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Simultaneous Contact and Non-contact Measurements of the Melting Temperature of a Ni–C Fixed-Point Cell

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Abstract A novel fixed-point cell design that allows simultaneous measurements using contact and non-contact thermometers was developed and investigated at PTB to realize the nickel-carbon (Ni–C) fixed-point. The melting temperature indicated by the LP3 radiation thermometer amounted to (1328.86 ± 0.52)°C ($k = 2$). The melting temperature of the Ni–C fixed-point cell was also calculated by extrapolating the emf-temperature characteristics of two Pt/Pd thermocouples based on their calibrations at conventional fixed points of the ITS-90. The melting temperature of the Ni–C eutectic amounts to (1328.44 ± 0.45)°C using thermocouple Pt/Pd 01/04, and to (1328.53 ± 0.46)°C using thermocouple Pt/Pd 01/05, with uncertainties for $k = 2$. The contact and non-contact thermometers agree well within the combined uncertainties.

Keywords Eutectic · Melting temperature · Ni–C · Pt/Pd thermocouple · Radiation thermometer

1 Introduction

The International Temperature Scale of 1990 (ITS-90) extends from 0.65 K to the highest temperatures measurable with the aid of Planck's radiation law. It is based on 17 fixed points—realized by means of phase transitions—to which certain temperature values are assigned. The temperatures between the fixed-point temperatures are measured by means of defined measuring devices which are calibrated on the basis of the fixed points. The temperatures above the freezing point of silver at 961.78°C have to be measured by means of radiation thermometers, whereby the ratio of the unknown

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radiance to the radiance of a blackbody at the temperature of freezing silver (961.78°C), gold (1064.18°C) or copper (1084.62°C) is determined [1]. At the same time, these fixed points serve to calibrate contact thermometers that are used to approximate the ITS-90 in a large temperature range, also above the freezing point of silver.

The different measuring principles of contact and radiation thermometers bring about different requirements for the fixed points. Fixed points for the calibration of contact thermometers are operated vertically. Fixed points for the calibration of radiation thermometers are constructed as cavities with an emissivity close to 1 and are usually operated horizontally. Therefore, a direct comparison of fixed-point cells intended for the calibration of contact thermometers with cells intended for radiation thermometry is generally impractical, if not impossible. The traceability of contact thermometers to ITS-90 is often only possible through time-consuming and metrologically complex comparison measurements in furnaces specifically developed for this purpose.

This problem has now been solved based on the development of the combined fixed-point cell [2], by means of which the phase-transition temperature of the corresponding fixed-point material can be determined with low uncertainties and simultaneously (or consecutively) with a non-contact and/or a contact thermometer. In this way, it becomes possible, for the first time, to calibrate contact thermometers and radiation thermometers simultaneously at a fixed-point temperature in accordance with the ITS-90. First results of measurements of Ni–C eutectic fixed-point cells are presented in this article.

2 Design and Preparation of the Combined Ni–C Fixed-Point Cell

The concept of the combined fixed-point cell is depicted in Fig. 1. A photograph is shown in Fig. 2. In the fixed-point cell, a vertically mounted thermometer well and a horizontally arranged cavity are accommodated. These are positioned perpendicularly to each other and are entirely enclosed by the fixed-point material. The tube and the cavity are designed in such a way that they comply—with regard to their constructional properties and their dimensions—with the respective measurement requirements for contact (sufficient immersion depth to prevent heat flux effects) or non-contact (emissivity) temperature-measuring methods. All components of the combined fixed-point cell are made of pure graphite in order to maintain the purity of the fixed-point material inside the cell. To reduce the risk of a failure of the combined fixed-point cell due to breakage of the crucible, the cell was constructed with a double wall.

The nickel and graphite powders used to fill the cell were supplied by Alfa Aesar and had nominal purities of 99.996% (Ni) and 99.9999% (graphite), according to the supplier. The powders were mixed at approximately 3.0% by weight carbon in nickel. The crucible, filled with the powder mixture, was heated in an argon atmosphere until a temperature about 15 K above the melting temperature of the Ni–C eutectic was reached and maintained for about 40 min. After that, the temperature of the furnace was lowered by about 25 K and kept at this temperature until solidification was completed, and then decreased further to room temperature. This procedure was repeated until the crucible was almost completely filled (six runs were required). The thermometer well was inserted during the melt of the last filling step. The total mass of the

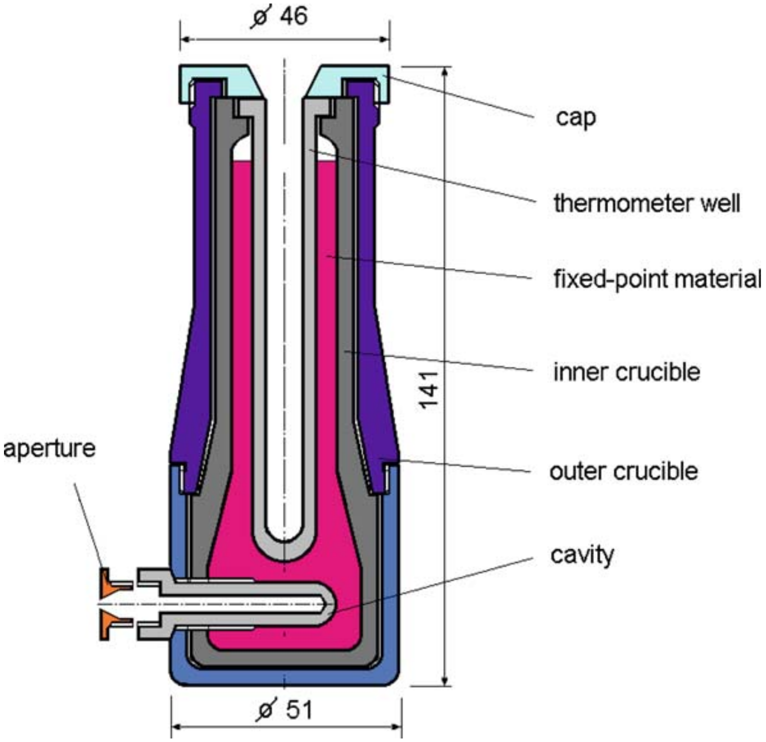


Fig. 1 Schematic diagram of the setup of the novel fixed-point cell for the simultaneous calibration of radiation thermometers and contact thermometers (dimensions in mm)

eutectic alloy in the combined eutectic fixed point cell was 357 g. The inner volume of the Ni–C cell amounts to about 50 cm^3.

3 Measurements

3.1 Measurement Setup

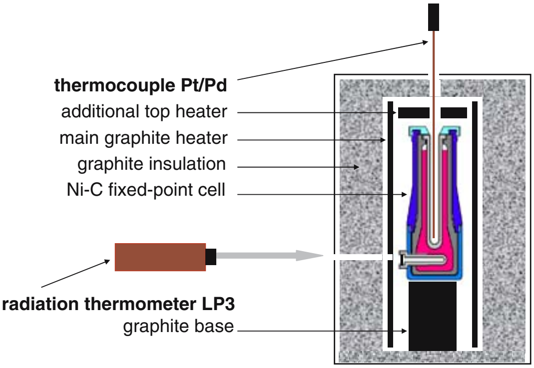
The investigation of the combined Ni–C eutectic fixed-point cell was performed in the High-Temperature Furnace (HTF-R) of PTB, described elsewhere [2]. A graphite-based heating system that consists of a central main heater and an additional top heater was used in an argon atmosphere. The Ni–C cell was placed vertically and axial-symmetrically with respect to the main heating system on a graphite base. The top heater was adjusted so that an axial symmetric temperature distribution was ensured around the fixed-point cell. The temperature inhomogeneity over the combined fixed-point cell amounts to about ±2.5 K The measurement set up is illustrated in Fig. 3.

3.2 Measurement Devices

Two Pt/Pd thermocouples (No. 01/04 and 01/05) were chosen as contact thermometers to perform the measurements. Their design and performance are described elsewhere

Fig. 2 Picture of the combined Ni–C eutectic fixed-point cell

Fig. 3 Measurement setup of the HTF-R



[3,4]. The emf measurements were performed using a Keithley 2182 voltmeter; data acquisition was performed using a Labview program.

The radiation thermometer used was an LP3 (No. 80-05) radiation thermometer, which combines a high quality interference filter and a selected silicon photodiode, resulting in an effective wavelength around $\lambda_{\text{eff}} = 650\,\text{nm}$. For the non-contact measurements, the LP3 was focused directly, at a distance of 690 mm, onto the aperture of the horizontal blackbody cavity which is part of the Ni–C cell. The radiation temperature measurements were performed without a window in the optical path of the HTF-R, in order to avoid additional uncertainties which might arise due to an unknown and changing transmittance of the glass.

3.3 Fixed-Point Realizations

To begin the fixed-point realization, the furnace with the combined Ni–C eutectic fixed-point cell was evacuated at room temperature, filled with argon, and heated to 900°C at a heating rate of about $10\,\mathrm{K{\cdot}min^{-1}}$. At 900°C, the heating rate was reduced to about $5\,\mathrm{K{\cdot}min^{-1}}$. At a temperature of about 3–5 K below the expected melting temperature, the furnace temperature was kept stable for about 15 to 20 min. Subsequently, in order to initiate the melt, the temperature was increased—at a heating rate of $5\,\mathrm{K{\cdot}min^{-1}}$—to temperatures between 8 and 18 K above the melting temperature of the Ni–C eutectic. After the melt, and after a stabilization time of 10 to 20 min at the offset temperature, the temperature of the furnace was decreased at $5\text{–}7\,\mathrm{K{\cdot}min^{-1}}$ to initiate the freeze.

A total of 15 melting and freezing cycles were realized. The first five cycles were performed only with the Pt/Pd thermocouple 01/04; 10 of the 15 cycles were recorded simultaneously with the LP3 and one of the two Pt/Pd thermocouples. Pt/Pd thermocouple 01/04 was used for seven cycles, Pt/Pd thermocouple 01/05 for eight cycles. After the fifth measurement, a horizontal crack appeared at the lower part of the cell. An additional sleeve, also made of pure graphite, was slipped over the outer crucible and the Ni–C cell could be used further without any problems.

Typical melting and freezing curves, measured by means of the Pt/Pd thermocouple 01/05 and the LP3, are presented in Fig. 4. The initial part of the melting curve obtained with the LP3 indicates a vertical temperature gradient along the fixed-point cell, with a lower temperature at the top of the cell. The run-off indicated by the LP3 reveals that the cavity is not uniformly surrounded by a solid/liquid interface, indicative of additional horizontal gradients in the cell. According to the consensus of the temperature community, and based on the high reproducibility that has been found for the inflection point of a melting curve, the emf measured at the inflection point was assigned to the melting temperature of the Ni–C eutectic.

3.4 Measurement Results

On the basis of the seven and eight measurements carried out by means of the two Pt/Pd thermocouples 01/04 and 01/05, respectively, the melting temperature of the Ni–C fixed point cell was calculated by extrapolating the emf-temperature characteristic of the corresponding thermocouple based on its calibration at the conventional freezing points of the ITS-90—Cu, Ag, Al, and Zn—and at the melting point of gallium and the ice point. A detailed description of this method can be found elsewhere [5]. In this way, the melting temperature of the Ni–C eutectic is predicted to be (1328.44 ± 0.45)°C using thermocouple Pt/Pd 01/04 and (1328.53 ± 0.47)°C

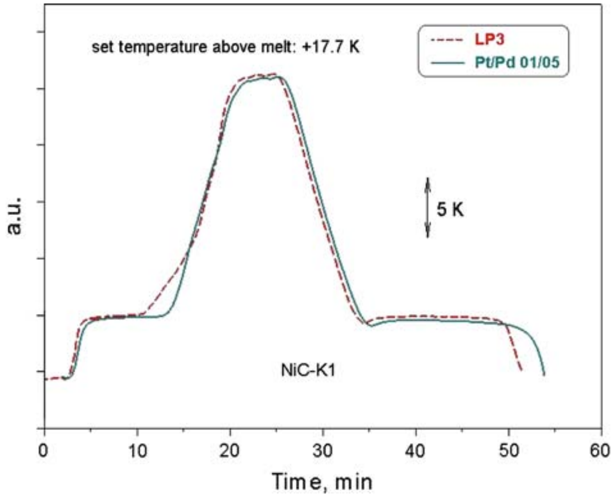


Fig. 4 Simultaneous melting and freezing curves at the combined Ni–C eutectic fixed-point cell obtained using the LP3 and the Pt/Pd thermocouple 01/05

using thermocouple Pt/Pd 01/05. The mean temperature obtained using the LP3 was (1328.86 ± 0.52)°C. The given uncertainties are for a coverage factor of $k = 2$.

3.5 Measurement Uncertainties

The following uncertainty contributions have to be taken into account to estimate the uncertainty of the extrapolated melting temperatures using the Pt/Pd thermocouples: the uncertainty due to the calculated deviation functions in the temperature range between 0 and 1100°C—this is the uncertainty of the calibration of the thermocouples at the conventional fixed points, including their inhomogeneities as measured at the freezing point of Ag—which amounts to a value of 0.2 K; the uncertainty due to the extrapolation of the calculated emf/temperature relationship of the temperature range between 0 and 1100°C to the temperature of the Ni–C melting point, which amounts to a value of 0.03 K (Pt/Pd 01/04) and 0.05 K (Pt/Pd 01/05); the uncertainty of the measurement at the Ni–C eutectic itself (one-point-calibration) of 0.06 K; and the uncertainty of the Pt/Pd thermocouple reference function [6] at the melting temperature of the Ni–C eutectic of 0.08 K. These uncertainty contributions are for $k = 1$. Therefore, the expanded uncertainties of the extrapolated melting temperatures for $k = 2$ amount to values of 0.45 K for Pt/Pd 01/04 and 0.47 K for Pt/Pd 01/05.

The uncertainty of the measurements performed using the LP3 is composed of four components: the calibration uncertainty of the LP3 with respect to the ITS-90 of 0.24 K, the uncertainty of the SSE correction due to the hot surroundings of the radiating opening of the Ni–C fixed point of 0.03 K, the standard deviation of the measurements of 0.02 K, and the uncertainty due to the stray-light originating from the tube-like lead-through inside the HTF-R of 0.1 K. The overall uncertainty of the temperature measurements of the Ni–C fixed point carried out using the radiation thermometer LP3 amounts to 0.52 K, at a coverage factor of $k = 2$.

4 Summary

The novel design of the combined Ni–C eutectic fixed-point cell was tested successfully. The traceability of its melting temperature to the ITS-90 was given by the measurements with the LP3. For the first time, simultaneous measurements of the phase transition temperature of a eutectic fixed-point cell are possible. The novel fixed-point design allows calibration laboratories a time-optimized, exact calibration, and traceability of radiation thermometers and contact thermometers in only one step.

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