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Assessment of Temperature Measurements Using Thermocouples at NIS-Egypt

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Abstract Thermocouples are increasingly used in industry and research. For many industrial heating processes, particularly those carried out at high temperatures, a thermocouple is the most convenient and simple instrument for temperature measurement. In some instances, it is the only feasible method. The aim of this study is to select and recommend the best thermocouples from both base and noble metals to users in industrial and scientific institutions. Different types of thermocouples and calibration methods are described. From this work, the Nicrosil–Nisil thermocouple has been proposed as the best base metal thermocouple and the Au/Pt thermocouple is the most recommended as a substandard up to 1,000 °C.

Keywords Calibration · Thermocouple · Uncertainty

1 Introduction

Thermocouples are the most widely used of all temperature sensors. Their basic simplicity, economic practicability, ruggedness, and low cost, compared with other temperature sensors such as platinum resistance thermometers, have an obvious appeal for many industrial and scientific applications.

Platinum metals and their alloys have long been recognized as the most reliable sensing elements for high-temperature requirements. Among their outstanding properties are their high melting points, repeatability, and sensitivity over a wide temperature range, resistance to corrosion, and stability after calibration.

Thermocouples play no direct role in the realization of the International Temperature Scale of 1990 (ITS-90) [1], unlike in the International Practical Temperature

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Scale 1968 (IPTS-68). They have been replaced in the ITS-90 by high-temperature standard platinum resistance thermometers (HT-SPRT) in the temperature range from 600 °C to 960 °C and by radiation thermometers above the silver freezing point of 1234.93 K. However, due to their simplicity, good accuracy, and repeatability, thermocouples are often used for temperature measurements in the temperature range up to about 2,450 K.

There are different types of thermocouples, both base metal and noble metal. However, they differ in terms of stability, sensitivity, repeatability of readings, homogeneity, and chemical resistance to oxidation. In our laboratory, we have made a study of these properties on several types of thermocouples, two thermocouples per each type. In the present article, we have summarized the results of this study.

2 Stability of Nicrosil–Nisil versus Chromel–Alumel Thermocouples

As Nicrosil–Nisil (type N) thermocouples show enhanced thermoelectric stability, particularly at high temperatures, compared to the other base metal thermocouples, at NIS we have investigated their stability as compared with Chromel–Alumel (type K) thermocouples [2]. Two pairs of type N and type K thermocouples were investigated. Each thermocouple had a 60 cm long and 4.0 mm diameter alumina sheath. The thermoelements were fabricated and supplied from Italcoppie Co., Italy. Type K thermocouples have been widely used in industry for temperature measurement and control, especially in the range from 500 °C to 1,100 °C. This is because the thermocouple is resistant to oxidation and has a large Seebeck coefficient. However, its characteristics show two main mechanisms of change in use [2]. First, upon heating between 250 °C and 500 °C, a relatively rapid change in the thermoelectric voltage is produced by short-range ordering effects in the nickel–chromium arm. Second, at higher temperatures, long-term drifts in the output are produced by the effects of internal oxidation. These may amount to changes in the indicated temperature of around 10 °C for each 1,000 h of operation around 1,000 °C.

Due to a large demand for accurate temperature measurements in the range from 0 °C to 1,000 °C and instabilities in the use of base-metal thermocouples, new possibilities have arisen with the recently introduced type N thermocouple [2]. It was developed to avoid or at least lessen the two main drawbacks of the type K thermocouples discussed above.

At NIS, we have investigated the long-term drifts in the thermal emf outputs involved after exposure of test thermocouples at a constant high temperature of 900 °C using a Heraeus thermocouple calibration furnace. The uniformity of the central test region of this furnace is ±0.2 °C over 40 cm at 900 °C.

The results of calibration of both type-N and type-K thermocouples before and after thermal aging at a temperature of 900 °C for 50 h against the Pt–10 % Rh/Pt (type-S) thermocouple were obtained and are presented in Fig. 1. From this study and from the literature [2, 3], it can be concluded that type-N thermocouples are more stable than type-K ones. Its use should not only reduce the uncertainties in temperature measurement but alleviate the need for frequent inspection, maintenance, and replacement of the sensor.

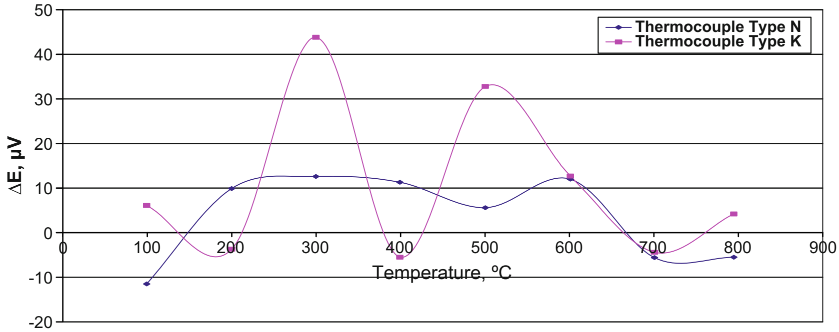

Fig. 1 EMF differences (ΔE) in µV versus temperature in °C, of calibration data of type K and type N thermocouples, before and after 50 h heating

3 Platinum Metal Thermocouples

3.1 Performance of Noble Metal Thermocouples

Platinum-based thermocouples are the most commonly used as reference standards in the comparison calibration of thermocouples because of their ability to be used to carry out simple and accurate temperature measurements. Type-S and type-R are stable thermocouples and usually calibrated in the temperature range from 0 °C to 1,100 °C, with typical uncertainties from 0.2 °C to 0.3 °C. According to Bedford et al. [4], in the Monograph of the CCT/WG2, typical uncertainties of the types R and S thermocouples are 0.3 °C from −50 °C to 1,000 °C and 0.2 °C above 1,000 °C using a fixed-point calibration technique.

The platinum from which the negative element of the thermocouple is prepared must be the purest metal produced commercially. Contamination of the pure platinum leg of the thermocouple during use is perhaps the main reason for loss of calibration, in addition, the following factors may also limit the life and stability of calibration:

1. diffusion of rhodium from the thermocouple bead into the platinum leg;
2. volatilization of the Pt or Rh;
3. contamination of the thermocouple from the environment.

All of these factors are temperature dependent. The second and third are also dependent on the nature of the insulating tubes and the atmosphere in which the thermocouple is used [5].

Although the best achievable calibration uncertainty obtained from the platinum–rhodium alloys thermocouples is 0.1 °C at 1,100 °C as described before, the in-use uncertainty increases to 0.3 °C, mainly because of reversible hysteresis due to preferential oxidation of rhodium over the temperature range from 500 °C to 900 °C. Thermocouples constructed from pure elements do not suffer from this preferential oxidation problem and do not require adjustments of alloy composition to match a reference function. Being pure elements, these thermocouples are inherently more thermoelectrically homogeneous and their stability is not limited by shifts in alloy composition caused by preferential oxidation [6].

In the case of Au/Pt thermocouples, homogeneity and initial calibration tolerances are superior to those of type-S or type-R thermocouples by over an order of magnitude, and no long-term drift has been detected over 400 h of use at NIS—Egypt [7]. The accuracy achievable with Au/Pt thermocouples was comparable to that of a high-temperature Pt resistance thermometer above about 500 °C.

3.2 Fabrication of the Noble Metal Thermocouples

The type S, Au/Pt, and Pt/Pd thermocouples used in this study were prepared from reference grade Pt, Pt–10 % Rh, Au, and Pd wires, and all wires were 0.5 mm in diameter and 150 cm in length purchased from Johnson Matthey. The gold wire had a purity of 99.995 %, the platinum wire of 99.999 % purity, and palladium wire of 99.997 % purity. The Pt, Pt–10 % Rh, and Pd wires were first annealed electrically at 1,300 °C for approximately 10 h, cooled rapidly to room temperature, and then annealed for 1 h at about 450 °C to reduce the lattice vacancies that may be quenched into the wires during cooling from the high-temperature anneal. The gold wires 1.5 m in length were installed in high-purity Al2O3 tubes and heated for 10 h at 980 °C in a 90 cm long conventional tube furnace, which had a 70 cm effective working length over which the temperature uniformity was within ±3 °C. After heat treatment of the first 70 cm of the length, the gold wire was then shifted by 70 cm and heated in the same way.

Thus, the overall length of the gold wire was heat treated. After heat treatment, the wire was cooled in the furnace, and then vacancy annealed overnight at 450 °C. The annealed wires were assembled by threading the thermoelements into the bores of a twin bore high-purity alumina tube with an overall diameter of 4.5 mm and a length of 75 cm. Before use, all alumina tubes were baked at 1,200 °C. For each of the Au/Pt and Pt/Pd thermocouples, a five-turn coil of 1 mm diameter constructed from 0.15 mm diameter platinum wires was used to connect the thermoelements at the measuring junction [7], and a pair of insulated copper wires was soldered to the other ends of the thermoelements to form the reference junction.

Although a Au/Pt thermocouple is the most accurate thermocouple, it is limited in use to temperatures below 1,000 °C, and the Pt/Pd thermocouple is suitable for use to 1,500 °C.

3.3 Measurement Equipment and Procedure

For calibration and evaluation of the noble metal thermocouples in the temperature range from 232 °C to 962 °C, the freezing points of Sn, Zn, and Ag were used in this study. All the fixed-point cells were supplied by NPL. The furnace used for realization of the freezing-point cells was made by Carbolite—UK. Its maximum temperature was 1,000 °C, and it utilized a double-walled sealed sodium heat pipe. The temperature of the furnace was controlled by a unit that has a set point resolution of 0.1 °C.

The reference junction of the thermocouple was maintained at 0 °C in a Dewar filled with distilled water and crushed ice (ice bath). The reference junction was inserted into the closed-end glass tube and was immersed 20 cm in the ice bath. A digital nanovoltmeter with an internal resistance higher than $10^9\ \Omega$ was used to measure the emf. Its resolution corresponds to a temperature resolution of 1 mK.

Calibrations of the thermocouple were also performed from 500 °C to 970 °C by comparison with a high-temperature standard platinum resistance thermometer (HTSPRT) calibrated according to ITS-90 using the same three-zone furnace mentioned before. The alumina insert, in which the thermocouple and HTSPRT were compared, is made of ultra-pure alumina with 50 mm outer diameter and 55 cm length.

The thermocouple was placed very close to the center of the resistance thermometer coil. To prevent heat losses by convection, the furnace tube was closed with an alumina cover with a central hole through which the insert was inserted inside the furnace. The temperature gradient along the furnace was about 0.05 °C over the length of 20 cm. Resistance measurements of the HTSPRT were made with a Model F18 AC-bridge manufactured by Automatic Systems Laboratories Inc.

3.4 Thermocouple Calibration Methods

In the following, we describe the calibration methods for type-S thermocouples as an example for NIS thermocouple calibration techniques. The results of a comparison between techniques for calibration of type-S thermocouples by fixed points and by intercomparison against high-temperature platinum resistance thermometers in the temperature range from 100 °C to 960 °C is summarized.

A calibration run consisted of determining the thermocouple emf using metal freezing-point cells. The run started at the melting point of gold by using the gold wire method [7], then it went to the Ag, Al, Zn, and Sn freezing points; the results are given in Table 1. In the table, E_R is the emf calculated using the reference function given by Burns et al. [6], while E_T is the experimentally measured emf of the Pt–Pt 10 % Rh thermocouple. The technique used for realization of the metal freezing points were similar to those developed by Mclaren and Murdock [8] and given in the Supplementary Information for ITS-90.

For comparison between calibration techniques, the thermocouple was calibrated by comparison with a calibrated high-temperature standard platinum resistance thermometer in the temperature range from 90 °C to 950 °C. The results are given in Table 2. The treatment of experimental data was the same as that mentioned in the case of calibration with a fixed point.

Table 1 Summary of type S thermocouple calibration at the fixed points

Metal	Equilibrium state	Freezing temp. (°C)	E_T^a (μV)	E_R (μV)	$\Delta E = E_T - E_R$ (μV)
Tin, Sn	Freezing	231.928	1717 ± 0.4	1716	+1
Zinc, Zn	Freezing	419.527	3447 ± 1.4	3447	0
Aluminum, Al	Freezing	660.323	5859 ± 1.4	5860	−1
Silver, Ag	Freezing	961.78	9149 ± 1.4	9148	+1
Gold, Au	Melting	1064.18	10336 ± 3	10334	+2

[a] All presented standard deviations in the above table for E_T are expressed as 1σ

Table 2 Experimental results obtained for the calibration of the thermocouple by intercomparison with HT-SPRT in the range from 90 °C to 965 °C

T (°C)	E_T (Aver. exper.) (μV)[a]	E_R (μV)	$\Delta E = E_T - E_R$ (μV)
90.3	575.5 ± 0.5	574.4	−0.9
193.00	1379.3 ± 0.6	1382.0	−2.7
296.42	2289.0 ± 1.2	2290.8	−1.8
395.05	3209.0 ± 1.8	3212.0	−3.0
563.48	4869.2 ± 2.1	4868.0	+1.2
631.79	5566.0 ± 2.4	5565.0	+1.0
892.7	8372.9 ± 2.7	8367.7	+5.2
962.3	9160.4 ± 3.0	9154.3	+6.1

[a] All presented standard deviations in the above table for E_T are expressed as 1σ

This study and other studies [7, 9] given in the literature have shown that calibration of standard Pt–10 % Rh versus Pt thermocouples by using the freezing points of high purity metals used for the realization of ITS-90 is better than calibration by intercomparison against HTSPRT. The study had also shown that the accuracy, which can be achieved by type-S thermocouples, is not better than 0.2 °C.

In Table 2, T is the temperature as was measured by HT-SPRT, E_T is the average experimentally measured emf, E_R is the emf corresponding to the temperature given in column (1), and ΔE is the difference, $\Delta E = E_T - E_R$.

4 Comparison Between the Stability and Sensitivity of the Noble Metal Thermocouples

In Table 3, comparisons are made between stabilities of the three thermocouples, Au/Pt, Pt/Pd, and Pt–10 % Rh/Pt at the Ag freezing points after thermal treatment for 50 h, 100 h, 200 h, and 300 h. From Table 3, it is clear that Au/Pt and Pt/Pd thermocouples have better repeatability than type S thermocouples as observed from the standard deviation of measurements at the Ag freezing point for the three thermocouples.

Figure 2 shows the emf variations relative to the initial values (expressed in °C) as a function of the annealing time at the freezing point of Ag (960 °C). $V(t)$ represents the emfs at time t, and $V(0)$ is the initial emf. From the curve, it is clear that after 200 h of anneal the emf changes of thermocouples became within an order of magnitude of the standard deviation of results, given in Table 3. This shows that after 300 h at 960 °C, the three types of thermocouples became sufficiently stable. Comparing the Au/Pt and Pt/Pd with the type S thermocouple, both the pure noble metal thermocouples show improved thermoelectric stability. However, Gotoh [10] noticed that the Au/Pt thermocouple is slightly more stable than the Pt/Pd thermocouple above 1,000 °C.

Ancsin [11], Ripple and Burns [12], and Izuchi et al. [13] have reported that soaking a noble metal thermocouple at the Ag freezing point for about 200 h for stabilization was sufficient so that it became reproducible. Thus, the initial variation of emf in this work can be considered as part of the stabilization process. From 200 h to 300 h,

Table 3 Variation of the measured emf at the Ag freezing point (961.78 °C) with time for the Au/Pt, Pt/Pd, and type S thermocouples after thermal treatment

Time (h)	Au/Pt		Pt/Pd		Type S	
	Emf (μV)	Std. dev.[a] (±μV)	Emf (μV)	Std. dev.[b] (±μV)	Emf (μV)	Std. dev.[b] (±μV)
1st use	16106.20	2.26	10792.55	3.84	9162.04	4.62
50	16105.71	1.72	10793.51	2.51	9160.58	3.91
100	16107.71	0.48	10794.19	1.34	9160.59	1.54
200	16107.28	0.42	10793.57	0.60	9159.38	0.94
300	16107.45	0.32	10793.96	0.42	9159.52	0.72
Average	16106.9		10793.6		9160.4	
[b]Total stability of thermocouples Std. dev. (±μV)	±1.73 μV (=about 69 mK)		±1.30 μV (=about 68 mK)		±2.14 μV (=about 188 mK)	

[a] All presented standard deviations in the above table are expressed as 2σ (five freeze experiments per each stage)

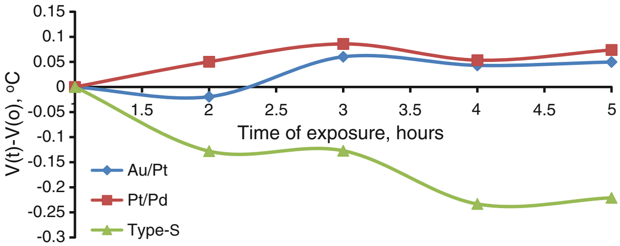

Fig. 2 Emf variations relative to the initial values (expressed in °C) as a function of the annealing time at the freezing point of Ag (960 °C)

the emf variations were small, as seen from the standard deviation shown in Table 3 and from the curves in Fig. 2.

Wei and Weixin [14] found that the best stability of Pt/Pd thermocouples at the Ag freezing point was within 40 mK after 230 h annealing at 1,000 °C, which compares with our results after 200 h of anneal as shown in Fig. 2.

The Seebeck coefficients $\mathrm{d}E/\mathrm{d}t$ at the Ag freezing point for the thermocouples investigated are as follows:

$$\mathrm{d}E/\mathrm{d}t \text{ for Au/Pt} = 24.94\,\mu\mathrm{V}\cdot{}^\circ\mathrm{C}^{-1},$$
$$\mathrm{d}E/\mathrm{d}t \text{ for Pt/Pd} = 19.2\,\mu\mathrm{V}\cdot{}^\circ\mathrm{C}^{-1},$$
$$\mathrm{d}E/\mathrm{d}t \text{ for type S} = 11.4\,\mu\mathrm{V}\cdot{}^\circ\mathrm{C}^{-1}.$$

The above values of $d\mathrm{E}/dt$ indicate that the Au/Pt thermocouple is more sensitive than the Pt/Pd thermocouple, and the Pt/Pd thermocouple is more sensitive than type S. For Pt/Pd thermocouples, Wei and Weixin [14] observed that the Seebeck emf increases, which can be attributed to oxidation of the Pd wire in the temperature range from

500 °C to 800 °C. Bentley [15] states that dissolved interstitial oxygen may affect the Seebeck emf.

5 Evaluation of the Inhomogeneity of Thermocouples

Inhomogeneity effects are measured by exposing the thermocouple to defined temperature gradients. Different methods can be applied.

The common method used to investigate assembled thermocouples, is to submerge them slowly into a fixed-point cell during freezing. Thus, any deviation of the measured emf is an indication of inhomogeneities. Most of the previous research studied thermocouple inhomogeneity on assembled thermocouples [16]. We have used this technique as summarized in the following.

5.1 Assessment of the Thermoelectric Homogeneity of Au/Pt and Type S Thermocouples

The insertion–withdrawal technique was carried out for Au/Pt and Pt/10 % Rh versus Pt thermocouples at the freezing points of Zn, Al, and Ag using sealed cells [17]. During the fixed-point calibrations, the thermocouple was inserted into thermometric wells of the cells and its measuring junction was positioned approximately 1 cm below the surface of the metal. In sequence, they were moved down until reaching the bottom with 1 cm steps. After the measuring junction was held at full immersion, then it was withdrawn from the bottom of the thermometric wells of the cells in steps of 1 cm, and the emf was measured at the same locations as for immersion.

For the freezing point of Zn at deep quantifiable immersion, the results indicate inhomogeneity along the length of a type-S thermocouple equivalent in temperature to ≤6 mK and along the length of a Au/Pt thermocouple to ≤4 mK, as shown in Fig. 3.

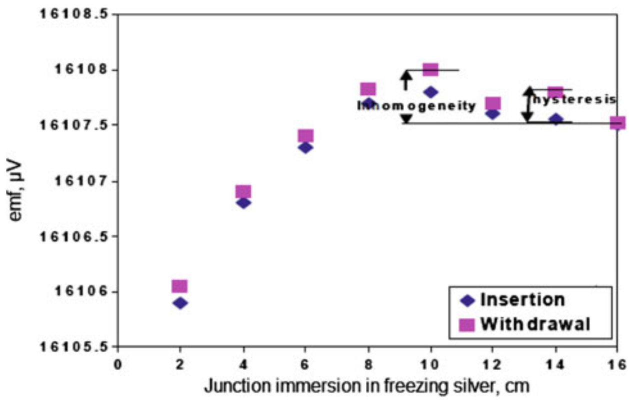

Fig. 3 Values of emf measured for Au/Pt thermocouples on insertion into and withdrawal from Ag freezing-point cell versus depth of immersion [7]

Table 4 Inhomogeneity values for Au/Pt, Pt/Pd, and type S thermocouples derived from thermocouple immersion profiles in a silver freezing-point cell during a freeze

Type of thermocouple	Inhomogeneity	
	(μV)	(mK)
Au/Pt	0.46	19
Pt/Pd	0.44	23
Type S	2.46	219

At higher temperatures, much larger thermoelectric changes occur for the type-S thermocouple. For the Al and Ag freezing points, the results indicate inhomogeneities equivalent to temperatures of ∼40 mK and 60 mK, respectively.

To investigate the inhomogeneity, immersion/withdrawal thermal emf profiles were investigated for three types of thermocouples at the Ag freezing point following 300 h anneal at 960 °C. The results are given in Table 4.

From this test, it is clear that Au/Pt and Pt/Pd have inhomogeneity values that are less than type S. Ogura et al. [18] found that the drift and homogeneity of Pt/Pd thermocouples depend on prior heat treatment and that heat treatment at 850 °C or 1,030 °C for 100 h decreased the drift and inhomogeneity to values less than 0.5 μV over 150 h. This result implies that optimal heat treatment can reduce the uncertainty of the Pt/Pd thermocouple calibration.

6 Uncertainty Budget in Thermocouple Calibration

Table 5 shows a summary of how the uncertainty budget for a Pt/Pd thermocouple calibration at the Ag freezing point had been calculated according to GUM recommendation [19, 20].

Table 5 Uncertainty budget for the calibration of Pt/Pd thermocouples at Ag freezing point

Source of uncertainty	± Value	Probability distribution	Divisor	c_i ($\mu V \cdot {}^{\circ}C^{-1}$)	u_i (T) (mK)
Repeatability	35.0 mK	Normal	1	1	35.0
Uncertainty of fixed point	1.5 mK	Normal	1	1	1.5
Uncertainty due to inhomogeneity of Pt/Pd thermocouple	0.44 μV	Rectangular	$\sqrt{3}$	0.052	13.2
Uncertainty due to thermocouple stability	0.65 μV	Normal	1	0.052	34
Uncertainty of voltmeter	0.77 μV	Normal	2	0.052	20.2
Uncertainty of ice point	8.65 mK	Rectangular	$\sqrt{3}$	1	5.0
Combined standard uncertainty, U_c		Normal			54.7
Expanded uncertainty U ($k = 2$)		Normal			109.4

Table 6 Combined uncertainty budget for the calibration of Pt/Pd, Au/Pt, and Type S thermocouples at Ag freezing point

Source of uncertainty	Au/Pt (mK)	Pt/Pd (mK)	Type S (mK)
Repeatability	6.5	35	33.0
Uncertainty of fixed point	1.5	1.5	1.5
Uncertainty of thermocouple inhomogeneity	11.0	13.2	125.0
Uncertainty due to thermocouple stability	34.5	34.0	94.0
Uncertainty of voltmeter	20.0	20.2	43.0
Uncertainty of ice point	5.0	5.0	5.0
Combined standard uncertainty, U_c	42.2	54.7	165.6
Expanded uncertainty $U(k=2)$	84.4	109.4	331.2

The type A uncertainty U_A (repeatability) is taken as the calculated standard deviation of the mean of the series of experiments that we carried out. The type B uncertainties include the maximum estimated allowances U_B for the other factors contributing to the uncertainty budget as given in Table 5. Table 6 gives the uncertainties of the other types of thermocouples calculated as given in Table 5.

7 Conclusions

This study shows that the best base metal thermocouple for routine temperature measurements and control at the temperature range from −40 °C to 1,200 °C is the Nicrosil–Nisil (type N) thermocouple compared to Chromel–Alumel (type K) thermocouples, which have a sensitivity of $39\,\mu V \cdot {}^\circ C^{-1}$ at 1,000 °C.

Among the noble metal thermocouples, the Pt/Au thermocouple is the most recommended as a substandard for routine calibration of other types of thermocouples working in the range up to 1,000 °C; it has a sensitivity of $25\,\mu V \cdot {}^\circ C^{-1}$ near 900 °C and good repeatability.

The Pt/Pd thermocouple can be used as a substandard for routine measurements up to 1,500 °C and also for calibration purposes. It has good sensitivity ($19\,\mu V \cdot {}^\circ C^{-1}$ at 1,000 °C) and good repeatability.

The stability and repeatability of the three types of noble metal thermocouples were improved after annealing at 950 °C for 300 h.

The insertion–withdrawal inhomogeneity test shows that the pure element thermocouples are more homogenous than alloyed thermocouples.

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