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A PARADIGM CHANGE IN HIGH TEMPERATURE METROLOGY

Prof Graham Machin, Head of Temperature Standards

National Physical Laboratory, Hampton Road, Teddington, Middlesex, TW11 0LW, UK, graham.machin@npl.co.uk

Abstract: The measurement of temperatures above 1100 °C is undergoing a step change improvement with the development of high temperature fixed-points and improved thermocouple types such as Pt/Pd. This paper reviews international developments in these areas that could lead to an improved way of realising ITS-90 above the Ag point, reduced thermocouple uncertainties by factors of two or more, and improved dissemination and measurement of high temperatures to industry.

Keywords: pyrometry, high temperature fixed-points, metal-carbon eutectics, thermocouples

1. INTRODUCTION

The accurate measurement and control of temperature above 1100 °C is vital for the success of many industrial processes, such as aerospace, advanced materials and alloys, power generation and ceramics. This is generally performed by radiation thermometry, through noble metal thermocouples Types S, R or B above 1200 °C, or W-Re types if very high temperatures (i.e. >1800 °C) are required. In the past few years significant developments have taken place in this field that promise to radically improve primary and industrial high temperature thermometry and hence improve product quality, reduce waste and optimize energy use.

Firstly, there has been rapid progress in high temperature fixed point research [1, 2, 3]. Here, through the utilization of binary eutectic alloys of metal and carbon, new fixed-points up to 2500 °C have been developed. They are often known as metal-carbon eutectic, or M-C eutectic fixed points. These have proven repeatability of <100 mK from melt to melt, much better than the scale realisation uncertainties. It is envisaged that these new fixed points will be used for the calibration of radiation thermometers, in radiometry and for improved scale dissemination to industry. Ultimately they may provide a means of realising a temperature scale above the silver point with significantly lower uncertainties than are currently possible.

Secondly, work on Pt/Pd thermocouples has significantly advanced [4, 5]. These elemental thermocouples are considerably better in terms of stability and repeatability than the platinum-rhodium alloy thermocouples currently deployed. In principle they could be used to transfer the temperature scale to industrial measurement laboratories from national measurement institutes with very low uncertainties up to 1500 °C. These thermocouples with the new high temperature fixed-points make a powerful combination for reducing uncertainties and increasing the reliability of high temperature measurement.

This paper reviews the activity that has been taking place in world metrology in these areas and looks forward to where these developments might lead in the next five years.

2. HIGH TEMPERATURE FIXED POINTS AND Pt/Pd THERMOCOUPLES

In this section an outline of the construction of high temperature fixed-points is given followed by progress in their research in the fields of non-contact thermometry, absolute radiometry and contact thermometry.

2.0 Construction

Yamada of the National Metrology Institute of Japan (NMIJ) first reported development of these high temperature fixed-points [1]. They consist of a high purity graphite crucible in which is cast an ingot of binary metal-carbon eutectic alloy. For both the metals and the graphite the purest materials available are used in construction, typically 0.9999+ for the metals and 0.99999+ for the graphite. There are on-going concerns about the reliability of manufacturers assays of the metals and it would be wise to obtain an independent assay before use of metals in ingot construction. A photograph of a typical fixed-point for non-contact thermometry is shown in Figure 1. An ingot of eutectic alloy surrounds the reentrant well. This forms the blackbody radiator.

Fig. 1: Photograph of a typical high temperature fixed point crucible

To ensure good filling of the cells it is necessary to begin filling with lower graphite concentration than required for the eutectic composition (i.e. using a hypoeutectic composition). The initially metal rich ingot attains the eutectic composition through dissolving the required graphite from the inner protective sleeve. This process can take a number of filling cycles, typically ~10 mainly due to the fineness of the powders used.

The alloys that can be used, with their nominal melting temperatures, are given in Table 1.

Table 1: Metal-carbon and metal-carbide-carbon eutectics with nominal temperatures [6]

Eutectic	Approximate temperature / K	Approximate temperature / ºC
Metal-carbon		
Fe-C	1426	1153
Co-C	1597	1324
Ni-C	1602	1329
Pd-C	1765	1492
Rh-C	1930	1657
Pt-C	2011	1738
Ru-C	2226	1953
Ir-C	2565	2292
Re-C	2747	2474
Metal-carbide-carbon		
B_4C-C	2659	2386
δ(MoC)-C	2856	2583
TiC-C	3032	2759
ZrC-C	3155	2882
HfC-C	3458	3185

In addition to metal-carbon fixed-points ultra-high temperature fixed-points can be constructed using metal-carbide-carbon (MC-C) eutectic alloys. The details of these are also given in Table 1.

Cell robustness has been a problem. This has largely been overcome by a) introducing the inner protective sleeve (see Fig. 1) and b) ensuring, if possible, that the coefficient of thermal expansion of the graphite is lower than that of the ingot material.

2.1 Non-contact thermometry and radiometry

Nearly all the early work on high temperature fixed points was performed using non-contact thermometry methods.

The measurement of the fixed-point transition temperature became the immediate focus of the research field. A typical transition curve is given in Figure 2 [taken from 2]. All the metal-carbon fixed-point transitions follow a similar pattern.

These initial studies showed that the point of inflection of the melting point was a very repeatable temperature. Typical repeatabilities from melt to melt of any individual cell were much less than 100 mK even at the Re-C point. This feature on the melt curve has, at least for now, become the de-facto way of specifying the transition temperature of these fixed points.

After the repeatability of individual cells had been clearly established the focus of research shifted to how reliably to construct and test fixed-point cells so that cells made by different groups nevertheless have equivalent radiance temperatures, within the measurement uncertainties.

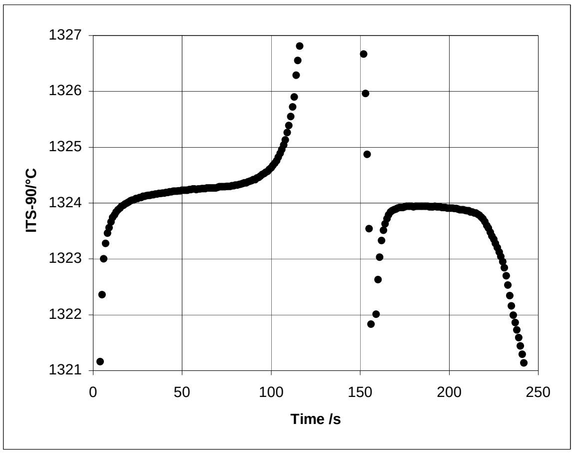

Fig. 2: A typical Co-C fixed-point melt-freeze cycle [2]

The bulk of this work has been performed in Japan and Europe, with significant activity particularly at higher temperatures (with the metal-carbide carbon eutectics) for radiometry applications at VNIIOFI (All-Russian Institute for Optical and Physical Measurements) [7]. In the EU the work has been coordinated through the recently completed EU Framework 5 project HIMERT [8].

From the proof of concept studies reported by Yamada [1], research has progressed sufficiently to construct reliable metal-carbon cells with reproducibilites of order 200mK at 2500 °C [9, 10]. In the study reported in [9] high temperature fixed-point cells were constructed by the NPL, NMIJ and Bureau International des Poids et Mesures (BIPM), of Pt-C and Re-C and compared at the NPL. In this case the three Pt-C cells agreed to better than 150 mK and the three Re-C cells agreed to better than 250 mK, the uncertainties of the measurements were <250 mK. This very promising result led to a further more extensive study at Physikalisch-Technische Bundesanstalt (PTB), Berlin in May 2004 [10]. Here M-C eutectic cells of Co-C, Pd-C, Pt-C, Ru-C and Re-C constructed by NPL, NMIJ and the then BNM-INM (Bureau National de Metrologie- Institut National de Metrologie) were melted using quasi-identical high temperature furnaces (type Nagano [11]) and compared using two similar high performance pyrometers (one of PTB and the other of INM).

The results of this comparison (with the PTB pyrometer) are shown in Figure 3 below. For Co-C, Pt-C and Re-C they were excellent with intra-cell agreement of less than 200mK, lower than the uncertainty of the comparison. The agreement between the three Pd-C and Ru-C cells was somewhat worse but this is almost certainly due to problems with residual impurities in the Ru and Pd.

These and subsequent results [12] clearly indicate that it is possible, with care, to construct high temperature fixed-point cells with a high degree of reproducibility.

Further work is still required to understand and mitigate the effects of a) residual trace impurities in the metal powders; b) the effects of furnace temperature gradients; c) different filling procedures; d) structural effects due, for example to freezing rate; to ensure that <100 mK reproducibilities for all high temperature fixed-points of interest can be routinely achieved.

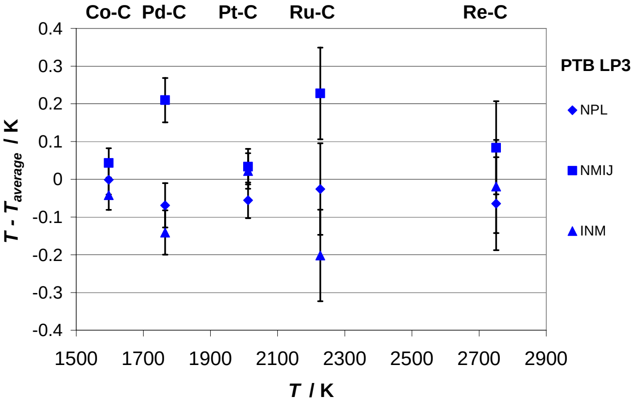

Fig. 3: Radiance temperature differences between M-C eutectic fixed-point cells constructed by NPL, BNM-INM and NMIJ, May 2004 [10]

Once reproducibility and long term stability has been reliably demonstrated the final step in this work is to assign thermodynamic temperatures to the fixed-points. This is one of the main goals of a coordinated programme of research planned by the CCT-WG5[1] [13]. In this programme a two-step comparison is envisaged. First participating National Measurement Institutes (NMIs) will perform a preliminary comparison of their absolute radiometric thermometry capabilities using a selection of high temperature fixed-points (currently envisaged in 2007-2008). This will allow the NMIs to identify any weaknesses in their absolute radiometry capabilities and undertake improvements. Then in ~2009-2010 a second measurement sequence will take place. This time high quality fixed-point cells will be circulated between participating NMIs and definitive thermodynamic temperatures will be determined.

2.1.1 An improved high temperature scale

The advent of high temperature fixed points leads to the interesting possibility of improving high temperature scales both in realisation and also in dissemination. Improved dissemination will be dealt with in Section 3.

The high temperature part of the ITS-90 is currently derived by using Planck's law in ratio form referenced to a defined fixed-point of Ag, Au or Cu. As this is an extrapolation the uncertainty necessarily increases as T^2. This leads to scale realisation uncertainties of approximately 1~2°C in the best laboratories at 2500 °C. However once the transition points of high temperature fixed-points have been definitively measured and assigned then the way is opened to allow alternative improved methods of realizing and disseminating high temperature scales. It has been shown [14] that there is significant scope to greatly reduce scale uncertainties. Two approaches could be followed:

a) The temperatures of the fixed-points could be defined as in the current ITS-90. These would formally have no uncertainty. Then using a prescribed interpolation function [e.g. 14], in direct analogue to the ITS-90 for lower temperatures (i.e. using standard platinum resistance thermometers) a predefined function could be fit to a radiation thermometer output at the defined temperatures. This method of scale realisation would give very low uncertainties, require possibly as few as two fixed-points (one of which could be one of the already defined ITS-90 points), and give laboratories flexibility as to the temperature range over which they desired to realize a scale.

b) A second approach would be to use directly the measured thermodynamic temperatures of the high temperature fixed-points. In this case the fixed-points could be used in the same way as in a) above but now a thermodynamic temperature scale could be established, mediated by the fixed-points.

Either path would lead to a scale which is consistent with the current ITS-90 but would have improved reproducibility and lower uncertainties. This approach will, in the next few years, have to be rigorously qualified before it could be adopted in practice. Eventually alternate but consistent means of high temperature scale realisation through the mise-en-pratique for the kelvin [15] are envisaged.

2.2 Contact thermometry

High temperature fixed-points have an immediate utility in improving the calibration of high temperature noble metal thermocouples [e.g. 16]. These have long been calibrated at conventional fixed-points and at the high end generally using the Pd wire-bridge method. This limits the primary level calibration uncertainty in an NMI to about 1-1.5 °C (*k*=2) at the Pd point, 1553 °C. As these new fixed-points have repeatabilities of <100mK it is clear that significant improvements could be achieved by introducing high temperature fixed points into the calibration chain.

As part of the HIMERT project [2, 8] Morice [5] constructed M-C eutectic cells of Co-C, Ni-C, Pd-C, Pt-C and Ru-C. These cells were considerably larger than those used for radiometric applications to facilitate adequate immersion of the thermocouples. The fixed-points at temperatures of Pd-C and below were investigated with standard type B and type S thermocouples. The Pt-C and Ru-C were investigated using specially constructed W-Re thermocouples. The results of these studies indicated that M-C eutectic fixed-points could successfully be implemented into calibration chains at high temperatures, replace the Pd wire-bridge method and lead to improved uncertainties for

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[1] CCT-WG5 is a working group of the Comite Consultatif de Thermometrie responsible for radiation thermometry

the calibration. A typical melt and freeze cycle with a thermocouple obtained by [5] is given in Figure 4.

An active international programme of M-C eutectic research into their application to thermocouple applications is underway [17, 18, 19].

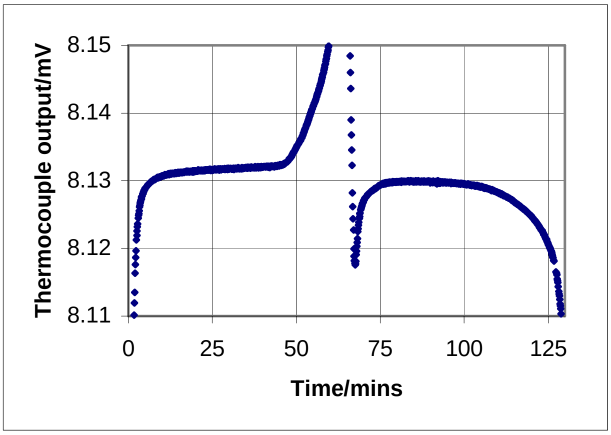

Fig. 4: Melt and freeze curve for a Co-C point using a type S thermocouple [5]

Besides the gains that can be realized in the calibration of conventional noble metal thermocouples further improvements can be made by using high temperature fixed points in conjunction with the superior pure metal thermo-element Pt/Pd thermocouples. Combining the benefits of high temperature fixed-points with the performance of Pt/Pd thermocouples [4, 20] is the subject of a new Euromet research project 857 [21], between PTB, LNE and NPL and an extra-EU partner NMIJ. This project is set to run for the next five years. The project will examine in detail the metrological potential of Co-C and Pd-C fixed points for calibrating noble metal thermocouples generally and exploring the limitations of Pt/Pd thermocouples as stable thermometers.

The final outcomes of this project are expected to be:

- The production of robust Pt/Pd thermocouples to facilitate full transfer of their benefits to industry
- A new methodology for calibrating noble metal thermocouples using fixed-points of Co-C and Pd-C, including the superseding of the current wire bridge method
- A reduction in the routine calibration uncertainty of noble metal thermocouples by a factor of 2 to 3

2.3 Scale comparisons

Another immediate benefit of high temperature fixed-points is the comparison of primary realisations of high temperature scales with unprecedented accuracy. These have, until the advent of high temperature fixed-points been performed using tungsten ribbon lamps of transfer radiation thermometers/radiometers. Both are subject to unpredictable drifts during transport, radiometers have size-of-source corrections that must be applied and lamps are fragile, have a small target size (typically 1.5 mm) and only operate over a limited range of wavelengths.

A comparison of scales between NIST and NPL was performed in October 2003 using NPL high temperature fixed-points of Co-C, Pd-C, Pt-C, Ru-C and Re-C. The difference in scales between NIST and NPL was shown to be <0.5 °C over the range 1300 °C to 2500 °C, with an uncertainty U(*k*=2) of 0.5 °C at 1300 °C and 2 °C [22], Figure 5. Such good agreement was only demonstrable because the transfer artifacts were of themselves inherently robust and as far as can be demonstrated driftless.

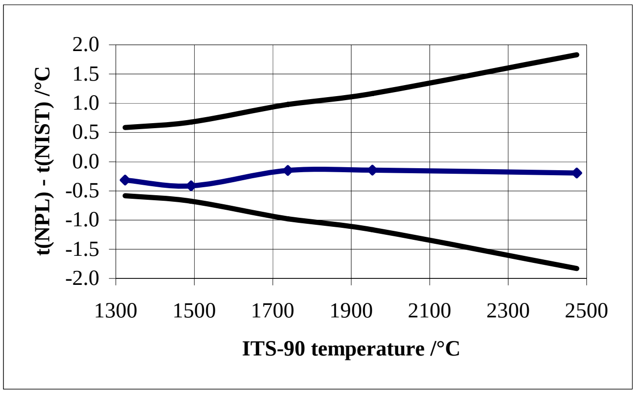

Fig. 5: The comparison of ITS-90 realised by NPL and ITS-90 equivalent as realized by NIST [21]

These cells were subsequently transported to PTB and their thermodynamic temperature measured. Thus the artifacts were used to compare the absolute radiometric scales of NIST and PTB. The radiance temperature agreement between the two institutes was shown to be in agreement to 0.5 °C with a combined U(*k*=2) of less than or equal to 0.5 °C [23]. The great utility of high temperature fixed-points in confirming the veracity of primary scale realisations has thus been demonstrated.

3. INDUSTRIAL APPLICATIONS

The application of the new fixed-points in industry could potentially be widespread, improving energy efficiency and improved/more consistent product quality (reducing waste) through helping optimize process control. The benefits would first be seen in industrial and accredited calibration laboratories, but in the medium term it is envisaged that high temperature fixed-points could be used directly "in-process" to improve temperature control through monitoring and facilitating corrections for control sensor drifts.

3.1 Improved industrial calibrations and dissemination

High-level industrial laboratories accredited for the calibration of noble metal thermocouples usually perform their calibration by comparison of the thermocouples under test to a reference noble metal thermocouple. Alternatively they use a fixed point of Ag or Cu and the Pd-wire bridge method fitting a known reference function between two or more fixed-points. This approach is prone to error because of the considerable temperature gap between the copper point at 1084 °C and the Pd wire point at 1553°C.

A third, eminently practical, alternative will become available shortly utilizing high temperature fixed-points. When the temperature of the fixed-points has been internationally agreed then any of the fixed-points listed in Table 1 can be used to generate a high temperature scale for calibrating thermocouples. However in this interim period there is no reason why high temperature fixed points cannot be used for thermocouple calibration provided they have been pre-qualified by an appropriate NMI. This is the aim of a current project between NPL and UK manufacturer of noble metal thermocouples Consolidated Ceramic Products (CCP) [24]. In this project a Co-C cell will be manufactured and qualified by NPL with low uncertainties using radiation thermometry. This fixed-point will then be incorporated into CCP's calibration facility. When the suitable tests have been performed CCP will be able to offer special calibrations with lower uncertainties (±1 °C) at the Co-C point (~1324°C). This is a factor of two or more better than CCP's current best measurement capability. Demanding aerospace applications such as the heat treatment of a new generation of turbine blades are requiring these small uncertainties of thermocouple manufacturers.

Radiation thermometer calibration traditionally has relied upon tungsten ribbon lamps being transferred from an NMI to the calibration laboratory. However in recent years the temperature scale has been transferred between NMIs and industrial calibration laboratories through the use of calibrated radiation thermometers. Whilst these are an improvement on tungsten ribbon lamps, they are still subject to undetected drift/shifts in calibration. To ensure this doesn't invalidate the calibrations an elaborate traceability route with multiple cross-checking is required. In the future this could radically change. The scale could be disseminated to industrial laboratories through the supply of driftless high temperature fixed-points such as those in given in Table 1. The laboratory, dependent on requirements, could generate a range of scales. For instance if the laboratory only requires a scale to 1500 °C with low uncertainties then it could fit a suitable interpolation equation [14, 25] to the output of a low target size radiation thermometer at the Cu point, the Co-C and Pd-C point with no a-priori knowledge required as to its spectral response. If an extended range is required then the laboratory could use the Re-C point at the upper end. It has been shown [26] that very low uncertainties are possible for a scale obtained in this way, provided that the temperatures are known.

Efforts by NMIJ [27] and NPL [2] have led to experimental adoption of M-C eutectics in high-level industrial radiation thermometry calibration laboratories. In particular a large area Co-C point has been installed in the test laboratories of Land Instruments International Ltd, UK to check the performance and calibration of non-contact thermometers.

In summary, the dissemination of high temperature scales to industry by NMI's 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 for ISO17025. This approach would require much less checking and calibration to be performed by NMI's, reducing costs to industry on the one hand whilst improving dissemination and calibration quality on the other.

3.2 Optimising process control and energy use

The question could then be asked "Why stop at the calibration laboratory?" "Why not disseminate these improvements directly to the industrial process?"

Significant improvements could be made through the *in-situ* checking of thermocouple drifts – particularly tungsten rhenium type, and noble metal type in critical applications.

In addition improvements can be made for processes that are controlled by radiation thermometers through windows subject to progressive contamination. By incorporating a high temperature fixed-point in the process a normalization of the thermometer could be performed correcting for the window contamination and allowing the process to run optimally, again improving energy efficiency and product quality.

4. CONCLUSION

A step change improvement in high temperature metrology is to be anticipated in the next 5-7 years as the benefits of the new high temperature fixed-points and improved thermocouple types are realized. Use of energy in high temperature processes will be optimized leading to significant energy efficiency gains, improved product quality and reduced scrap levels.

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