

Construction and Investigation of Pt/Pd Thermocouples in the Framework of the Euromet Project 857

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Published online: 29 January 2008
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Abstract The objective of the EUROMET Project 857 in the field of thermometry, “High-temperature fixed points for improved thermocouple calibrations,” is the development of robust high-temperature fixed points based on metal-carbon eutectic alloys for the calibration of thermocouples above 1,084°C. This paper describes the construction and investigation of Pt/Pd thermocouples to be used to compare different cobalt-carbon (Co-C) fixed-point cells constructed by the three participants of this project. A set of three Pt/Pd thermocouples was prepared by PTB and NPL and a set of four by LNE. Their metrological performances in terms of thermoelectric stability and homogeneity were assessed in different ways. The results of these investigations, as well as the results of first measurements of local Co-C eutectic fixed-point cells by using Pt/Pd thermocouples, are presented.

Keywords Cobalt-carbon eutectic · Melting temperature · Pt/Pd thermocouple

1 Introduction

Research has been undertaken to develop novel high-temperature fixed points based on metal-carbon eutectic alloys. These new fixed points were constructed mainly for non-contact applications because of their high-melting temperatures (up to 3,185°C, HfC-C) [1–4]. Recently, their potential for the calibration of contact thermometers,

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especially of thermocouples, was recognized [5–8]. The actual calibration uncertainty of thermocouples at temperatures higher than the freezing point of copper (1,084.62°C) rises from about ± 0.3 K at 1,100°C to about ± 1.5 K at 1,550°C [9], due to the absence of reliable high-temperature fixed points in this temperature range. For some industrial applications, this is unacceptably large and there is a strong need to reduce this uncertainty by at least a factor of two.

The melting temperature of the Co–C eutectic of about 1,324°C is midway between the freezing point of copper and the upper limit of use of Pt/Pd thermocouples at about 1,500°C, which are considered to be the most stable and reliable thermocouples for measurement of temperatures up to this limit. Therefore, Pt/Pd thermocouples are ideally suited to the investigation of Co–C eutectic fixed-point cells designed for measurements using contact thermometers and to compare their melting temperatures, provided that the thermoelectric performance, specifically the stability and homogeneity, of the chosen thermocouples are demonstrably sufficient, i.e., of the order of magnitude of about 0.1 K or better.

2 Pt/Pd Thermocouples

2.1 Material

The platinum and palladium wires of 0.5 mm diameter used to construct the thermocouples were supplied by Alfa Aesar. The nominal purities of the wires were 99.997% and 99.99+% (metals' basis) for the Pt- and Pd-wires, respectively. Besides the certificates of analysis provided by Alfa Aesar, two further independent impurity analyses of the thermoelements were performed using glow discharge mass spectroscopy (GDMS) by H.C. Starck GmbH and AQura GmbH. The results of the three analyses are listed in Table 1. The two additional analyses indicate lower purities of the thermoelements than stated by the supplier, Alfa Aesar. Furthermore, it should be mentioned that both GDMS analyses of the external service providers agree better with each other than with the results of the supplier. Nevertheless, some strong discrepancies of the impurity concentrations were found in the different analyses, for instance, for Si in Pd (92 ppm, Alfa Aesar) and for In in Pt (30 ppm, AQura). The total concentration of impurities in the thermoelements can be estimated from the three analyses to be about 60 ppm (mass fraction) in the Pt wire and about 170 ppm (mass fraction) in the Pd wire. This is about a factor of two higher than that stated by the supplier, but it should be kept in mind that the uncertainty of the GDMS analyses is at least a factor of two.

2.2 Construction of the Pt/Pd Thermocouples

2.2.1 Procedure at PTB

Three Pt/Pd thermocouples designated as Pt/Pd-PTB-01/06, Pt/Pd-PTB-02/06, and Pt/Pd-PTB-03/06 were constructed from the pure platinum and palladium wires described above. The thermoelements were cleaned using ethyl alcohol and then distilled

Table 1 Results of impurity analyses of the platinum and palladium thermoelements in ppm (mass fraction) (bold characters are the main impurities)

| Element | Platinum | | | Palladium | | |
|---------|------------|-------------|------------|------------|-------------|------------|
| | Alfa Aesar | H.C. Starck | AQura | Alfa Aesar | H.C. Starck | AQura |
| Ag | <1 | 0.85 | 1.1 | <1 | 14 | 6.1 |
| Al | <1 | 5 | 6.5 | 8 | 2.5 | 3.2 |
| Au | – | – | <0.3 | <1 | – | 5.4 |
| C | – | – | <2.8 | – | – | <8.3 |
| Ca | <1 | 0.25 | 1 | 3 | 1.8 | 4.9 |
| Cr | <1 | 0.26 | 0.8 | <1 | 0.49 | 1.3 |
| Cu | 7 | 2 | 4.4 | <1 | 40 | 31 |
| Fe | 1 | 12 | 24 | 12 | 39 | 44 |
| In | <1 | <0.05 | 31 | – | <0.05 | 10 |
| Ir | 2 | 4 | 4.7 | <3 | 1.4 | 1.4 |
| Mg | <1 | 0.06 | 0.2 | 12 | 45 | 62 |
| Mn | <1 | 0.12 | 0.3 | <1 | 2.3 | 1.2 |
| Na | – | – | 1.6 | – | – | 1.9 |
| Ni | <1 | 0.24 | 0.9 | <1 | 1.8 | 0.9 |
| O | – | – | <5.6 | – | – | <4.5 |
| Pd | 10 | 4 | 9.5 | – | – | – |
| Pt | – | – | – | <2 | – | 21 |
| Rh | 7 | 7 | 15 | 5 | 5 | 7.9 |
| Ru | – | 2 | 3.5 | <1 | 1.5 | 3.8 |
| Sb | – | <0.05 | <0.05 | 9 | 0.33 | 0.09 |
| Si | <1 | 0.81 | 2.6 | 92 | 5 | 2.6 |
| W | <30 | <0.05 | 0.04 | – | 1.2 | 0.7 |
| Zn | <10 | 0.4 | 0.6 | 1 | 3 | 0.9 |
| Zr | <1 | 1.6 | 2.5 | – | 0.08 | 0.2 |
| | 27 | 40.6 | 110 | 142 | 164.4 | 210.5 |

water. The Pd wires were annealed electrically in air for 10 h at about 1,300°C (5 h for Pt/Pd-PTB-02/06) and the Pt wires for 10 h at about 1,300°C (5 h for Pt/Pd-PTB-02/06) and for 30 min at about 750°C [10]. They were cooled to ambient temperature by switching off the current. Afterward, the wires (length: 2,100 mm) were inserted into a twin-bore capillary tube (outer diameter: 4 mm, bores: 1.2 mm, length: 710 mm) protected by a one-end-closed protection tube (diameter: 7 × 5 mm, length: 700 mm), both made of pure alumina (Al₂O₃: 99.7%). The sections of the thermoelements from the measuring junction over a length of about 850 mm underwent a further furnace annealing for 3 h at a temperature of 1,100°C. The measuring junctions of the three thermocouples were prepared in different ways: the thermoelements of Pt/Pd-PTB-01/06 were welded directly, the thermoelements of Pt/Pd-PTB-02/06 were welded in the usual way using a four-turn coil of 0.2 mm Pt wire, and the Pt thermoelement of Pt/Pd-PTB-03/06 was bent into a U-shape and welded directly to the Pd thermoelement so that the measuring junction was located some mm inside one hole of the capillary tube. The thermoelements emerging from the alumina tubes were insulated with flexible plastic tubes and soldered to a pair of Cu wires of 0.3 mm diameter to form the reference junction in a tube made of aluminum.

2.2.2 Procedure at LNE

Two Pt/Pd thermocouples designated Pt/Pd-LNE-3 and Pt/Pd-LNE-4 were constructed from the pure platinum and palladium wires described above. Two other Pt/Pd thermocouples designated Pt/Pd-LNE-1 and Pt/Pd-LNE-2 were constructed from pure platinum and palladium wires drawn from the existing stock of LNE (wires were supplied by Engelhard-CLAL in 2001). All thermoelements were cleaned with a cloth and then in an ultrasonic bath for 15 min using high-purity ethyl alcohol. The wires were removed from the bath and carefully cleaned using a dry cloth. Both Pd- and Pt-wires were annealed electrically in air for 10 h at about 1,300°C and 1 h at about 450°C. All wires were cooled to ambient temperature by switching off the current. One of the Pd wires (Alfa Aesar) broke halfway after a few hours annealing. The cause could not be identified. It was replaced by a new wire prepared in the same way. The wires were inserted in a twin-bore capillary tube protected by a one-end-closed protection tube as described above. Afterward, the measuring junctions of all thermocouples were welded using a four-turn coil of 0.2 mm Pt wire. The construction and initial annealing of the thermocouples were performed in two different ways. The thermocouples Pt/Pd-LNE-1 and Pt/Pd-LNE-4 were annealed in a furnace at a temperature of 1,100°C for 3 h over a length of about 400 mm starting from the measuring junction; the thermocouples Pt/Pd-LNE-2 and Pt/Pd-LNE-3 were annealed in another furnace over a length of about 1,000 mm starting from the measuring junction. The thermoelements emerging from the alumina tubes were insulated with flexible plastic tubes, soldered to a pair of Cu wires to form the reference junction, and inserted in a glass tube (outside diameter: 7 mm, length: 350 mm).

2.2.3 Procedure at NPL

Three Pt/Pd thermocouples designated NPL/PtPd1, NPL/PtPd2, and NPL/PtPd3 were constructed using pure platinum and palladium wires as described above. After cleaning the thermoelements with ethyl alcohol and distilled water, the wires of NPL/PtPd1 and NPL/PtPd3 were subjected to electrical annealing for 8 h at approximately 1,300°C, with the Pt subjected to an additional 30 min at 750°C. Both wires were then annealed at 450°C for 30 min before being cooled to ambient temperature by switching off the current. The wires of NPL/PtPd2 were subjected to electrical annealing at 1,300°C for 100 h. After 100 h, the Pd thermoelement broke halfway along its length. After the electrical annealing, the wires, of length 2,000 mm (except NPL/PtPd2, with a length of 1,100 mm), were then inserted in a twin-bore capillary tube (outer diameter: 4 mm, bores: 1.2 mm, length: 710 mm) protected by a one-end-closed protection tube (diameter: 7 × 5 mm, length: 700 mm), both made of pure alumina (Al₂O₃: 99.7%). The sections of the thermoelements 1,000 mm from the measuring junction were annealed at 1,100°C in a horizontal tube furnace for a further 3 h. All three measuring junctions were constructed in the same way, by welding the two thermoelements directly to each other, then pushing the junction inside the bore containing the Pd leg by a distance of 10 mm from the end of the twin-bore tube. The thermoelements emerging from the tubes were insulated with flexible plastic tubes and twisted together with a pair of Cu wires of diameter 0.3 mm to form the reference junction. The insulated wires were

Table 2 LNE platinum/palladium thermocouples

| Thermocouple | PtPd-LNE-1 | PtPd-LNE-2 | PtPd-LNE-3 | PtPd-LNE-4 |
|---|---------------------|---------------------|---------------------|---------------------|
| Wires | Engelhard-CLAL | Engelhard-CLAL | Alfa-Aesar | Alfa-Aesar |
| Annealed length at 1,030°C | 400 mm | 1,000 mm | 1,000 mm | 400 mm |
| First measurement at the silver point after initial annealing of 3 h at 1,100°C and then 100 h at 1,030°C | | | | |
| emf | 10793.5 μ V | 10784.1 μ V | 10816.4 μ V | 10812.0 μ V |
| Immersion profiles ^a | 0.4 μ V (21 mK) | 0.6 μ V (31 mK) | 0.6 μ V (31 mK) | 1.4 μ V (73 mK) |
| Second measurement at the silver point after further annealing for 100 h at 1,030°C | | | | |
| Immersion profiles ^a | / | 0.3 μ V (16 mK) | / | / |
| Third measurement at the silver point after annealing for 140 h at 1,340°C | | | | |
| Immersion profiles ^a | 1.0 μ V (52 mK) | 1.2 μ V (62 mK) | 0.8 μ V (42 mK) | 1.6 μ V (83 mK) |

^a Maximum emf variations of immersion profiles in the LNE silver-point cell

further insulated by enclosing them inside a flexible plastic tube, of diameter 8 mm, up to approximately 300 mm from the reference junction. The reference junction was then inserted into a glass tube of length 300 mm.

2.3 Annealing of the Pt/Pd Thermocouples

After complete mounting of the thermocouples, furnace annealing was performed in the three laboratories to remove any physical defects introduced during the assembly and to improve the stability and homogeneity of the thermoelements. The annealing was performed at temperatures of about 1,340°C, slightly above the melting temperature of the Co–C eutectic (1,324°C) and additionally at about 1,030°C (PTB and LNE) to improve the homogeneity of the thermocouples [11]. The annealing was performed over a length of about 500 mm from the measuring junction in horizontal tubular furnaces whose temperature homogeneity of the hot zones—at PTB and NPL—was about ± 3 K. The NPL/PtPd1 thermocouple was annealed in a vertical three-zone tube furnace over a length of about 450 mm. At LNE, the thermocouples were annealed in two horizontal furnaces over different lengths (see Table 2) with the temperature homogeneity of the hot zones being about ± 16 K. After annealing, the thermocouples were removed from the furnace slowly (1–2 min) without performing additional vacancy annealing. Annealing periods of different durations were applied. In between, the emfs of the Pt/Pd thermocouples were measured at the freezing point of silver.

3 Stability and Homogeneity of the Pt/Pd Thermocouples

3.1 Investigations at PTB

The thermoelectric stability and homogeneity of the three Pt/Pd thermocouples were checked by repeated measurements at the freezing point of silver. The results are

presented in Fig. 1a. After 60 h annealing at 1,340°C, the emf at the freezing point of silver was measured for the first time. Starting from this value, a total increase of the emfs between 0.5 and 1 μV was observed during two further 40-h annealing periods at 1,340°C, but of decreasing amounts. This corresponds to a temperature equivalence of about 25–50 mK. During the next annealing step, a failure of the power supply of the furnace caused a slow decrease of the furnace temperature overnight and a fast increase to 1,340°C again. As a result of this unregulated annealing, a change of the emf at the freezing point of silver of about 0.5 μV was observed. The effect of the further annealing steps at temperatures of 1,030 and 1,340°C can be summarized as follows: an annealing at a temperature of 1,030°C, applied to improve the homogeneity of the thermoelements, caused an increase of the emf at the freezing point of silver by about (1–2) μV , and an annealing at 1,340°C caused a decrease of the emf by about the same value. The expanded uncertainty of the repeated measurements at the freezing point of silver was about 1.3 μV .

The homogeneity of the three Pt/Pd thermocouples was tested by measuring immersion profiles over a length of about 10 cm at the freezing point of silver. The maximum emf variations (and corresponding temperature equivalents) found after about 340 h total annealing time at 1,340 and 1,030°C are listed below:

Pt/Pd-PTB-01/06 : 1.2 μV (62 mK)

Pt/Pd-PTB-02/06 : 1.0 μV (52 mK)

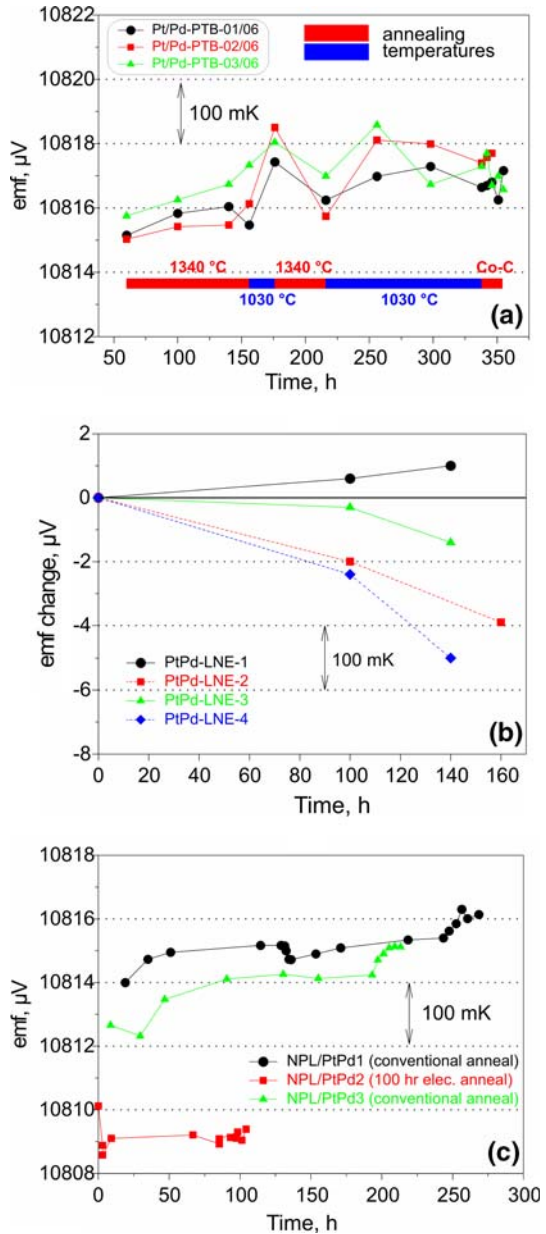
Pt/Pd-PTB-03/06 : 1.3 μV (68 mK)

It should be noted that the different measuring junctions had no significant influence on the thermoelectric stability and homogeneity of the thermocouples.

3.2 Investigations at LNE

After 100 h annealing at 1,030°C, the thermocouples were measured at the freezing point of silver. The two thermocouples constructed from the same wire lot, but annealed over a different length, showed a difference in emf of $-9.4 \mu\text{V}$ (Engelhard-CLAL wires) and $+4.4 \mu\text{V}$ (Alfa Aesar wires), as shown in Table 2. The measuring junction of the Pt/Pd-LNE-2 thermocouple was damaged when measurements are carried out at the freezing point of silver. Its thermoelectric homogeneity being affected, the measuring junction was replaced and a further 100 h annealing at 1,030°C was performed over 400 mm. The maximum emf variations of the immersion profiles over a length of 8 cm measured at the freezing point of silver before and after 140 h annealing at 1,340°C are also given in Table 2. The first annealing at 1,340°C was performed over 100 h. The second annealing of 40–60 h led to a change in the emf of $+0.4 \mu\text{V}$ ($+21 \text{ mK}$) for PtPd-LNE-1 thermocouple, $-1.1 \mu\text{V}$ (-57 mK) for PtPd-LNE-3 thermocouple, $-1.9 \mu\text{V}$ (-99 mK) for PtPd-LNE-2 thermocouple, and

Fig. 1 (a) Thermoelectric stability of the Pt/Pd thermocouples PTB-01/06, PTB-02/06, and PTB-03/06: measured emfs at the freezing point of silver, (b) thermoelectric stability (after annealing at 1,340°C) of the four Pt/Pd thermocouples of LNE: measured emfs at the freezing point of silver, and (c) thermoelectric stability of the Pt/Pd thermocouples NPL/PtPd1, NPL/PtPd2, and NPL/PtPd3: measured emfs at the freezing point of silver. Furnace annealing performed at 1,350°C up to the indicated measurements during tests at the Co–C point. ‘Co–C’ indicates measurements at the silver point in between measurements at the Co–C point



–2.6 μV (–135 mK) for PtPd-LNE-4 thermocouple (Fig. 1b). The lower stability observed for the PtPd-LNE-2 thermocouple can be explained by the problem encountered during the earliest measurements at the freezing point of silver. The behavior of PtPd-LNE-4 thermocouple is under assessment.

Although based on a limited number of thermocouples, measurements at the freezing point of silver after the first annealing at 1,030°C showed the influence of the annealing furnace and wire source on the thermocouples' initial performances. As a result of the annealing steps at 1,340°C, the good performances of PtPd-LNE-1 and PtPd-LNE-3 thermocouples in terms of thermoelectric stability and homogeneity will allow the assessment at LNE of the cobalt-carbon eutectic cells.

3.3 Investigations at NPL

The emf of each thermocouple was measured at regular intervals at the freezing point of silver. The results are presented in Fig. 1c. For NPL/PtPd1 and NPL/PtPd3, an annealing period of 150 h at 1,340°C resulted in an increase of emf of approximately 2 μV at the Ag point, corresponding to a temperature difference of 100 mK. Subsequently, the thermocouples were stable to within approximately 10 mK. The annealing of NPL/PtPd2 (subjected to 100 h electrical annealing beforehand) shows stability within 20 mK after only approximately 10 h annealing at 1,340°C. The lower emf (by about 5 μV) measured with NPL/PtPd2 is probably due to the longer time of electrical annealing.

The homogeneity of the three thermocouples was also tested at the freezing point of silver by measuring immersion profiles over a length of approximately 10 cm. The maximum emf variations after 200 h for NPL/PtPd1 and NPL/PtPd3, and 100 h for NPL/PtPd2, are as follows:

NPL/PtPd1: 3.9 μV (200 mK)

NPL/PtPd2: 2.3 μV (120 mK)

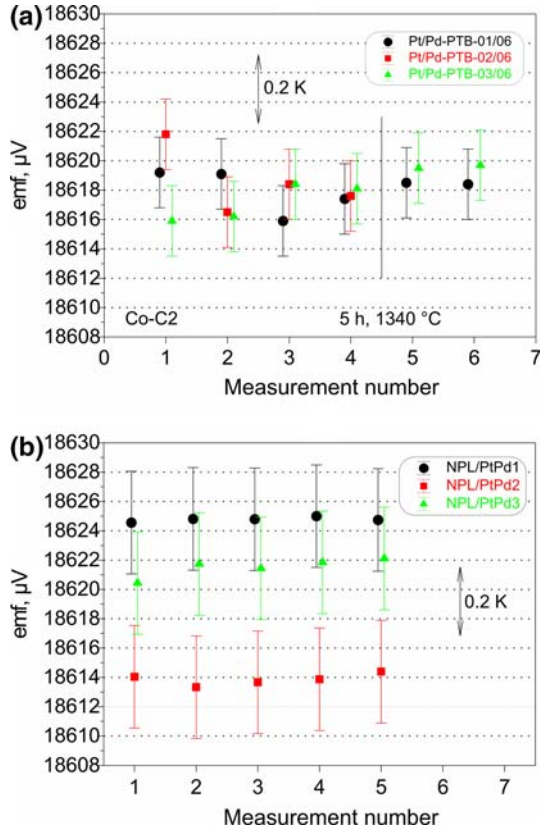
NPL/PtPd3: 3.9 μV (200 mK)

4 First Measurements of Co–C Eutectics

4.1 Measurements at PTB

The emfs measured at the melting point of the eutectic fixed-point cell Co–C2 of PTB are shown in Fig. 2a. After the 4th measurement of each Pt/Pd thermocouple, additional annealing for 5 h at a temperature of 1,340°C was performed. The Pt/Pd-PTB-02/06 thermocouple failed after this short-duration annealing caused by the breakage of the Pd thermoelement near the measuring junction (Pt-coil). No significant drift in the emf at the melting point of the Co–C eutectic was found using Pt/Pd-PTB-01/06, and only a slight increase in the emf of Pt/Pd-PTB-03/06 was observed. The changes in the measured emfs were almost always within the expanded uncertainties of about $\pm 2.4 \mu\text{V}$

Fig. 2 (a) Measured emfs at the melting point of the local cobalt-carbon eutectic Co–C2 of PTB with expanded uncertainties ($k = 2$) and (b) measured emfs at the melting point of the local cobalt-carbon eutectic NPL/Co–C1 of NPL with expanded uncertainties ($k = 2$)



(± 0.1 K) of a single measurement. Therefore, the Pt/Pd-PTB-01/06 and Pt/Pd-PTB-03/06 thermocouples are considered to be thermoelectrically stable, which was also shown by the repeated emf measurements at the freezing point of silver.

4.2 Measurements at NPL

The emf measurements at the melting point of the Co–C eutectic are shown in Fig. 2b. No significant drift in the emfs was found. No additional annealing was performed during these tests. After each measurement at the Co–C fixed point, the emf was measured at the freezing point of silver. Use of the NPL/PtPd1 and NPL/PtPd3 thermocouples at the Co–C eutectic resulted in a small drift (50 mK) at the freezing point of silver which stabilized after approximately five measurements at the Co–C point. These thermocouples can therefore be considered thermoelectrically stable. The slight increase of the emf measured at the freezing point of Ag is probably caused by a change of homogeneity of the thermocouples over their length during exposure to the high-temperature furnace used to realize the melting points of the Co–C eutectic.

We note particularly the high stability of NPL/PtPd2, which was subjected to 100 h electrical annealing prior to the conventional furnace annealing. This thermocouple

exhibits stability comparable to the other conventionally annealed thermocouples and does not exhibit the small drift of 50 mK at the freezing point of silver after use at the Co–C fixed point that is observed with NPL/PtPd1 and NPL/PtPd3. Furthermore, it is a factor of two more homogeneous than the other two conventionally annealed thermocouples.

5 Summary

The thermoelectric stability of the 10 Pt/Pd thermocouples, constructed and investigated in the three participating laboratories and exposed to temperatures up to about 1,350°C, was found to be within the temperature equivalent of about 50 mK, measured at the freezing point of silver (with the exception of Pt/Pd-LNE-2 and Pt/Pd-LNE-4). This thermoelectric stability was about a factor of two worse than observed after 250 h annealing of Pt/Pd thermocouples at 1,100°C [12]. The thermocouples that have undergone different annealing schedules generated significantly different emfs (at NPL and LNE), whereas those that have undergone similar annealing generated similar emfs (at NPL and PTB). These results suggest that annealing conditions at a given temperature influence thermocouple characteristics. The Pt/Pd thermocouples of PTB and LNE are characterized by better homogeneity (factor of 2–3) than the NPL thermocouples, probably due to the additional annealing at 1,030°C at PTB and LNE. Nevertheless, the homogeneity of most of these thermocouples was a factor of two worse than the homogeneities observed after annealing Pt/Pd thermocouples at only 1,030°C [11], possibly due to different thermoelement materials and/or to the previous higher annealing temperature of 1,340°C in this work. Furthermore, the annealing at 1,030°C caused a reversible change of the emf by about 1–2 μV, measurable at the freezing point of silver.

The thermocouples of PTB and NPL assessed up to now at the melting points of local Co–C eutectics after finishing the annealing process in the laboratories (PTB: 340 h at 1,340 and 1,030°C, NPL: approx. 200 h at 1,350°C) caused no further significant changes of the emf measured at the freezing point of silver. The thermoelectric homogeneities of the best of the investigated Pt/Pd thermocouples are within temperature equivalents of about 50 mK. It should be mentioned that annealing of the Pt/Pd thermocouples for a few hours at 1,340°C did not change the emf measured at the melting point of the Co–C eutectic (PTB). Taken together, these results suggest that additional annealing around 1,030°C may deteriorate the stability of these thermocouples, and so should be avoided, during the intercomparison of Co–C fixed points which is planned in the frame of the Euromet project 857.

Acknowledgments R. Morice acknowledges D. Jouin for his help in the assessment of LNE thermocouples as well as the fruitful collaborative work of M. El Gourdou and J. O. Favreau.

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