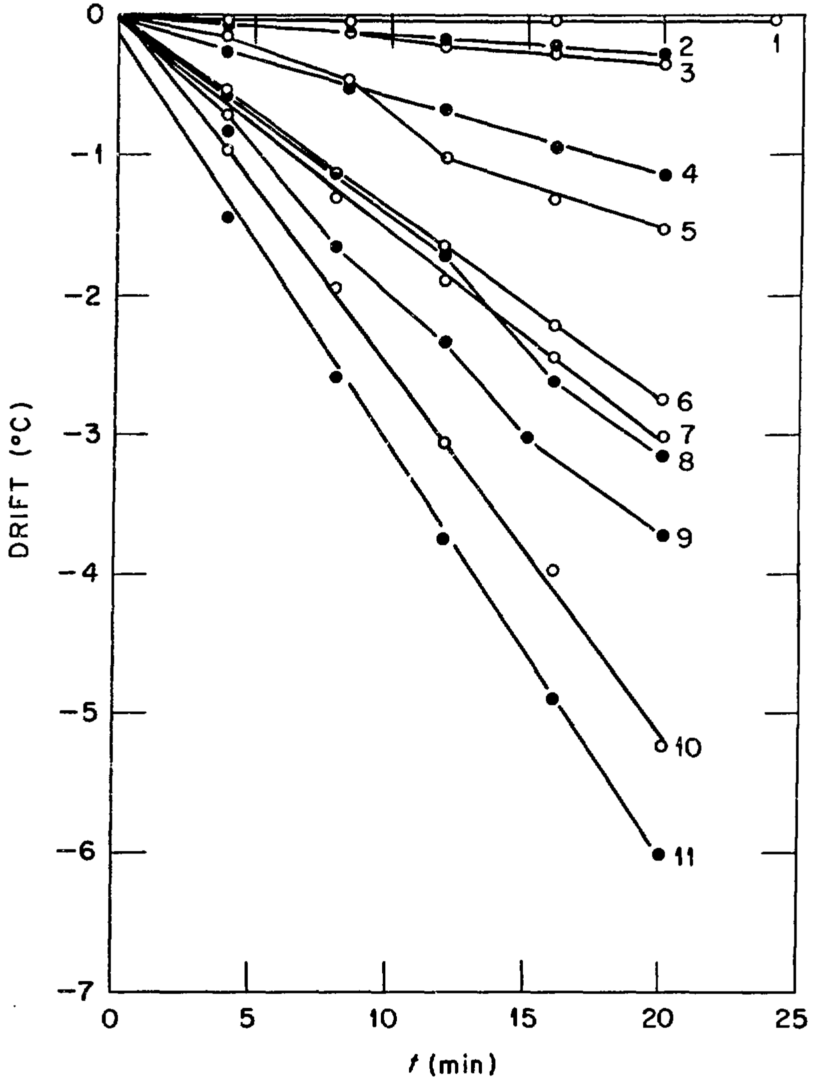

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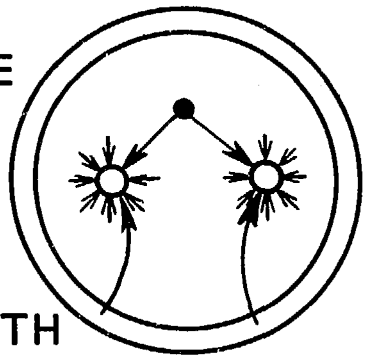
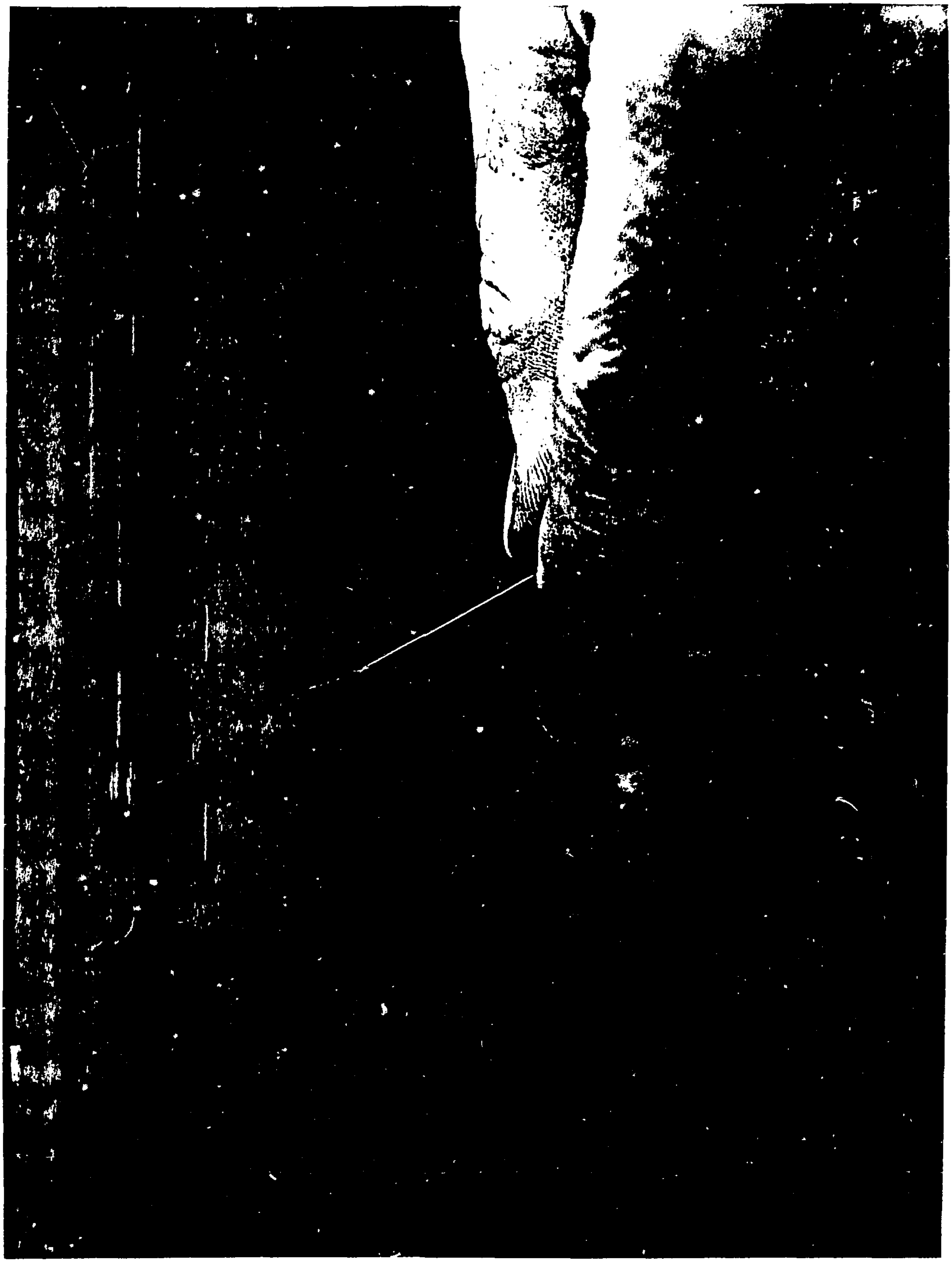
CHEMICAL REACTIONS CAN AFFECT BOTH ELEMENTS OF THE THERMOCOUPLE

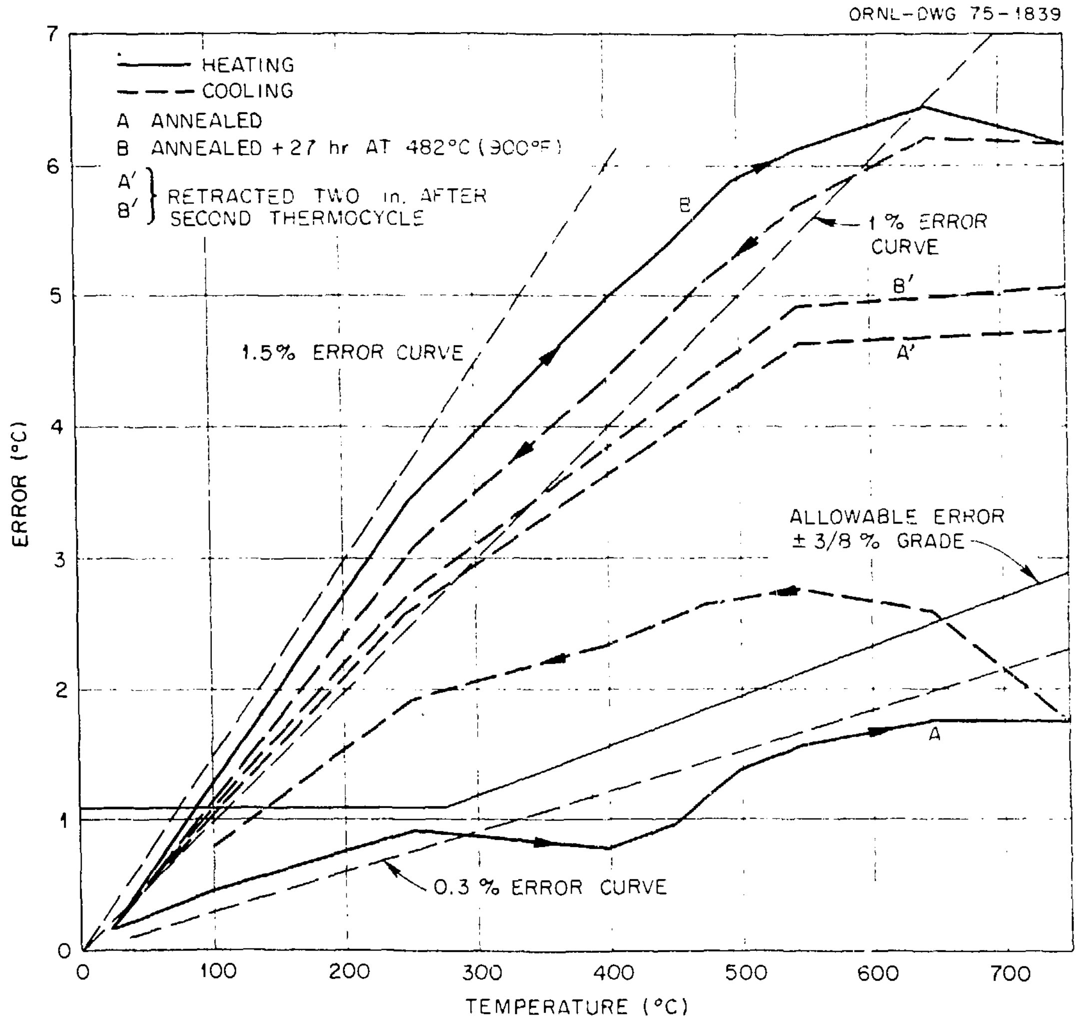


REACTIONS MAY TAKE PLACE BETWEEN THE THERMOCOUPLE WIRES AND:

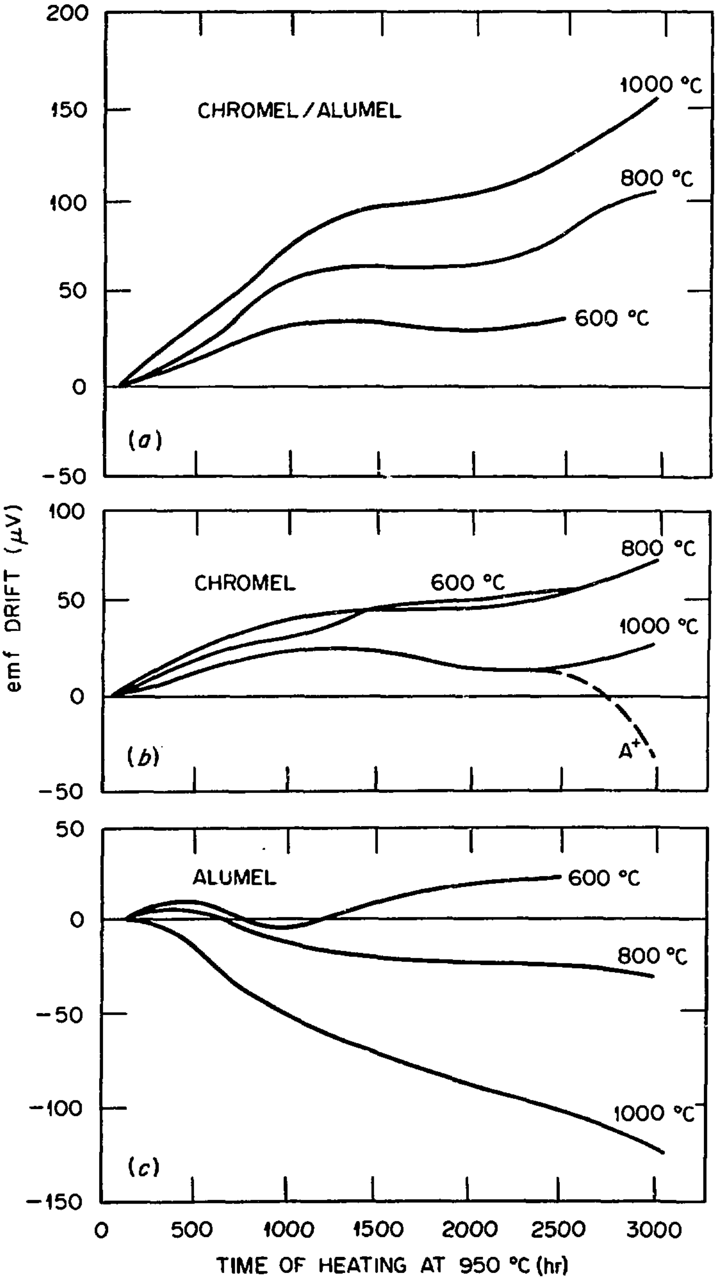
1. THE INSULATION
2. IMPURITIES IN THE INSULATION
3. THE ATMOSPHERE INSIDE THE SHEATH
4. THE SHEATH

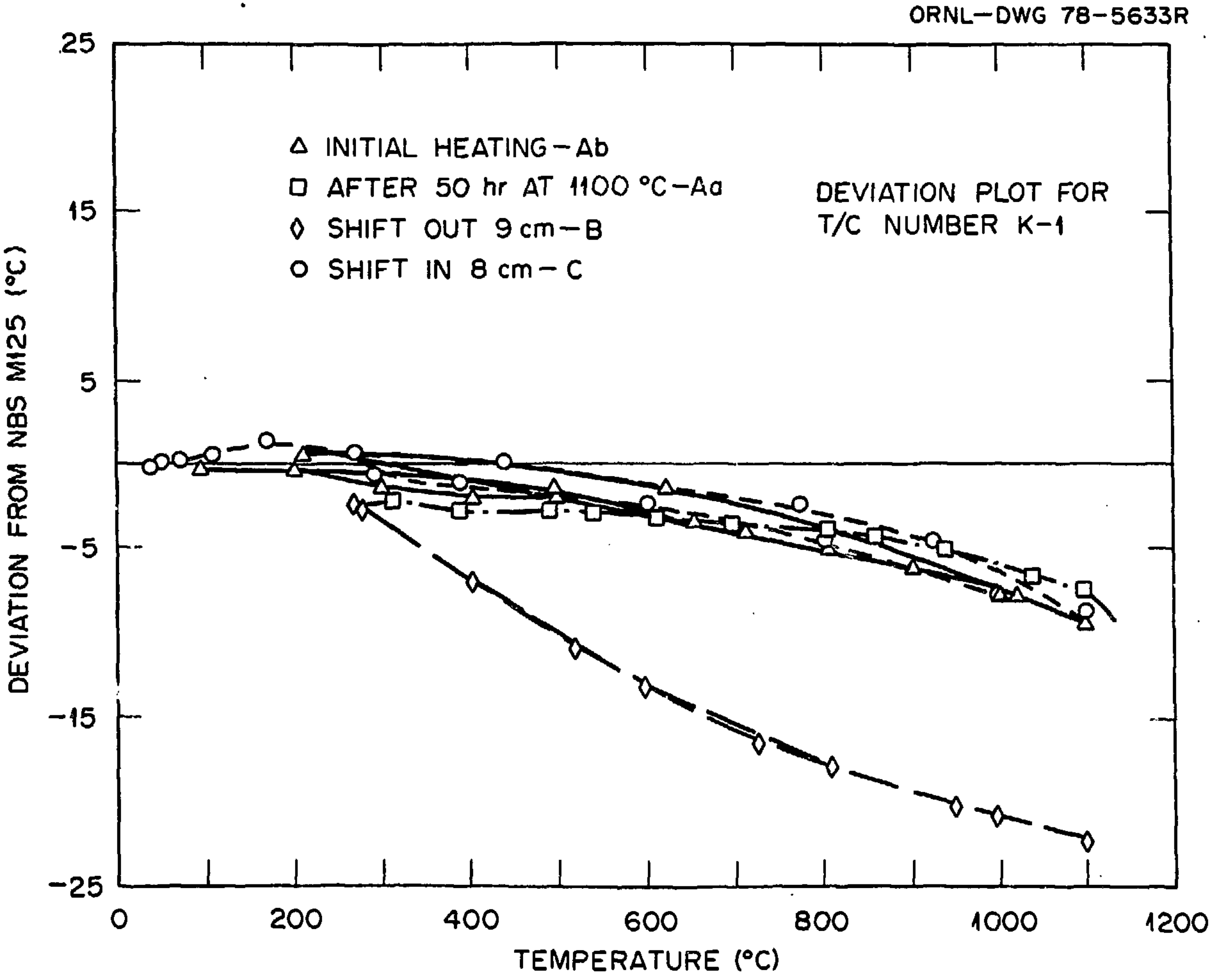
RATES OF CHEMICAL REACTIONS, AS A GENERAL RULE, DOUBLE FOR EACH 10°C RISE IN TEMPERATURE.




ORNL-DWG 75-1839
HEATING
COOLING
A ANNEALED
B ANNEALED + 27 hr AT 482°C (900°F)
A′ B′ RETRACTED TWO in. AFTER SECOND THERMOCYCLE
B
1 % ERROR CURVE
B′
A′
1.5% ERROR CURVE
ALLOWABLE ERROR ± 3/8 % GRADE
A
0.3 % ERROR CURVE
ERROR (°C)
TEMPERATURE (°C)
0
1
2
3
4
5
6
7
100
200
300
400
500
600
700

ORNL–DWG 78–3947R




ORNL–DWG 78–5633R
△ INITIAL HEATING – Ab
□ AFTER 50 hr AT 1100 °C – Aa
◊ SHIFT OUT 9 cm – B
○ SHIFT IN 8 cm – C
DEVIATION PLOT FOR
T/C NUMBER K-1
DEVIATION FROM NBS M125 (°C)
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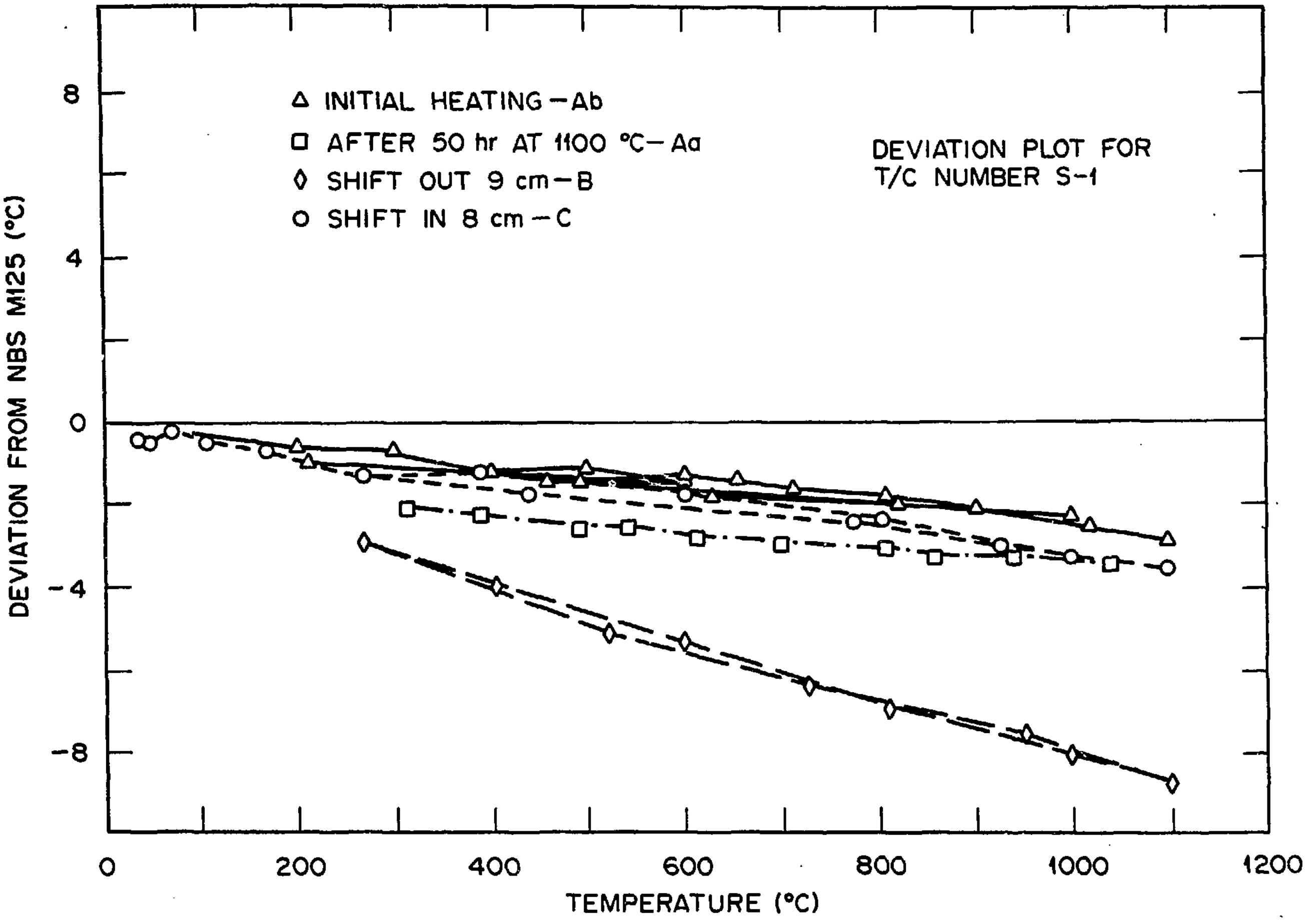
15
5
−5
−15
−25
0
200
400
600
800
1000
1200
TEMPERATURE (°C)

ORNL-DWG 78-5632



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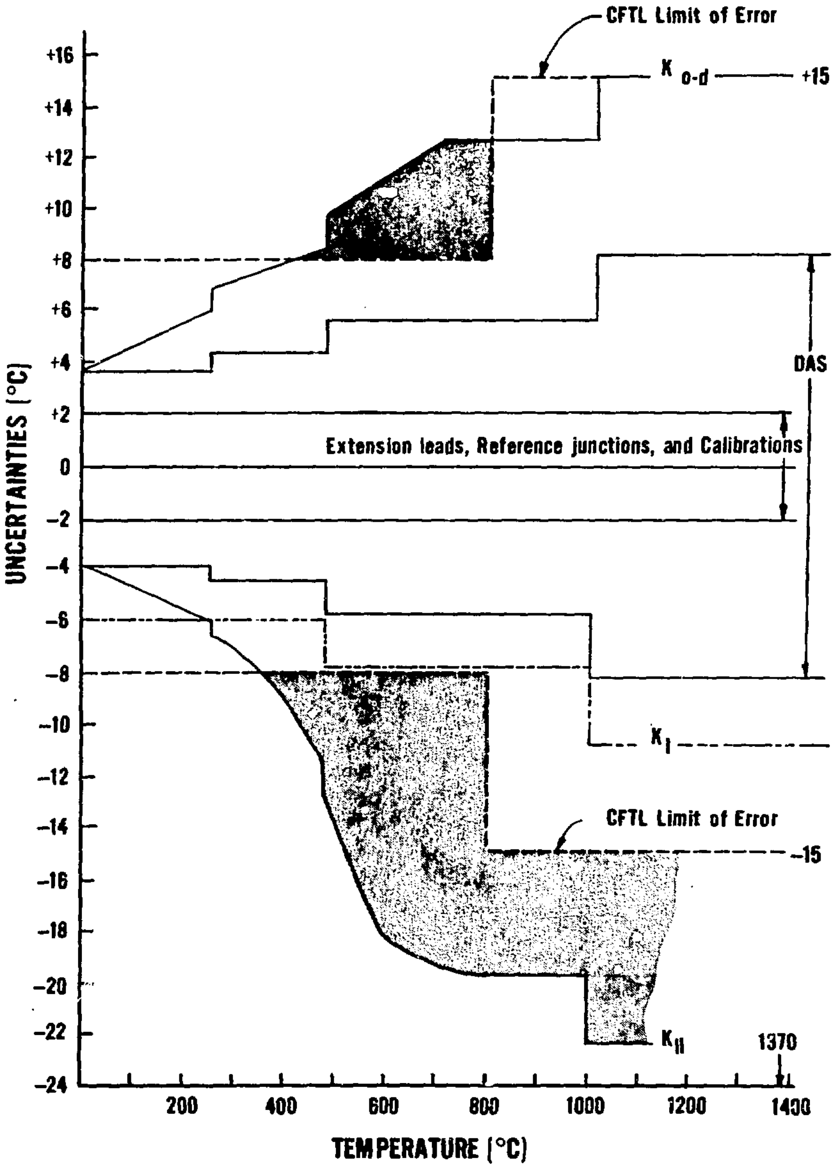

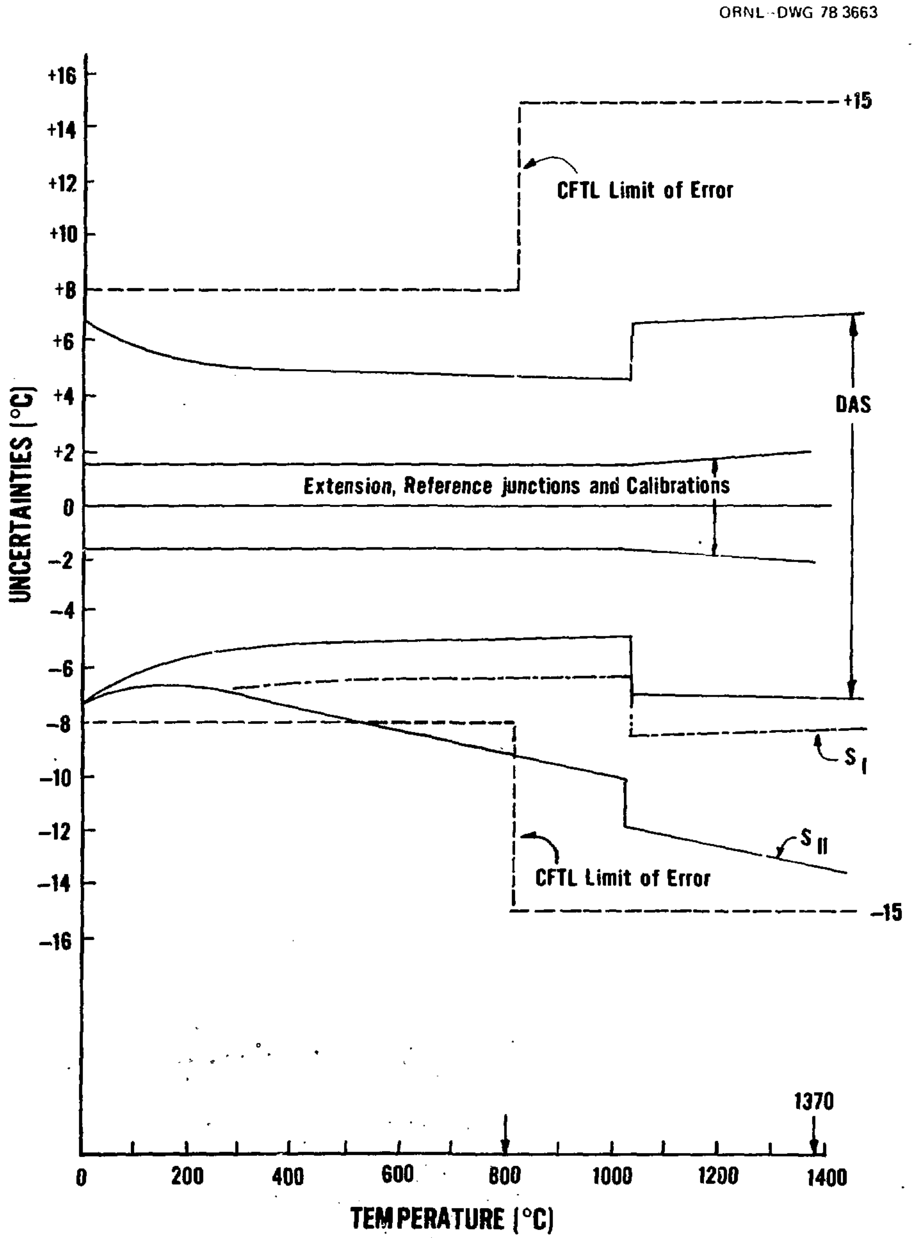

ORNL-DWG 78 3663
+16
+14
+12
+10
+8
+6
+4
+2
0
-2
-4
-6
-8
-10
-12
-14
-16
UNCERTAINTIES (°C)
+15
CFTL Limit of Error
DAS
Extension, Reference junctions and Calibrations
S_I
S_{II}
CFTL Limit of Error
-15
1370
0
200
400
600
800
1000
1200
1400
TEMPERATURE (°C)

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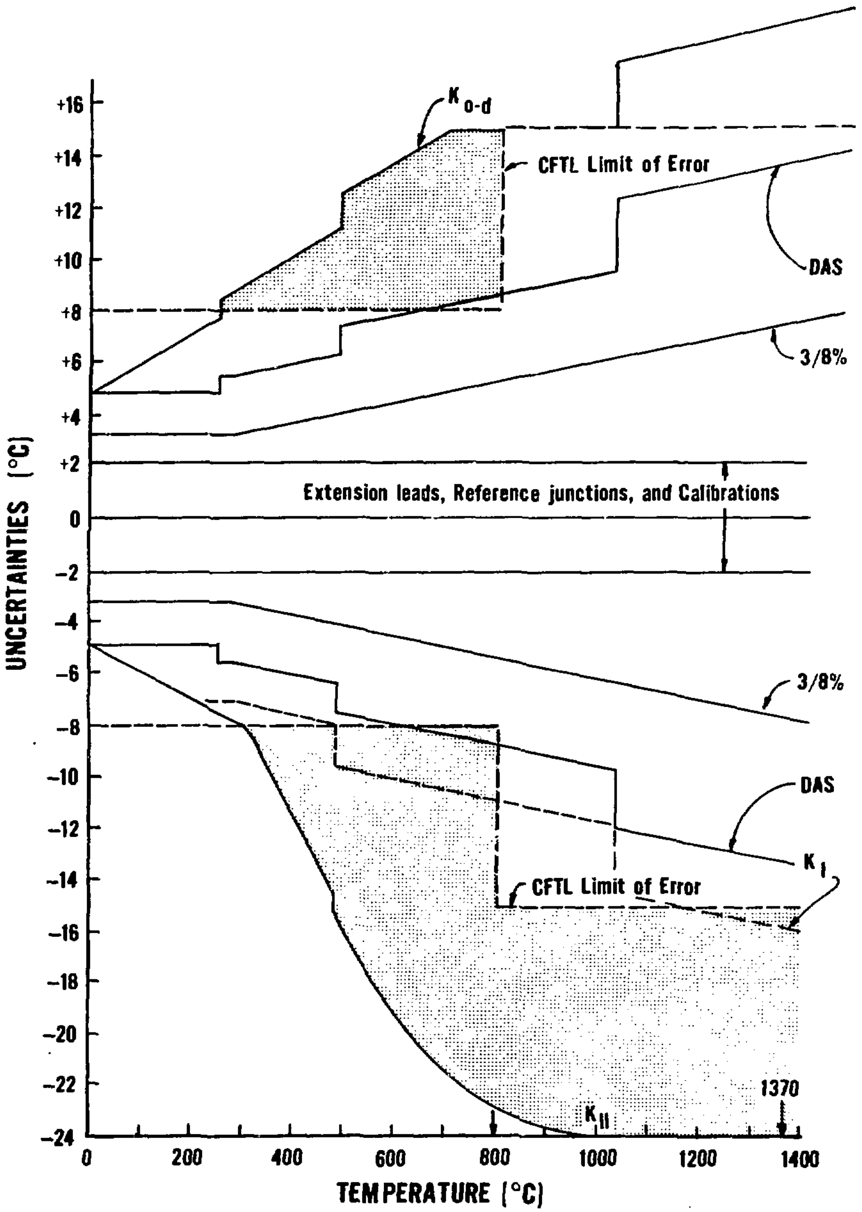

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Cumulative Uncertainties in CFTL FRS thermocouple temperature measurements using a Type S thermocouple in a platinum-10% rhodium sheath