XIX IMEKO World Congress
Fundamental and Applied Metrology
September 6–11, 2009, Lisbon, Portugal

COMPARISON OF THERMOCOUPLE TEMPERATURE SCALES REALIZED BY FIXED-POINT AND RADIATION METHODS

Yong-Gyoo Kim, Inseok Yang and Yong Shim Yoo

Korea Research Institute of Standards and Science, Daejeon, Korea, dragon@kriss.re.kr

Abstract – We have developed a blackbody block system for comparing thermocouple temperature scales realized using fixed-point and radiation methods. Platinum/palladium, type S and type B thermocouples were calibrated using the freezing points of Ag, Cu, Fe-C, Co-C, and Pd (only for Type B). The radiation thermometer used was an LP4 linear pyrometer operated at a central wavelength of 650 nm. Two scales were compared from 962 to 1544 °C, and it was found that the two scales were consistent within 0.5 °C up to 1400 °C, but the discrepancy increased to 2.0 °C at 1544 °C for the type B thermocouple. In terms of the measurement uncertainty, the thermometric and radiometric scales realized at KRISS were in agreement up to the freezing temperature of Pd.

Keywords: thermocouple thermometry, radiation thermometry, fixed-point calibrations, eutectic temperature, scale comparison

1. INTRODUCTION

Dissemination of temperature standards through thermocouples is achieved by calibrating thermocouples at metal fixed points to obtain the best uncertainty. The pure-metal fixed-point cells normally used for such calibrations are: Pd, Cu, Ag, Al, Zn, Sn, and Ga. Thermocouples can be calibrated using these fixed points, and the deviation function can be interpolated. However, the temperature difference between the freezing point of Cu (1084.62 °C) and Pd (1544.8 °C) is about 470 K. To reduce the interpolation error in this temperature range, many metal–carbon eutectic points were studied in several National Metrology Institutions [1]. The behaviour of these deviation functions may vary for different thermocouples as well. In this study, we built a blackbody block system to compare thermocouple temperature scales calibrated using fixed points and a radiation thermometer in the temperature range 962 to 1544 °C, and verified the accuracy of our temperature scale and thermocouple reference functions. The comparison was carried out using type S and type B thermocouples, which are widely used in accurate temperature measurements, and a Pt/Pd thermocouple, a type that is gaining in importance in research into high temperature thermometry. Our aim was to improve temperature standards using thermocouple thermometry based on the results of our comparison

2. EXPERIMENTS

One type S thermocouple (designated as HSTC), one type B thermocouple (designated as HBTC), and two Pt/Pd thermocouples (designated as HPtPd_1 and HPtPd_2) were prepared for this work. The thermocouple wires had a diameter of 0.5 mm, and were purchased from the Heraeus Co. The Pt and Pd wire thermoelements were electrically annealed at 1450 °C and 1300 °C, respectively, for a period of 1 h, and then at approximately 400 °C overnight. The type B and type S thermoelements were both annealed electrically at 1700 and 1450 °C, respectively. After annealing, the thermoelements were inserted into high-purity twin-bore alumina insulators (length = 600 mm, diameter = 3 mm, and bore diameter = 0.8 mm), which were pre-baked at high temperatures. The measuring junction of the type S and Pt/Pd thermocouples were made by connecting the legs using a 0.1 mm diameter Pt wire to reduce the strain caused by the different degrees of thermal expansion. The type B thermocouple had a normal welded junction. After assembly, the thermocouples were annealed at 1000 °C for a period of 20 h in a vertical electric furnace.

The thermocouples were calibrated using the freezing points of Ag and Cu and at melting points of Fe-C and Co-C alloys [2, 3]. The type B thermocouple was also calibrated using the freezing point of Pd [4]. The melting temperatures of the Fe-C and Co-C alloy eutectics were 1153.67 [5] and 1324 °C [1], respectively. The reference junction was a normal ice–water mixture, and pure copper wires were connected to thermoelements. The thermal emf was measured using a nanovoltmeter (Keithley, Model 2182). The deviation emf was calculated by subtracting the reference emf from the measured emf at each fixed point. The reference functions used were obtained from ASTM standards [6, 7].

The radiation thermometer used in this work was an LP4 linear pyrometer (IKE, Germany) calibrated using the freezing point of Cu employing a 650 mm optical filter. The highest comparison temperature was limited to 1544 °C. The uncertainty of the calibration (k = 2) was 0.08 °C at the Cu point, which increased to 0.21 °C at 1544 °C.

Figure 1 shows a schematic diagram of the inter-comparison blackbody block made of high-purity graphite. The right-hand side of Fig. 1 shows the blackbody with a diameter = 15 mm and an aperture = 1.5 mm. The left-hand side shows a thermocouple well designed for a 7 mm outer

ISBN 978-963-88410-0-1

diameter protecting tube of a thermocouple. The thermocouple well was located below the tilted face of the blackbody, off-centre of the block. Originally, the block was designed for a 5 mm outer diameter platinum protecting tube. However, in preliminary experiments, the platinum became contaminated and melted because of unknown reactions that appeared to occur in the furnace. Therefore, an alumina protecting tube for the thermocouples was employed. The temperature profile of the horizontal furnace used for the comparison was within ±0.3 °C over the 100 mm length of the blackbody block. A small volume of high-purity argon gas was used during the experiments to prevent oxidation of the graphite.

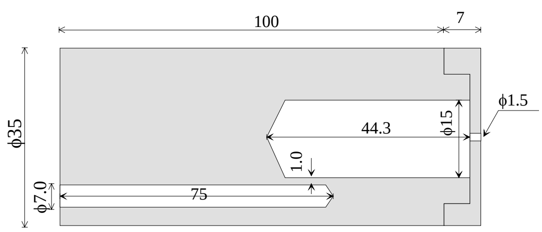

Fig. 1. A schematic diagram of the inter-comparison blackbody block. All dimensions shown are in mm.

3. RESULTS

Before comparison, the thermocouples were calibrated using various high-temperature fixed points. Table 1 shows the results of the fixed-point measurements. Calibrations were carried from high to low temperature in sequence. A vertical furnace with Kanthal superheaters was used to obtain temperatures above the Cu point, and a sodium heat-pipe furnace was used to reach the Ag and Cu points. Figure 2 shows the deviation emf and interpolating curves with temperature for each thermocouple. The deviation curves were smooth, and thus, it was inferred that the fixed-point calibrations had been carried out properly.

Table 1. Thermal emf measured at various fixed points.

Fixed points	Temperature /°C	Measured emfs /μV			
		HPtPd_1	HPtPd_2	HSTC	HBTC
Ag	961.78	10783.2	10780.9	9154.5	4479.3
Cu	1084.62	13242.9	13240.3	10581.0	5619.0
Fe-C	1153.67	14714.4	14714.4	11401.3	6303.3
Co-C	1324	18574.1	18571.9	13455.4	8100.3
Pd	1554.8	-	-	-	10735.0

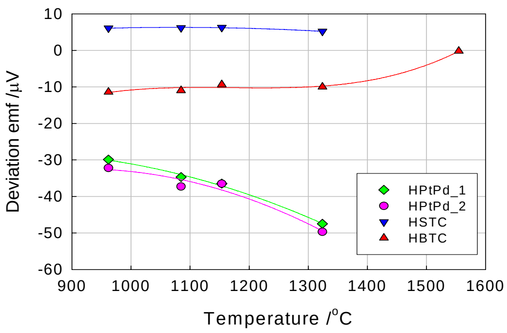

Fig.2 Deviation emf and curves for each thermocouple.

Table 2. Comparison of results at various temperatures.

Temperature /°C	Measured emfs /μV			
	HPtPd_1	HPtPd_2	HSTC	HBTC
961.78	10780.9	10779.7	9152.5	4475.1
1084.62	13240.6	13239.6	10579.5	5614.8
1153.67	14710.5	14709.2	11398.56	62998.3
1250	16854.7	16853.5	12557.2	7296.7
1324	18570.7	18569.3	13454.5	8095.1
1400	20391.6	20389.9	14377.1	-
1450	-	-	-	9503.7
1544	-	-	-	10584.3

After the fixed-point measurements, the thermocouples were installed in the horizontal blackbody comparison system. The comparisons were performed from 960 to 1544 °C, near to the temperatures listed in Table 2. For the Pt/Pd and type S thermocouples, the highest comparison temperature was limited to 1400 °C, which is the maximum temperature of continuous usage. In case of the type B thermocouples, the calibration temperature of 1400 °C was replaced by 1450 °C, and the highest temperature used was 1544 °C. Because the furnace set temperatures did not exactly coincide with the fixed-point temperatures shown in Table 2, the actual measured temperature values were corrected to the freezing and melting temperatures for comparison with the fixed-point measurement results. The furnace was set to remain at the calibration points for a period of 1 h while the radiation thermometer and the thermocouple readings were obtained. The comparison tests were performed three times. Table 2 shows a comparison of the results of calibration of thermocouples with the radiation thermometer data.

Figure 3 shows the results of calibration compared with the radiation thermometer data. The results of the fixed-point calibration are also shown for comparison. In Fig. 3, the error bars denote the expanded calibration uncertainty for k = 2. The closed circles show the comparison results with the radiation thermometer. The diamond, open circle, reverse triangle, and triangle symbols denote the measurement data at the various fixed points. The solid lines denote the least-square interpolation fits for the comparison results. The Pt/Pd thermocouples showed gradual decrease in the deviation emf values with increasing temperature, and it was found that these thermocouples had a very small difference between the two calibration methods in the temperature range 962 to 1400 °C. The type S thermocouple also showed a good agreement over the entire comparison temperature range. In the case of the type B thermocouple, the two scales were nearly same up to 1320 °C, but the discrepancy increased as the temperature increased. As shown in Fig. 3(d), the variation in the deviation emf of the type B thermocouple in the fixed-point calibration increased with increasing temperature, but the comparison results showed a slightly decreasing behaviour above 1320 °C. At 1554 °C, the scale difference was 2.0 °C for the type B thermocouple.

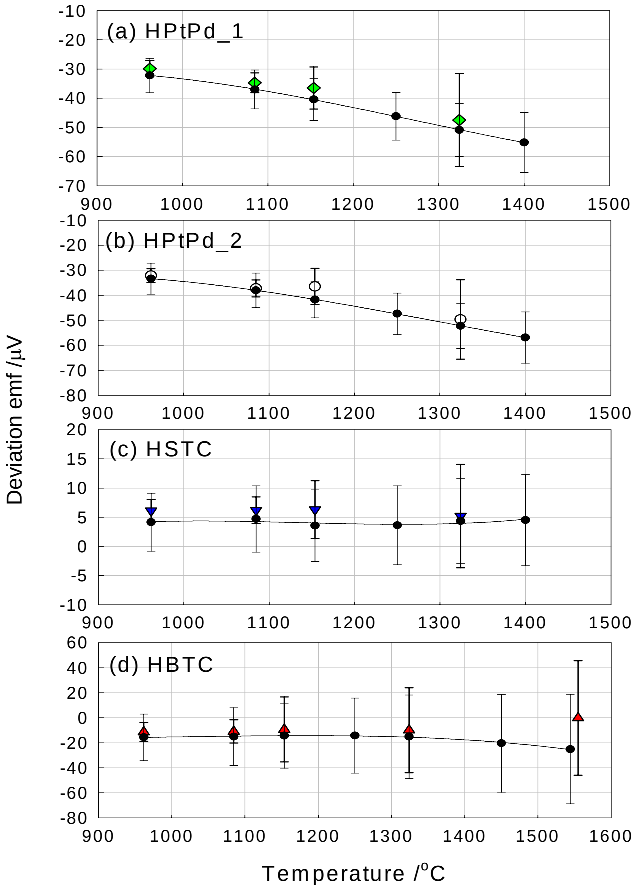

Fig.3 Variation in the deviation emf for each thermocouple. The results from the fixed-point calibrations are also shown.

4. DISCUSSION

Table 3 shows the difference in temperature between fixed-point calibration and a comparison with radiation pyrometer data. For the Pt/Pd thermocouples, the difference was small, < 0.2 °C, over the entire comparison range. The type S thermocouple also showed a small temperature difference as well. In the case of the type B thermocouple, the two scales agreed within 0.5 °C up to a temperature of 1324 °C, but the discrepancy increased to 2.0 °C at 1544 °C.

Table 3. Temperature difference between the fixed-point calibration and the comparison calibration at each temperature.

Temperature /°C	Temperature difference /°C			
	HPtPd_1	HPtPd_2	HSTC	HBTC
961.78	0.07	- 0.01	0.10	0.48
1084.62	0.07	- 0.03	0.15	0.41
1153.67	0.15	0.20	0.23	0.47
1250	0.15	0.13	0.17	0.33
1324	0.15	0.09	0.06	0.44
1400	0.09	0.02	- 0.08	-
1450	-	-	-	1.15
1544	-	-	-	2.00

Table 4. Uncertainty budgets for the fixed-point calibration at the Cu freezing temperature for each type of thermocouple.

Factors	Standard uncertainties /μV		
	Pt/Pd	S	B
Reference temperature			
(1) Fixed-point temperature	0.12	0.07	0.06
(2) Plateau determination	0.12	0.07	0.06
Emf measurement			
(1) Inhomogeneity	1.63	1.08	4.62
(2) Reproducibility	0.21	0.12	0.10
(3) Ice point	0.17	0.10	0.08
DVM calibration and stability			
(1) Calibration uncertainty	0.08	0.06	0.03
(2) Stability	0.33	0.27	0.14
Expanded uncertainty /μV(*k*=2)	3.39	2.26	9.25

To validate this discrepancy, the measurement uncertainty of the two scales was calculated, and this was plotted in Fig. 3. Table 4 shows the uncertainty budgets for the fixed-point calibration. For simplicity, the uncertainty analysis is shown at the Cu freezing temperature as representative data for each type of thermocouple. To calculate the measurement uncertainty of a thermocouple, it is essential to perform an inhomogeneity test of the thermocouple. In our work, inhomogeneity scanning tests were performed using a liquid bath operating at 180 °C [8]. The thermocouples were scanned before the fixed-point calibration and after comparison with the radiation thermometer. The inhomogeneity curves before and after measurements over the entire temperature range were nearly the same. Figure 4 shows the inhomogeneity results after the comparison.

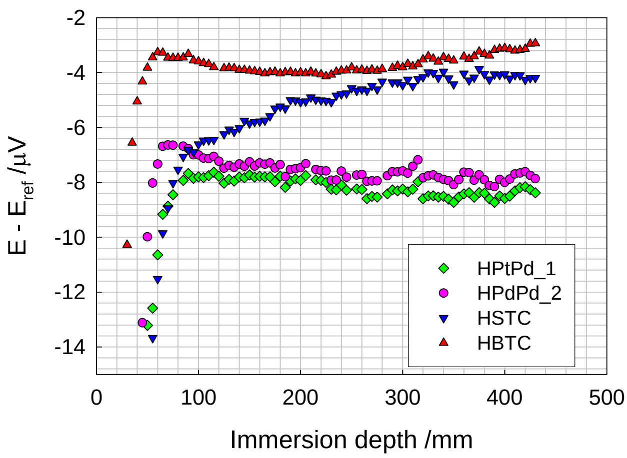

Fig. 4 Inhomogeneity results after comparison with the radiation thermometer. The terms E and E_{ref} indicate the measured and reference emf at 180 °C for each thermocouple, respectively.

The Pt/Pd thermocouples showed a smaller variation with immersion depth than the other thermocouples did. The uncertainty factor due to the thermoelectric inhomogeneity was calculated by considering the temperature gradient of the furnace and the inhomogeneity scan as a function of the immersion depth [9].

The uncertainty analysis method used of comparing the calibration with the radiation thermometer data is similar to a fixed-point calibration except for the reference temperature determination. The uncertainty of radiation thermometer LP4 and the uncertainty caused by alignment errors were factors affecting the reference temperature. Finally, it was found that the uncertainty of the comparison calibration was larger than that of the fixed-point calibration, as shown in Fig. 3.

When considering the calibration uncertainty, it should be noted that the temperature scale of a thermocouple using fixed-point calibration and comparison with the radiation thermometer were within the uncertainty, even though the temperature difference was as large as 2.0 °C for the type B thermocouple at 1544 °C. It is thought that this large difference was caused by an error in determining the freezing temperature of Pd realized in our laboratory. Therefore, there is a need for further study on the determination of the freezing temperature of Pd.

Furthermore, in this work, the melting temperature of the Fe-C alloy was assigned to 1153.67 °C, which was determined from a work on thermocouple thermometry by Edler [5]. However, the melting temperature of Fe-C in the literature varies from 1153 [1] to 1154.17 °C [10]. From this study, our Fe-C eutectic cell would be close to the melting temperature of Edler’s result. In the near future, we intend to fabricate radiometric Fe-C cells and measure the melting temperature using the same materials used in the cells for the thermocouple thermometry.

5. CONCLUSIONS

The temperature scale of thermocouples realized using a fixed-point calibration was compared with a radiometric temperature scale realized from calibration at the freezing temperature of Cu using a blackbody comparison block system. Two Pt/Pd, one type S, and one type B thermocouple were compared in a horizontal furnace in the temperature range 962 to 1554 °C. From 962 to 1400 °C, the two temperature scales agreed within 0.2 °C for the Pt/Pd thermocouples, and 0.3 °C for the type S thermocouple. The type B thermocouple showed agreement within 0.5 °C up to 1324 °C, but the temperature difference between the two scales increased as the temperature increased. At 1554 °C, the discrepancy of the type B thermocouple was as high as 2.0 °C. However, this temperature discrepancy was less than the measurement uncertainty over all the temperature range. Therefore, it is concluded that the temperature scale realized by the thermocouples from 962 to 1554 °C agreed well with the ITS-90 value realized by a radiation thermometer in our laboratory.

REFERENCES

[1] E. R. Woolliams, G. Machin, D. H. Lowe and R. Winkler, “Metal (carbide)-carbon eutectics for thermometry and radiometry: a review of the first seven years”, Metrologia, vol, 43, pp. R11-R25, 2006

[2] Y.-G. Kim, I. Yang, S. Y. Kwon and K. S. Gam, “Features of Co/C and NI/C eutectic transitions for use in thermocouple thermometry”, Metrologia, vol. 43, pp.67-70, 2006

[3] I. Yang, Y.-G. Kim and K. S. Gam, “The dependence of the melting temperatures of the eutectics Fe-C and Co-C, designed for thermocouple thermometry, on the furnace offset temperature during the preceding freeze”, Meas. Sci. Technol. vol. 19, p. 015106, 2008

[4] Y.-G. Kim, K. S. Gam and K. H. Kang, “Realization of the palladium freezing point for thermocouple calibrations”, *Metrologia,* vol. 36, pp. 465-472, 1999

[5] E. Edler, P. Ederer, A. C. Baratto and H. D. Vieira, “Melting temperature of eutectic fixed-point cells usable for the calibration of contact thermometers”, Int. J. Thermophys., vol. 28, pp. 1983-1992, 2007

[6] ASTM Standard E 230-03, Standard specification and temperature-electromotive force (EMF) tables for standardized thermocouples, ASTM International, West Conshohocken, PA, 2003

[7] ASTM Standard E 1751-00, Standard Guide for temperature electromotive force (EMF) tables for non-letter designated thermocouple combinations, ASTM International, West Conshohocken, PA, 2000

[8] Y.-G. Kim, I. Yang, and K. S. Gam, “Measurement of the inhomogeneity of Pt/Pd thermocouples using an immersion-into-liquid method”, Instru. Sci. Technol., vol. 36, pp. 257-266, 2008

[9] Y.-G. Kim, C. H. Song, K. S. Gam and I. Yang, “Change in inhomogeneity with temperature between 180 ^{o}C to 950 ^{o}C in base-metal thermocouples”, Meas. Sci. Technol., vol. 20, p.075102, 2009

[10] P. Bloembergen, Y. Yamada, N. Sasajima, Y. Wang and T. Wang, “The effect of the eutectic structure and the residual effect of impurities on the uncertainty in the eutectic temperatures of Fe-C and Co-C”, Metrologia, vol. 44, pp. 279-293, 2007