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A Concerted International Project to Establish High-Temperature Fixed Points for Primary Thermometry

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Abstract Research into high-temperature fixed points above 1,100°C has made significant progress since they were first reported in 1999. In particular, it has been established that single cells are repeatable at the sub-50 mK level, and intra-cell reproducibility at the 100 mK level has been demonstrated even at temperatures as high as 2,500°C. The fixed points have been used to compare temperature and radiometry scales over a wide temperature range, and are being developed and established as secondary references for thermocouple calibrations. However, before they can be fully accepted as primary temperature references, much work remains to be done, namely: (1) Establishment of long-term stability of the fixed-point temperature; (2) Development of robust procedures for the reliable construction of the fixed-point cells (to ensure routine intra-cell reproducibility of 100 mK); (3) Demonstration of long-term robustness of the fixed-point cells; (4) Assignment of thermodynamic temperatures to a selected set of fixed points; (5) Agreement and acceptance of these temperature values by the CCT; (6) Agreed methods on how to take full utility of these new fixed points into any future International Temperature Scale (ITS)—or the current ITS-90

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via an addendum to the *mise en pratique* for the definition of the kelvin. To ensure that this work progresses to completion in a reasonable time frame, a research project, under the auspices of the CCT-WG5, has been formulated and is currently in progress with the aim of achieving the above mentioned targets by 2012. This article will describe this project and detail partner contributions.

Keywords Fixed points · High temperatures · International research · Metal-carbon eutectics

1 Introduction

Progress in research into high-temperature fixed points (HTFP), also known as metal-carbon, or metal (carbide)-carbon eutectics (M(C)-C eutectics) and metal carbide-carbon peritectics (MC-C), since their inception [1–3] has been rapid and sustained. A summary of progress to date can be found in [4,5]. The progress would not have been as rapid or significant if the research had not been undertaken on a collaborative basis. Examples of formal collaborative research are the EU project HIMERT [6] and Euromet 857 [7], both undertaken in close formal co-operation with NMIJ.

Currently, these fixed points have been shown to be repeatable to much less than 100 mK; the best fixed points are now reproducible to ∼100 to 200 mK [5], even at the highest temperatures. They have been used for non-contact thermometry scale comparisons (e.g., [8–10]) and are currently been developed as new standards for thermocouple calibrations [7,11,12].

The aim of the project described here is, in the next five years, to build on and bring to full fruition the research that has taken place previously. The overall goal of the project is to establish high-temperature fixed points as routine metrological tools for the high-temperature measurement community. This is a multi-partner project involving nine National Measurement Institutes (NMIs): NPL (UK), PTB (Germany), LNE-INM/Cnam (France), NMIJ (Japan), NIST[1] (USA), VNIIOFI[1] (Russia), KRISS[1] (Republic of Korea), NIM[1] (China), and NMIA[1] (Australia) as well as a number of associate NMIs and research institutes. This is also a cross-disciplinary project with expertise in thermometry, radiometry, thermal modeling, and materials science required to make it a success.

This project grew out of the deliberations of the CCT-WG5[2] [13] and is an agreed joint research project within the "implementing Metrology in the European Research Area" (iMERA) framework [14].

The work program, described below in detail, has been divided into six technical work packages with the assigned work package leaders being authors of this article. In brief, the following will be performed.

[1] NIST: National Institute of Standards and Technology. VNIIOFI: All-Russian Research Institute for Optical and Physical Measurements. KRISS: Korea Research Institute for Standards and Science. NIM: National Institute of Metrology. NMIA: National Measurement Institute of Australia.

[2] Consultative Committee for Thermometry Working Group 5 – Radiation Thermometry. The CCT is a consultative committee of the CIPM (Comité International des Poids et Mesures)

(a) Determination of the long-term stability and robustness of the high-temperature fixed-point cells.
(b) Construction of high-performance high-temperature fixed-point cells for thermodynamic temperature assignment.
(c) The optimum fixed-point operational conditions will be identified and specified.
(d) An assessment of absolute radiometry capabilities among the project partners will be performed, and definitive thermodynamic temperature measurements subsequently assigned to the high-performance cells.
(e) Recommendations to the CCT[2] will be made on fixed-point temperatures and how these developments can contribute to step change improvements in high-temperature thermometry [15].

2 Elaboration of Technical Work to be Performed in this Project

As described above, the project is divided into six technical work packages. A seventh concerns project coordination and will not be elaborated here.

2.1 WP1: Establish Long-Term Stability and Robustness of High-Temperature Fixed-Point Cells

This work-package is being led by LNE-INM/Cnam (hereafter INM), with partners PTB, NPL, NMIJ, NIM, and NIST and is planned to run from 2007 to June 2009. The purpose of this work-package is to definitively establish the long-term stability of the high-temperature fixed points (HTFPs), which is an essential pre-requisite to them being accepted by the thermometry and radiometry communities. Also, the cells' intrinsic robustness will be tested during the stability tests, allowing for design refinements to improve the cell's longevity.

The long-term stability and robustness will be tested for a representative sample of HTFP cells, namely, those of Co-C[3] (four to be supplied by INM), Pt-C[3] (two each to be supplied by NPL and NMIJ), and Re-C[3] (four to be supplied by NMIJ). These will all be of a similar design with a maximum outer diameter of 24 mm and an overall length of 40 mm. These will then be sent to the laboratories performing the stability tests, namely: PTB (Co-C), NIM (Pt-C), and NIST (Re-C). Precautions must be taken to prevent cell contamination. Temperature differences between the four cells, to be designated A, B, C, and D, will then be determined. One cell (A) will then undergo a 50-h heat treatment, which means a 50-h continuous run of melt-freeze cycles. Its temperature difference to one of the batch of reference cells (D) will then be re-determined. At this point, cells A, B, and C will be sent to the coordinator of WP4 to be used in that work package. Cell D will not be circulated and will serve as the reference cell against which the drift of the circulated cells is measured. After the measurements for WP4 are completed (this is envisaged to be around December 08), cells A, B, and C are to be returned to the laboratories involved in WP1 for final stability

[3] Approximate transition temperatures are: Co-C ∼ 1,324°C, Pt-C ∼ 1,738°C, Re-C ∼ 2,474°C

measurements against reference cell D. It is envisaged that the report on stability tests will be sent to CCT-WG5 by June 09.

2.2 WP2: Construction of HTFP Cells for Definitive Thermodynamic Temperature Measurements

This work package is being led by NMIJ with partners PTB, NPL, INM, NIM, and VNIIOFI, and will run from early 2008 to March 2009. The purpose of this work package is to ensure that a set of three "primary" HTFP cells will be produced for measurement by absolute thermometry in WP5. Each WP2 participant will produce a selection of Co-C, Pt-C, and Re-C; additionally a set of Cu cells will also be produced to tie the definitive measurements in WP5 to the current ITS-90. The coordinator will specify the general design, minimum material assay requirements, and agree with participants as to which cells they will construct to ensure that a good mix of cells is produced. A set of objective selection criteria will be developed, by the coordinator, in conjunction with all project participants, to select which cells will go forward into WP5 for thermodynamic temperature assignment. It is anticipated that the selection criteria will be agreed by October 08 and that the cells will be selected for WP5 by the end of March 09.

2.3 WP3: Specifying Fixed-Point Operational Characteristics

This work package is being led by NMIJ, with partners PTB, INM, NPL, and VNIIOFI, and will run from early 2007 to October 2008. The aim of this work package is to provide guidance on how to obtain the best performance from HTFPs. Issues to be considered will include furnace-gradient effects, micro-structural effects, the role of impurities, calculation of temperature drop across the crucible wall, and emissivity. Some of these aspects have been considered previously (e.g., [16–19]) but need further elaboration. Guidance will be given on how to calculate an a priori uncertainty budget for HTFPs.

2.4 WP4: Assessment and Improvement of Absolute Thermometry Capability

This work package is being led by PTB, with partners NPL, INM, VNIIOFI, NIST, KRISS, and NMIA and will run from early 2008 to 2009. The purpose of this work package is to assess and provide guidance on how to improve the absolute thermometry capabilities of the participating laboratories.

The cells A, B, and C for the three fixed-point materials Co-C, Pt-C, and Re-C (see Sect. 2.1 above) will be sent to the coordinator by the laboratories performing the stability tests. Cells A will be kept as spares at PTB in case of breakage during the measurement cycle. Two measurement loops will be established. These are; loop 1: PTB, NIST, NMIA, KRISS, PTB and loop 2: PTB, INM, NPL, VNIIOFI, PTB. Each participant will be provided with information concerning the cell. Crucially, this will include a reference curve (without temperatures) for a fixed point in a good (i.e., uniform) furnace. Participants should aim, through optimizing their furnace conditions, to reproduce as nearly as possible that plateau shape. After the furnace has been optimized, each participant will determine the absolute temperature of the supplied cell, including a rigorous uncertainty analysis. The cell will remain with each participant for 2 months. It is envisaged that the participant will provide a measurement report (including full experimental details, the measurement results, and the estimated uncertainties) to the coordinator within 2 months of completing their measurements. After completion of the measurements and when the coordinator has completed his analysis, guidance on possible improvements to absolute-radiometry capabilities will be issued—at least 6 months before the commencement of WP5, the definitive assignment of HTFP temperatures.

The coordinator will then return cells A, B, and C to the laboratories involved in WP1 for the final stability evaluation.

2.5 WP5: Assignment of Definitive Thermodynamic Temperatures

This work package will be led by NPL, with partners PTB, INM, NIST, VNIIOFI, KRISS, and NMIA. The purpose of this work package is (a) to implement improvements in absolute thermometry facilities identified in WP4 and (b) to assign definitive thermodynamic temperatures to a set of HTFP cells selected from those produced in WP2. These measurements will provide the baseline thermodynamic temperatures for the selected HTFPs. The target uncertainty for these measurements is $<200\,\text{mK}$ ($k = 1$) at the Re-C point, and lower for the lower-temperature HTFPs. The measurements are currently scheduled to run from mid-2009 to Spring 2011.

2.6 WP6: Redefining Temperature Above the Silver Point

This work package is being led by NPL with all partners contributing. It is anticipated to run from November 2010 to July 2012. The overall aim of this work package is to allow the dissemination of T (that is, thermodynamic temperature, rather than T_{90}) above the silver point mediated by HTFPs. The theoretical framework for this has already been elaborated [20,21]. It is clear, provided two or more fixed points (one could be the Cu point) are used in conjunction with a reasonable-quality radiation thermometer, that suitable robust interpolation equations are available that would yield scale interpolation errors much less than 100 mK. In addition, these interpolation equations will work over a very wide range of temperatures, e.g., from the Cu point to, say, Re-C or above, with minimal increase in interpolation error. Of course, other uncertainties would need to be accounted for, such as the instruments size-of-source effect (SSE) and the overall uncertainty of the HTFP, but the prospect is for a step-change improvement in the realization and dissemination of high-temperature scales through these developments.

A suitable addendum to the *mise en pratique* for the kelvin [22] would be developed, giving alternative formalisms for realizing the scale above the silver point. Three clear options should be elaborated: traditional ratio pyrometry (i.e., the ITS-90 formalism), primary absolute radiation thermometry, and T mediated by HTFPs. This would include definitive temperatures of the HTFPs measured in WP5 above, along with a comparison of the uncertainties routinely achievable by each method.

3 Discussion

What are the implications of the research proposed here? There will be significant impact in both non-contact and contact thermometry, as well as in primary radiometry and photometry. For example, in non-contact thermometry the fixed points will provide a simple and robust method of disseminating thermodynamic temperature above the silver point through absolute radiometry mediated by the use of (these) high-temperature fixed points. The advantages of doing this are:

(a) Thermodynamic temperature (T) can be directly disseminated to the user community via short traceability lines.
(b) The uncertainty in scale realization and dissemination would be ~5 to 10 times less than in the current scale.
(c) A methodology of scale realization by interpolation is established that is a direct analog of contact thermometry, giving much greater flexibility than the current ITS-90.
(d) This would enable most NMIs throughout the world to realize the new scale, which would be fully thermodynamic, without having to incur the cost of establishing absolute radiometry facilities.
(e) Easy to disseminate the scale to user community through the supply, or self-construction to a prescribed recipe, of a limited number of HTFP cells.

In contact thermometry, uncertainty reductions of a factor of two or more are envisaged [7] as the wire bridge method is supplanted by the use of HTFPs for thermocouple calibration. In addition, improving high-temperature thermometry would have a significant benefit because thermometry is a key industrial control parameter essential for product quality, process control, and optimal energy use. Specific examples include: improving thermal processing of super-alloys for the next generation of turbine blades where uncertainties of 1°C ***in process*** at temperatures in excess of 1,300°C are required, improved and more reliable sintering of fuel for nuclear-power applications at ~1,750°C, and optimized production of composites (e.g., carbon-carbon, carbon-ceramic) whose production often takes place at temperatures in excess of 2,500°C.

In summary, the dissemination of high-temperature scales to industry by NMIs could undergo a radical change in the next few years. Instead of the scale being disseminated to calibration laboratories using instruments requiring regular recalibration, it could instead be disseminated by a set of once-only validated driftless high-temperature fixed points. These could then be used to perform the calibration of reference standards, and also the routine checks required to retain ISO 17025 accreditation. This approach would require much less checking and calibration to be performed by NMIs, reducing costs to industry on the one hand while improving dissemination and calibration quality on the other.

4 Conclusions

An ambitious, multi-center, cross-disciplinary research project has been put in place for which the end goal is step-change improvements in high-temperature thermometry above the silver point, in both primary contact and non-contact thermometry, with commensurate and possibly greater improvements down the supply chain. In 5–7 years, it is anticipated that NMIs will be able to routinely disseminate T above the silver point, with lower uncertainties than the current ITS-90, mediated by HTFPs.

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