

Development of high- and medium-temperature fixed points for use in Radiometry and Thermometry

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Abstract

This paper describes the reason for, and progress in, development of high- and medium-temperature metal (carbide) - carbon eutectic fixed point cells for use in the Radiometry and Thermometry laboratories of NMISA.

High-temperature eutectics (around 2500 °C and 2600 °C) will improve the accuracy of temperature measurement of the variable-temperature blackbody to be used as the primary source for realisation of spectral units, by providing very stable sources for calibration and drift checks.

Medium-temperature eutectics above 1085 °C (around 1300 °C and 1500 °C) will improve the accuracy of thermocouple and radiation thermometer calibrations, by increasing the number of available temperature fixed points.

NMISA's capabilities will be extended and its uncertainties reduced in the existing calibration areas of radiation thermometry, spectral radiance and spectral irradiance.

1. Introduction

Fixed-point blackbodies based on pure metals are widely used in radiometry and radiation thermometry due to their extremely high reproducibility and uniformity of the radiating cavity, but are limited to a temperature of 1357.77 K (the freezing point of copper), whereas present-day radiometry, photometry and radiation thermometry measurements use blackbody sources working at temperatures up to 3500 K. Extrapolation to such high temperatures from the Cu point significantly increases the uncertainty. Hence, the investigation of new high temperature fixed points above the defining fixed points of ITS-90 is one of the most pressing areas.

Recent attention has been focused on fixed-point blackbodies based on metal-carbon eutectics [1, 2].

The first experiments with metal-carbon eutectic fixed points at NMISA were performed in 2006 to investigate the reproducibility of the Re-C (2747 K) and $\delta(\text{MoC})\text{-C}$ (2856 K) points [3]. The project was conducted in collaboration with VNIIOFI (Russia) [4]. One cell of Re-C and one of $\delta(\text{MoC})\text{-C}$ were investigated. Initial experiments showed that reproducibility of melting and freezing temperatures of the Re-C cell were 0.07 K, and 0.17 K respectively. For the $\delta(\text{MoC})\text{-C}$ cell those values were 0.08 K and 0.04 K [5].

There is a similar shortage of pure-metal fixed points for the calibration of noble-metal thermocouples between Cu (1084.62 °C) and Pd (1553.5 °C). In addition, the Pd melting point is usually realised using the wire-bridge method, which is less accurate than using a larger sample of Pd enclosed in a cell. For this reason, work has begun to develop larger Ni-C

(1329 °C) and Pd-C (1492 °C) [3] cells for use with both thermocouples and radiation thermometers.

2. Preparation of fixed point cells

Following the design of VNIIOFI [6], two types of graphite crucibles with different dimensions have been designed at NMISA (fig.1). Both types contain cylindrical blackbody cavities with conical bottoms. The smaller cell, “R”-type, has a cavity with inner diameter of 4 mm, outer diameter of 24 mm and cavity depth of 34 mm. The bigger one, “CT”-type, has a cavity of 9 mm diameter, 32 mm outer diameter and cavity depth of 89.5 mm. The “CT”-type crucible design can be used with contact thermometers as well as for irradiance applications.

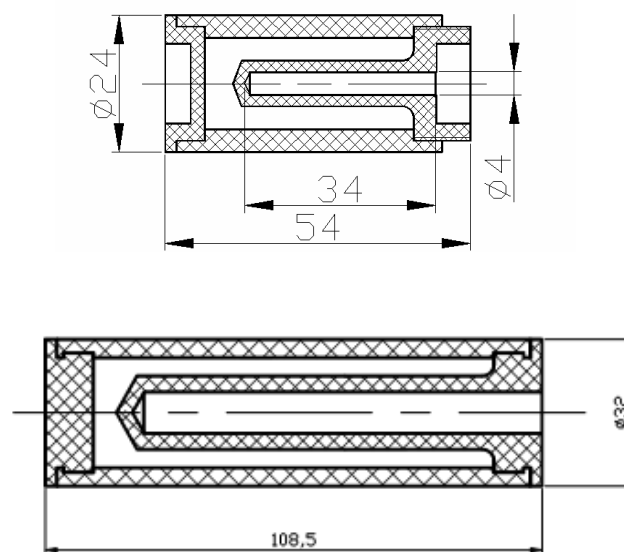


Figure 1. “R”-type (above) and “CT”-type (below) crucibles for eutectic fixed points.

Effective emissivities of the cavities were estimated to be 0.9999 and 0.9996 for “R”-type and “CT”-type cells respectively, assuming graphite emissivity of 0.85 and uniform temperature distribution [7].

“R”-type crucibles were designed to be used with high-temperature windowless blackbody furnace BB3200pg [8], designed at VNIIOFI. Cells are held between the pyrolytic graphite heating rings of the blackbody radiator (fig.2), using an additional holder. Although BB3200pg is normally operated horizontally, it was used vertically during the procedure of filling of crucibles with eutectic.

The “CT”-type crucibles (to be used for medium-temperature eutectics such as Ni-C and Pd-C) are intended to be used in a larger horizontal furnace with an alumina worktube. Such a cell has not yet been filled and evaluated.

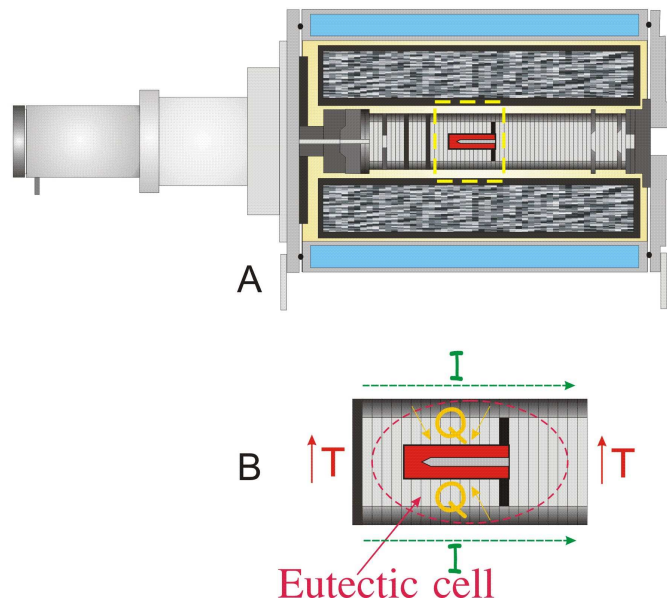


Figure 2. High-temperature blackbody BB3200pg with eutectic fixed point cell inside its radiating cavity.

Two types of eutectic fixed points have been made: Re-C and $\delta(\text{MoC})\text{-C}$. Pure carbon and metal powder were used for the preparation of the eutectic mixtures. The purities of the starting materials were 99.999 %. Metal and carbon powders were mixed and a crucible was filled with the mixture in an argon atmosphere. Then the crucible was placed vertically in the BB3200pg furnace and the mixture was melted to form the eutectic alloy. From 8 to 25 cycles of mixture adding and melting were necessary to fill the crucible completely.

3. Investigation of fixed points

Radiation thermometer LP-4 with a central wavelength of 650 nm was used for temperature measurements and reproducibility investigations [9]. Fig.3 shows typical melting and freezing curves of the Re-C cells.

To observe a phase transition plateau, the blackbody was originally heated to between 20 K and 25 K below the melting temperature of the eutectic. Once it had stabilised at this temperature, the current to the blackbody was increased and the eutectic melt was observed. After the melt, the eutectic was stabilised for five to 15 minutes at 20 K to 25 K above the melt temperature. Following this, the current to the main blackbody was decreased and the freeze plateau observed. Finally, the current was altered to hold the eutectic approximately 20 K to 25 K below the freeze temperature for at least five minutes before the next cycle was started. The transition rate could be controlled by varying the current step used to force the eutectic into melt and freeze.

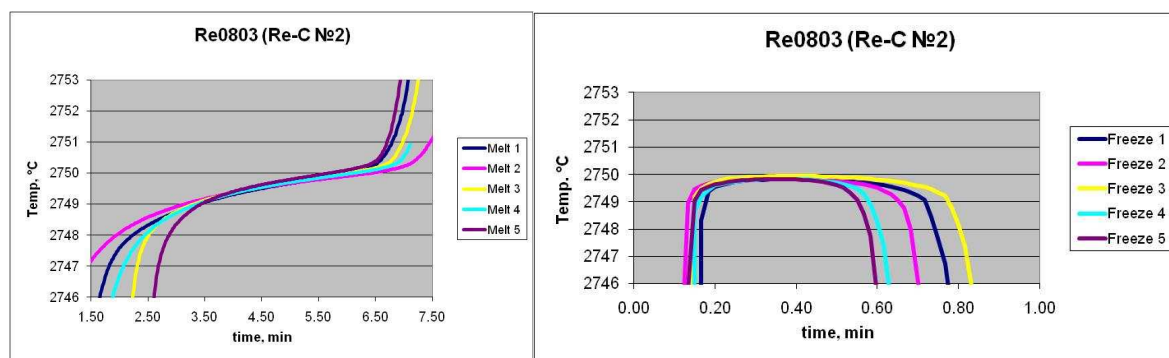


Figure 3. Typical melting(left) and freezing(right) curves of the Re-C cells.

Table 1 presents measurements of Re-C melting and freezing temperatures, showing intra- and inter-cell reproducibility.

Re0603			Re0803		
Cycle	Melt T, K	Freeze T, K	Cycle	Melt T, K	Freeze T, K
1	2749.72	2749.80	1	2749.77	2749.83
2	2749.76	2750.10	2	2749.91	2749.86
3	2749.81	2749.67	3	2749.91	2749.94
4	2749.76	2750.18	4	2749.95	2749.84
5	2749.87	2749.77	5	2749.96	2749.83
6	2749.79	2749.24			
mean	2749.79	2749.79	mean	2749.90	2749.86
std. dev	0.05	0.34	std. dev	0.08	0.05

4. Conclusion and future plans

Fixed point cells of Re-C and $\delta(\text{MoC})\text{-C}$ have been constructed at NMISA, and their temperatures measured and reproducibilities evaluated. (Results for $\delta(\text{MoC})\text{-C}$ are not presented in this paper: these cells crack easily and require a more robust crucible design and careful treatment.)

Observed inter-cell agreement is 0.1 K, while reproducibility of each cell (traditionally measured as a standard deviation) is approximately the same. Measurements of the phase transition temperatures on ITS-90 are currently handicapped by the 2 K calibration uncertainty of the radiation thermometer at these temperatures.

It is planned to construct larger cells of Ni-C and Pd-C, for use with both thermocouples and radiation thermometers.

The effect of temperature (non-)uniformity along the crucible on the plateau shapes of both high- and medium-temperature cells will be investigated.

The long term stability of the constructed cells will be further evaluated and compared to the characteristics of eutectics produced by other researchers.

5. References

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