A Comparison of Re-C, Pt-C, and Co-C fixed-point cells between NIST and NMIJ

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Abstract. A comparison of Re-C, Pt-C, and Co-C eutectic fixed-point cells was conducted between NIST and NMIJ at NIST. In the comparison, two high temperature furnaces, two radiation thermometers and one gold-point blackbody were used. Nagano furnace and an LP-3 radiation thermometer were transferred from NMIJ and were used in conjunction with a Thermogauge furnace and an AP-1 radiation thermometer of NIST to check the differences in measurement equipment. Agreement of Re-C and Co-C between NMIJ and NIST cells was better than 100 mK. The melting temperature of NIST Pt-C cell was 270 mK lower than that of NMIJ cell due to the poor purity of Pt powder.

Introduction

Above the freezing temperature of silver (1234.93 K), the International Temperature Scale of 1990 (ITS-90) is defined using Planck's law of blackbody radiation. The temperature is determined by ratio of its emitted radiance against the radiance from Ag, Au, or Cu freezing-point blackbody. Because the temperatures above these fixed points are extrapolated from these lower temperature blackbodies, the uncertainties increase dramatically with increasing temperatures. Recently, metal (carbide)-carbon (M(C)-C) eutectic blackbodies have been developed in several national laboratories to reduce the uncertainties of high temperature region of the ITS-90. Even if eutectic cells could be made and these cells showed good repeatability, their performance can only be confirmed by international comparison by use of high temperature furnaces with good temperature uniformity and stable radiation thermometers [1].

NIST has also started development of M(C)-C eutectic high-temperature fixed points. At NIST, absolute radiometry has already been used to determine the thermodynamic temperature of the freezing-temperatures of gold and silver. The NIST-determined values of the eutectic fixed-point temperatures would contribute for the discussion of the next ITS.

The NIST has constructed three eutectic cells for the first time: Re-C, Pt-C, and Co-C. To confirm the performance of these cells, a comparison was conducted between NIST and NMIJ at NIST. Nagano M furnace (VR10-A23) and an LP-3 radiation thermometer were transferred from NMIJ and were used in conjunction with a Thermogauge (TG) furnace and an AP-1 radiation thermometer of NIST to check the differences in measurement equipment. Gold-point blackbody of NIST was also used to check the short term and long term stability of radiation thermometers during the comparison. A Cu-point cell that can be placed into Nagano furnace was also measured by LP-3 to check the stability during comparison and before and after transportation from NMIJ.

This paper presents the preparation method of eutectic cells including information of metal sources and crucible materials, comparison results of three eutectic cells constructed by NIST and NMIJ, and stability of radiation thermometers.

Filling of eutectic cells

NIST: The crucibles containing M-C eutectics are same in size: outer diameter of 24 mm and a length of approximately 50 mm. The internal dimensions of the blackbody cavity are: aperture diameter: 4 mm; length: 33 mm; bottom shape: conical with an apex angle of 120°. Wall thickness of the cavity: 2 mm. The crucible with nominal purity of 99.9995% was machined and purified by Carbone of America.

The NIST M-C cells were filled as follows: rhenium or cobalt powder and graphite powder were mixed at approximately 1% hypo-eutectic composition. For the Pt-C cell, graphite powder was not added. Purities and suppliers of metals and graphite powder are summarized in Table 1. The crucible was filled with the mixture and placed in the BB3200cc furnace for vertical operation. It was then heated under argon atmosphere to just above the eutectic temperature. For a complete filling the filling process was repeated approximately 10 to 15 times.

Several Co-C cells were broken during filling at NIST. The possible origin is difference in density between liquid and solid of eutectic alloy and temperature nonuniformity of the furnace. To avoid freezing of the molten alloy from the surface of the crucible, which result in the cracking of crucible due to the expansion of the alloy during freezing, the furnace temperature was decreased quickly until the end of freezing. During the measurement of the Co-C, however, the NIST cell was again broken. The main reason might be the crucible material.

NMIJ: The dimensions of crucibles containing M-C eutectics were the same in size: outer diameter of 24 mm and a length of 45 mm. The internal dimensions of the blackbody cavity are: aperture diameter: 3 mm; length: 34 mm; bottom shape: conical with an apex angle of 120°. Wall thickness of the cavity: 2 mm. For Co-C and Pt-C, crucible with a nominal purity of 99.9995% was supplied by Toyo Tanso KK. For Re-C, the crucible was supplied by SGL carbon in order to prevent cracking of crucible due to difference of thermal expansion between metal and crucible material.

Nominal purities of powders and suppliers are described in Table 1. The filling procedures were almost the same as those of NIST procedures except for the furnace and filling atmosphere: The crucible was filled with the mixture and placed in the Nagano VR20-A10 vertical furnace. It was then heated under argon atmosphere for Re-C and under vacuum atmosphere for Co-C and Pt-C cells.

Table 1: Nominal purity of materials and their suppliers

	Graphite	Co	Pt	Re
NIST	99.9999 % Alfa Aesar	99.998 % Alfa Aesar	99.999 % Alfa Aesar	99.995 % Alfa Aesar
NMIJ	99.9999 % Alfa Aesar	99.999 % Nikko Materials	99.999 % Tanaka Kikinzoku	99.999 % Alfa Aesar

Apparatus

NIST TG furnace and NMIJ Nagano furnace were used for the comparison. The TG furnace has graphite furnace tube of 25.4 mm inner diameter. To improve the temperature uniformity, radiation shields, whose design was almost the same as those used in the Nagano furnace, were placed on front and back of the cell. The fixed-point cells were wrapped with graphite sheet to fit into the center of the furnace.

Nagano furnace has CC composite heater elements around a 27 mm inner-diameter furnace tube, made of the same material. The M-C cells were wrapped with graphite felt and put in the furnace. Radiation shields were used on front and back of the cells. Detailed description is in [2].

Two radiation thermometers of different design were used in the comparison to check the stability or other radiation-thermometers related effects: an AP-1 supplied by NIST and an LP-3 from NMIJ. The operating wavelength of both thermometers was 650 nm.

A daily measurement was done as follows: 1) melting plateau of Au-point blackbody was measured by AP-1 and its freezing plateau was measured by LP-3. 2) Melting and freezing plateau of M-C eutectic cell in TG furnace was measured by AP-1, and M-C cell in Nagano furnace was measured by LP-3 simultaneously. 3) The M-C cell in Nagano furnace was measured by AP-1. 4) M-C cell in TG furnace was measured by LP-3. 5) Au-point blackbody was measured by use of both radiation thermometers with the same procedure described in 1).

Results and discussion

Fig. 1 and 2 show typical melting curves of Pt-C and Re-C realized in Nagano furnace. Agreement of Re-C and Co-C between NMIJ and NIST cells was better than 100 mK. The melting temperature of NIST Pt-C cell was approximately 270 mK lower than that of NMIJ cell.

The melting temperature of Pt-C cell constructed by NPL using nominal purity of 99.999 % powder provided by Alfa Aesar has also showed 330 mK lower than NMIJ cell [3]. Even though nominal purity is the same, the real purity of Pt might worse than Pt provided by Tanaka Kikinzoku.

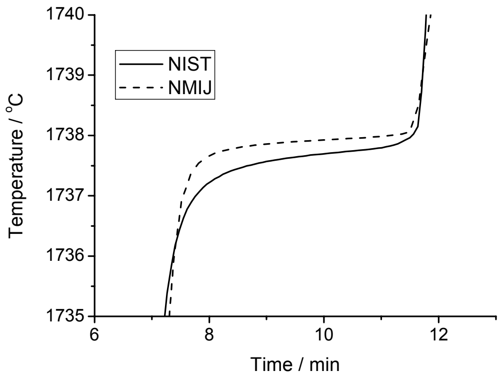

Figure 1. Typical melting curves of NIST and NMIJ Pt-C cells in Nagano furnace measured by LP-3. Time scale was adjusted to become the same plateau duration.

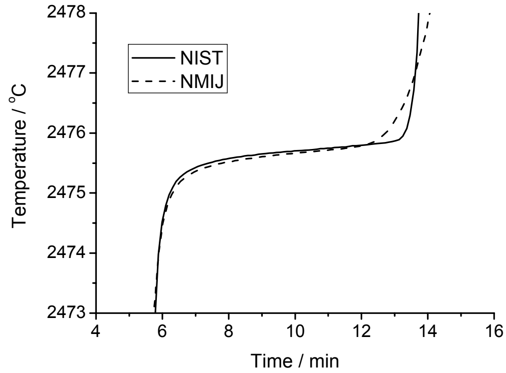

Figure 2. Typical melting curves of NIST and NMIJ Re-C cells in Nagano furnace measured by LP-3. Time scale was adjusted to become the same plateau duration.

Stability of thermometers during 2 weeks comparison by use of Au point was within +/- 50 mK: The freezing temperature of Au-point blackbody measured by LP-3 after eutectic cell measurement was always higher than the temperature before measurement in the same day. There is no apparent drift for both radiation thermometers.

These results show that high temperature M-C eutectic cell like Re-C cell can be used as stable radiance source; it is possible to make these cells at independent laboratories with exchange of information of material sources, filling procedures, crucible materials and so on.

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References

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