High-Temperature Fixed-Point Blackbody with Temperature of about 2856 K Based on Melting Phase Transition of $\delta$(MoC)-C

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Large-area HTFP blackbody of $\delta$(MoC)-C type with temperature of about 2856 K has been developed. The $\delta$(MoC)-C HTFP has been investigated using several $\delta$(MoC)-C cells of different sizes. Thermodynamic temperature of the $\delta$(MoC)-C fixed point (POI) has been determined equals to 2856.93 K with standard uncertainty of 0.35 K, while the temperature of the large-area blackbody has been measured to be 2856.25 K.

INTRODUCTION

Creation of high-temperature fixed-points (HTFPs) and inclusion them in the Mise en pratique for the definition of the kelvin (MeP-K) have significantly contributed to the development of the source-based (namely, blackbody-based) radiometry/photometry by means of increasing accuracy and reliability of temperature measurements of high-temperature blackbodies (HTBB).

The radiometry/photometry can further benefit from the development of large-area HTFP cells and, thus, creation of high-temperature fixed-point blackbodies with relatively large apertures capable of operating in irradiance mode and realizing such quantities as spectral radiance or luminous intensity.

VNIIOFI traditionally has been dealing with development of large HTFP cells. The last research presented the development and investigation of Re-C and WC-C large cells [1] and announced the development of $\delta$(MoC)-C. The point $\delta$(MoC)-C, first suggested by Sasajima [2], with melting temperature of about 2856 K is particular interesting for photometry as a base of the photometric Type-A standard blackbody.

Recently at VNIIOFI the $\delta$(MoC)-C blackbody based on a large cell was created. The several $\delta$(MoC)-C cells, large and small (“normal”) size, were built and investigated. Thermodynamic temperature of the $\delta$(MoC)-C fixed point was determined. This paper presents design of the developed cells and the blackbody as well as the results of the investigations.

DESIGN

Design of new $\delta$(MoC)-C blackbody is similar to that described in [1] for Re-C and WC-C. Its cross-sections is shown in Fig.1. The blackbody is based on the largest VNIIOFI-developed high-temperature furnace BB3500MP [3] with the cavity diameter of 59 mm, which contains a large $\delta$(MoC)-C fixed-point cell. The cell has a blackbody cavity with the diameter and length of 14 mm and 45 mm, respectively.

Although the cell cavity is not deep, together with the furnace it has an emissivity of 0.9996. The dimensions of the cell and furnace allow using an outer aperture in front of the blackbody of about 5 mm, when calibrating a sensor of 5 mm at the distance of 500 mm. In this configuration the blackbody reproduces luminous intensity and illuminance of about 400 cd and 1600 lx.

To study the fixed point itself, tree small cells, shown in the bottom right of Fig.2, have been built and investigated: one with cavity diameter of 3 mm and two with cavity of 5 mm and opening of 3 mm.

The same materials with nominal purity of 99.999 % were used for all the cells, small and large: molybdenum powder from American elements and graphite R4550 from “SGL Group” (Germany).
MELTING PLATEAUS

The melting plateau shape of $\delta$(MoC)-C and repeatability were investigated using the temperature stabilized radiation thermometer of LP5 type. The plateau of the large-cell $\delta$(MoC)-C blackbody was also observed by a photometer head in “irradiance” mode. Fig.3 shows the typical melting/freezing plateaus of a small cell measured by LP5 and a melting plateau the large-cell blackbody recorded by the photometer. The cells showed high-quality melting plateau with melting range, varied for the small cells from 120 mK to 250 mK, which is less than that of the well-studied Re-C fixed point. The “best” cell, MoC-C7, demonstrated typical melting range of (120-150) mK that comparable with WC-C. The melting range of the large $\delta$(MoC)-C cell was a bit larger, 200 to 350 mK, which is explainable for the bigger ingot. The good shape of the melting plateau allows determining of the point of inflection (POI) with accuracy within $\pm$20 mK. Repeatability of melting temperature, determined as the standard deviation of POI, was 10 to 20 mK, which is typical for HTFPs.

![Figure 3. Left: melting/freezing plateaus of small cell. Right: melting plateau of large cell measured by photometer in irradiance mode.](image)

$\delta$(MoC)-C TEMPERATURE MEASUREMENT

Thermodynamic temperature of all tested $\delta$(MoC)-C cells has been measured as POI temperature by means of comparison with Re-C fixed point, whose thermodynamic temperature was determined as 2747.84 K with standard uncertainty of 0.18 K at the international complain [4]. The same Re-C cell was compared with all the $\delta$(MoC)-C cells. When comparing, a $\delta$(MoC)-C cell was placed in BB3500MP, while the Re-C cell – in another HTBB furnace. Both furnaces stood next to each other in front of the radiation thermometer LP5 and mounted on the translation stage. Relative spectral responsivity of LP5 was measured against a trap-detector using a 1-m double monochromator. To avoid possible drift, the cells were compared at the same day, wherein the Re-C cell was measured twice, before and after the $\delta$(MoC)-C cell.

The standard uncertainty of the $\delta$(MoC)-C thermodynamic temperature has been estimated to be 0.35 K. The major components have been related to the realisation of the Re-C fixed point.

The measured values are presented in Table 1. For the small cells the temperature varied from 2856.81 K to 2856.93 K with the average value of 2856.85 K. The cell with the smallest melting range showed the highest temperature. The large cell was measured as it was and with an additional aperture of 3 mm. Temperature shown with the aperture equalled to that average for small cells. Without any aperture the 14mm cell $\delta$(MoC)-C blackbody showed the temperature slammed by 0.6 K, similar to Re-C and WC-C large-area blackbodies [1].

Table 1. Measured thermodynamic temperatures of $\delta$(MoC)-C blackbodies.

<table>
<thead>
<tr>
<th>Cell ID</th>
<th>Cavity/opening diameter, mm</th>
<th>T, K</th>
</tr>
</thead>
<tbody>
<tr>
<td>MoC-C7</td>
<td>3/3</td>
<td>2856.93</td>
</tr>
<tr>
<td>MoC-C8</td>
<td>5/3</td>
<td>2856.84</td>
</tr>
<tr>
<td>MoC-C9</td>
<td>5/3</td>
<td>2856.81</td>
</tr>
<tr>
<td>MoC-C10D14</td>
<td>14/3</td>
<td>2856.85</td>
</tr>
<tr>
<td></td>
<td>14/14</td>
<td>2856.25</td>
</tr>
</tbody>
</table>

ROBUSTNESS

The cavity walls of all built cells cracked during the filling or in the early stage of use. Fortunately, no one cell has leaked. All the measurements described above were done with the cracks. Probably the thermal expansion of the graphite used does not match well to the Mo-C alloy. Therefore, proper type of graphite has to be tested searched and selected for the $\delta$(MoC)-C fixed point.

REFERENCES

1. B. Khlevny et al., Development of large-area high-temperature fixed-point blackbodies for photometry and radiometry, Metrologia, 55, S43-S41, 2018.
4. E. Woolliams et al., Thermodynamic temperature assignment to the point of inflection of the melting curve of high-temperature fixed points, Philosophical Transactions of the Royal Society A, p. 374: 20150044, 2015.