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Thermal Effects in the HTBB-3200pg Furnace on Metal-Carbon Eutectic Point Implementation

M. Sadli · K. Anhalt · F. Bourson · S. Schiller · J. Hartmann

Abstract The general statement that a temperature fixed-point cell will show better melting and freezing plateaux with better temperature uniformity along the dimensions of the fixed point is understood to be valid for metal-carbon (M-C) eutectics as well as for pure metal fixed points. In this article, it is shown that improved temperature uniformity in the central part of the high-temperature blackbody BB3200pg (HTBB), where the M-C fixed point is implemented, results in flatter and longer plateaux. Pyrolitic graphite rings, clamped together by a spring, form the heated cavity of the HTBB. As a first step, the relative electrical resistivity of each pyrolitic graphite ring was measured using a method advised by the furnace manufacturer. Next, the ring positions were optimized, taking into account their relative resistivities, in order to obtain a more homogeneous temperature distribution. Subsequent measurement of the temperature uniformity at the furnace walls confirmed the improvement. Measuring the melting plateaux of the Pt-C eutectic with different arrangements of the rings, and thereby operating the fixed-point cell in different temperature distributions, confirmed the influence of the temperature distribution on the plateau shape, with the best plateau shape corresponding to the most homogeneous temperature distribution.

Keywords High temperatures · Metal-carbon eutectics · Temperature uniformity · Radiation thermometry

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1 Introduction

Metal(carbide)-carbon eutectic fixed points have been in use for several years in different National metrology institutes [1–3] and have proven to be serious candidates to become new fixed points [4–6] of a future temperature scale at the highest temperatures. To achieve this stature, certain capabilities must be demonstrated; for example, the long-term stability and robustness of the cells must be assessed and all parameters affecting the reproducibility and accuracy of the melt/freeze temperature measurement must be understood.

EUROMET project 864 represents a collaboration between PTB, NPL, and LNE-INM that aims to improve the thermodynamic temperature measurement of high-temperature blackbodies, especially of metal-carbon (M-C) eutectic points. PTB and LNE-INM both use a similar furnace, the Vega HTBB (BB3200PG). Originally designed to be used as a blackbody source for radiometry purposes [7,8], it can be adapted to metal (carbide)-carbon eutectic fixed points [9]. The PTB and the LNE-INM have jointly characterized and improved the temperature uniformity of their furnaces. The results of the investigations carried out in the two laboratories are presented here.

2 Measurement and Improvement of Temperature Uniformity

2.1 Methodology

One of the most important factors affecting the reproducibility of the melting temperatures of metal(carbide)-carbon eutectics is the temperature uniformity of the furnace, which can influence the temperature of the phase change as well as the interpretation of the plateau. Figure 1 shows the variation of the plateau shape and the shift in temperature observed when the same Re-C cell is operated in the same furnace—but in different positions, and therefore in different thermal conditions. The cell studied in this case is a Re-C cell 50 mm long, the usual dimension of the early cells developed at LNE-INM. The cell was positioned first in the center of the furnace and then pushed back by 8 cm, where the temperature uniformity was obviously poorer. The figure shows a very important difference in the plateau shape for the two cases. Of course, the length of the cell also has an influence—a longer cell is more likely to suffer from the temperature non-uniformity of the furnace—and this is why the cells developed recently at LNE-INM are now shorter, at 40 mm.

The measurement of the temperature distribution in the HTBB was done with a slightly tilted (5° with respect to the optical axis of the cavity) radiation thermometer viewing the heated cavity over about 20 cm within the center of the furnace (Fig. 2), where the temperature distribution should be the most uniform and where the cell is most likely to be positioned to benefit from the best temperature uniformity. This method has already been applied to the calculation of the emissivity of the HTBB cavity [10]. To scan the temperature distribution, the front lens of the radiation thermometer is equipped with an aperture of 10 mm diameter in order to reduce the risk of vignetting at the entrance of the furnace, thereby maximizing the scan width. The

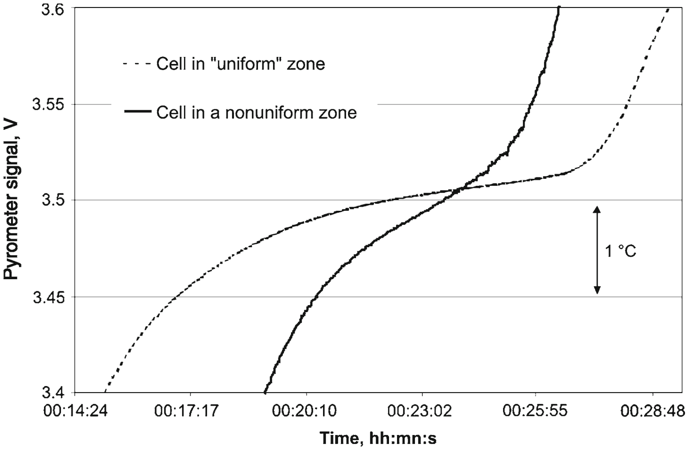

Fig. 1 Re-C melting plateaux for two different temperature distributions around the cell

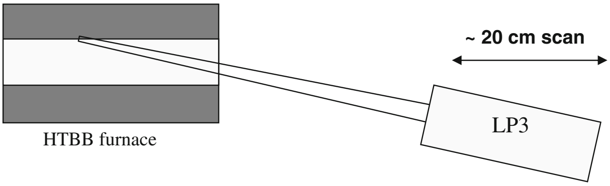

Fig. 2 Schematic view of the HTBB temperature distribution measurement

temperature distribution is deduced from the relative change in the measured photocurrent of the radiation thermometer's photodiode.

At the start of the measurements, the temperature distribution was measured for the three temperatures, 1,550 K, 2,000 K, and 3,040 K. Figure 3 summarizes the typical temperature distributions at these temperatures obtained initially with the HTBB. One can notice that the temperature uniformities become worse and the hottest part is pushed toward the rear of the furnace as the furnace temperature increases. This is caused by the heat lost from the front opening of the furnace, which cools the front part of the heated cavity.

2.2 Measurement of the Resistance of the Rings

Vega's HTBB furnace heating element is composed of a series of pyrolytic-graphite rings assembled to form a heating cylinder of about 350 mm length and 37 mm inner diameter. These rings are clamped together by a spring pushing from the rear of the furnace. The positioning of the individual rings can be organized in different configurations. By measuring the electrical resistivity of the rings and arranging them appropriately according to their resistivities (resistance per unit length), the temperature uniformity can be improved. In other words, putting the high-resistivity rings at both ends of the heater is likely to compensate somewhat for the heat losses and "flatten"

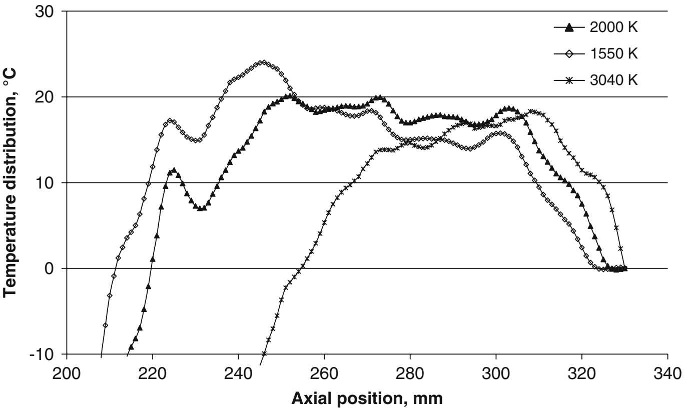

Fig. 3 Initial temperature distribution measurements at 1,550 K, 2,000 K, and 3,040 K in PTB's HTBB 3200pg

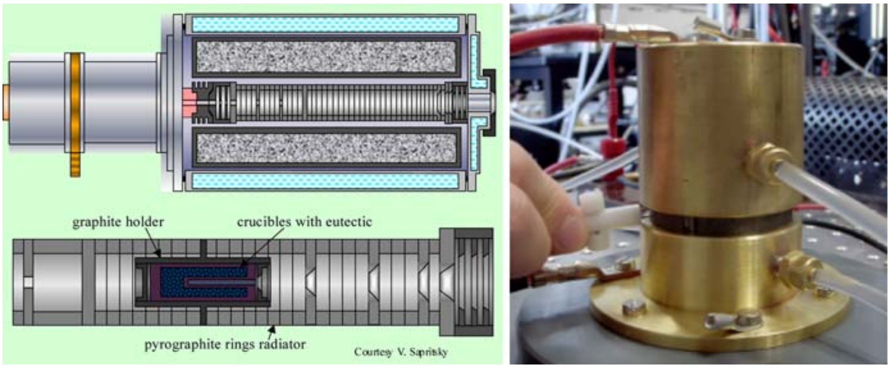

Fig. 4 Left: schematic view of the HTBB, detail of the cavity equipped with a cell holder containing a cell. Right: photograph of the apparatus used at PTB for the measurement of the resistivity of the rings

the temperature distribution in the central part of the cavity. A resistivity-determining apparatus was constructed following the advice of the furnace manufacturer [11].

Figure 4 shows a schematic of the HTBB in cross section and a photo of the ring resistance-measuring apparatus used at PTB. This apparatus consists of two massive water-cooled brass metal pieces, through which a current of about 10 A is applied perpendicular to the ring plane and the resistance is deduced from the voltage measured with a pair of pin electrodes applied along the side (width) of the ring. The critical point is to ensure good electrical contact between the ring and the brass electrodes. Of course, high-accuracy resistance measurement is not the aim of this work. Only the ranking of the rings in terms of resistivity and, consequently, thermal dissipation is important. Arranging the rings according to their resistivity can improve the temperature uniformity, i.e., by having the higher resistivity rings at the ends to compensate for the heat losses.

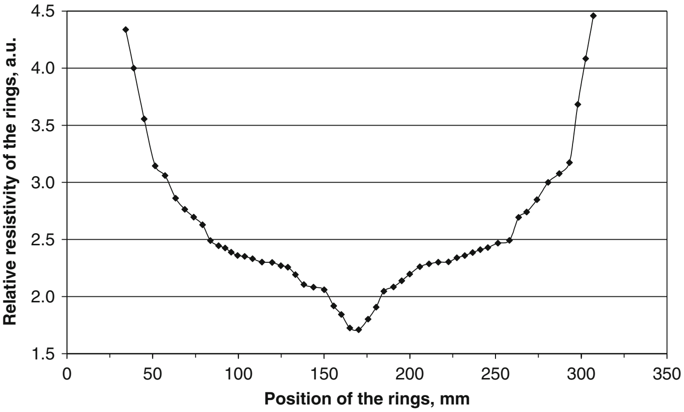

Fig. 5 Rearrangement of INM's HTBB rings with the high resistivity ones at the ends and the low resistivity ones in the center of the cavity. The position is with respect to the front flange plane

This ordering of rings has been done at LNE-INM for the HTBB3200pg and the arrangement of the rings according to their relative resistivity can be seen in Fig. 5. In Fig. 6, the corresponding temperature distributions obtained near the Co-C eutectic temperature and the Pt-C eutectic temperature are shown. As can be seen, over the 4 cm length of the cell, a temperature uniformity of 2 °C can be achieved. The difficulty is to maintain this temperature uniformity with the cell inside the furnace. It is quite probable that the temperature uniformity changes when the cell is positioned inside the furnace because its two faces will radiate to the outside of the furnace and lose heat. This effect will most probably degrade the temperature homogeneity. As can be seen in Figs. 3 and 7 referring to the measurements done on PTB's HTBB, the presence of a cavity wall facing the entrance of the furnace will always induce a temperature drop in the nearest region. Moreover, with the same arrangement of rings, the position of the most uniform region where the cell should be positioned is pushed toward the back of the furnace with increasing furnace temperature. This is most probably due to the fact that the heat losses from the entrance of the furnace are higher than the losses from the rear flange.

2.3 Temperature Uniformity in the Cell Holder

The M-C eutectic fixed-point cell is usually positioned inside a cell holder that is adapted to the outer diameter of the cell and is about 3 mm to 4 mm smaller in diameter than the heater (Fig. 4). One might expect that the cell holder itself would have an effect on the temperature distribution. This has been checked at two temperatures, 1,550 K and 2,000 K, and the results (Fig. 7) clearly show that this is not the case. The temperature is noticeably lower at the bottom of the cell holder (as it is lower at the bottom of the heated cavity). This is due to the heat lost by radiation to the (colder) outside of the furnace. It may be interesting to compensate this heat loss by putting high-resistivity rings at both ends of the cell holder [11].

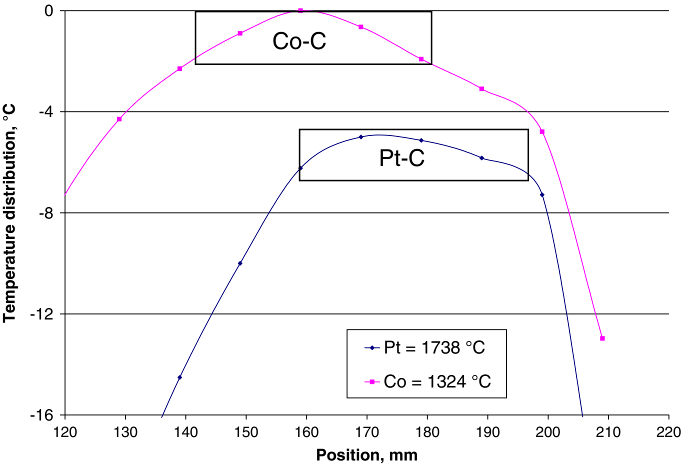

Fig. 6 INM's HTBB temperature distributions measured at the Co-C and Pt-C eutectic temperatures. The position is with respect to the front flange plane

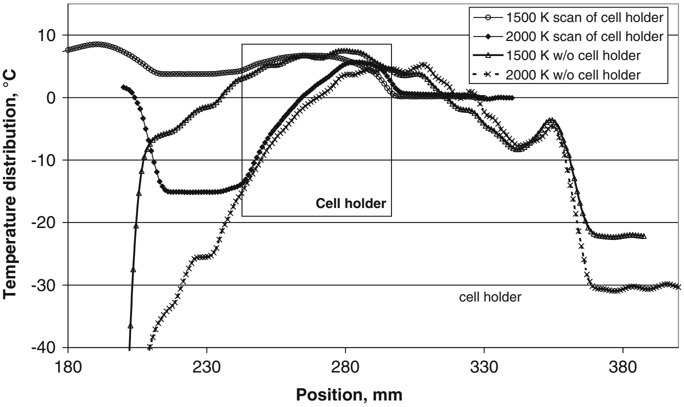

Fig. 7 Temperature distributions with and without the cell holder at 1,550 K and 2,000 K in PTB's HTBB 3200pg

2.4 Effects on the Shape of the Melting Plateau

The most important result of this work is that the temperature uniformity improvement can be very useful in obtaining better melting curves, i.e., flatter curves and more reproducible melting temperatures. In Fig. 8, the improvement of the plateau shape at the Pt-C eutectic point is obvious, especially in terms of the sharpness of the run-off, when the higher resistivity rings have been placed at the ends and the lower resistivity ones in the center of the furnace, near the cell, and when the cell is positioned exactly within the most uniform part of the furnace.

It is recommended that the temperature uniformity of the furnace be checked prior to installing the fixed-point cell and that the cell be positioned within the most uniform

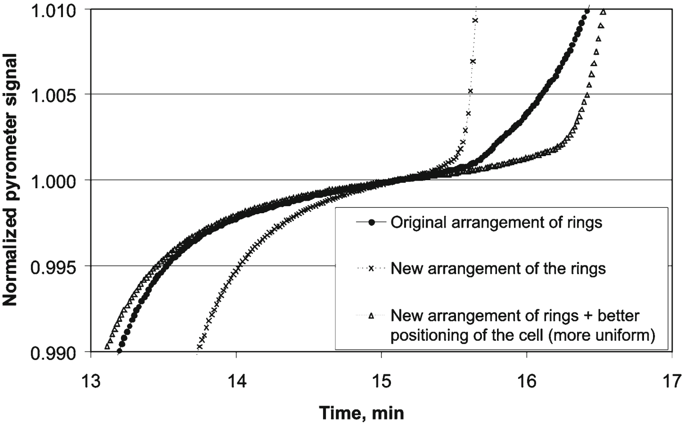

Fig. 8 Effect of temperature-uniformity improvement by optimizing the order of the rings, and the position of the Pt-C fixed point, on the plateau shape

part of the cavity. Only with these precautions can the plateau shape be improved. However, the potential for further improvement is quite limited for the BB 3200pg because the cavity diameter (37 mm) leaves little room for further insulation or thermal confinement of the cell, as has been proven to be helpful by the work on the large-diameter cavity of the BB 3500YY [11]. At this stage of the study, this does not limit the utility of the BB 3200pg for the implementation of M-C eutectic fixed points. Indeed, the repeatability of the melting curve is satisfactory and can be, for some cells, as good as a few millikelvin. The anticipated comparisons in this field will certainly give valuable input regarding the evaluation of the furnace effect [5], although it is a well-known fact that the temperature uniformity of the furnace can affect the plateau shape and the melting temperature.

3 Conclusion

Measuring the resistivity of the rings forming the heating cavity of the HTBB and ordering them appropriately is a suitable method to avoid large temperature gradients along the cell. Besides this, several important conclusions can be drawn from this work:

- plateau shape and repeatability are critically affected by the position of the cell inside the furnace and the temperature uniformity along the crucible,
- with no additional insulating elements, the graphite cell holder does not improve the temperature gradient,
- ensuring better temperature uniformity yields more repeatable plateaux (for instance, for a single Co-C cell, the repeatability was improved by a factor of three),
- the optimum position of the cell inside the furnace is a function of the temperature of the heated cavity,

- having a cavity bottom too close to the cell holder negatively affects the temperature uniformity and the plateau shape.

These characterizations and improvements to the temperature uniformity are a necessary prerequisite for obtaining better reproducibility of the fixed-point cell melting temperature. It is also important to improve the quality of the cell construction by using higher-purity materials and improved "clean" filling methods. Only after taking into account all of these factors will it be relevant to measure the thermodynamic temperature of the M-C eutectic fixed points. If independent thermodynamic temperature determinations are shown to be equivalent for several fixed-point realizations, then this will be the last step required for the CCT to include the M-C eutectic fixed points under the auspices of a new temperature scale or within the current international practice via an addendum to the "mise en pratique" for the definition of the kelvin.

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