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A Slimline Integrated Self-Validating Thermocouple: Initial Results

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Abstract NPL, in collaboration with CCPI Europe, have designed a slimline integrated self-validating ("inseva") thermocouple with the same external form factor as conventional thermocouples, with the aim of making them suitable as direct replacements for existing thermocouples in process. Type S thermocouples have been manufactured in recrystallized alumina-sheathed assemblies, with Cu and Co–C reference ingots, with an outer diameter of 7 mm. The new slimline inseva thermocouple is, in principle, suitable for use in the same positions and conditions as the conventional thermocouple which it replaces. This paper reports the initial reference ingot melt and freeze plateaus successfully observed using the first inseva thermocouples, and demonstrates observation of furnace sensitivity and ramp rate sensitivity of the plateau temperatures.

Keywords Calibration · HTFP · Integrated · Self-validating · Thermocouple

1 Introduction

Currently, thermocouples, when used at high temperatures (>1000 °C), exhibit drift from a known calibration state, which if unmonitored causes loss in both

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knowledge and confidence of the temperature being measured. This creates the need for mitigation or adjustments to the process concerned, which may need to be made using information from other sources. This is of particular concern for long-term, high-temperature monitoring in high-value manufacturing processes such as heat treatment of components where the repeatability of thermal processes is key. Such mitigation steps often rely only on previous experience, and there is presently no way of validating the efficacy or appropriateness of such adjustments.

The concept of using a very small reference artifact integrated with the thermocouple wires was first developed by Tischler in 1982 [1]. This has been followed by a series of trials of various cells, and alternative designs by several teams [2–7]. For example, Augustin et al. [8] presented an industrially applicable design which was based on a miniaturized traditional temperature fixed-point: maintaining the conventional thermowell design. This was reported to have a plateau duration of up to 1 min, using alumina crucible and reference temperatures around 600 °C.

It is known that the pure metal or eutectic temperature fixed-points can also be used in a graphite crucible [6]—but this had not, until now, been successfully achieved within the dimensions of a typical thermocouple sheath (at high temperature) due to the apparent limitations of the robustness of the crucible, reactivity of carbon with thermocouple elements and need to widely separate the thermoelements around the cell.

In this paper, the initial results of a successful integrated self-validating (“inseva”) thermocouple for use up to 1350 °C is reported. NPL have designed, with advice and assistance from CCPI Europe, and within the framework of the European Metrology Programme for Research and Innovation project 14IND04 *EMPRESS* [9], a new inseva thermocouple. This contains a cell based on a pure metal or binary eutectic alloy ingot integrated within a standard alumina thermocouple design. The cell is designed to act as a fixed temperature reference point (through its melting and freezing transitions, as with traditional pure metal and eutectic fixed-points used for calibration purposes), and the new inseva thermocouple is able to be used within oxidizing furnace atmospheres. This design develops the technology proven by earlier work at NPL [10], to create a like-for-like replacement sensor which can be immediately deployed in temperature monitoring and potentially control situations. Other self-validating techniques have been proposed in the past, but those which have been deployed industrially have required bespoke physical installation [2,3].

This paper presents the successful observation of the phase transition for both Cu and Co–C inseva thermocouples, which provides immediate feedback on the performance of the thermocouple by allowing *in-situ* validation of the reading. Results of a single point calibration on the inseva thermocouples are presented. Results of initial performance testing are then given and discussed, including the sensitivity of the inseva thermocouples to furnace temperature offset and ramp rate during the melting and freezing transitions.

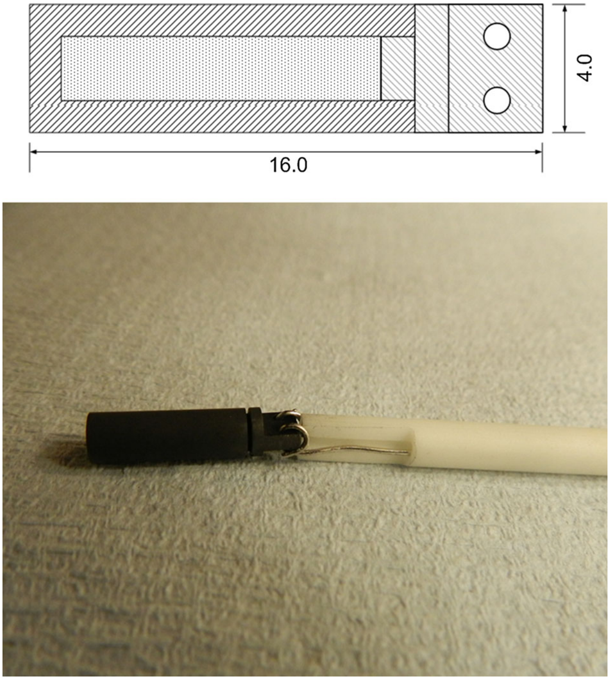

Fig. 1 A schematic of the inseva graphite cell (dimensions in mm) and *photograph* showing the inseva cell, thermocouple hot junction and twin-bore insulator

2 The Inseva Design

The inclusion of a reference fixed-point cell inside the thermocouple allows *in situ* validation of the thermovoltage reading (emf) to be made, enabling thermocouple drift to be mitigated in real time.

Figure 1 shows a photograph and schematic of the graphite cell at the measuring junction end of the thermocouple. The thermocouple is connected at the cell and inserted, with the twin-bore, into a high-purity recrystallized alumina sheath (OD = 7 mm, L = 700 mm).

Four inseva thermocouples have been made, each with Type S thermocouple wires (outer diameter 0.5 mm), using a standard alumina twin-bore and sheath arrangement. The first pair each contains a Cu ingot (whose reference temperature is approx. 1084 °C). The second pair each contains a Co–C eutectic ingot (∼1324 °C). The alumina sheath of each has been sealed at the cold end, preserving a protective argon atmosphere around the thermoelements and cell. The construction of the cells and pre-treatment of the alumina parts and thermocouple wires were completed at NPL. Construction of the complete thermocouple was performed at CCPI Europe. All measurements reported here (excluding the calibration at Co–C) were performed with the outer sheath exposed externally to an atmosphere of air. The calibration at Co–C was performed under argon, to protect the National Standard reference Co–C cell.

The outer dimensions of the inseva cell are 16.0 mm in length and 4.0 mm in outer diameter. The purity of the Cu and Co ingot metals and graphite was stated by the manufacturer as 99.999 %, 99.995 % and 99.9999 %, respectively. To manufacture the fixed-point ingot, a total of 0.28 g of Cu and 0.22 g of Co are used. The graphite cell was made of high-purity Poco DFP-3-2 graphite.

3 Initial Measurements

3.1 Observation of Ingot Melt and Freeze

To begin testing the inseva thermocouples, they were each, in turn, suspended into a single-zone, vertical furnace at NPL (an Elite Thermal Systems TSV18/15/100 furnace). They were positioned such that the inseva cell was at the hottest part of the furnace (which is uniform within 0.5 °C over 2 cm).

Figure 2 shows the typical Cu and Co–C inseva cell melt and freeze plateaus. During this cycle, the furnace was ramped (up and down) at a rate of 1 K/min. The furnace settings used for the Cu inseva thermocouple were: 2 K above the melting plateau to realise the melt (+2 K) and then 2 K below the melting plateau, for the freeze (−2 K). For the Co–C inseva thermocouple in this example, the furnace settings used were +2 K and −10 K.

It is found that the melt plateau generally occurs at a significantly higher emf than the freeze plateau. This is due to the furnace affecting the thermocouple reading directly, creating an artificially high melting temperature reading and artificially low freezing temperature reading. (The cell does not provide the full thermal immersion which is achieved in full-size calibration cells.) The undercool of the Cu cell is considerably longer lasting than that of the Co–C cell, which is to be expected due to the high-purity nature of the Cu ingot which delays initiation of the freezing process.

3.2 Calibration

The thermocouples were calibrated using the standard NPL procedure for UKAS-accredited thermocouple calibrations, in the NPL reference cell type corresponding to the inseva ingot.

For the Cu inseva thermocouples, each was measured for at least 20 min over the stable part of the NPL reference Cu cell freeze plateau (realizing an assigned temperature of 1084.62 ± 0.02 °C) using the local ISO 17025-accredited procedure. The results (average over 20 min) are shown in Table 1. No sign of the freezing of the inseva thermocouple was observed during the calibration.

For the Co–C inseva thermocouples, three melts and freezes were carried out with the NPL reference Co–C cell (1324.09 ± 0.44 °C). The results (average of the three repeats) are also shown in Table 1. The melting of the inseva cell was not visible in addition to the reference cell plateau, but the freeze of the inseva cell was visible (at ∼50 µV lower than the NPL reference cell)—an example is shown in Fig. 3. This type of feature has been observed before in trial cell manufacture (although not reported), where it was assumed that the reference metal has formed in two separate

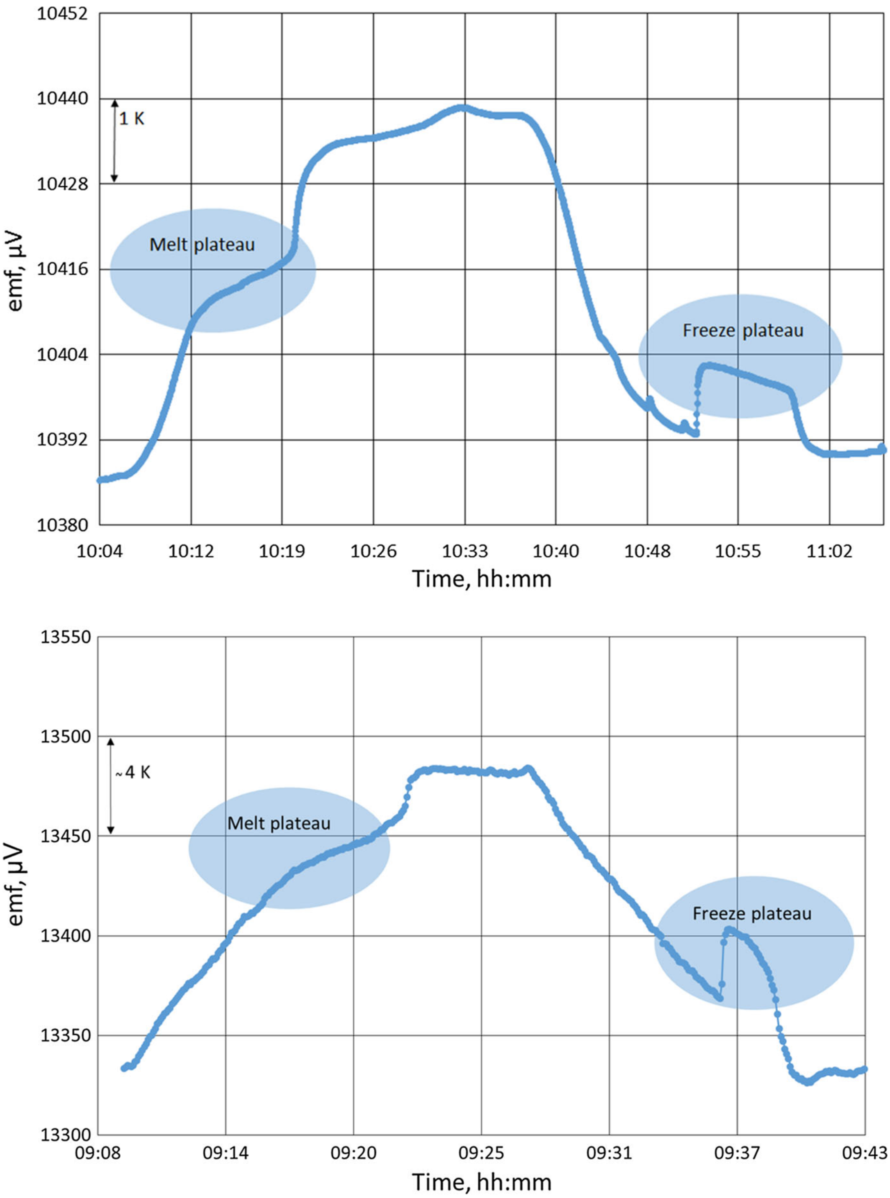

Fig. 2 Typical Cu (*top*) and Co–C (*bottom*) melt and freeze plateaus (1 K/min ramp rate)

ingots (such cells are typically discarded). This is therefore expected to be due to late onset of initiation of the inseva ingot, with a lower temperature recorded due to the overwhelming temperature of the surrounding large fixed-point cell. It is likely that the inseva ingot was effectively insulated, until the freezing of the reference cell was complete.

Table 1 Calibration results for the Cu and Co–C inseva thermocouples, after ramp rate testing

Thermocouple	Calibration emf (μV)	Calibration uncertainty °C ($k = 2$)
Cu#1	10 413.74	0.21
Cu#2	10 390.69	0.21
Co–C#1	13 444.86	0.53
Co–C#2	13 442.26	0.53

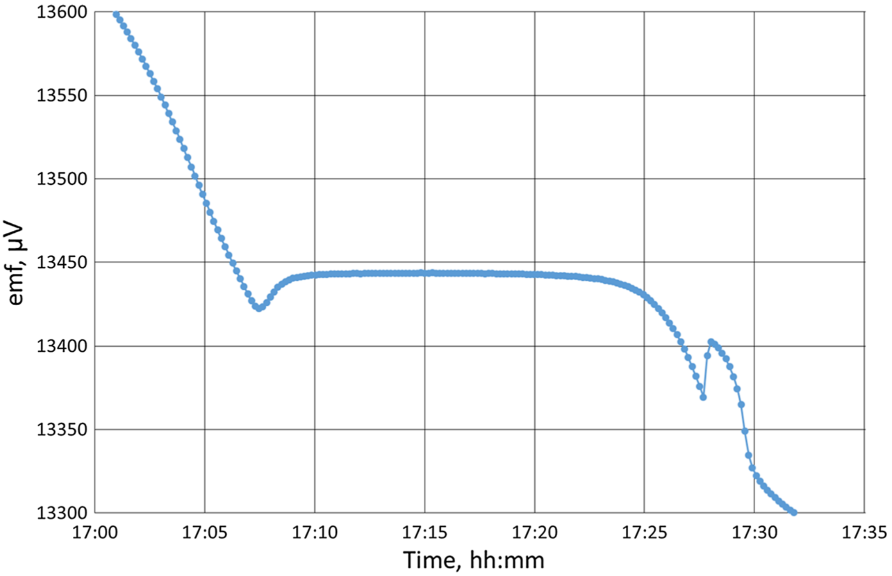

Fig. 3 Freezing of the inseva Co–C thermocouple in a reference fixed-point cell; both ingot freezes are visible (the first from ∼17:08 to ∼17:25 and the second from ∼17:28 to ∼17:30)

3.3 Furnace Sensitivity

To begin to assess the sensitivity to varying the furnace temperature, while in the vertical orientation, both Co–C inseva thermocouples and both Cu inseva thermocouples were repeatedly run through melt and freeze cycles, with varying upper furnace offset temperatures. A ramp rate of 1 K/min was maintained for each setting.

The offset temperature for freezing was kept at −10 K, but for melting, the upper furnace offset temperature was set to +2 K, +5 K, +8 K, +12 K and +20 K in turn, with three repeats made at each setting. In the case of +2 K, the melt plateau had not completed before the furnace reached the set point: this resulted in an extended plateau length (typically 9 min). In each other case, the melting had completed before the furnace reached the set point (the plateau length in each case, typically reduced to 4 min).

Figure 4 shows the average of three repeat Cu and Co–C inseva cell melt and freeze emf measurements, as the furnace offset temperature was changed. The thermocouple calibration emf and calibration uncertainty are also indicated for reference. The difference between the highest and lowest measurement at each setting (i.e., repeatability of each point in the graph) varied between 0.3 μV (0.025 K) and 16.0 μV (1.3 K) with four outliers, with a difference up to 48 μV (4.0 K)—these are marked with a star in the figure.

It can be seen that for the Co–C inseva thermocouples, the melting and freezing temperatures measured are repeatable (within the uncertainty, which would be dominated by the repeatability). The plateau measured for the set point of +2 K does not differ from the other values; therefore, no clear dependence on the furnace set point is found. The melting temperature recorded is very close to the calibration of the thermocouple. The freeze temperature measured with both inseva thermocouples is suppressed, by around 36 μV. This is smaller than the 50 μV observed when calibrating the thermocouple, but still indicates that the external temperature is having a strong impact on the freezing temperature reading.

The suppression of the freezing temperature, with respect to the melting temperature is also seen for the Cu inseva thermocouples. The measurements varied quite considerably (up to 24 μV) both within the set of three repeats and between measurements at differing set points—no clear dependence on the furnace setting is possible to conclude. However, the two Cu inseva thermocouples tested did demonstrate quite different (positive) offsets from the thermocouple calibration (albeit with a wide repeatability)—with the melting temperature also affected, unlike in the case of the Co–C inseva thermocouples.

These differences in measurement quality (repeatability) and apparent temperature may be due to differing extents of thermal contact between the cell and the thermoelements (which were prepared by hand, and subject to change with thermal expansion/contraction).

3.4 Ramp Rate Sensitivity

Although the measurement has not been found to be sensitive to changes in the furnace set point, the warm environment around the thermocouple when recording the melting temperature does appear to create a constant offset of the melt temperature recorded (both apparent in Fig. 4). For this reason, the sensitivity to varying the furnace ramp rate was also investigated, for the melting plateaus of the Co–C and Cu inseva thermocouples. In all cases, the melting plateau was complete before the furnace reached the set point temperature.

The Co–C inseva thermocouples were assessed in the vertical orientation, using rates of 1 K/min, 5 K/min and 10 K/min. The average of three melting plateaus (at each point) are shown in Fig. 5, with a +20 K furnace offset. The Cu inseva thermocouples were assessed in the horizontal orientation, using rates of 1 K/min, 5 K/min and 10 K/min; the average of three melting plateaus (at each point) are also shown in Fig. 5, with a +20 K furnace offset. For clarity, in each case, the difference in emf from the value obtained for a ramp rate of 1 K/min is shown. The difference between

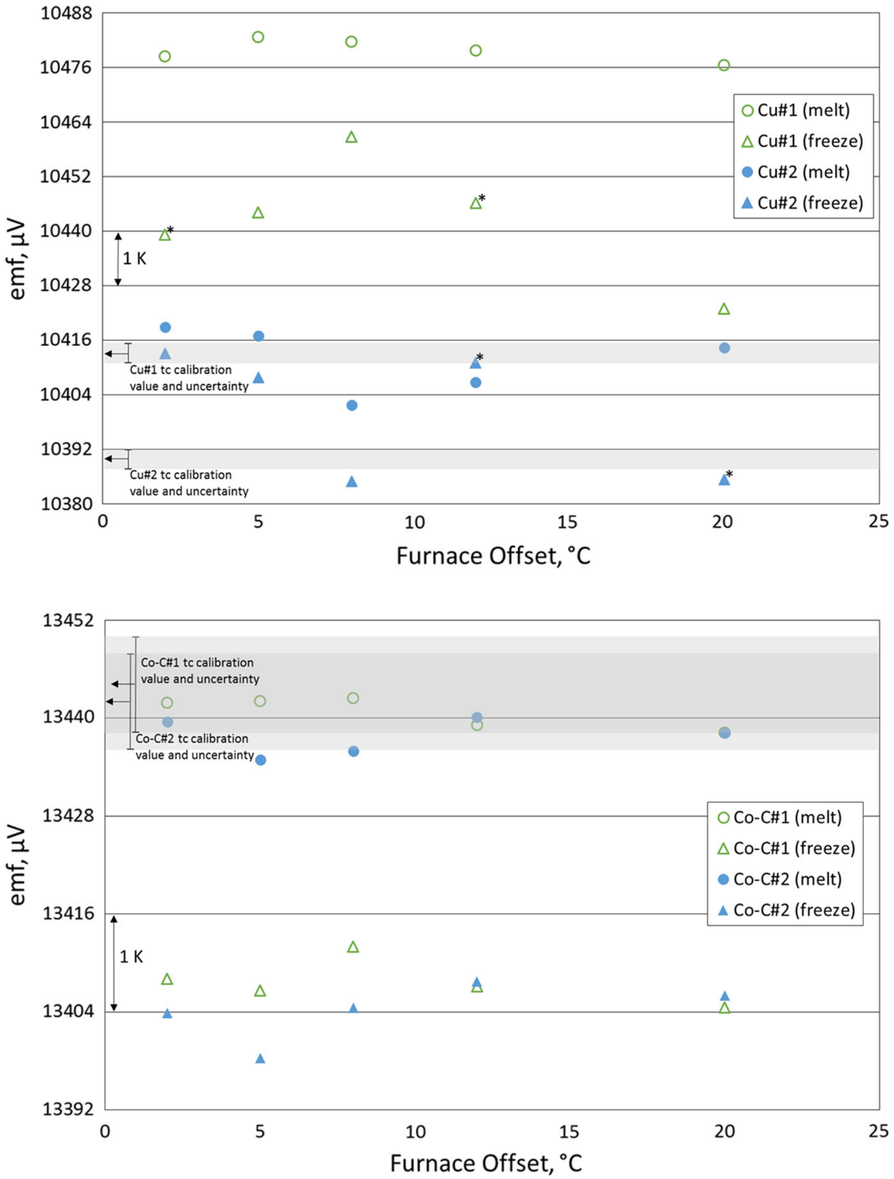

Fig. 4 Measured inseva thermocouple emf at the Cu (*top*) and Co–C (*bottom*) melting and freezing temperatures, with varying furnace offsets

the highest and lowest of the three plateaus measured at each point was (at or) below 9.5 µV (∼0.8 K), except the value at 10 K/min for the second Cu inseva thermocouple which was found to be 15 µV (this is marked with an asterisk in the figure).

The results shown in Fig. 5 clearly show a sensitivity to the ramp rate used. A general increase in the melting emf with the furnace ramp rate was found, excluding the second

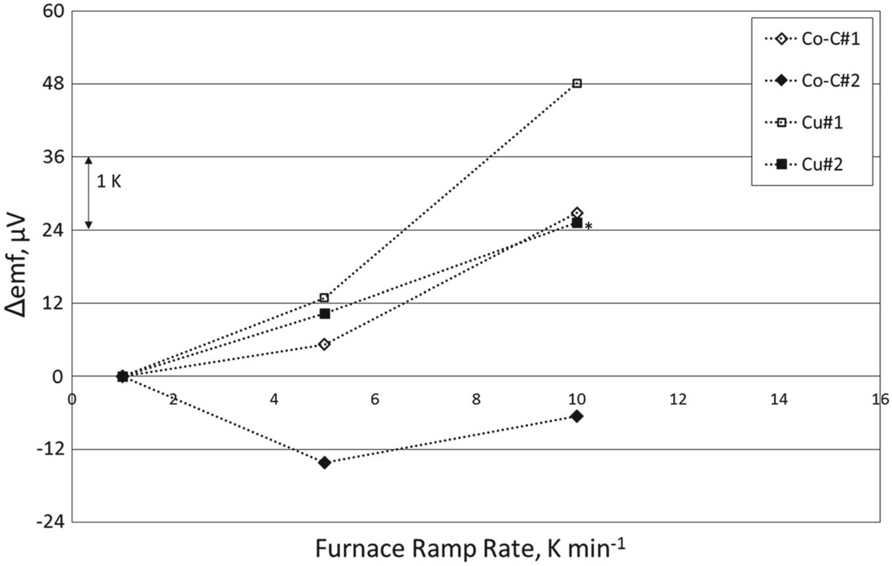

Fig. 5 The Cu and Co–C ramp rate sensitivity (melting emf shown as the difference from the value measured at 1 K/min)

Co–C thermocouple (which was essentially unaffected by the ramp rate, within the observed repeatability). This is most likely due to the furnace providing more heat than the cell could absorb into the melting process—and thereby leading to excess heating of the thermocouple elements at the hot junction. This effect will therefore need to be taken carefully into account in assessing the measurement uncertainty when the thermometers are in use.

4 Conclusions and Further Work

An integrated self-validating thermocouple, which incorporates a reference ingot into standard thermocouple dimensions has been designed and shown to operate successfully.

Clear melt and freeze plateaus are easily observable using the inseva design, lasting typically for 5 min. The apparent temperature is affected by the furnace temperature (with the melt consistently occurring higher in apparent temperature than the freeze), although not found to be sensitive to the offset furnace setting used. The measurement results are found to be different from the calibration temperature—which may be due to differing extents of thermal contact between the ingot and the thermoelements, and the poor immersion characteristic of this design. The recorded melting temperature is generally found to be sensitive to the ramp rate, increasing with ramp rate, which is likely to be due to increased direct heating of the thermocouple elements when the ramp rate is increased.

Further work is ongoing to extend the testing to additional conditions, including a more thorough repeatability assessment, horizontal operation (which is typical for industrial use), and fast ramp rates (quenching up to 100 K/min) to prove that the design is robust for use in industrial environments. The overall aim is to show this working in industrial trials, which is supported through the EMPRESS project.

For these thermocouples to be of value to industrial users, the repeatability of both the melt and freeze plateaus of the HTFP should be at least on a par with the tolerance of the thermocouple itself, which is ±1.0 °C (class I at 1084 °C) and ±1.7 °C (class I at 1324 °C). Therefore, it is very encouraging that this design repeatedly provides a repeatability of better than 1.0 °C in the tests described here—enabling *in-situ* validation of the temperature reading and monitoring of long-term drift. Further improvement to ensure robustness and longer term behavior is intended.

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