A possible BIPM project on metal-carbon eutectic fixed points

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This paper gives an overview of the interest of metal-carbon eutectic fixed points for high temperature metrology. The available results are briefly summarised. A possible contribution of the BIPM radiometry section to this field is discussed.

1. Current situation : Temperature measurements according to the ITS-90 in the pyrometry range lead to increasing uncertainty with increasing temperature, especially above 2000 K. The main reason for this is that the use of a much lower reference temperature requires an effective wavelength of about 600 nm or higher. All uncertainty contributions expressed as $\Delta L_\lambda / L_\lambda$ (emissivity, non-linearity,…) lead to a temperature uncertainty which increases with the square of the temperature, according to

$$\Delta T = \frac{\lambda T^2}{c_2} \frac{\Delta L_\lambda}{L_\lambda}, \quad c_2 = 0.014388\ K\ m$$

For higher temperatures it would be advantageous to measure the spectral radiance at shorter wavelengths. The much lower reference temperature in pyrometry, however, excludes this option.
An additional uncertainty is introduced by the reference temperature, whose uncertainty is estimated as ΔT_{ref}~50 mK, and which is propagated as

$$\Delta T = \left(\frac{T}{T_{ref}} \right)^2 \Delta T_{ref}$$

Non-linearity is a problem, because the ratio of the spectral radiances at the two temperatures becomes very large for $T >> T_{ref}$.

As a consequence of these difficulties, the measurement uncertainty at 3200 K is of the order of 1 K.

To overcome these problems, the CCT recommended in 1996 the development of high temperature fixed point black bodies above 2300 K with a reproducibility of better than 100 mK [1]. This would allow to calibrate radiation thermometers by interpolation or by comparison with a nearby fixed point. A determination of the thermodynamic temperature of a high temperature fixed point with an uncertainty of about 100 mK would also allow to determine subsequently by pyrometry the temperature of the Ag, Au and Cu points with uncertainties comparable or even below the current estimated uncertainties.

Spectral radiance scales are *disseminated* by tungsten ribbon lamps and pyrometers which both are not ideal transfer standards. The radiance temperature of tungsten ribbon lamps depends strongly on the measurement wavelength. The repeatability after careful alignment is typically 0.15 %. A simple-to-operate high temperature fixed point black body might replace lamps as transfer standards on the longer term.

2. Eutectic high temperature fixed points: In 1999 Y. Yamada (NMIJ) presented first results on eutectic metal-carbon fixed points in graphite cells [2]. In these cells the carbon from the crucible is a component of the fixed point material, so that the material is inherently free of contamination from the crucible material. The following table shows a list of eutectics with approximate transition temperatures. [3].

Eutectic material	Melting temp. / °C
Fe-C	1153
Co-C	1320
Ni-C	1326.5
Pd-C	1504 +/- 16
Rh-C	1694 +/- 17
Pt-C	1705 +/- 13
Ru-C	1940
Ir-C	2296 +/- 16
Re-C	2505 +/- 15
Os-C	2732+/- 22
Ti-C	2755

Table 1: Metal-Carbon eutectics above the copper point and their approximate melting temperatures.

For a number of materials from this list melting and freezing plateaux were observed with radiometers or with high-temperature thermocouples of type R [2,4]. Recently repeatabilites of melting points of better than 100 mK or even 70 mK were reported [5, 6].

Possible applications are the use as additional defining fixed points of a future ITS-20XX and use as secondary fixed points for pyrometer or thermocouple calibration. Another possibility is the replacement of tungsten ribbon lamps as transfer standards by fixed point black bodies.

Research on metal-carbon eutectics is currently one of the main research fields related to temperature metrology. Euromet has started a co-operation project in late 1999 (BNM-INM, BNM-LNE, PTB, NPL) to investigate the suitability of these materials for high temperature references. Other work is done at the NMIJ and the NPL [6]. Besides the present document three other working documents (CCT/01- 9, -17, -21) treat the same subject.

3. A possible contribution of the BIPM: The next step towards a possible application of eutectic fixed points in a future ITS-20XX would be a systematic study of the optimal operation conditions and a determination of their thermodynamic temperatures. The most promising technique for this is radiation thermometry based on a cryogenic radiometer. The latter requires a well-equipped radiometry laboratory as it exists at the BIPM. Such measurements would make use of nearly all radiometric techniques available at the BIPM: laser-based cryogenic radiometer, spectral comparator and aperture measurements. The planned second cryogenic radiometer for monochromator-radiation would also contribute to this project, since the existing system is not ideally suited for filter radiometer calibrations near or below 400 nm, the optimal wavelength range for radiation thermometry above 2000 K. The existing heat-pipe black body could be used to test filter radiometer calibrations against PRTs carrying the ITS-90, although only for wavelengths above 600 nm due to the limited temperature range of this black body.

According to the 'Blevin – report' [8] one of the principle tasks of the BIPM is 'to undertake scientific research related to measurement units and standards, including appropriate fundamental research and the determination of physical constants'. A contribution of the BIPM to the problems identified above would be in line with this statement and would contribute to an active research field. At the same time it would allow the BIPM staff to gain experience in a new, but related, field.

Such a project would consist of the following work packages:

A) Development of filter radiometers operating near or below 400 nm and measurement of their spectral responsivity. The principal difficulties are to find sufficiently stable interference filters and to compare them against a trap detector with a very different spectral responsivity curve. The uncertainty of the effective wavelength can be estimated as 20 pm, that of the absolute responsivity as about 0.04 %. Time scale: Construction of filter radiometers: < 6 months, Calibration of filter radiometers: < 6 months

B) Development of a high temperature furnace for temperatures up to about 2800 °C. A suitable commercially available furnace has to be found and installed. An important aspect is the temperature uniformity. Time scale: 9 - 12 months.

C) Development of graphite crucibles with blackbody cavities for the fixed point material and development of a filling technique for the metal and carbon powder. The crucibles should be optimised for uniform temperature and for a long lifetime. The available experience shows that cells can survive more than 20 runs without breaking. The melting/freezing behaviour has to be studied and optimal operation conditions leading to the best reproducibility have to be found. Materials from different supplies should be compared. Time scale:12 months or longer, depending on the results

D) Radiometric measurements on the fixed points. An optical system has to be set up to measure the radiance temperature of the cells. This technique is in principle well known [7]. Time scale: 6-12 months.

Several of these activities are independent and could overlap with others. Results should be available after three years.

It is clear that this project could not be carried out within a reasonable time with the current staff of the radiometry and photometry section (2 physicists + 1 assistant).

A collaboration with a laboratory having already experience in the preparation of the eutectic fixed points in the form of a guest scientist project at the BIPM would allow to bring together experiences from both the thermometry and the radiometry side. A guest scientist without a solid background in thermometry could also be helpful since he could be trained by one of the expert laboratories. An installation at the BIPM would allow to diffuse largely the knowledge about this technique and to promote the possible application of this technique for an improved temperature scale.

Summary

The new metal-carbon eutectic fixed points seem to be very promising for future application in high temperature metrology. Propositions range from the replacement of tungsten ribbon lamps as transfer standards to the use as defining fixed points for a future temperature scale. Results available up to now indicate that the repeatability of the melting temperatures can be better than 70 mK. The melting and freezing curves are, however, less flat than is known from pure metals. Further studies are needed to understand and optimize the behaviour of the melting or freezing plateaux and to determine the thermodynamic temperatures.

The BIPM would be willing and able to contribute to this work building on its great experience in radiometry. The most efficient form of such a project could be the stay of a guest scientist at the BIPM.

References

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