The Practical Significance of Seebeck Inhomogeneity in Measurement Interoperability of Thermocouple Calibration

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Abstract

For thermocouples, measurement interoperability, "*assurance that calibrations are repeatable, comparable between laboratories, and traceable to an authoritative reference,*" can be subverted by Seebeck inhomogeneity that is often unrecognized in calibration. The well-developed and reported, but rarely applied, **S**eebeck **I**nhomogeneity **M**ethod **Of** **T**est (SIMOT) has provided data, understanding, and concrete answers to practical questions concerning the actual prevalence and severity of Seebeck inhomogeneity in present day thermoelectric thermometry. Complementary SIMOT measurements and traceable calibrations on applied thermocouples have demonstrated conclusively that traceability of thermocouple calibration, unaccompanied by authentic assurance of commensurate Seebeck inhomogeneity, can not definitely assure calibration uncertainty as implicitly is presumed. Users are skeptical that calibration is reliable. [1]

"... Ye blind guides, which strain at a gnat and swallow a camel !"
Matthew 23:24 KJV

1 Introduction

The admonition above was a harsh reproof of authorities who overlook a much greater kind of error in myopic preoccupation with a manifest error of lesser consequence. Of course, it was not directed toward present day arbiters of thermocouple measurement quality. Nevertheless, it might appropriately be applied to any who perform and certify "traceable calibration" to purported very small uncertainty yet merely presume, untested, that possible inhomogeneity of a thermocouple under calibration is commensurate with certified calibration uncertainty. Well-developed **S**eebeck **I**nhomogenity **M**ethods **O**f **T**est (SIMOTs) are an essential complement to authentic calibration. It is unfortunate that such tests now are rarely practiced. Too often, calibrators of thermocouples "strain" at longitudinal heat conduction yet "swallow" unrecognized, possibly more significant, inhomogeneity (non-uniformity of sensitivity along a thermocouple.)

1.1. Thermocouple Calibration is Consequential

Temperature does matter broadly in science and in industry, therefore thermometry matters. Because thermometry matters, accuracy matters. Because users trust calibration to assure accuracy, calibration must be authentic and comprehensive. Because the majority of high temperature measurements are made with thermocouples, often in harsh environments, thermocouple calibration is consequential.

The Program Office of NIST sponsored an independent study of the beneficial economic impact of NIST calibration. [1] That study noted that the cost of the most commonplace industrial thermocouples is relatively low. Therefore the indirect economic benefit of the relatively significant cost of calibration is not appreciated. There is huge worldwide annual economic cost and consequence of even small thermocouple thermometry error. Thermocouple errors of just one to three Celsius degrees were conservatively estimated annually to cost various industries hundreds of thousands or even millions of US dollars. (In thermocouple characterization for standardization, even errors of milli-Kelvin order can be significant. Note that expanded uncertainties for thermocouple calibration above 700°C in NVLAP accredited laboratories approach 3°C.) Apart from the estimated direct cost of such moderate thermometry inaccuracy, the loss of time, product, opportunity, and the risk of damage to facilities and harm to personnel can be of even greater practical consequence. These are compelling reasons for insisting on authentic quality of thermocouple calibration.

Definite assurance of thermocouple calibrations demands complementary testing of Seebeck inhomogeneity. Present thermocouple calibration rarely examines inhomogeneity to assure that it is consistent with the claimed calibration uncertainty. Inhomogeneity that passes unrecognized in calibration and in use often results in hidden error of similar or even much greater magnitude than claimed traceable calibration uncertainties or even standardized commercial tolerances.

1.2 Measurement Interoperability

This 2002 NCSL International conference is addressed by theme to justifying "*confidence that measurements made in one location in the world are equivalent to those made in other locations on the same or related products*." [2] The functional approach to accomplishing this laudable goal is: "*Measurement interoperability, assurance that measurements and calibrations are repeatable, comparable, between laboratories, and traceable to both national and international standards.*" Significant components of the measurement interoperability approach are reliance on the authentic traceability of formal calibrations and the standardized sophisticated mathematics of statistical uncertainty. [3]

This paper focuses on one significant, ubiquitous, and usually hidden source of error. That error is due to unrecognized significant Seebeck inhomogeneity. That obscure problem is unique to thermocouples. In extreme instances, though hidden, inhomogeneity can greatly exceed the sum of all the many other error sources that degrade temperature measurement and that are considered in traditional sophisticated uncertainty analysis. Despite significant error that often results from Seebeck inhomogeneity, that occurrence usually is not revealed by conventional thermocouple calibration.

1.3 Calibration Uncertainty vs. Measurement Uncertainty

Conventional calibration of homogeneous thermocouples can reduce thermoelectric uncertainty but that alone is not sufficient to assure accurate measurement. In thermometry, it is overall measurement uncertainty that matters. Some authors have appropriately distinguished *calibration* traceability from *measurement* traceability, recognizing that many of the most significant (often the predominant) measurement errors are not and cannot be addressed by calibration of the instruments of measurement alone. [4-7] There are many well-understood reasons why temperature measurements made with any physical thermocouple have much greater uncertainty than is represented by the thermocouple calibration uncertainties alone. Some measurement degrading factors relate to heat transfer characteristics of the sensor while others, less quantifiable, relate nebulously to the individual installation and use. Calibration traceability applies to the systems of measurement rather than directly to a measurement result obtained with them.

However, it is much less obvious that even carefully performed, competent, fully traceable thermocouple calibration can systematically overlook consequential thermoelectric uncertainty. The usually ignored uncertainty component from Seebeck inhomogeneity, unnoticed, can exceed or even dwarf the uncertainties routinely reported for conventional calibration. Accepting thermocouple calibration traceability as a *panacea* while ignoring, untested, the possibility of significant inhomogeneity is tantamount to "admiring the naked emperor's fine clothes".

1.4 Traceability

NIST recommends the formal definition of traceability that is asserted in the *ISO International Vocabulary of Basic and General Terms in Metrology, viz.*:

> ***Traceability** – "…The property of the result of a measurement or the value of a standard whereby it can be related to stated references, usually national or international standards, through an unbroken chain of comparisons, all having stated uncertainties."* [8,9]

That definition has been accepted internationally by BIPM, IEC, ISO, and OIML. [10] Of course, the definition intends that traceability of a calibration would definitely authenticate a quality-assuring measurement of thermocouple sensitivity. As currently practiced, conventional thermoelectric calibration, though technically traceable, does not accomplish that intent for thermocouples.

2 The Basic Thermoelectric Principle

The present paper briefly reviews technical facts of thermoelectricity that are known to most readers. To be definite, this paper concisely addresses the well known, but widely ignored significance of the simplest thermoelectric principle as it affects calibration: *the real physical source of Seebeck emf.* The authentic few principles of thermoelectricity as applied to thermometry, though now often misrepresented, have long been known. [10-24] Since 1900, it has been correctly understood (and taught by and to many) that the net Seebeck terminal voltage that is observed as a measure of thermocouple junction temperatures, occurs only from the source emf distributed along all non-isothermal segments of thermoelements. In calibration, it is segments of the thermoelements (the thermocouple "legs") not the junctions, that are the sources of the voltage, Fig.1. It is only localized non-isothermal portions of the thermocouple, not the thermocouple as a whole, nor of its junctions, that are being calibrated. [11-13,15-24]

Regardless of this definite elementary fact, contemporary calibration, diagnosis, and description of degradation usually is still performed as if the thermocouple is a "black-box" sensor with voltage sources localized at the junctions. This harmful, widely propagated, misconception is nurtured by the fact that, *if but only if* the thermoelements are of strictly uniform Seebeck sensitivity from end to end, the location of the emf sources is immaterial. The *critical* qualification, that this “rule of thumb” (ROT) applies *only* to perfectly homogeneous thermoelements, in effect, has been carelessly dismissed by many in the thermocouple user community (and by most who now write tutorial articles.)

Presently, most never-used thermocouples would allow measurement initially within standard tolerances despite their undetermined inhomogeneity. [19,24] However, no physical thermocouple is ideally homogeneous. Significant inhomogenity should always be recognized as a realistic possibility. Authentic individual calibration is appropriate for the more critical applications but requires assurance of tolerable inhomogeneity.

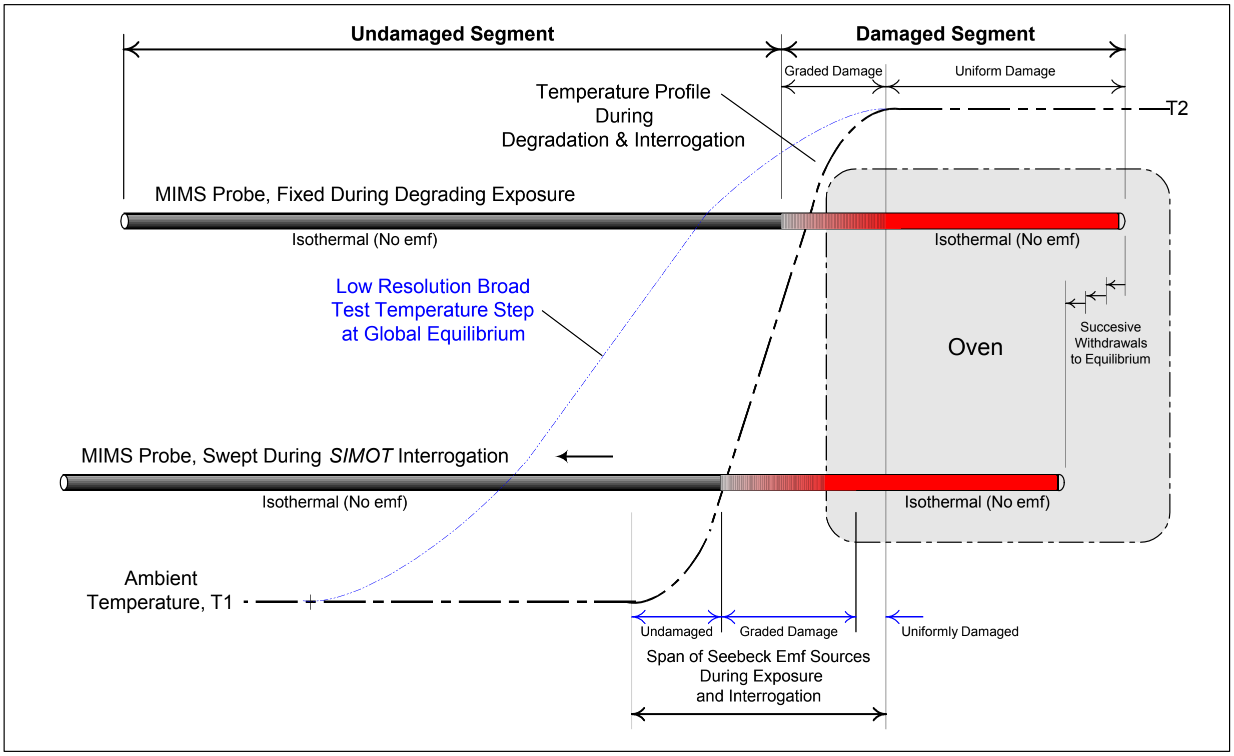

Fig. 1 Sources of Seebeck emf during damaging thermal exposure and in a SIMOT procedure

As illustrated in Fig. 1, the predominant voltage observed in calibration is from the specific, usually undocumented, region of thermoelements that happen to span the temperature step that is imposed for calibration. The terminal voltage observed is only from non-isothermal segments of thermoelements that might be 1) locally homogeneous and undamaged, or 2) from a segment that is non uniformly degraded, or else 3) partly from damaged and partly from undamaged material. Because the calibration-imposed temperature profile as well as the degradation profile are non-linear, the observed terminal voltage is a misleading unevenly-weighted sampling of a range of differing sensitivities. For that reason (contrary to current practice), it is important always to document for the customer the specific location and the width of the applied calibration temperature step.

3 Seebeck Inhomogeneity Defined

The *Seebeck coefficient,* **σ(T) ≡ dE(T)/dT** is the thermometric sensitivity that relates temperatures and the corresponding thermocouple terminal voltage on which thermometry with homogeneous thermocouples relies. *Seebeck inhomogeneity* is spatial non-uniformity of the temperature-dependent *Seebeck coefficient*, σ , along a thermoelement. [17-29]

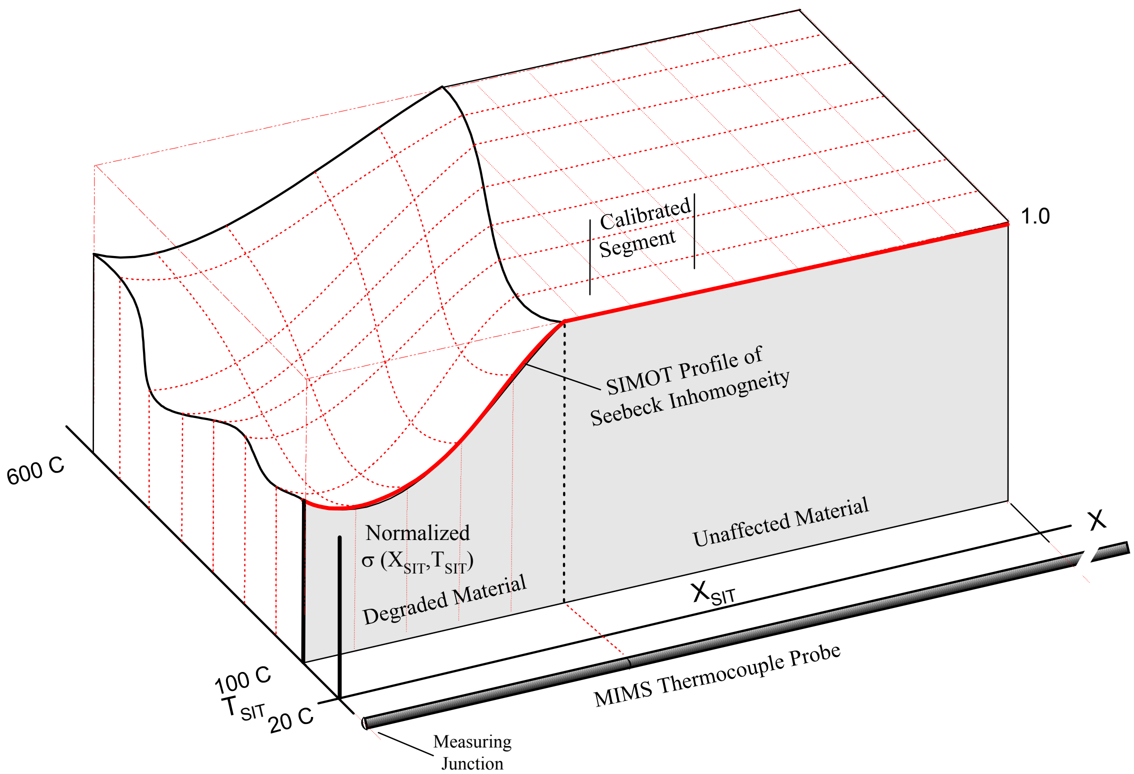

Fig. 2 The temperature dependent variation of Seebeck profile of a tip-degraded thermocouple

As seen in Fig. 2, for an *inhomogeneous* thermocouple the distributed Seebeck coefficient actually is **σ(T,X)** , a function of longitudinal position, **X**, along the thermocouple as well as of temperature, **T**. [17-29] Therefore, the terminal voltage observed in calibration of a thermocouple that is inhomogeneous depends on the particular region across which the calibrating temperature step was applied as well as on the shape of the temperature profile imposed. A generally applicable thermocouple calibration can not be performed to smaller uncertainty than is allowed by any such inhomogeneity anywhere along the thermocouple. The maximum error from such inhomogeneity is the difference between the extreme locally observed sensitivity and the normal calibration of the undamaged material.

A calibration result is well defined only if the location and shape of the imposed temperature step is defined. This information is rarely stated. A thermocouple calibration is definite if the thermocouple is everywhere inhomogeneous strictly within limits that are commensurate with the claimed calibration uncertainty. Too often, it is merely presumed that the actual inhomogeneity of the test subject is consistent with the level of calibration uncertainty that is required or is claimed. In precise traceable calibrations performed in qualified calibration laboratories (preceded and followed by authentic SIMOT test) it has been demonstrated that inhomogeneity does often pass the most careful calibration unrecognized and even results in severe insidious and costly thermometry error. [30]

4 Seebeck Inhomogeneity

4.1 Why Inhomogeneity Occurs

Seebeck sensitivity of a thermocouple can be locally degraded in manufacture, in calibration, or in use by thermal, nuclear, or chemical exposure, by mechanical deformation, by metallurgical diffusion or state change, or by a combination of them. Severe interaction can occur at high temperature in apparently benign exposure to adjacent components. [30] Thermometry typically exposes the measuring junction at the tip to the highest temperature and the harshest environment along the thermoelements. Therefore, damage most often occurs most severely at and near the tip and extends many centimeters away from the junction, Fig. 1. In less common instances, the degrading environment and damage occur over one or more regions well apart from the tip.

4.2 Inhomogeneity in Contemporary Thermoelectric Thermometry

Thermocouples now produced by reputable manufacturers, as delivered are expected to adhere to standard grade production tolerances or else to special grade tolerances for premium products even where those formal standards are not explicitly invoked. [19,24,31,32] Suppliers are convinced that most thermocouples produced in the US do now initially conform to these limits. The standardized tolerances applied to the relations between Seebeck emf and temperature are consensus values that manufacturers have accepted as appropriate limits for manufacture. Implicitly, the standardized tolerances are expected to encompass inhomogeneity. (Inhomogeneity is not now tested.) For routine measurement of ordinary consequence where standard uncertainty limits are acceptable, no initial calibration by the user should be required. Nevertheless, thermocouples for critical applications should be qualified by assurance of tolerable inhomogeneity and by appropriate calibration.

Eventual inhomogeneity in damaging use can not be avoided by the manufacturer. Error from inhomogeneity can not be avoided nor corrected by the most sophisticated traceable calibration nor by SIMOT data. Most incidences of significant Seebeck inhomogeneity and those of greatest severity in alloyed thermoelements occur during prolonged use at temperatures above 300°C and, in smaller amount, occasionally even during brief high-temperature calibration. Inhomogeneity can sometimes be minimized or avoided by the user by control of the environment. Thermally or mechanically induced inhomogeneity of elemental thermocouples can sometimes be removed by thermal treatment. [10,14,16,18,19,22,24] Elemental thermocouples are particularly susceptible to irreversible chemical or metallurgical contamination that is not removed by annealing.

4.3 Anecdotal Evidence of Inhomogeneity

A special study prepared for the Program Office of NIST addressed the economic value of thermocouple calibration. [1] It was based on anecdotal evidence from many personal interviews of selected expert specialists. They all were considered to be broadly representative of the community of manufacturers, suppliers, and users of thermocouple products and of thermocouple calibration services in the USA. The NIST-assigned focus of the study was on the economic impact of NIST calibration and also of the implementation of the ITS-90 temperature scale. Unexpected incidental findings implicitly suggested the costly and pervasive adverse effect of Seebeck inhomogeneity and the consequence in contemporary thermometry of ignoring its effect in calibration.

Despite the existence of many excellent thermocouple standards, in that broad and representative sampling of knowledgeable practitioners of thermoelectric technology industrial users, many expressed skepticism, frustration, and annoyance about the perceived inaccuracy of commercial industrial thermocouples and even about the reliability of costly calibration. [1] Several respondents reported having to calibrate each thermocouple and even having to perform redundant calibrations that, when repeated, conflicted with calibrations on the same thermocouple performed by different accredited laboratories.

Candid views expressed by knowledgeable users clearly questioned that *"measurement interoperability"* presently applies to thermocouples. Of course, from such anecdotal evidence presented in the report, it is impossible to know whether user's perceptions and expectations are unrealistic or whether current manufacture and the conventional calibration process actually are flawed. Regardless, customer's perceptions should be seriously considered by hardware suppliers and by purveyors of calibration services. Specific experience with SIMOT measurements strongly suggests that such commonplace user experience and sentiment can reasonably be attributed to unrecognized and misunderstood Seebeck inhomogeneity. An authentic SIMOT, complementary to calibration and applied to customer-questioned thermocouples, would resolve such issues with definitive data. Lacking such information and understanding, users now just move from supplier to supplier randomly seeking perceived quality.

4.4 Historic Observation of Significant Inhomogeneity

Apart from user subjective perceptions, there is much concrete experimental evidence of commonplace and substantial inhomogeneity that has systematically passed unrecognized in calibration. Both effective primitive and advanced hardware and software tools for the direct observation of thermoelectric inhomogeneity are well-developed and reported. [11-13,16,22,25-40] The long-available hardware and software concepts are essential to document the particular inhomogeneity of an individual thermocouple. They are required to demonstrate its variable effect under different temperature distributions. Assembled SIMOT units are not now commercially available to thermocouple users or to calibration laboratories. Each of the diverse designs has been implemented by the user according to special needs. A general design is possible but manufacturers and calibrators respond only to customer demand. Customers don't recognize the critical need so there is no demand. There is strong economic incentive, for suppliers of thermocouple hardware and calibration services, to ignore the obscure problem of inhomogeneity, lacking customer demand. Likewise, users (most with only occasional need for the SIMOT or even for a calibration facility) have little incentive, therefore little motivation, to tool up and to broadly document the actual occurrence of inhomogeneity in application. Central calibration laboratories are logical sites to efficiently implement SIMOT services. Lacking recognition of the practical inhomogeneity problem, there is little systematic hard documentation of the widespread problem of inhomogeneity in thermocouple use. Nevertheless, some data does exist.

Inhomogeneity was long ago recognized by a few as a significant practical problem in thermocouple thermometry. [11-13] It remains as an obscure problem today. [28] Before wire materials specialized to thermocouple thermometry were readily available, knowledgeable users selected thermoelements for accurate thermometry by effective, though primitive, inhomogeneity tests. Later, in support of critical thermocouple thermometry for nuclear reactors, formal quantitative testing was performed in England at the Risley Engineering and Materials Laboratory UKAEA and in the United States by the Oak Ridge National Laboratory (ORNL.) [16,33-35] The Australian NML/CSIRO, for many years, has routinely used a SIMOT procedure to establish an inhomogeneity uncertainty components for characterization and for calibration reports. [22,36,37,40] Historic inhomogeneity data on unused thermocouple calibration candidates has lead that laboratory to add a small 0.1% inhomogeneity component to calibration uncertainty statements. [22] Of these laboratories, only NML routinely supports calibration with a formal SIMOT. Primary standards laboratories that accept only unused thermocouple material for calibration often depend on thermal pre-treatment to avoid significant inhomogeneity. To those laboratories, working under such idealized conditions, inhomogeneity may not be a problem.

An advanced prototype SIMOT apparatus was implemented at NASA Langley Research Center. [25,26,38,39] That developmental unit was used to perform tests of several hundred thermocouples of different MIMS construction. There, several unexpected, costly, and puzzling thermoelectric inhomogeneity problems were diagnosed and resolved. [26,30,39] That versatile SIMOT demonstration unit was developed in cooperation with Sandia National Laboratories/Albuquerque and an ASTM E20 Thermometry Standards exploratory task group. Improved, the unit is now in use in the NVLAP-accredited DOE Temperature Primary Standards Laboratory at Sandia National Laboratories/NM.

No investigator who was properly equipped by equipment and understanding to quantitatively measure Seebeck inhomogeneity has failed to observe significant to substantial Seebeck inhomogeneity in as-delivered commercial material, in new post-calibrated thermocouples, and most frequently and severely in used thermocouples.

From the numerous complaints of users who were considered expert and from direct SIMOT measurement, there is no doubt that inhomogeneity is still commonplace and can be locally severe and of significant consequence even though it usually is not recognized. Observed inhomogeneity magnitudes have ranged from tolerable, to levels comparable to the tolerance values of special and ordinary grade thermocouple material, to the extraordinary local degradation of several select special grade Type R thermocouples in unusually severe instances. [30] Clearly, the mere presumption of probably adequate homogeneity is disproved, not justified, both by experimental and implicitly by user experience. Tolerable inhomogeneity levels can not safely be presumed, untested.

4.5 Authentic Tests for Inhomogeneity

As illustrated in Fig. 1, an *authentic* SIMOT applies a temperature profile of known shape, amplitude, width, and spacing that is swept along successive localized segments of a thermoelement or thermocouple. [25-29,33-40] It relates the Seebeck voltage observed at the terminals to corresponding longitudinal positions along the test subject. The result is a profile of thermoelectric sensitivity versus lengthwise location. The significance and spatial resolution of test results differ substantially as a function test temperature profile and sweeping motion. Apparent sensitivity can usefully be qualitatively relative or else a good approximation of actual Seebeck coefficient.

Authentic and useful tests of inhomogeneity can be performed by very different scanning profiles that impose one or more temperature steps. [11-13,25-29] Some are simple, *ad hoc*, and qualitative. Some are less simple and are precisely quantitative. Some SIMOTs relate to individual thermoelements. Others apply to the paired thermoelements of a thermocouple. The objectives and interpretation of results differ. In all SIMOTs, test variables are the motion and the width, temperature span, distribution, and spacing of the interrogating temperature step(s). For a particular thermocouple test subject, these conditions depend on the properties of the immersion medium, the test subject, and the motion imposed.

If the interrogating temperature step is very narrow, Fig. 1, and the magnitude of the temperature step is moderate, the observed profile of terminal voltage well represents the actual distribution of Seebeck coefficient, $\boldsymbol{\sigma(X_{SIT}, T_{SIT})}$, for the particular test temperature condition, $\mathbf{T_{SIT}}$, and tested span, $\mathbf{X_{SIT}}$. In advanced methods, fine spatial resolution (resolution to less than 1 cm) is available. Of course, the actual temperature distribution in thermometry is rarely known. However, from this quantitative measured profile of Seebeck coefficient, the voltage that would result from any arbitrary temperature distribution can be accurately calculated. This allows retrospective estimation of the credible measurement uncertainty for an inhomogeneous thermocouple that has generated data. At the other extreme, just for screening, simpler lower resolution methods impose known standardized temperature steps that are broad yet narrower than any likely to be encountered either in general thermometry or in a particular application. This directly yields in screening a simple measure of the likely practical effect on thermometry.

Narrow spatial resolution is important because the most commonplace inhomogeneity occurs in the region nearest the measuring junction and often for a short distance of 10 to 25 cm from that junction where it lies isothermally concealed during calibration. With appropriate controlled motion, the distorting effect of significant longitudinal conduction is reduced so the sensitivity can be well resolved from the junction onward. While precise computer-controlled motion is much preferred, manual immersion in simply heated baths has been used effectively for screening.

The higher resolution methods employ immersion into an isothermal liquid bath of favorable heat transfer properties. With liquid media it is easiest to use vertical immersion into the bath. Tap water, silicone fluids, liquid metals, molten salts, and other liquids have been used effectively for high resolution tests. The bath medium favored for highest resolution is a liquid metal eutectic alloy of gallium, indium, and tin (GITE) that conveniently has a freezing point below room temperature (~5-10°C). GITE readily wets MIMS thermocouple sheaths and has favorable thermal diffusivity for enhanced heat conduction to thermoelements. Normally used at a bath temperature of only 100°C, it can be used at elevated temperatures to above 1000°C.

Less desirable, but more readily implemented with commercial components, is insertion into a gaseous medium, such as into a lengthy air-environment isothermal tube furnace. The tube furnace can be oriented with its axis horizontal. Unavoidably, the temperature step imposed by conduction and radiant heating in gas is much broader in air than in liquids. More than from liquid immersion, ovens broadly smear and under-represent the magnitude of inhomogeneity features. Nevertheless, this commercially available form of manual-controlled SIMOT apparatus is adequate for efficient qualitative inhomogeneity screening complementary to calibration. Tube furnace heating in air is used routinely by CSIRO. [22,36,37,40]

Ordinary temperature measurements properly are made after a prolonged dwell for the thermocouple globally to reach thermal equilibrium. Therefore, it is counterintuitive, that an inappropriately prolonged dwell at a fixed immersion depth actually tends adversely to broaden the SIMOT-imposed temperature step profile and so substantially to reduce spatial resolution.

Consider that upon an abrupt short incremental advance of a thermoelement to a new immersion depth, thermal conduction occurs over the short quickly immersed segment both radially and longitudinally. For slender test subjects, such as Mineral-Insulated Metal-Sheathed (MIMS) probes, the temperature suddenly applied to the radial boundary quickly heats the thermoelements locally while temperature from longitudinally remote segments concurrently produces slower delayed superposed heating or cooling. Consequently, there is a preferred intermittent immersion motion that abruptly advances a segment into the heating region in short steps, each followed by only an optimum brief dwell.

This intermittent motion results in the fastest local thermal equilibrium, the narrowest step, and the best spatial resolution by reducing the broadening effect of undesired longitudinal conduction. This intermittent computer controlled motion is more effective than motion either at a continuous rate or intermittently with too long dwells. It is cost-effective if automatically used for frequent SIMOT application. It is accomplished with conventional, mostly commercial available, hardware components but not as a complete facility.

Motion at a fixed but selectable rate is less effective but is simpler to implement for casual use. It can also reduce operator time for frequent operation. Manual advance of a test subject at an approximate constant rate between fixed limits can be used for *ad hoc* evaluation and is least costly to set up. Traditional manual insertion with prolonged dwells to equilibrium at many fixed positions requires little special setup. However, it yields very poor resolution, is misleading, and is very costly of operator time and overall test time, if frequently performed.

4.6 The Effect of SIMOT Test Temperatures

It is appropriate to question the dependence of observed SIMOT inhomogeneity profile on the interrogating test temperature profile, $\mathbf{T_{SIT}}$. Even thermoelements designed especially for thermometry are significantly non-linear in $\boldsymbol{\sigma}\mathbf{(T)}$. The damaging occurrence of inhomogeneity generally results in Seebeck coefficients that are unpredictably exaggerated in non-linearity. Therefore, the inhomogeneity profile of a degraded test subject must depend on the test temperature profile, $\mathbf{T_{SIT}}$. A screening scan imposes steps of particular widths that bridge isothermal plateau. For convenience, one reference temperature, $\mathbf{T_1}$, is usually ambient laboratory temperature. An ideal complete SIMOT result would explore the $\boldsymbol{\sigma}(\mathbf{X_{SIT}}, \mathbf{T_{SIT}})$ surface, Fig. 2, over a broad range of temperatures. It is impractical and unnecessary to measure this entire surface function over the full temperature range of use. Instead, for practical screening application, the profile is sampled in response to only one or a few different temperature steps between $\mathbf{T_1}$ and an isothermal immersion temperature $\mathbf{T_2}$. Each SIMOT profile represents a cut from the full inhomogeneity surface.

A single SIMOT cut can not prove general homogeneity for all temperature distributions. However, a SIMOT can conclusively demonstrate any intolerable inhomogeneity. Like calibration, a SIMOT scan relates only to a particular tested span, Fig. 2. For most commonplace thermocouples, that SIMOT span, X_{SIT}, can usually include all of the thermometry-relevant segments. [25] As knowledge of inhomogeneity can not generally allow correction of data, the conservative SIMOT working presumption must be that a thermoelement that is excessively inhomogeneous under any one test condition must be presumed also to be excessively inhomogeneous at most others. [25-28] In one standardized SIMOT procedure the nominal temperature step is between ambient 20°C and a 100°C bath. [38,39] This temperature test range is benign and is adequate for SIMOT testing of all thermoelements and thermocouples. It provides excellent resolution using commonplace digital multimeters.

4.7 The Compelling Need for Quantitative Seebeck Inhomogeneity Tests

The availability of the telescope and of the microscope first allowed the visual resolution of vast unseen microscopic and cosmic-scale domains that previously could be explored only via inaccurate speculative imagination. Progressively advanced methods have allowed such augmented observation with ever-finer resolution of detail. Quantitative observational tools dispel myths and ignorance, but only if applied. Resolving actual detail enhances comprehension.

So it is with direct observation of Seebeck inhomogeneity by SIMOT. Those who discount the commonplace occurrence of inhomogeneity have not taken the opportunity to factually observe it. No investigator equipped with proper apparatus and understanding has failed to observe significant inhomogeneity in used thermocouples and sometimes in as-delivered material. Without a capable SIMOT tool of observation the particular distribution of degradation is obscure. Inhomogeneity then tends to be misperceived as a nebulous "black-box" phenomenon. Also, without some acquaintance with the simple but unfamiliar physical and mathematical convolution process that distorts profiles the specific consequence of a revealed distribution is poorly visualized and comprehended. [41] The appropriate SIMOT tools have been described and demonstrated since 1900, more than a century. The commonplace occurrence and practical consequence of thermoelectric inhomogeneity is factual.

4.8. The Proven Value of the Seebeck Inhomogeneity Test

The SIMOT has been presented primarily as an essential complement to traceable calibration. However, it also has several closely related applications that might not be apparent. [27,28] Typical applications, as suggested by Fig. 3, are: 1) in characterization of the Seebeck property of new materials, 2) in the study of the detailed process of instability in thermometry materials, 3) in supporting the surrogate calibration of material batches, 4) in the initial calibration of individual thermocouples for critical applications, and 5) in the screening of a used thermocouple to economically assess the suitability for re-use, re-work, or re-calibration to reduced uncertainty. It may be desirable to quickly scan the thermocouple both before and after calibration at high temperature or in hazardous environments. For critical calibrations, as in characterization of new materials by calibration at many high temperatures, it is desirable to confirm inhomogeneity cyclically between each possibly damaging high temperature exposure. Instability testing necessarily involves a progression of inhomogeneity. The progressing inhomogenity should be monitored periodically by SIMOT as degradation proceeds. Traditional thermoelectric instability studies that observe superficial drift of terminal voltage without SIMOT are very costly yet they have little general practical value for thermometry. [42] Using advanced SIMOT, well-resolved patterns of inhomogeneity structure might be useful in thermoelectric material studies.

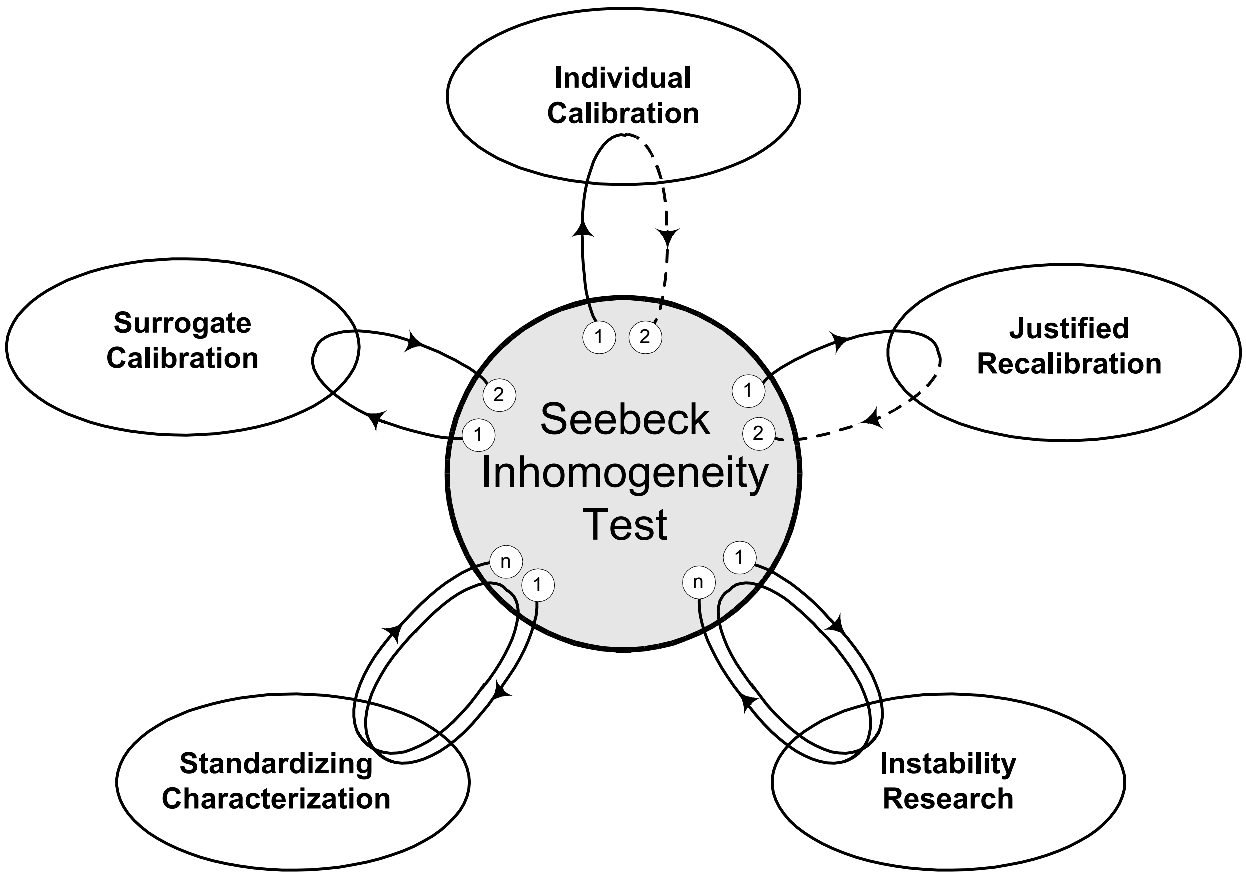

Fig. 3 Some alternative applications of the SIMOT in R&D, calibration, and thermometry

An authentic computer-controlled SIMOT procedure is quick and inexpensive to perform (less than 5 minutes per scan. A single scan can examine several test subjects.) It can have a definite quantitative inhomogeneity result. It can provide, incidentally, a temperature single-point comparison calibration. SIMOT procedures could be cost effective if thermocouples needlessly are being discarded, are producing faulty data, or could be reliably re-used or re-worked without costly re-calibration. Previously SIMOT-proved and calibrated thermocouples that continue to test as adequately homogeneous by SIMOT do not require re-calibration.

The authentic SIMOT test provides that essential objective thermoelectric observational tool to displace the myths, ignorance, and subjective bias that remain in the present practice of thermoelectric thermometry. Depending on the test objective, it is possible to implement the SIMOT at various levels of simplicity or sophistication that are practically adapted to tasks from simple screening to advanced high-resolution materials R&D. Traceable calibration and Seebeck inhomogeneity testing are complementary. Neither alone is sufficient to assure the authenticity of thermocouple thermometry.

Where applied independently in actual practice, the SIMOT has quantitatively shown the continued homogeneity of some suspect used competent thermocouples so that needless more costly re-calibration was avoided. It has made it possible to re-work expensive thermocouples by reliably removing just documented localized degraded regions identified by SIMOT. It has assured *a priori* that more costly calibration to a desired uncertainty was warranted by commensurate inhomogeneity.

These quantitative methods have allowed the authentic practically applicable study of progressive thermoelectric degradation (not merely the superficial "drift") and the accurate analysis of its predicted effect on thermometry calibration and measurement. Regrettably, such useful practical methods, though technically advanced and increasingly practical, have not been widely adopted by the modern thermometry community.

Authentic quantitative SIMOT tests of Seebeck inhomogeneity of several actually applied thermocouples have clearly proved that competent conventional calibration very often fails to, and indeed can not, reveal even huge local inhomogeneous degradation of sensitivity that degrade practical thermometry. Perversely, even the very act of high temperature calibration can locally degrade thermoelectric sensitivity by amounts that are comparable to the difference between standardized ordinary and special grade tolerances. [16,25,34-36,40] An authentic test of Seebeck inhomogeneity as a complement to calibration has been shown to be essential to *bona fide* quality assurance of thermoelectric thermometry.

4.9 Spurious Inhomogeneity Tests

A reason that inhomogeneity is often dismissed as insignificant is the commonplace use of misleading very insensitive spurious tests that actually under-represent or even conceal inhomogeneity thereby suggesting that a tested thermocouple is only insignificantly inhomogeneous or apparently is essentially homogeneous.

4.91 The "Flame Test". The worst of these "practical" tests is the popular so-called "flame test" that sweeps the narrowest possible sharply peaked heated zone along the thermocouple while monitoring the terminal voltage. Two very narrow back-to-back opposed temperature ramps differentially compare emf from immediately adjacent thermoelement segments that are *most similar* in Seebeck coefficient, even if very inhomogeneous.

This simple "flame test" is a truly useful test but it is not an inhomogeneity test. It is effective in locating hidden junctions (abrupt step discontinuities between dissimilar materials). [43] However, it actually is impractical as an inhomogeneity test as it is extremely insensitive so is grossly deceptive. It is harmfully counterproductive. Unfortunately, many who rely on this non-test have been convinced by it that inhomogeneity is usually negligible or is very uncommon.

4.92 The Longitudinal Conduction Test. A second, conditionally misleading, test is sometimes used by the more careful and well funded calibrators. Some precede calibration by advancing the thermocouple in small steps to progressive immersion depths over a range to assure that the measuring junction is actually at the isothermal calibration temperature, isolated from longitudinal heat conduction. (Actually, the appropriate minimum depth in the isothermal zone can be determined once for each thermocouple size and construction in a particular oven. Individually repeating the slow procedure should usually be unnecessary.) The test usually is performed manually, dwelling for a long period at each position to assure global thermal equilibrium.

This appropriate procedure to avoid longitudinal conduction, recorded at overall thermal equilibrium at several locations (with result compared between immersion and withdrawal), is also sometimes incorrectly viewed as a definitive test of inhomogeneity. The test purports to document inhomogeneity that might have been introduced before or during the calibration. This cautious procedure, particularly used in an air-environment tube furnace, imposes a very broad and undocumented test temperature profile, Fig. 1. The test step usually encompasses mostly pristine material and relatively little calibration-degraded material. Therefore, the result also can be deceptively insensitive. The fractional contribution from degraded material is usually smaller than the emf contribution from unaffected material. (Note, also that in calibration the observed effect of typical inhomogeneity is not separable from longitudinal heat transfer.) As usually conducted, the test for longitudinal conduction is inappropriate as a measure of inhomogeneity.

4.93 Surrogate Calibration Tests. Lengthy bulk thermoelement and MIMS thermocouple batches are routinely quality assured by the manufacturer. Typically, one short sample from each end is calibrated (often at a single temperature) and assured to conform within standardized tolerances. [31] Adequate agreement of both samples with the norm properly demonstrates conformity. However mere calibration agreement between the two samples, whether within or beyond standard tolerance, is incorrectly viewed by manufacturers as an adequate demonstration of tolerable inhomogeneity as well. This test is *not* an inhomogeneity test.

Insensitive pseudo tests of inhomogeneity often improperly lead to continued use of a thermocouple that is significantly degraded in uncertainty. A thermometry error in application could range from insignificant to excessive, depending indefinitely on the momentary temperature profile experienced in use. More broadly, naïve reliance on such tests misleads users to misunderstand inhomogeneity and its commonplace occurrence in commercial thermometry.

4.94 Avoidance By Presumption. Finally, a risky presumption, not a test, is often used in an attempt to avoid the consequence of unseen inhomogeneity. Calibration is often avoided as being thought too costly (i.e., more costly than the presumed consequence of invalid thermometry.) A so-called ”practical” approach is to routinely discard thermocouples after a prescribed number of uses or arbitrary duration of use. This logically seductive approach is based entirely on implicit presumptions of initial homogeneity and on rate of degradation. This naïve presumption supports unwarranted confidence. Realistically, for critical applications there is no reliable assurance without a SIMOT that the inhomogeneity of even a new thermocouple initially is tolerable. There is no reliable way other than through a simple SIMOT to judge after use how long an initially homogeneous thermocouple remained within inhomogeneity tolerance before replacement.

5 Calibration

The value of appropriate thermoelectric calibration is not questioned. Proper calibration, and its traceability through an unbroken measurement chain to a single authoritative reference touchstone, is an appropriate foundation on which contemporary measurement quality is based. [4-10] Nevertheless, it may not be apparent that a very precise, yet invalid, "calibration" of an *inhomogeneous* thermocouple is often just as "traceable", by international definition, as is a valid calibration of an adequately *homogeneous* thermocouple. [8,9] The most rigorous traceability of a conventional thermocouple calibration without associated evidence of commensurate inhomogeneity can not provide real assurance of the relevance of that calibration. A draft NVLAP Handbook that will include suggestions for thermometer calibrations now suggests only that a thermocouple "… calibration is traceable to NIST…" without mention of inhomogeneity. [44]

Too often, perversely, the very formalization of routine procedures, impressive certification to finely resolved uncertainty, and sophisticated statistics that are intended to guarantee accuracy can psychologically subvert actual measurement quality by leading to unwarranted confidence and misdirecting the attention from uncertainty issues that may be even more significant. [4,7]

5.1 Decalibration Misunderstood

Thermocouple degradation is often suspected by the user only because a temperature indication that was expected to be nearly constant was observed to "drift" progressively. Infrequently, it is rather a strange and unexpected pattern of temperature variation that seems implausible that rouses suspicion. [25-28,30] Suspect thermocouples are usually said only to be *"decalibrated".* To many, "decalibration" is a simplistic "black-box" concept that incorrectly perceives a single sensitivity, $\boldsymbol{\sigma(T)}$, as a uniform global property of the entire thermocouple. To the contrary, unlike other typical thermal sensors, an apparent progressive "drift" of thermocouple calibration in service, as in instability tests, necessarily is a very insensitive indication of significant and likely of substantial inhomogeneity and possibly hidden error. [27,28]

5.2 Re-calibration

The economic study of calibration for NIST asserted that " ... disagreement exists among thermocouple experts on the ability to effectively recalibrate a used thermocouple." [1] Should the author's assertion be accurate, that would simply reveal the ignorance of some experts about the true nature and consequence of Seebeck inhomogeneity. If *excessively* inhomogeneous, the damaged thermocouple is incapable of calibration for general use. Any apparent calibration is illusory.

Some measurement quality plans, coordinators, and auditors and a few cautious users (accustomed to re-calibrating other types of sensors) require unnecessary periodic re-calibration of thermocouples, even if they were not used. Once a thermocouple has been calibrated in a homogeneous state, re-calibration to the same uncertainty is not beneficial while it remains homogeneous as demonstrated by SIMOT. However, re-calibration to a reduced uncertainty consistent with its measured inhomogeneity level could be useful. SIMOT screening can also reveal what portions of a locally damaged thermocouple can usefully be reworked by removing degraded material without calibration. Based on authentic SIMOT results, it is definite beyond subjective bias when a particular thermocouple is incapable of being calibrated or used to a given minimum level of uncertainty.

Many calibration labs routinely refuse to recalibrate, or to re-certify for use, thermocouples that appear likely to be inhomogeneous. All operational personnel of thermometry calibration laboratories must understand specifically why significantly inhomogeneous thermocouples can not be usefully calibrated for re-application. Occasionally, used thermocouples are accepted for re-calibration. Used thermocouples that are visibly discolored or distorted by heat, chemical attack, or mechanical distortion, and so appear to be damaged, usually are not re-calibrated even if actually still usable. Therefore, thermocouples that are reported to be newly calibrated or re-calibrated in effect are presumed by the calibrating laboratory, and so by the customer, to be adequately homogeneous. Often, unrecognized, they are very inhomogeneous so any re-calibration certificate is invalid.

5.3 Why Do Traceable Calibrations Usually Fail to Recognize Inhomogeneity?

Peculiarly, thermocouples that are not recognized to be severely degraded often are certified by proficient calibration laboratories using conventional traceable calibration to be of tolerable uncertainty. [30] On re-calibration, the calibrated sensitivity may even remain unchanged from the as-delivered values or in its prior deviation from the norm. If so, the defective thermocouples routinely are certified for re-use. It is not the careless fault of the performing laboratory nor of failure of traceability. The calibration of the unidentified local non-isothermal *segment* that contributed emf conditionally is truly traceable (but only if explicitly identified). However, *segments* not involved in the calibration clearly are not calibrated. Therefore, the global *thermocouple* calibration is not actually traceable. The reason for such calibration oversight is the inherent inability of conventional calibration to recognize and to quantify inhomogeneity.

As presently practiced, such traceability of a conventional *thermocouple* calibration measurement does not accomplish the implicit goal with definite uncertainty. This strong assertion is factually attested by several costly experiences with calibration of premium grade thermocouples. For example, traceable pre- and post-use calibrations of several MIMS Type R thermocouples were very precisely and correctly performed by competent laboratories. All calibrations reported the sensitivities to lie mid-range in the standardized *special tolerance* band (±0.10%) after use. [30] However, definitive complementary SIMOTs performed before and after repeated certified calibrations revealed characteristic degradation profiles over extended regions adjacent to the measuring junction. Prompt systematic damage in an apparently benign laboratory experiment environment had resulted in obviously peculiar behavior in thermometry. The SIMOT profiles immediately showed the distributions, magnitude, and even the progress with exposure time, of damage to the samples as used. Some of the SIMOT results proved Seebeck coefficients for the 20-mil diameter platinum-sheathed isolated junction Type R probes to be reduced locally by as much as 60%! Under the most unfavorable possible temperature distribution, a 1000°C junction temperature would appear to be only about 400°C! That is an exceptional error. Quickly conducted SIMOT procedures immediately explained the puzzling behaviors of the several thermocouples that had delayed projects for weeks. The definitive SIMOT data allowed simple immediate solution of the problem of degradation. Consequentially, the local thermoelement *segments* that repeatedly were traceably calibrated were different than those exposed in measurement. [30]

Reasons that even such extraordinarily severe inhomogeneity usually is not discovered by conventional calibration are simple and definite. Proper calibration precautions to avoid longitudinal heat conduction tend routinely to conceal commonplace inhomogeneity. Thermocouple calibrations relate only to usually unidentified local segments of the calibration subject. The segments exposed in use are often different from those in calibration that tend to remain in as-received state. Also, popular *ad hoc* inhomogeneity pseudo tests mislead their practitioners by concealing or severely under-representing suspected inhomogeneity.

Conventional calibration of the familiar MIMS-style thermocouple probe is performed by immersing the measuring junction of the test subject to a fixed depth into an isothermal region (e.g., an oven, dry well, stirred bath, or fixed point reference cell.) The usual rule of thumb for MIMS thermocouples is to insert the measuring junction to a depth that is *at least* 10 to 20 sheath diameters inside the isothermal zone of the calibration oven. To assure that the immersion depth is at least adequate, the terminal voltage often is monitored at thermal equilibrium at several insertion depths to an immersion where the terminal voltage will remain constant at greater depths. However, to avoid the considerable delay and cost of making multiple manual readings, a single conservative, larger than necessary, immersion depth is usually employed. For probes with sheaths of very small diameter, the actual immersion depth typically is very much deeper than necessary to avoid longitudinal heat conduction.

It is unusual to determine or to report in the result of thermocouple calibration the depth of immersion, or the location, shape, and width of the temperature distribution along the probe. The actual temperature step profile along the thermoelements depends on the probe materials and diameter. The step width is broader for probes of the larger diameters. These unreported test parameters are essential to the interpretation and use of calibration results of a possibly inhomogeneous thermocouple.

Significantly, in junction fabrication and in use it is that extended region adjacent to the junction where the thermoelements are most likely to be degraded. It is that most likely damaged region that is carefully held isothermal in calibration. Paradoxically, particularly in abrupt transient or slower dynamic thermometry, it is the possibly degraded region near (but not necessarily including) the junction that is non-isothermal and that produces the predominant fraction of emf during measurement, Figs. 1 and 2. Irrelevant calibration, deceptively, may apply to segments that were not used in measurement. Consequently, even thermocouples that happen in use to be severely degraded near the junction or elsewhere, in careful traceable calibration very often appear to be pristine within normal narrow tolerance range or as-delivered.

5.4 Conscientious Traceable Thermocouple Calibration

A thermocouple calibration that is not accompanied by an authentic complementary assurance of inhomogeneity is *necessarily indefinite* even if it is certified as “traceable.” Such calibrations without assurance of acceptable inhomogeneity, are not necessarily *“unique, repeatable, or* *comparable between laboratories,"* even if conventionally traceable to an authoritative reference. [2,8,9]

Actual SIMOT measure of inhomogeneity is certainly preferred. However, few calibration laboratories or users are now equipped or inclined to perform an authentic SIMOT. A forthright alternative to definite experimental assurance is candor about the implicit assumption of homogeneity and an explicit statement of the particular calibration conditions. The longitudinal temperature distribution along the thermocouple at the entry to the calibration furnace should be determined. A generic temperature distribution could be determined from thermocouples that are thermally like the calibration subject under like conditions. On this basis, a frank disclosure of test condition could use wording equivalent to the following:

> *"This thermocouple calibration, referenced to 0°C , was performed by imposing a temperature step between laboratory temperature, 21°C , and the 1000°C calibration temperature beginning 35 cm from the measuring junction and extending for an additional distance of 20 cm.. This certified calibration and its uncertainty traceably apply to this particular segment only. Possibly unrecognized inhomogeneity of other segments of the thermocouple, either during calibration or in use could result in greater uncertainty than reported when exposed under other temperature distributions in calibration or in application."*

Such non-traditional overt recognition of the inherent limitation on traceability of thermocouple calibration would at least alert the customer to the real possibility of unrecognized uncertainty. Typical customers presume (reasonably) that the traditional calibration applies globally to the entire thermocouple. For them, such a notice would provides a proper caution. In extreme instances, such a cautionary disclaimer might even protect the calibration laboratory from product liability should a merely-conditional traceable calibration be relied on by a customer in a critical application that resulted in loss.

6 SUMMARY

Regardless of inhomogeneity problems that remain obscure without testing, properly used, thermocouples remain the temperature sensor of choice for multitudes of applications. Seebeck inhomogeneity of thermocouples (longitudinal non-uniformity of Seebeck coefficient) has long been suspected, sometimes recognized. By authentic Seebeck Inhomogeneity Methods of Test (SIMOTs) troublesome inhomogeneity has been conclusively shown to be commonplace in applied thermoelectric thermometry. Inhomogeneity, that usually is unrecognized in calibration, can cause thermometry errors that dwarf those suggested by conventional traceable calibration uncertainty. Even when inhomogeneity is severe and causes significant error, it is noticeable under only special thermometry conditions. It is rarely recognized and is never quantified by customary traceable calibration. Therefore, its commonplace occurrence in thermometry is acknowledged only in principle. It is largely ignored in calibration practice. Uncertainty of traceable calibration without assurance of commensurate homogeneity is necessarily indefinite. Well developed and reported authentic SIMOTs have been developed and reported but are used in very few laboratories. Traceable thermocouple calibration and SIMOTs are complementary. Neither alone is a sufficient basis for certifying sensor uncertainty. Less satisfactorily, at least an explicit disclaimer should accompany each thermocouple calibration report. The disclaimer should describe the particular temperature profile imposed in calibration and acknowledge the inherent inhomogeneity limitations of the thermoelectric calibration that was conducted without complementary SIMOT. In calibration, it is appropriate to *strain* at longitudinal heat conduction but traceable thermocouple calibrations should not *swallow* the hidden uncertainty contribution of inhomogeneity.

Acknowledgements

Radford M. Carrol and Robert L. Shepard (decd.), as members of a task group of the ASTM E20 task group on the Testing for Thermoelectric Inhomogeneity. shared their extensive hands-on experience with inhomogeneity testing. Both applied SIMOT methods before retirement from Oak Ridge National Laboratories, Willard A. Bauserman, jr. (ret.), also a member of that task group, gained NASA support and, with Cecil G. Burkett, jr., implemented the prototype SIMOT test hardware and initial control software at the Langley Research Center. There they generated a mass of instructive SIMOT data that solved practical thermometry problems at NASA and allowed insight into the practical occurrence and specific nature of Seebeck inhomogeneity in modern skilled thermocouple practice. Dr. Thomas F. Wunsch, Sandia National Laboratories Primary Standards Laboratory, Albuquerque, NM, has gained support for continued development and useful application of that SIMOT apparatus in calibration. With the assistance of Mario Delgado, he has made advances in software and hardware that further refine the operation, convenience, and utility of the advanced SIMOT tool. The author appreciates the opportunity to work closely with these productive individuals.

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