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Triple-Fixed-Point Blackbody for the Calibration of Radiation Thermometers

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Abstract A new triple-fixed-point blackbody containing the fixed-point materials aluminum (freezing point 660.323°C), zinc (FP 419.527°C), and tin (FP 231.928°C) in one device has been developed at the Ilmenau University of Technology. It enables calibration of a radiation thermometer with direct reference to the ITS-90 at three fixed points after a single adjustment of the calibration object. The setup significantly reduces the technical effort and the time for the calibration procedure. Measurements of the phase-transition temperature and the time-dependent blackbody temperature made with a transfer radiation thermometer, the Linearpyrometer LP5 of the IKE Stuttgart, are presented in the article.

Keywords Aluminum · Calibration · Phase transformation · Radiation thermometry · Tin · Triple-fixed-point blackbody · zinc

1 Introduction

In metrological institutes and calibration laboratories, radiation thermometers are often calibrated using variable-temperatures blackbodies. In this secondary calibration method, the radiance of the blackbody is adjusted and its temperature determined with a reference thermometer of some sort that must be calibrated periodically to maintain traceability to the ITS-90 [1].

The calibration of radiation thermometers can directly reference the International Temperature Scale by using blackbodies at the defining fixed points, such as aluminum or silver [2], for example. At least three different fixed-point blackbodies are necessary to calibrate radiation thermometers over a broad temperature range to take into account

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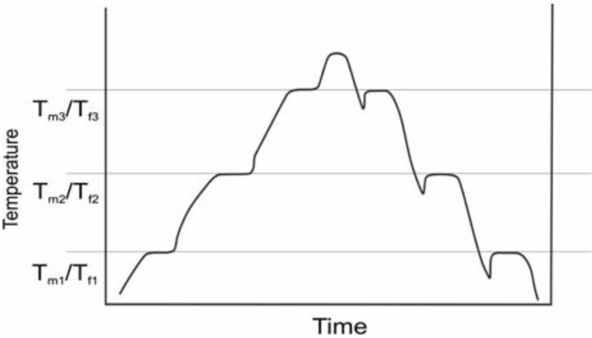

Fig. 1 Schematic diagram of the blackbody temperature/thermometer output signal during a triple-fixed-point calibration cycle

the nonlinearity of the thermometer characteristic. So, three fixed-point setups are usually needed, including a controller, furnace, and heat pipe for each fixed-point cell. Therefore, three fixed-point materials were integrated into a single blackbody to reduce the technical effort and time required for the calibration. This triple-fixed-point blackbody provides three calibration signals (S_{m1}–S_{m3}) during the melting and also three signals (S_{f1}–S_{f3}) during the freezing processes within a single calibration procedure, consisting of the adjustment of the calibration object and one heating cycle. A typical curve of the radiation thermometer's output signal during the calibration is shown schematically in Fig. 1.

1.1 Design of the Triple-Fixed-Point Cells

The shape of the temperature plateau is strongly affected by the design of the cell and the properties of the materials that are employed. The material of the crucible especially has to meet high demands; it needs a high thermal conductivity to ensure low temperature gradients in the inner part of the blackbody cavity and a high mechanical strength to resist the strains during heating, cooling, and the phase transformations of the filling material. Furthermore, the crucible has to be very pure and non-reactive with respect to the fixed-point materials to avoid introducing impurities that may influence the phase-transition temperature. These properties must be maintained at high temperatures as well. Therefore, the triple-fixed-point cell is made of highly pure alumina (Al_2O_3 99.7%) [3] to ensure that the required properties are maintained over a broad temperature range and in a form that is relatively easy to handle.

The design and dimensions of the fixed-point cell were evaluated by means of several thermal calculations using the finite-element method (FEM). The aim was to optimize the cell to achieve three clear, reproducible, and easily analyzed temperature plateaux in the blackbody cavity. Ideally, each of them should have low and similar uncertainties. In this calculation, we considered that the properties of the materials change with the temperature and during the transition between the solid and the liquid state.

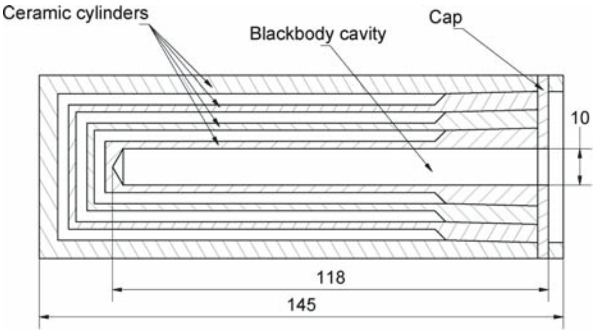

Fig. 2 Cross-sectional view of the triple-fixed-point body (dimensions in mm)

During the calculations, it became clear that only a coaxial arrangement of the chambers containing the fixed-point materials would provide the required attributes. Hence, all versions of the triple-fixed-point cells were constructed according to this principle. The latest model of the triple-fixed-point cell is shown in Fig. 2. It consists of four cylinders of alumina that form three coaxial chambers with volumes from 15 to 38 cm^3 where the fixed-point materials are contained. The conical pieces in front of the cell center the cylinders and prevent leakage of the molten fixed-point materials. A pasted cap additionally locks the fixed-point cell. The cap also forms the aperture of the blackbody cavity with a diameter of 10 mm. The bottom of the cavity is conical with an angle of 60°, and its length is 118 mm. The cavity was painted with HE23, a high emissivity paint produced by Rolls Royce for high temperatures, because of the strongly varying spectral emissivity of alumina.

1.2 Fixed-Point Materials

In general, this triple-fixed-point blackbody design can be used from room temperature to high temperatures, e.g., the solidification point of gold. However, the dimensions of the cell have to be adapted to the thermal and mechanical properties of the selected fixed-point materials. In the present design (Fig. 2), the focus is on the temperature range from 230 to 660°C, which is important for the calibration of radiation thermometers with InGaAs detectors, for example. This range is ideal to validate the functional principle of the triple-fixed-point blackbody because difficulties in filling the chambers and producing the cell, which occur at higher temperatures, can be avoided. Table 1 lists a variety of possible pure fixed-point materials with their melting points in the considered temperature range.

Tin, zinc, and aluminum suit our purpose best. They have a high heat of fusion, good thermal conductivity, and their melting points are equally spaced. Therefore, they were used as fixed-point materials in the triple-fixed-point blackbody presented here. Lead was used as a fixed-point material in an earlier design of the triple-fixed-point blackbody [5], but fast phase transformations with only short temperature plateaux occurred during the measurements at this fixed point due to lead's low latent heat of fusion, and that is why lead is no longer applied. In addition, the elements antimony,

Table 1 Material properties of possible pure fixed-point materials with their melting points in the temperature range from 200 to 660°C [4]

Fixed-point material	Melting temperature[a] (°C)	Heat of fusion ($J \cdot g^{-1}$)	Thermal conductivity[b] ($W \cdot m^{-1} \cdot K^{-1}$)	Specific heat capacity[c] ($J \cdot kg^{-1} \cdot K^{-1}$)
Sn	231.928	59.6	67	243
Bi	271.402	52	7.9	127
Cd	321.069	57	97	241
Pb	327.462	23.2	35	134
Zn	419.527	111	116	402
Sb	630.628	163	24	213
Al	660.323	388	237	951

[a] Temperature of the melting point of pure materials at normal pressure [1]
[b] Thermal conductivity at room temperature (25°C)
[c] Specific heat capacity at 125°C

Table 2 Arrangement of the fixed-point materials in the assembled blackbodies

	Inner chamber ($15\,cm^3$)	Middle chamber ($26\,cm^3$)	Outer chamber ($38\,cm^3$)
Variant A	Sn	Zn	Al
Variant B	Al	Zn	Sn

cadmium, and bismuth were not seriously considered because of their toxicity or low thermal conductivity.

1.3 Assembled Blackbodies

The three selected materials could be filled from the inner to the outer chamber in six different orders. In practice, the filling process limits the number of variations. In order to assure reproducible fillings with minimal impurities, the materials should be filled in descending order of their melting points. From this, it follows that there are only two variants of the realized triple-fixed-point cell (Table 2). The purity of the materials used in these blackbodies is 4N (99.99%).

2 Measurements

2.1 Measurement Setup

The main part of the measurement setup is a tubular furnace (1) with a maximum operating temperature of 1,300°C and containing a sodium heat pipe (2) as shown in Fig. 3. The triple-fixed-point blackbody (3) is placed in the heat pipe to gain better thermal conductivity and to achieve a higher homogeneity of the temperature field in the blackbody. The heat pipe is fixed in the furnace by a rack of thermally stable high-grade steel (4). The special construction of this rack minimizes the displacement of the blackbody aperture due to thermal expansion during the heating cycles. In front of the blackbody are several layers of ceramic fiber plugs (5) to minimize the heat

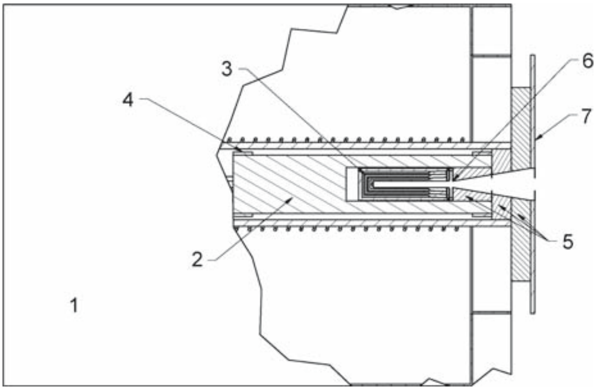

Fig. 3 Measurement setup with tube furnace, heat pipe, and triple-fixed-point blackbody

loss at the furnace front. Three baffles are inserted between the insulation layers to reduce the heat loss by radiation. The inner baffle (6) with a diameter of 10 mm is mounted directly at the front of the blackbody as an aperture stop. The front baffle is part of a cooled front panel (7), which blocks stray radiation from the furnace and the surroundings. An inert gas inlet for argon with a purity ≥99.999% and a positioning stage for the radiation thermometer complete the setup.

The radiation thermometer that we used was a Linearpyrometer LP5, manufactured by the Institut für Kernenergetik und Energiesysteme, IKE, of Stuttgart University. It is a further development of the transfer radiation thermometer LP4 [6,7], equipped with an achromat F/143 and InGaAs detector. The nominal wavelength of the interference filter is 1,550 nm (30 nm bandwidth), and the resolution of the radiation thermometer is 1 mK.

2.2 Measurement Procedures

The LP5 was used to detect the time-varying blackbody temperatures during the phase transformations. At each fixed point, a similar heating procedure was used to gain reproducible and comparable phase transitions. The furnace was heated to a stable temperature T_L about 5 K below the melting point of the fixed-point material. Melting was induced by raising the furnace temperature at a rate of $1\,K \cdot min^{-1}$ to a temperature T_H, 5 K above the melting point. After melting was completed, the furnace temperature was lowered again at a rate of $-1\,K \cdot min^{-1}$ to a temperature about 5 K below the freezing point. This temperature was maintained until freezing was completed. This procedure was repeated at least 10 times at each of the three fixed points. Both variants of the triple-fixed-point blackbody configurations of Table 2 were tested.

It is remarkable that, during the phase transformation of aluminum, the two other fixed-point materials are overheated by 250 and 440 K above their melting points.

2.3 Plateau Forms

The main idea of the development was the achievement of precise and analyzable plateaux in the blackbody temperature during the three phase transformations. The measurements of the blackbody variant A in Figs. 4–6 indicate that the goal was reached.

In variant A, the aluminum filled the outer chamber at the thermally worst position. Between the fixed-point material and the blackbody cavity's wall are several layers of ceramic and metals that hinder the formation of the measured plateau. Nevertheless, a well formed melting and freezing plateau developed because of the optimal dimensioning of the crucible and the appropriately calculated quantity of fixed-point material (Fig. 4).

The mean length of the melting plateau is about 2,000 s. It depends on the heating cycle and can be varied by changing the temperature T_H. The mean slope of the melting plateau is 3 mK$\cdot$ min^{-1}. Furthermore, the freezing plateau lasts for 2,000 s and shows the typical supercooling.

The plateaux of the zinc fixed point are also distinctly formed and are of a similar duration. The freezing plateau does not show any supercooling for zinc (Fig. 5). Although the melting point of tin is outside the working range of the sodium heat pipe, the plateaux develop very clearly. The reason for this result is the favorable position of the material near the cavity. The unbalanced heat insertion from the outside is thereby compensated. A disadvantage, however, is the large and variable supercooling of the tin freeze that reduces the plateau length to a few hundred seconds. The depth of the supercool varies from 6 to 9 K. This prevents optimization of the plateau length by

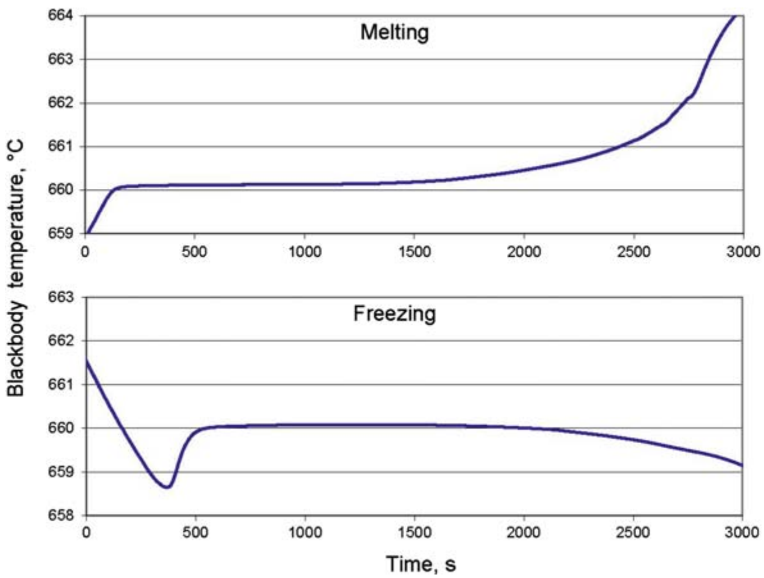

Fig. 4 Phase transformation plateaux at the aluminum fixed point (variant A)

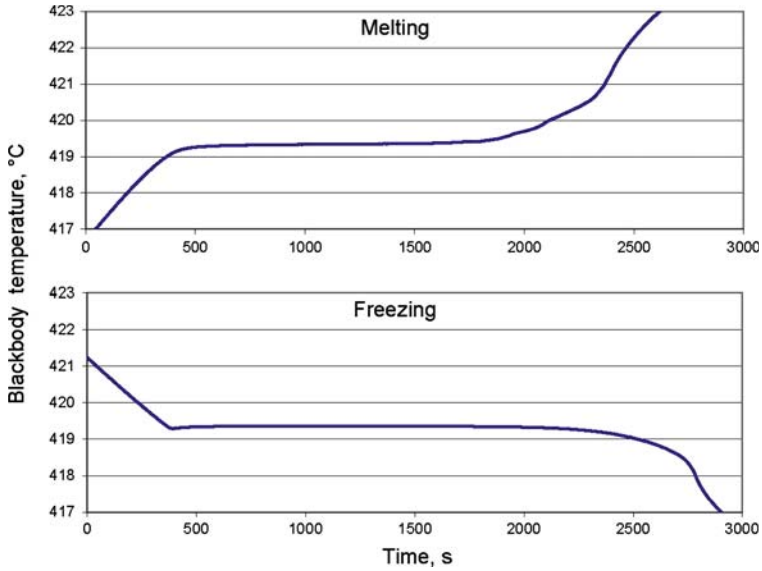

Fig. 5 Phase transformation plateaux at the zinc fixed point (variant A)

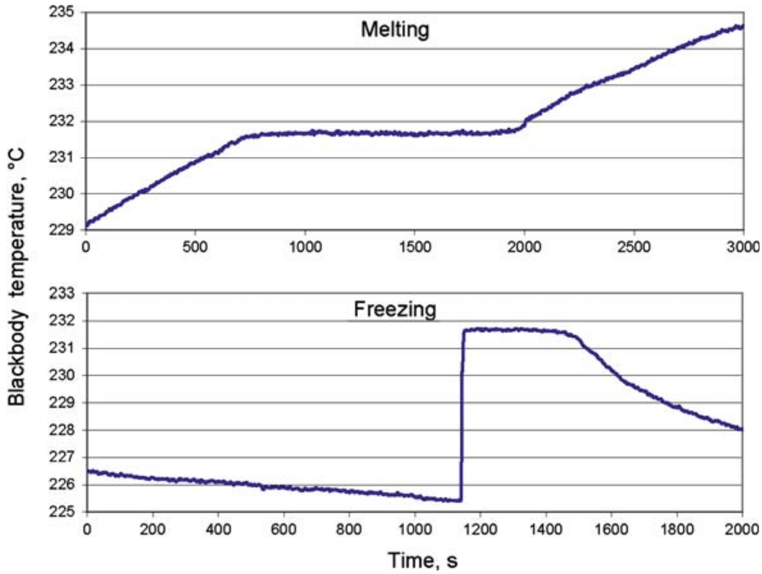

Fig. 6 Phase transformation plateaux at the tin fixed point (variant A)

changing the furnace set-point parameters. Nevertheless, the maximum temperature of the freezing plateau is reproducible (Fig. 6).

Variant B of the blackbody configuration did not show such good results. Although the aluminum and zinc fixed points were developed in a similar manner, the temperature of the tin fixed point was too low (Fig. 7). On one hand, this might be caused by the inhomogeneous heat introduced during melting and could be improved by the use of

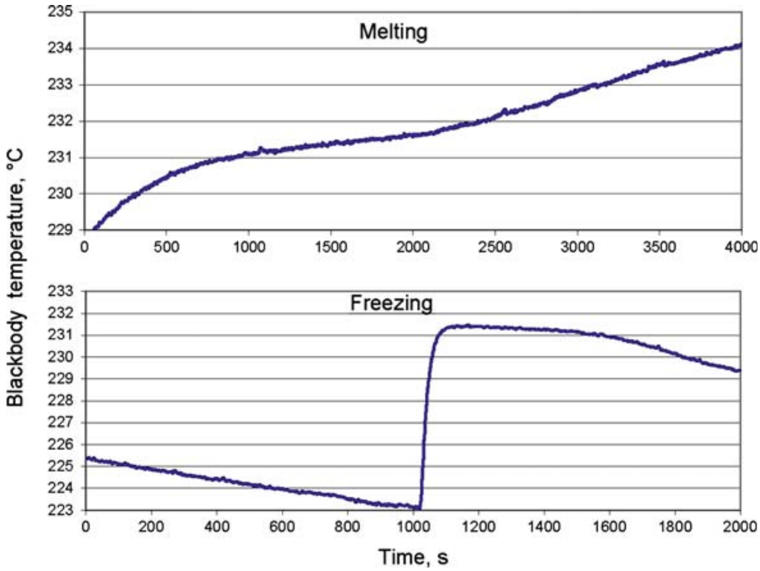

Fig. 7 Phase transformation plateaux at the tin fixed point (variant B)

a different heat pipe. On the other hand, the heat of fusion released during freezing is insufficient to heat the whole cell and the cavity to the freezing temperature. The noticeably negative slope of the plateau is evidence of this.

According to these results, variant A, with aluminum in the outer chamber and tin in the inner chamber, is better than variant B and was used for further measurements.

2.4 Analysis

At each of the three fixed points of variant A, at least 13 phase transitions were measured. The estimated phase transformation temperatures shown in Fig. 8 were calculated from the measured values. The inflection point of the melting plateau (minimum of the second derivative) was used as the best estimate of the melting point. The maximum of the freezing plateau was the estimate of the freezing temperature. As one can see, very stable fixed points occurred that were only influenced by stochastic effects.

It is obvious that the utility of the triple-fixed-point blackbody strongly depends on the repeatability of these fixed points. Therefore, the standard deviation of each series of measurements made under repeatable conditions was calculated. It includes the repeatability of the fixed point, the evaluation of the fixed-point temperature, and the repeatability of the pyrometric measurement itself. Table 3 gives an overview of the results obtained from measurements with the LP5.

As can be seen, the repeatabilities (standard deviation of the realizations) are very low. They increase at the tin fixed point because of the InGaAs detector's low and noisier signal. The radiant temperature of the fixed points depends on factors such as the effective spectral blackbody emissivity, temperature gradients in the cavity,

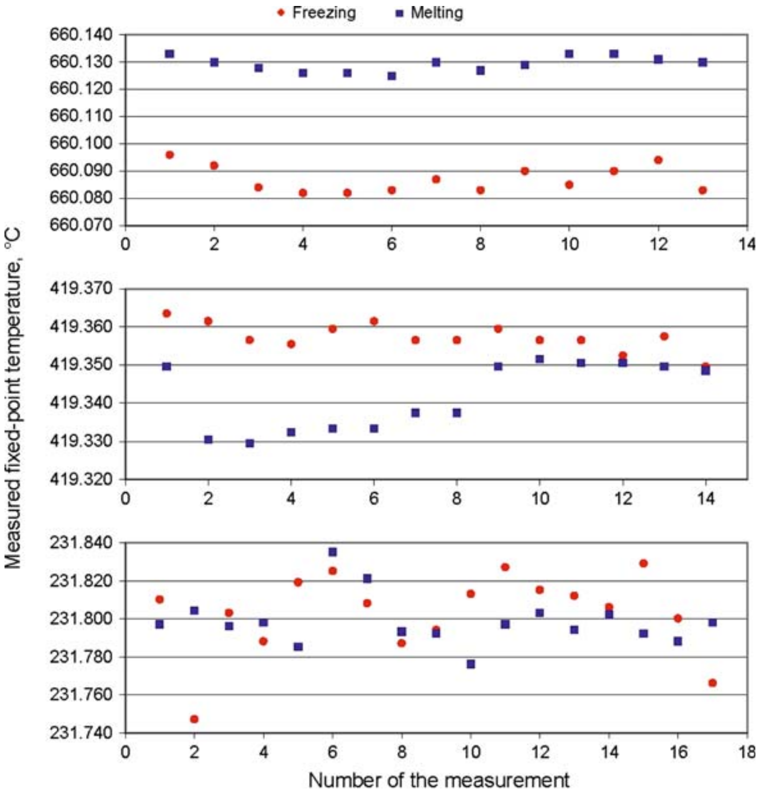

Fig. 8 Estimated fixed-point temperatures at the three fixed points (variant A)

Table 3 Mean temperatures and repeatabilities ($k = 2$) of the measured fixed points of variant A

	Al Mean fixed-point temperature (°C)	Repeatability (mK)	Zn Mean fixed-point temperature (°C)	Repeatability (mK)	Sn Mean fixed-point temperature (°C)	Repeatability (mK)
Melting	660.129	6	419.342	18	231.799	26
Freezing	660.088	10	419.357	8	231.803	44

impurities of the fixed-point materials, the calibration of the radiation thermometer, and several other contributions [7]. Correction of these effects was not attempted here. The assessment of these systematic errors is not yet complete for the three fixed points; however, our initial estimates are in the range of 100–150 mK, depending on the fixed point. This should lead to a residual deviation between the corrected temperatures and the ITS-90 values of some tens of millikelvin.

We expect to reach an uncertainty ($k = 2$) in the fixed-point temperature of about 100 mK. Hence, the deviation from the ITS-90 values should be within the uncertainty of the realized fixed-point temperature.

3 Summary

A triple-fixed-point blackbody made of alumina ceramic was designed on the basis of thermal calculations using the finite-element method. It includes three coaxially arranged chambers that were filled with the fixed-point materials, tin, zinc, and aluminium. Two variants of this design were assembled, and thermally and mechanically tested. The time-dependent blackbody temperature during periodic heating and cooling of the triple-fixed-point cells was measured with a Linearpyrometer LP5 of the IKE Stuttgart. Readily analyzable melting and freezing plateaux developed at the fixed points when tin filled the inner, zinc the middle, and aluminum the outer chamber. The slopes of the measured plateaux were in the range of some millikelvin. The repeatabilities of the estimated fixed-point temperatures range from 6 to 44 mK ($k=2$). These measurements show that both the functional principle and especially the realized design of a triple-fixed-point offer a good possibility to implement a simplified three-point calibration of a radiation thermometer.

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