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Progress Report for the CCT-WG5 High Temperature Fixed Point Research Plan

G. Machin[1], K. Anhalt[2], P. Bloembergen[3],
M. Sadli[4], Y. Yamada[5] and E. R. Woolliams[1]

[1]*National Physical Laboratory (NPL), Hampton Road, Teddington, Middlesex,TW11 0LW, UK*
[2] *Physikalisch-Technische Bundesanstalt (PTB), Abbestrasse 2-12, 10587 Berlin, Germany*
[3]*National Institute of Metrology (NIM), Bei San Huan Dong Lu No. 18, Beijing, 100013, China*
[4]*Laboratoire Commun de Métrologie (LNE-Cnam), 61, rue du Landy, 93210 Saint-Denis, La Plaine, France*
[5]*National Measurement Institute of Japan (NMIJ), AIST, Tsukuba, Ibaraki, Japan*

Abstract. An overview of the progress in High Temperature Fixed Point (HTFP) research conducted under the auspices of the CCT-WG5 research plan is reported. In brief highlights are: Provisional long term stability of HTFPs has been demonstrated. Optimum construction methods for HTFPs have been established and high quality HTFPs of Co-C, Pt-C and Re-C have been constructed for thermodynamic temperature assignment. The major sources of uncertainty in the assignment of thermodynamic temperature have been identified and quantified. The status of absolute radiometric temperature measurement has been quantified through the circulation of a set of HTFPs. The measurement campaign to assign low uncertainty thermodynamic temperatures to a selected set of HTFPs will begin in mid-2012. It is envisaged that this will be complete by 2015 leading to HTFPs becoming routine reference standards for radiometry and high temperature metrology.

Keywords: High temperature fixed points, radiation thermometry, pyrometry, radiometry

INTRODUCTION

High temperature fixed points (HTFPs) based on metal carbon eutectics, metal carbide carbon eutectics and metal carbide carbon peritectic alloys, have only recently been introduced into temperature metrology [1, 2]. However, recognising their far reaching potential, in 2007 WG5 (Radiation thermometry) of the CIPM[1] Consultative Committee for Thermometry (CCT) launched a large multi-partner project whose objective was to assign robust thermodynamic temperatures to a selected set of HTFPs; the metal-carbon eutectics of Co-C (1324 °C), Pt-C (1738 °C) and Re-C (2474 °C) [3].

The project's work was split into five workpackages (WP), each led by a different national measurement institute (NMI). The workpackages are:

- WP1 Assessment of the long term stability and robustness of the HTFPs, led by LNE-Cnam
- WP2 Development of optimum construction methods for HTFPs and construction of a set of HTFP cells for thermodynamic temperature assignment, led by NMIJ
- WP3 Operational characteristics of the selected HTFPs; data analysis and uncertainty budget, led by NIM
- WP4 Quantification of the world status of primary radiometry via an international comparison using HTFPs as transfer standards; recommendations for improvements, led by PTB
- WP5 Assignment of definitive thermodynamic temperatures to the selected set of HTFPs (developed in WP2) for use as temperature references, led by NPL

Progress has been made in all these workpackages and this paper describes the work that has been performed in each workpackage. It gives a forward look to the completion of the project, envisaged in 2015, where thermodynamic temperatures will be assigned to the three selected HTFP types so that they can then be used routinely, by the temperature and radiometry communities, as reliable high temperature references.

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[1] Comité international des poids et mesures

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WP1: LONG-TERM STABILITY AND ROBUSTNESS OF HTFPS

This WP is being led by LNE-Cnam, in cooperation with partners PTB, NPL, NMIJ, NIM and VNIIOFI (the All-Russian Research Institute for Optical and Physical Measurements). Establishing the long-term stability of the phase transition of HTFPs is of vital importance, as without stability they would be of no use as temperature references. Similarly HTFPs must be robust so that they are practical. Both of these aspects are investigated in this workpackage.

Four each of Co-C (4×NMIJ), Pt-C (2×NMIJ, 2×NPL) and Re-C (4×NMIJ) were constructed. These cells performed a dual role in the CCT-WG5 HTFP research project; they were used in this WP for stability trials and to investigate fixed point robustness and they were also used in WP4 for assessing the status of world radiometry, described below.

Long term stability studies of HTFPs

The starting point of the stability studies was to determine the temperature difference between the four HTFPs of a single type. Then a selected cell was aged for 50 hours at its melting point. The aging measurements were performed at NMIJ (Co-C), NIM (Pt-C) and VNIIOFI (Re-C). The difference between the aged cell and the designated reference cell was then re-measured to assess initial long term stability [4]. The results, reported as the temperature difference between post and pre aging, were presented in [5]; for Co-C (+8 ± 48) mK, for Pt-C (-52 ± 42) mK and for Re-C (-60 ± 44) mK. The Co-C was clearly stable during these measurements, whereas the Pt-C and Re-C exhibited a difference between pre and post aging slightly higher than the given $k = 2$ uncertainty, leading to an inconclusive result. Further investigations found that for both Pt-C and Re-C, the melting plateau shortened during the aging process. This was due to the erosion of the inner carbon/carbon (C/C) sheet liner. One effect of this plateau shortening was to cause the point of inflection (poi) of the melt to shift to lower temperatures, as observed [4]. In addition, for the Re-C fixed point, the aged cell was found to have experienced some leakage of material during the measurements which could also have contributed to the slightly larger difference than the $k = 2$ uncertainty.

In summary the first stability tests showed good stability for Co-C but the results were slightly inconclusive for the Pt-C and Re-C at the 50-60 mK level due to robustness problems with the aged cell,

The final step, which will be performed shortly, is to re-determine the temperature differences between the reference cell and the two HTFPs used to assess the status of absolute radiometric temperature measurement at NMIs worldwide (WP4). These latter two HTFPs will have experienced many 100s of hours of use in different furnace conditions – so this last step is a very strong test of HTFP stability. It is anticipated that these results will be available by late-2012.

Design for HTFPs robustness

The design of HTFPs has evolved over time; this is reviewed in [1]. The first HTFP cells were simple graphite crucibles. However this design was not sufficient to obtain optimum performance. A significant amount of development work was performed by the WP partners to improve both the robustness and performance of HTFPs. These improvements have led to a lower melting range (flatter melt plateau) and longer duration for the melt (in a given furnace) compared to the earlier design of HTFP.

Robustness was first improved by including a second thin graphite cylinder within the graphite crucible. Melt plateau shape was improved by including one or more wraps of C/C sheet within the graphite crucible. This improved the longitudinal temperature gradients along the cell. However its positive effect was limited in duration, as reported in [4], where plateau durations of Pt-C reduced by a factor of two during the 50 hour aging tests. This was found to be due to the erosion of the C/C sheet by contact with the molten metal ingot. Because of this erosion, the most recent design of HTFPs now includes a thin solid graphite cylinder for robustness, then one or more wraps of C/C sheet between this cylinder and the crucible wall to give the improved melt plateau shape and duration. This design is being used for all the HTFP cells constructed for the thermodynamic temperature assignment in WP5.

WP2: CONSTRUCTION OF HTFPS FOR THERMODYNAMIC TEMPERATURE ASSIGNMENT

This WP is being led by NMIJ with contributions from PTB, NPL, LNE-Cnam, INRIM (Istituto Nazionale di Ricerca Metrologica), NIM, VNIIM (All-Russia D.I. Mendeleyev Scientific and Research Institute for Metrology) and VNIIOFI. The purpose of this WP is to construct a number of high quality HTFPs, the parameters of which are set by a defined protocol [6]. These HTFPs are in addition to those constructed for the WP1 studies, and incorporate the improved design findings of that WP. From these cells four each of Co-C, Pt-C, Re-C (and to maintain links with ITS-90, Cu) will be selected and have their thermodynamic temperature determined by a number of institutes through the activities in WP5.

In total, seven Re-C cells were constructed by six institutes; six Pt-C cells were constructed by five institutes; seven Co-C cells were constructed by seven institutes and, in addition, six Cu cells were constructed by five institutes. The cells had to be 24 mm in diameter, 50 mm or less in length with a blackbody aperture of at least 3 mm. All HTFPs were constructed with a double layer of C/C sheet protected by an inner graphite sleeve as described above. The material purity should be where at all possible 99.999% metals basis, with the graphite crucibles purified to better than 10 ppm. Different metal lots and even suppliers of the materials were to be used where possible to test for variation caused by different residual impurities. Ideally high quality quantification of the level on impurities would be performed on the metals, separate from the assay supplied by the material manufacturer. Details of this are in [7].

Four project participants then assessed the quality of the HTFPs against a set of agreed objective criteria given in the protocol [6]. The Cu cells were assessed by INRIM, the Co-C cells by LNE-Cnam the Pt-C cells by NPL and the Re-C cells by NIM. Within the protocol [6] details were given to concerning purging and cleaning of the furnace to avoid any contamination of the HTFPs. The same set of measurements were performed on all the cells. The results of these will be used to facilitate an objective selection of the cells for thermodynamic temperature assignment. The measurements performed were: repeatability of the melt plateau (at least three melt plateaux with at least two different melting steps); determination of the melting range; the uncertainty in determining the point of inflection for a particular cell and the temperature differences between the cells.

These measurements will then be used, along with other information such as metal purity, melt/freeze plateau regularity, slow entry (or exit) into (or out of) the melt to identify four HTFPs (and Cu), of each type, to enter the thermodynamic temperature assignment WP. It is envisaged that this selection will be completed by Spring 2012.

WP3: OPERATIONAL CHARACTERISTICS OF HTFPS

This WP is being led by NIM with support from NPL and NMIJ. It is focused on investigating source aspects of the measurement problem. It seeks to understand the factors influencing the measurement results and, to ensure that the ensuing corrections and/or associated uncertainties in the determination of the eutectic temperatures, T_{E}, of the eutectics Co-C, Pt-C and Re-C in their pure state, in terms of thermodynamic temperature, *T,* are properly quantified (details of this are elaborated in [8]). Measurements of eutectic temperatures will be performed in WP5, where the overall uncertainty $u\left(T_{\mathrm{E}}\right)$ is established, which include the additional uncertainties inherent in detector based absolute radiometry [9].

Crucial to the measurements to be performed is the determination of the equilibrium liquidus temperature, $T_{\mathrm{liq}}(0)$, for the above mentioned eutectic systems, from which T_{E} can be derived as $T_{\mathrm{E}} = T_{\mathrm{liq}}(0) + \sum_i c_i m_i$, where c_i are the concentrations of the impurities in the liquid state of the sample in question, and m_i the associated liquidus slopes. Since the liquidus temperature $T_{\mathrm{liq}} = T_{\mathrm{liq}}(v)$ is dependent on the speed, *v,* of the preceding freeze, influencing the eutectic structure within the ingot, the so-called "(*micro*)*structure effect*", an extrapolation is needed to determine $T_{\mathrm{liq}}(0)$. This extrapolation is based upon the linear relationship that exists between $T_{\mathrm{liq}}(v)$ and $\sqrt{v}$, where v is proportional to the offset $T_{\mathrm{freeze}} - T_{\mathrm{furnace}}$ between the nominal HTFP freezing temperature T_{freeze} and furnace temperature T_{furnace} [10]. Note that $T_{\mathrm{liq}}(v)$ is deduced by extrapolation from $T_{\mathrm{infl}}(v)$, the inflection point of the melting curve, where the extrapolation procedure also yields the uncertainty $u\left(T_{\mathrm{liq}}(v)\right)$.

The uncertainty associated with impurities is accounted for by the Overall Maximum Estimate (OME) approach, i.e. disregarding its complement, the Sum of Individual Estimates (SIE) [11].

In addition to the (micro)structure effect, and the effect of impurities, other influence factors considered are typical source parameters such as the temperature drop between the liquid/solid interface and the cavity bottom, and the cavity emissivity. Their uncertainties are estimated in the protocol of WP3 [8] and in reference [9].

A complicating factor is the furnace effect, implying that the measurement results, i.e. the actual temperatures measured, appear to be locally biased, since they are to some degree influenced by the type of furnace used. Furnaces with non-uniform furnace temperature profiles cause the speed of the preceding freeze and of the subsequent melt to be dependent on the temperature gradient along the cell, resulting in irregularly shaped liquid/solid interfaces. This can cause the formation of a liquid eutectic thermal bridge (from the crucible wall to the cavity) before completion of the freeze/melt. When (or whether) this bridge is formed is critically dependent upon the severity of the thermal gradients within the furnace. Initially it seemed that the furnace effect acted via the temperature gradient through source parameters such as the structure effect, temperature drop and cavity emissivity and through the size-of-source effect. However as has been evidenced in [11], these four effects combined do not cover the overall measured effect.

Presently within the context of WP3 other factors are being investigated associated with the operation of the furnace on the one hand and with the characteristic properties of cell and ingot on the other. These are: The finite response time of the furnace to step-wise offsets and the procedure initiating freezing and melting. In addition alternatives to the current estimates (SIE and OME) of the uncertainty associated with the impurities are under consideration [13].

The Protocol to WP3 [8] will be available on request.

WP4: ASSESSMENT OF THE STATUS OF WORLD PRIMARY RADIOMETRIC TEMPERATURE ASSIGNMENT

This WP is piloted by PTB and involves participants NPL, NIST (National Institute of Standards and Technology, USA), NMIA (National Measurement Institute of Australia), LNE-Cnam, VNIIOFI and NRC-INMS (National Research Council-Institute for National Measurement Standards, Canada [NRC hereafter in this manuscript]). Its objective is to test and improve the local realizations of thermodynamic temperature measurement in the seven participating laboratories.

For implementation as primary temperature references the thermodynamic temperatures of the HTFPs have to be determined with measurement uncertainties as low as possible, ideally lower than what is currently achievable with the ITS-90 (when the thermodynamic uncertainty of the defining fixed points are taken into account). In the temperature range above 1000 °C this can be achieved by using absolute radiometry as a method of primary thermometry. A number of NMIs have established different experimental schemes that allow for the direct measurement of thermodynamic temperature by radiometric means [3, 14-18].

The WP commenced with PTB measuring the thermodynamic temperature for three HTFPs, namely, 3×Co-C, 3×Pt-C and 3×Re-C. These were the same cells as those constructed for use in WP1. One set of cells was kept at PTB for reference; the second two sets were then circulated in two loops between participants in the following sequence: loop 1; NPL, NIST, NMIA and loop 2; LNE-Cnam, VNIIOFI, NRC. Finally all cells were returned to PTB and re-measured.

Although the two loops principally could have run in parallel, they were in practice performed one after the other over a period of two years. This was to allow time for all the participating NMIs to prepare their thermodynamic temperature measurement capability. This was because that while for some NMIs high-temperature measurement based on absolute radiometry was well established, for others absolute radiometry was being used for the first time to determine the temperature of HTFPs.

An overview of the measurement uncertainties as quoted by the participating laboratories is given Table 1), where also the differences $\Delta(T-T_{\mathrm{ave}})_{\max}$ and $\Delta(T-T_{\mathrm{ave}})_{\min}$ to the average values for each HTFP are presented. More details are to be found in [19].

WP5: ASSIGNMENT OF THERMODYNAMIC TEMPERATURE TO HTFPS

WP5, due to start in June 2012, is the final technical WP of the CCT-WG5 HTFP research plan [3, 9]. Its objective is to determine definitive thermodynamic temperatures for the melting transitions of the metal-carbon eutectics Co-C, Pt-C and Re-C and, in addition, the freezing point of Cu.

TABLE 1. Overview of quoted measurement uncertainties for the local T measurement, and maximum and minimum differences to the average melting temperature for the three HTFP

HTFP	T_{nominal} / °C	$U_{\min}$ … $U_{\max}$ range, $k = 2$ / K	$\Delta(T-T_{\mathrm{ave}})_{\max}$ / K	$\Delta(T-T_{\mathrm{ave}})_{\min}$ / K
Co-C	1324	0.24 ... 0.36	<0.30	<0.10
Pt-C	1738	0.36 … 0.54	<0.50	<0.10
Re-C	2474	0.64 … 1.38	<0.75	<0.20

This WP is the culmination of the project and during WP5 measurements will be made of the HTFP cells selected from WP2 using filter radiometry developed and tested by WP4 and corrections made for different source effects determined during WP3.

Prior to the formal start of this WP, considerations have been given to the design and protocol of the measurement campaign and to how the results will be analysed. Four cells of each kind will be measured in two measurement loops.

Two of the cells, designated A and B will be measured by NPL, NIST, NRC, PTB, VNIIOFI and then again at NPL, and the other two cells, C and D, will be measured by NPL, LNE-Cnam, NMIA, PTB, NIM, IO-CSIC (Agencia Estatal Consejo Superior de Investigaciones Cientificas)/CEM (Centro Espanol de Metrologia) and finally at NPL. To ensure a strong link between the two groups of cells and to test for cell stability, NPL will measure all cells at the beginning and end of the measurement campaign and PTB will measure all cells during the campaign.

The protocol defines the operational conditions for the furnace and additional measurements required to ensure that the equilibrium liquidus temperature can be determined, that is the extrapolated melting temperature of a solid that was frozen with a freeze speed approaching zero. This extrapolation will remove the sensitivity of the melting temperature to the microstructure of the solid ingot determined from the speed of the previous freeze. Corrections will also be made (or uncertainties estimated) to account for ingot impurities, the furnace and cell dimensions and temperature profile.

The results of this measurement campaign will be combined in a generalised weighted mean accounting for the correlations of the different measured values of the different cells measured by the different participants.

At this stage the protocol is being finalised. The measurements are expected to start in September 2012.

Linkage to the European Metrology Research Programme[2] (EMRP) project "Implementing the new kelvin" (InK)

The work undertaken in this WP is to be performed as part of the larger EMRP "Implementing the new kelvin", the so called InK, project [20]. That project has a very broad scope seeking to perform primary thermometry from 0.9 mK to 3000+ K with the lowest uncertainties ever attained. Relevant to this project is the activity that focuses on assigning thermodynamic temperatures to HTFPs so that part of InK strongly overlaps with the objectives of WP5 of the CCT-WG5 research plan. The main difference is that the two Spanish institutes CEM and IO-CSIC will also take part besides those originally envisaged in WP5. According to the InK schedule thermodynamic temperatures will have been assigned to the HTFPs by July 2015.

SUMMARY

Good progress has been made in all aspects of the CCT-WG5 research plan. The design of HTFPs has been refined to ensure both robustness and optimum performance of the HTFP cells. Long term stability has been and is still being investigated. A good number of HTFPs (and Cu) have been constructed by different institutes and their performance is currently under evaluation. The operational characteristics of HTFPs have been studied in detail and corrections or uncertainty terms identified. The primary radiometric thermodynamic measurement capability of seven institutes has been assessed and recommendations for improvements currently being formulated. By September 2012 thermodynamic temperature assignment to the selected set of HTFPs (and the freezing point of Cu) will be underway culminating in definitive thermodynamic temperatures with ultra-low uncertainties for the four selected fixed points by the end of 2015. Once these temperatures are approved by CCT they will be incorporated into the developing *mise en pratique* for the definition of the kelvin (*MeP*-K, 21, 22). Then the HTFPs will be used in a variety of ways such as reference points for establishing thermodynamic temperature or a radiance scale or just as known reference points for confirming long term stability of filter radiometers.

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[2] Information about the EMRP can be found at: http://www.euramet.org/index.php?id=publicity

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