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The Combined Use of a Gas-Controlled Heat Pipe and a Copper Point to Improve the Calibration of Thermocouples up to 1100 °C

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Abstract The calibration of platinum-based thermocouples from 420 °C to 1, 100 °C is currently carried out at INRIM making use of two different apparatus: for temperatures below 930 °C, a potassium gas-controlled heat pipe (GCHP) is used, whereas a metal-block furnace is adopted for higher temperatures. The standard uncertainty of the reference temperature obtained in the lower temperature range is almost one order of magnitude better than in the higher temperature range. A sealed copper cell was investigated to see if it could be used to calibrate thermocouples above 930 °C with a lower uncertainty than our current procedures allowed. The cell was characterized with Type S and Pt/Pd thermocouples and with an HTPRT. The freezing plateaux were flat within 0.01 °C and lasted up to 1 h with a repeatability of 0.02 °C. The temperature of the cell was determined with a standard uncertainty of 0.04 °C. Hence, the copper cell was found to be superior to the comparator furnace for the calibration of platinum-based thermocouples because of the significant decrease in the uncertainty that it provides. An analysis was also carried out on the calibration of Pt/Pd thermocouples, and it was found that the combined use of the potassium GCHP and the Cu fixed-point cell is adequate to exploit the potential of these sensors in the range from 420 °C to 1,084 °C. A comparison with a fixed-point calibration was also made which gave rise to agreement within 0.07 °C between the two approaches.

Keywords Fixed point · Heat pipe · Thermocouple calibration

1 Introduction

Platinum versus platinum 10% rhodium (Type S) thermocouples are commonly used as secondary standards to disseminate the ITS-90 to industry. The calibration of these

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thermocouples from 420 °C to 1,100 °C is currently carried out at INRIM making use of two different apparatus, depending on the temperature range. For temperatures below 930 °C, the calibration is performed in a potassium gas-controlled heat pipe (GCHP) [1] by comparison against an Au/Pt thermocouple calibrated at the ITS-90 fixed points. The calibration accuracy takes advantage of the GCHP's performance, and a standard uncertainty of the reference temperature within 0.04 °C is obtained. Above 930 °C, the calibration is performed in a metal-block furnace by comparison against a reference Type S thermocouple calibrated at ITS-90 fixed points and, consequently, a higher uncertainty of the reference temperature must be accounted for.

Hence, the aim of the present work was to study whether a sealed copper cell is adequate to replace the comparator furnace, in order to improve the uncertainty level for calibrations above 930 °C. The performance and the weak points of the facility used for the thermocouple calibration are described in Sect. 2, while the characterization of the copper cell is presented in Sect. 3. Finally, in Sect. 4, a calibration of a Pt/Pd thermocouple with the "GCHP + Cu point" approach is presented.

2 Facility for Thermocouple Calibration

2.1 From 420 °C to 930 °C

In the Contact Thermometry Laboratory at INRIM, the calibration of platinum-based thermocouples in the temperature range from 420 °C to 930 °C is performed in a potassium GCHP. Nowadays, GCHPs are the most suitable devices to achieve a temperature stability and uniformity of the order of some m°C in a wide temperature range. These features are essential to perform very accurate temperature measurements; therefore, these devices are optimal for the calibration of platinum resistance thermometers (PRT) and thermocouples (TC). An update and complete review of the GCHP physical principles and the most recent results can be found in [2].

The device operating at INRIM since 2000 shows temperature stability of the order of 5 m°C for the whole temperature range and temperature uniformity within 0.01 °C over a length of more than 10 cm. Moreover, the device is equipped with six measuring wells, allowing up to five thermocouples to be simultaneously calibrated. The maximum difference among the wells is 0.02 °C.

The reference temperature is usually provided by an Au/Pt thermocouple, calibrated at ITS-90 fixed points up to the silver freezing point. This type of thermocouple is known to be very stable and accurate, and consequently its calibration uncertainty was estimated as less than 1 µV (i.e., 0.04 °C) at the silver freezing point. The standard uncertainty of the reference temperature at the maximum temperature of the heat pipe was evaluated to be 0.04 °C, the greater contributions to the uncertainty budget being the calibration uncertainty of the reference thermocouple and the temperature uniformity along the wells.

2.2 Above 930 °C

The thermocouple calibration above 930 °C is carried out in a comparator furnace by comparison with a reference Type S thermocouple. Initially, the comparator fur-

Table 1 Technical characteristics and performance of the apparatus used for thermocouple calibration

	Potassium gas-controlled heat pipe	Comparator furnace
Temperature range	(420–930) °C	(600–1,064) °C
Stability	0.005 °C	0.01 °C
Axial uniformity	0.01 °C	0.1 °C
Number of wells	6	4
Uniformity between different wells	0.02 °C	0.2 °C
Reference thermocouples	Au/Pt	Type S
Standard uncertainty of the reference temperature	0.04 °C (at 930 °C)	0.30 °C (at 1,060 °C)

nace was developed for the calibration of Type S thermocouples in the temperature range between 600 °C and 1,064 °C, as described in [3]. Subsequently, as a result of the introduction of the GCHP, its use has been limited to calibrations above 930 °C, where the potassium heat pipe could not be used for safety reasons.

The comparator consists of two Inconel cylinders: the main block, 20 cm long, covers the measuring zone, while the second block, 5 cm long, is placed 1 cm above the first one and hosts an auxiliary heater with the aim of maintaining the same temperature as the main block. This assembly achieves a temperature uniformity of about 0.1 °C over 8 cm at 1,060 °C. The uniformity between the four thermometer wells is better than 0.2 °C and the temperature stability is within 0.01 °C.

The standard uncertainty of the reference temperature at 1,060 °C is estimated to be 0.30 °C; also, in this case, the greater contributions to the uncertainty budget come from the calibration uncertainty of the reference thermocouple (which is 3 μV for a Type S thermocouple, i.e., 0.25 °C) and the temperature uniformity of the furnace, but both these factors are ten times the same factors evaluated in the case of the heat pipe. A summary of the technical characteristics and performances of both apparatus is presented in Table 1.

This is the reason why it is desirable to replace the comparator furnace with a fixed-point cell. The sealed copper cell seems to be the ideal solution to these problems, because (a) the freezing temperature of copper is high enough (1084.62 °C), (b) it is much cheaper than gold and presents a larger heat of fusion, which leads to more stable freezing curves, and, moreover (c) the sealed cell eliminates the need of a gas line to evacuate and introduce argon into the cell in order to protect the copper from oxygen contamination.

3 Copper Cell: Tests and Results

3.1 Description of the Cell

The copper cell under test was a sealed copper cell, labelled Cu JM-1, constructed at INRIM some years ago. A schematic of the cell is shown in Fig. 1 and details of

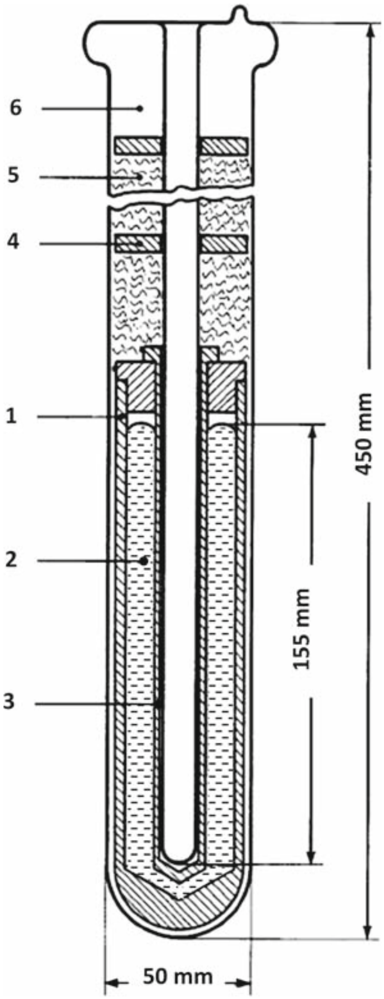

Fig. 1 Schematic of the sealed copper cell described in [4]: (1) graphite crucible, (2) copper ingot, (3) silica re-entrant well, (4) graphite-disk shields, (5) pure silica wool, and (6) argon atmosphere

the cell construction can be found in [4]. A mass of 1.2 kg of copper, with a nominal purity of 99.999 %, is contained in a very pure graphite crucible. The crucible is sealed in a silica-glass envelope, in an inert atmosphere of high-purity argon, with a pressure of 101,325 Pa at the melting point. The distance between the bottom of the well and the free surface level of the metal is 15 cm. The cell was inserted in a block-comparator furnace equipped with a main heater driven by a proportional-integrative-derivative (PID) controller and two end heaters that were manually controlled. The temperature profile was optimized with the copper ingot in the liquid state by adjusting the end heaters; uniformity within 0.1 °C over 7 cm was obtained.

The melting curves are obtained by increasing the temperature of the furnace to 1,090 °C, after the cell is kept at 1,079 °C for at least 1 h, while the freezing curves were obtained by means of the "induced-freeze" technique, so as to produce a constant temperature plateau.

The "induced-freeze" technique consists of the following procedure: (1) the metal melts overnight at a uniform temperature about 4 K above the melting point, (2) the temperature of the furnace is decreased by changing the set point of the controller to 2 K below the freezing point, (3) when the sensor inserted in the well detects an initial arrest on the cooling curve, it is withdrawn from the well and a cold inducing rod, a silica tube with about 15 cm of alumina rod at the bottom, is inserted for about 1 min into the cell, in order to induce the growth of a mantle of metal on the well, and (4) then, the inducing rod is withdrawn from the cell and replaced by the sensor for the measurements. The freezing procedure here described was adopted on the basis of previous studies carried out at INRIM [5], to obtain a stable liquid–solid interface around the thermometer well.

Typical melting and freezing plateaux are shown in Fig. 2. The melting curves showed a slope of approximately 0.1 °C, but, on the contrary, the freezing plateaux were constant within 0.01 °C and lasted (45–60) min.

3.2 Cell Characterization

The cell was characterized using a group of five thermocouples: a Type S thermocouple, calibrated at ITS-90 fixed points up to the gold freezing point, and four Pt/Pd thermocouples, calibrated up to 1,500 °C by comparison with the standard radiation thermometer [6].

Initially, a platinum shield was inserted in the thermometer well in order to minimize the thermal losses by radiation. Unfortunately, the presence of this shield affected all the measurements performed by different sensors, causing a decrease of the measured temperature. In fact, all the values of the emf measured by the thermocouples at the freezing plateaux give rise to calculated temperatures systematically below the temperature assigned by the ITS-90 to the copper fixed-point, i.e., 1084.62 °C: a difference of −0.09 °C was found for the Type S thermocouple, and an average difference of −0.14 °C, with a standard deviation of 0.08 °C, was found for the four Pt/Pd thermocouples. For the Pt/Pd thermocouples, the temperature was calculated using the reference function (now included in the ASTM E1751-00 standard [7]) and a deviation curve that was determined for each thermocouple from the calibration performed by comparison with the standard radiation thermometer [6]. The standard uncertainty of these two sensors was estimated to be 0.28 °C and 0.22 °C, for the Type S and Pt/Pd thermocouples, respectively.

This difference from the temperature assigned by ITS-90 to the copper freezing point was confirmed also by a comparison with an open copper cell, labelled Cu Lei1, with a crucible length of 20 cm and copper purity better than 99.9995 %, which was used in the EUROMET project No. 624 [8] with good results. A high-temperature platinum resistance thermometer (HTPRT), with a nominal resistance of 0.6 Ω and a sensitivity of $1.6\,\text{m}\Omega \cdot {}^\circ\text{C}^{-1}$ at 1,084 °C, was used to compare the two copper cells. The resistance measured at the freezing temperature of the Cu Lei1 cell was taken as the reference, R_{Cu}, and the deviations ΔR from this value were attributed to

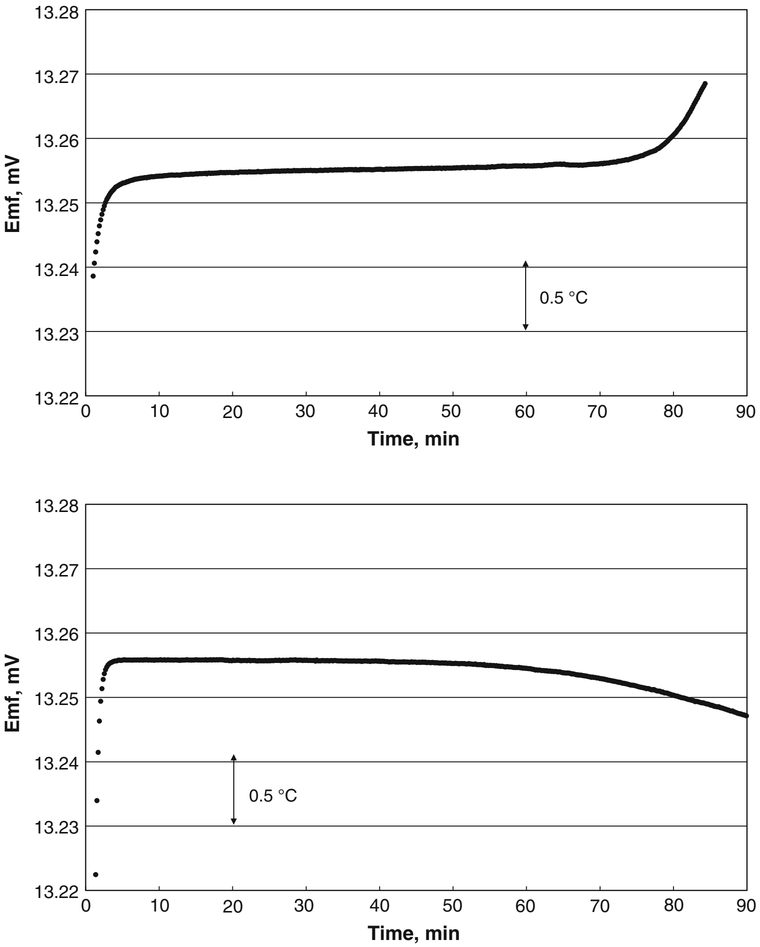

Fig. 2 Typical melting and freezing plateaux

temperature differences ΔT from $T_{Cu} = 1084.62\,°C$. The resistance measured on the freezing plateau in the Cu JM-1 cell with the Pt shield inserted in the thermometer well was lower than R_{Cu} by 0.23 mΩ for the first measurement, and by 0.20 mΩ for the second measurement; hence, a mean temperature difference of 0.14 °C from T_{Cu} was determined.

A temperature difference of 0.14 °C would be difficult to explain by accidental copper contamination or a loss of pressure in the sealed cell, because these factors should be so obvious, namely impurities at 10^{-4} levels or a pressure difference of about 4 MPa, that the freezing plateaux would be affected. Hence, at first sight, the temperature difference was believed to be due to heat loss caused by the small length of the cell, which is 30 % shorter than the cells recommended for the ITS-90 fixed points [9].

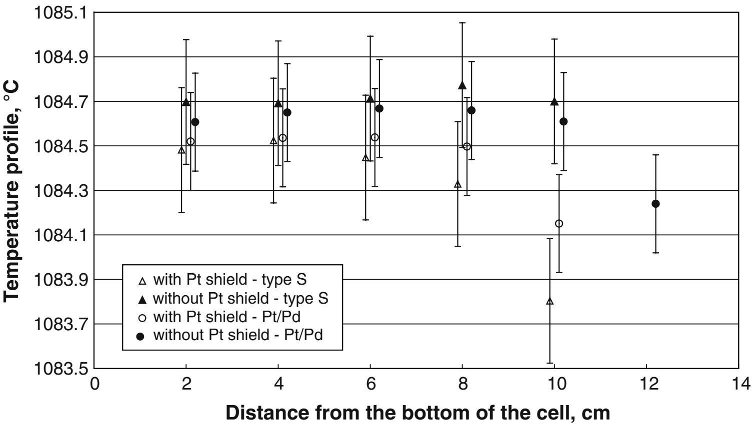

Fig. 3 Temperature profile measured during a freezing plateau by a Pt/Pd and a Type S thermocouple, with and without the Pt shield

Carrying out the cell characterization, the Pt shield was discovered to be responsible for the temperature difference of the cell with respect to the copper fixed-point value. Two thermocouples of the group of five were used to measure the immersion profile during a freeze, with and without the Pt shield. The measurement results are shown in Fig. 3 and indicate that (i) both sensors are in thermal equilibrium with the cell and, especially that (ii) the temperature of the cell without the Pt shield is clearly higher than the temperature measured with the Pt shield in place. More precisely, the temperature differences measured by each sensor with and without the shield were calculated from 2 cm to 10 cm from the bottom of the cell: an average temperature difference of 0.27 °C, with a standard deviation of 0.12 °C, was found for the Type S thermocouple, while the average difference obtained for the Pt/Pd thermocouple is 0.12 °C, with a standard deviation of 0.03 °C. The Type S thermocouple is more affected than the Pt/Pd thermocouple by errors generated by inhomogeneity of the wires; however, the two results are comparable within the estimated standard uncertainty for the two sensors and indicate that the sealed copper cell temperature is in agreement with the ITS-90 value.

In light of these results, it is likely that the Pt shield acted as a sort of "heat sink" that removed heat from the crucible; its length of 44 cm makes it protrude from the cell by approximately 2 cm.

3.3 Uncertainty Estimate

In order to validate the results obtained by the thermocouples with more accurate measurements, the immersion profile without the Pt shield was measured also with a HTPRT, with a nominal resistance value of 10 Ω and a sensitivity of 27.88 $\text{m}\Omega \cdot {}^\circ\text{C}^{-1}$ at the copper point.

Since the signal of this thermometer was too noisy to be measured with the current-comparator bridge, a high-accuracy digital multimeter was used for the measurement

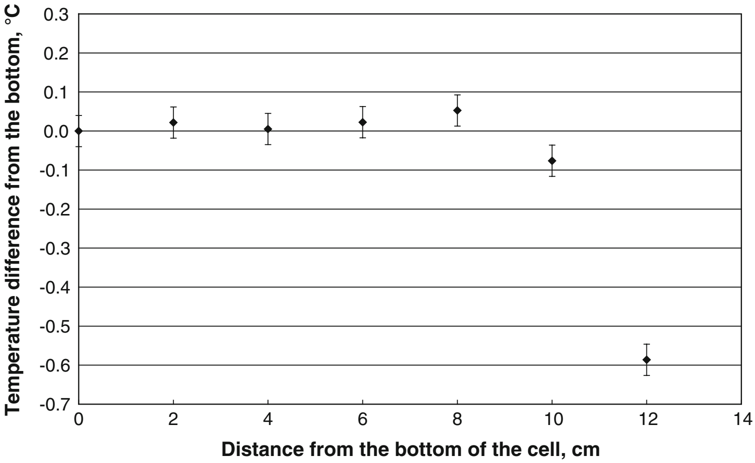

Fig. 4 Temperature profile measured during a freezing plateau by a HTPRT

of the immersion profile. Figure 4 shows the temperature difference from the bottom of the cell measured by the thermometer and allows us to conclude that the temperature in the cell is uniform within some hundredths of a degree for almost 10 cm. The standard uncertainty of the measurements with the HTPRT was estimated to be about 0.04 °C. The greatest contribution to the uncertainty budget is the short-term stability of the thermometer, which was evaluated as the repeatability at the triple point of water (TPW) multiplied by the ratio between the signal at the copper point and the signal at the TPW. Other uncertainty components considered for the uncertainty evaluation are the accuracy of the multimeter measuring a resistance in the 100 Ω range, the measurement noise, and the temperature stability during the plateaux.

Even if it is quite conservative, this uncertainty value is assigned to the sealed copper freezing-point cell, since only a few measurements have been performed with the HTPRT. Further investigations will be devoted to a better evaluation of the copper cell uncertainty with the help of a new HTPRT bought for this purpose. Still, the uncertainty value estimated for the sealed copper cell is about one order of magnitude better than the uncertainty of the reference temperature in the comparator furnace and is equal to the maximum uncertainty of the reference temperature evaluated for the measurements performed in the potassium heat pipe.

4 Analysis of Calibration Data

Some checks and analysis of calibration data were performed with the purpose of assessing the suitability of the Cu point itself and of the “GCHP + Cu point” approach. Both a Type S thermocouple and one of the Pt/Pd thermocouples used in the present investigations, and referred to as Ea in [6], were included in the analysis. Firstly, the sealed copper cell was used to calibrate the Type S thermocouple that was previously calibrated in the laboratory for primary thermometers at the copper fixed-point. The difference between the signal measured during this calibration and the signal value

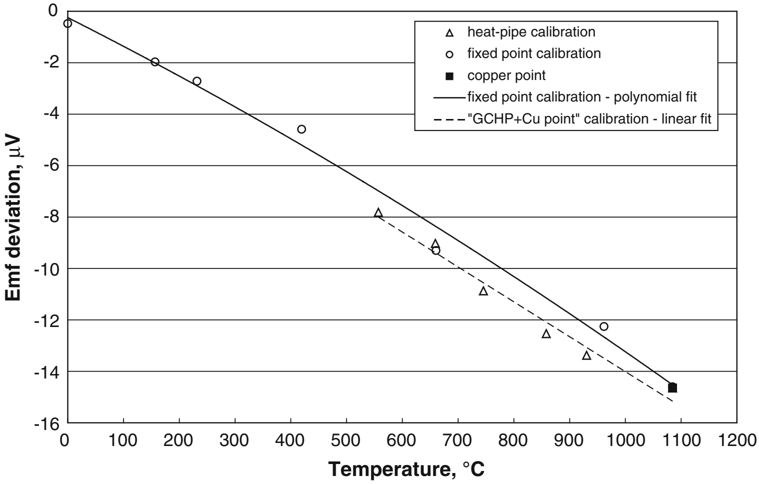

Fig. 5 Emf deviations from the Pt/Pd reference function measured in the potassium GCHP, at the copper point and in a fixed-point calibration

reported on the calibration certificate is less than 0.03 °C, a value which is within the estimated uncertainty assigned to the sealed copper cell.

The Pt/Pd thermocouple was originally calibrated at the fixed points of In, Sn, Zn, Al, and Ag and at the triple point of water. Successively, it was calibrated in the potassium GCHP from 550 °C to 930 °C and with the Cu fixed-point cell here described. Emf deviations for the different sets of data with respect to the reference function for Pt/Pd thermocouples are shown in Fig. 5. The emf deviations for the GCHP data were fitted with a linear equation and rms residuals of 0.016 °C were found. When the Cu point calibration data are added, the rms residuals slightly increase to 0.027 °C, substantially confirming that the Cu point does not introduce significant deviation with respect to the GCHP calibration data.

For the purpose of a direct comparison of the "GCHP + Cu point" calibration with the fixed-point calibration, the fitted emf deviations curve was compared with the second-order polynomial equation fitting the fixed-point calibration data. An average difference of about −0.07 °C, with a standard deviation of 0.02 °C, was found. Even if a systematic difference between the two approaches has been found that needs further investigation, it is worth noting that the difference is well within the estimated combined standard uncertainty of 0.12 °C, a figure obtained by adding in quadrature the uncertainty for the "GCHP + Cu point" calibration (0.09 °C) and that of the fixed-point calibration (0.08 °C).

5 Conclusions

A sealed copper cell was developed to replace the comparator furnace for the calibration of thermocouples above 930 °C with the purpose of setting up a facility also comprising a potassium GCHP for improved calibration of thermocouples.

The cell was characterized with different thermocouples and with a high-temperature platinum resistance thermometer. The freezing plateaux were flat within 0.01 °C and lasted up to 1 h with a repeatability of 0.02 °C. The estimated standard uncertainty in the temperature of the cell is 0.04 °C. The analysis of the calibration data of different sensors, both Type S and Pt/Pd thermocouples, indicated that a substantial reduction of the calibration uncertainty can be achieved. Such an achievement is especially important for the calibration of Pt/Pd thermocouples to adequately exploit their potential. A comparison was also made with the results obtained with a fully fixed-point calibration that achieved an agreement within 0.07 °C between the two approaches, well within the combined standard uncertainty of 0.12 °C. Nevertheless, a systematic difference between the two approaches was found that needs to be further investigated and resolved.

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