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Design and construction of a gallium fixed-point blackbody at CENAM

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For temperatures below silver fixed-point defined by the International Temperature Scale of 1990, a transfer radiation thermometer can be calibrated using either of two calibration schemes: a variable temperature blackbody with a standard platinum resistance thermometer as a reference, or with a set of fixed-point blackbodies. CENAM is presently working with the first scheme, and it is developing fixed-point blackbodies to have the capability to work with the second scheme too. For this purpose a gallium fixed-point blackbody to calibrate CENAM's transfer radiation thermometer was designed and constructed. The blackbody cavity has a cylindro-cone shape with effective emissivity equal to 0.9992 ± 0.0004 in the 8 μm to 14 μm wavelength range. The radiance temperature of the gallium fixed-point blackbody was estimated to have an expanded uncertainty of 54 mK, with a coverage factor $k = 2$.

Keywords: International temperature scale of 1990; fixed-point blackbody; radiation thermometry.

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1. Introduction

Radiation thermometers and thermal imagers are increasing their usage in the low temperature range (between 0 and 30°C) mainly for clinical laboratories and food industry applications. Due to their large field of view, most of those measurement instruments are difficult to calibrate directly with high-emissivity cavity radiators, so they are usually calibrated by comparison with a transfer radiation thermometer and a suitable radiating surface.

For temperatures below silver fixed-point (961.78°C) defined by the International Temperature Scale of 1990 (ITS-90) [1], a transfer radiation thermometer can be calibrated using either of two calibration schemes [2]: a variable temperature blackbody with a standard platinum resistance thermometer as a reference, or with a set of fixed-point blackbodies.

CENAM is presently working with the first scheme, and it is developing fixed-point blackbodies to have the capability to work with the second scheme too. For this purpose a gallium fixed-point blackbody to calibrate CENAM´s transfer radiation thermometer was designed and constructed. The melting point of gallium (29.7646°C) is a defining thermometric fixed-point of the ITS-90. Realization of this melting point is performed using high-purity gallium.

The objective of this work is to present a gallium fixed-point blackbody radiator made at CENAM for calibration of its transfer radiation thermometer. The uncertainty analysis of the blackbody radiance temperature and the results of a measurement with the transfer radiation thermometer are presented.

2. Gallium fixed-point blackbody

There is no fundamental restriction on the shape of a blackbody cavity [3]; the effective emissivity of the blackbody cavity mainly depends upon the emissivity of the walls, and the ratio of the area of the aperture to the total internal surface area of the cavity. For ease of fabrication, it was decided to make a cylindro-cone shape cavity. The cavity dimensions are 20.0 ± 0.1 mm inner diameter, 123.7 ± 0.1 mm length, and 120° cone angle. Fig. 1 shows a cross-section of the blackbody cavity.

The cavity is part of a crucible that contains the pure metal. Figure 2 shows the parts that form the crucible: (a) front cap, (b) blackbody cavity, (c) container and (d) back cap. All the parts were machined from virgin polytetrafluoroethylene (PTFE), and a 2.0 mm thick wall was used in the parts to provide the structural integrity to the crucible during the 3.1% expansion on solidification of the gallium.

Prior to filling the PTFE crucible, the crucible was cleaned to remove any contaminants from the machining process. The cleaning process was (1) clean in hot, soapy water;

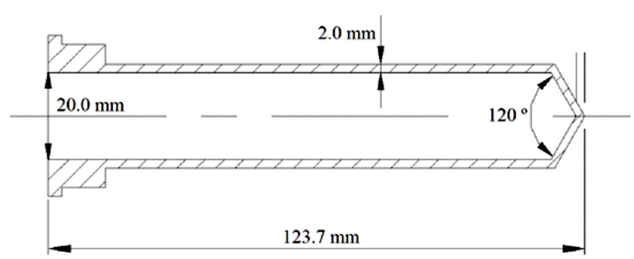

FIGURE 1. Cross-section of the blackbody cavity.

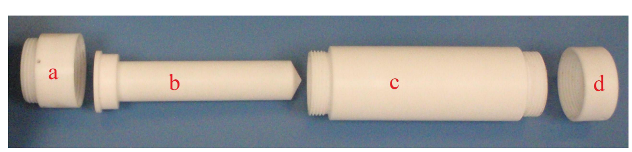

FIGURE 2. Crucible parts: (a) front cap, (b) blackbody cavity, (c) container and (d) back cap.

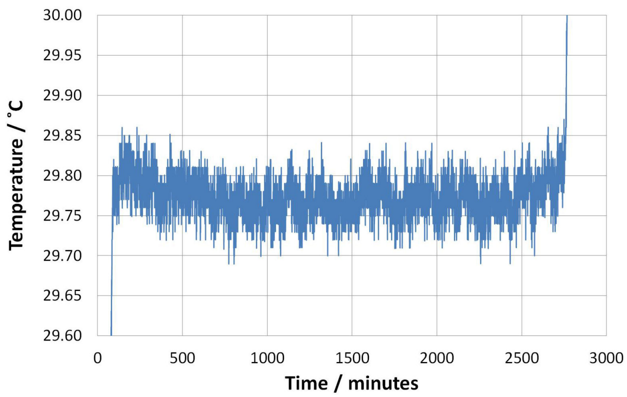

FIGURE 3. Measurement of the phase transition (fusion) of gallium with CENAM's transfer radiation thermometer.

(2) rinse with water; (3) soak in acetone for 20 hours; (4) rinse with distilled water, and then (5) air dried.

After all the parts were cleaned, they were placed in an argon-filled container. Dried argon gas was used to overpressure the container prior to melting the gallium and assembling the parts.

Approximately 316 g of high-purity (99.9999% wt. %pure) gallium metal from Alfa-Aesar was used to fill the crucible.

From the same PTFE bar where the pieces that form the cavity were cut, some samples (discs 50 mm in diameter) were manufactured to measure their emissivity at CENAM with the system described in [4,5]. With the emissivity measurement result, 0.850 ± 0.025 in the 8 μm to 14 μm wavelength band, an effective emissivity of 0.9992 ± 0.0004 was estimated for the blackbody cavity using a numerical Monte-Carlo method with STEEP3 software [6].

The liquid-bath furnace, where the fixed-point blackbody is inserted, can be set to temperatures in the range from -20°C to 40°C. The temperature uniformity of the furnace core is 0.02°C at 30°C. The bath fluid is a water/glycol, 1 to 1, mixture.

3. Fixed-point radiance temperature uncertainty analysis

For the uncertainty analysis of the blackbody radiance temperature, in the 8 μm to 14 μm working wavelength range, at the gallium fixed-point temperature, the influence variables described below were taken into account.

3.1. Impurities

The information on the impurities contained in the gallium was provided by the manufacturer (Alfa-Aesar) [7]. To estimate the uncertainty in temperature due to impurities, references [8,9] were followed. According to those references, the impurity uncertainty component value is calculated with Eq. 1 using the total mole fraction impurity concentration contained in the gallium sample and its first cryoscopic constant.

$$\Delta T = T_0 - T = \frac{x_2}{A}. \tag{1}$$

Where T_0 is the phase-transition temperature of a 100% pure sample, T is the observed realization temperature, x_2 is the mole-fraction impurity concentration, and A is the first cryoscopic constant equal to 0.00732 for gallium. From the impurity analysis for the gallium sample, the estimated impurity effect on the realized temperature of the gallium blackbody was estimated as 0.028 mK.

3.2. Plateau Identification

The fixed-point temperature can be estimated as the average between 25% and 75% fraction of the fusion plateau [2]. Due to the purity of the gallium used, the estimated uncertainty due to plateau identification was estimated as 10 mK.

3.3. Emissivity

The contribution of the effective emissivity was estimated with the experimental results of the surface emissivity of the PTFE discs assumed to represent the intrinsic emissivity of the cavity walls, and the geometrical data of the cavity. The maximum equivalent uncertainty contribution in the radiance temperature was estimated as 23 mK at a cavity temperature of 30°C.

3.4. Reflected ambient radiation

When viewing the blackbody, a small part of the radiation thermometer's signal arises from ambient radiation that enters the blackbody cavity's aperture, and subsequently emerges after multiple reflections. A contribution below 10 mK was estimated.

3.5. Heat loss

Two sources of heat loss of the cavity to the environment were considered: radiative heat loss and convective heat loss. For both, an effect of less than 1 mK was estimated on the

TABLE I. Uncertainty budget of radiation temperature gallium fixed-point blackbody.

Influence variable	Uncertainty / mK
Impurities	0.03
Plateau identification	10
Emissivity	23
Reflected ambient radiation	10
Heat loss	1.0
Combined uncertainty	27
Expanded uncertainty ($k = 2$)	54

basis that (i) radiative heat loss is very low near room temperatures [2], and (ii) no influence of convection cooling on cavity temperature was observed when covering the opening with a lid of synthetic rubber of low thermal conductivity and then opening to allow air convection to occur, so there was no significant effect of convection heat loss.

Table I shows all the contributions described above expressed as standard uncertainties in temperature. With all the contributions mentioned, the expanded uncertainty ($k = 2$) of the fixed-point blackbody radiance temperature was estimated to be 54 mK [10].

4. Measurement with CENAM's reference radiation thermometer

CENAM´s reference radiation thermometer has a working wavelength range of 8 to 14 μm in the -50°C to 300°C temperature interval. The size of source effect function of the radiation thermometer is experimentally known. It is estimated that 99.9% of the radiation energy received by the radiation thermometer comes from a circular area, 20 mm in diameter, at 385 mm distance from the thermometer.

The experiment started with the gallium at room temperature and the furnace was set to 31.0°C. It took approximately 84 minutes to start the solid to liquid phase transition. The fusion plateau lasted a little more than 43 hours. Laboratory conditions during measurements: 23.0 ± 0.5°C and 45 ± 5 RH. Figure 3 shows the results of the measurement of the phase transition (fusion) of gallium with the transfer radiation thermometer. The average temperature and standard deviation values calculated from the data were 29.77 ± 0.04°C. The measurements have been repeated several times during a two-month period with equivalent results confirming that the constructed gallium fixed-point blackbody is suitable for verification and calibration purposes.

5. Conclusions

A gallium fixe-point blackbody made at CENAM was presented. The blackbody cavity has a cylindro-cone shape with effective emissivity equal to 0.9992 ± 0.0004 in the 8 μm to 14 μm wavelength range.

Overall, the radiance temperature of the gallium fixed-point blackbody was estimated to have an expanded uncertainty of 54 mK, with a coverage factor $k = 2$, and it is traceable to the ITS-90.

Measurement results with CENAM's transfer radiation thermometer show that the fixed-point blackbody is suitable for verification and calibration purposes.

note

Reference to commercial product is provided for identification purposes only and constitute neither endorsement nor representation that the item identified is the best available for the stated purpose.

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