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Calibration Method of Radiation Thermometer Based on Rhenium-Carbon Eutectic Point

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Abstract: *A calibration method for high-temperature radiation thermometers based on the eutectic point of rhenium and carbon was adopted for radiation thermometers such as photoelectric pyrometers and infrared thermometers. The physical phenomenon of constant temperature during the eutectic process of rhenium and carbon was utilized to calibrate high-temperature radiation thermometers, raising the measurement uncertainty level to 1.6℃ (k=2), providing reliable data support for product test and design verification.*

Keywords: *Rhenium-carbon eutectic point, Calibration of radiation thermometers, Absolute radiation method, High-temperature calibration, Inflection point calculation method*

1. Introduction

In the field of high-temperature measurement and metrology, photoelectric pyrometers, infrared thermometers and other radiation thermometers are widely used to measure solid surfaces for high-temperature measurements, and for high-precision measurements or calibrations, absolute radiation-method thermometers are also used. In the current ITS -90, the transmission of the high-temperature temperature scale mainly relies on the high-temperature primary standard calibration work standard tungsten ribbon lamps[1], and the verification of the standard photoelectric high-temperature gauge for the work standard tungsten ribbon lamps[2]. This transmission method currently can only verify high-temperature gauges that are close to the wavelength of 0.66μm of the primary high-temperature gauge, and the temperature range is basically limited to between 800 ℃~2200℃[3]. Since there is no defined fixed point above the copper freezing point, the temperature above the copper point is extrapolated based on a fixed point of silver (961.78℃), gold (1064.18℃), or copper (1084.62℃) using Planck's law [4], which leads to a rapid increase in uncertainty as the temperature rises in a quadratic relationship . Due to multi-step transmission, the calibration uncertainty is far from meeting the requirements of the experiment. Using the high-temperature fixed point method will significantly reduce the uncertainty, as shown in the Figure 1.

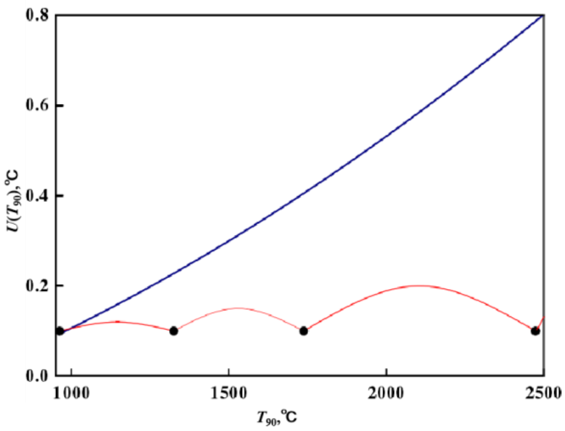

Figure 1. Uncertainty in the reproduction of the international temperature scale after the introduction of high-temperature fixed points

Led by the CCT-WG5 Radiation Thermometry Working Group of the International Committee for Weights and Measures, extensive research has been conducted around high-temperature fixed points[5].

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Participating institutions include the National Metrology Institute of Japan (NMIJ), the National Physical Laboratory of the United Kingdom (NPL), the Institute of Optics and Physics of Russia (VNIIOFI), the National Institute of Metrology of France (CNAM), the International Bureau of Weights and Measures (BIPM), the Korea Research Institute of Standards and Science (KRISS), the Physikalisch-Technische Bundesanstalt of Germany (PTB), the National Institute of Standards and Technology of the United States (NIST), Tamagawa University of Jap an, the National Institute of Metrology of China (NIM), the Institute of Metrology of Italy (INRiM), and the National Research Council of Canada (NRC), among other nation al metrology institutes[6,7].

This paper studies a calibration method for high-temperature radiation thermometers based on the rhenium-carbon eutectic point. The method utilizes the temperature-invariant physical phenomenon during the rhenium-carbon eutectic process to calibrate high-temperature radiation thermometers, forming a complete set of calibration methods for high-temperature radiation thermometers based on the rhenium-carbon eutectic point. This process uses absolute method calibration to improve calibration uncertainty.

2. Metal-carbon eutectic point calibration principle

Metal-carbon eutectic point calibration involves using a photoelectric high-temperature thermometer to measure the temperature platform of high-temperature fixed-point eutectic reactions, using the internationally agreed temperature values of the eutectic reactions as the standard to assign values to the high-temperature thermometer. The principle of eutectic point calibration is shown in the figure 2.

Figure 2. Schematic diagram of metal-carbon eutectic point calibration

The eutectic within the high-temperature fixed-point crucible melts (liquid–solid phase change) and solidifies (solid–liquid phase change) under constant pressure conditions, and during this process, the phase interface maintains isothermal characteristics. The blackbody cavity is isothermal during the phase change. The thermal radiation emitted by the isothermal blackbody cavity during the phase change passes through lens 1, apertures 3 and 4, converging lens 5, and interference filter 6 in the optical path of the photoelectric pyrometer to reach detector 7, where it is converted into photocurrent Ip. According to Planck's law, there is a uniquely determined relationship between the photocurrent Ip and the temperature of the blackbody cavity.

The photoelectric high-temperature meter achieves monochromatization based on interference filters, and the output photocurrent formula of its detector is:

$$I_p = \int_0^\infty \phi(\lambda) R(\lambda) d\lambda \tag{1}$$

In the formula, $\phi(\lambda)$ represents the spectral radiance flux received by the photodetector, and $R(\lambda)$ represents the spectral responsivity of the photodetector.

When the rhenium-carbon eutectic crucible is continuously heated from room temperature to the specified temperature. The output of the photoelectric pyrometer rises at a certain rate; however, when graphite and metallic rhenium form a eutectic and continuously absorb heat to melt into a liquid, the temperature of the crucible wall remains unchanged. Consequently, the slope of the photoelectric pyrometer output curve becomes more gradual, and under optimal conditions, a relatively horizontal section of the curve may be produced.

Calibration based on the metal-carbon eutectic point temperature platform effect has the following main advantages:

(a) Shortening the traceability chain by using the temperature platform effect to calibrate temperature sensors by the absolute method, thereby eliminating uncertainties introduced by the primary standard;

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(b) The metal-carbon eutectic crucible can be used multiple times after a single filling, offering good repeatability and introducing only a small uncertainty component during calibration;

(c) In the eutectic state, the temperature fluctuation at the bottom of the crucible is small and evenly distributed, serving as a repeatable radiation source;

(d) Using the inflection point calculation method to determine the start and melting points of the eutectic reaction, accurately determining the melting process and time.

3. Study on Calibration Methods

3.1 Mechanism of eutectic point formation in metal-carbon

The metal-carbon eutectic point calibration system consists of a high-temperature generation device, control system, water-cooling system, Inflatable and deflatable system, power supply system, etc., as shown in the figure 3.

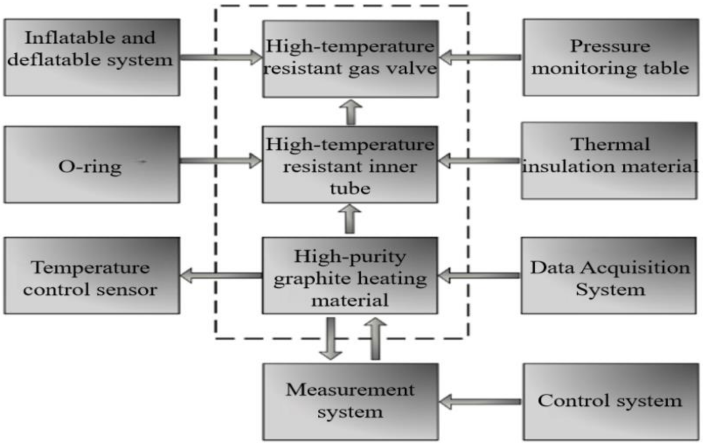

Figure 3. Calibration system block diagram

The structure of the calibration system is shown in the figure 4. The inflatable and deflatable system, pressure control gauge, and power supply are integrated into the cabinet, while the control system forms a separate control cabinet, capable of controlling the heating temperature and monitoring the heating current and voltage.

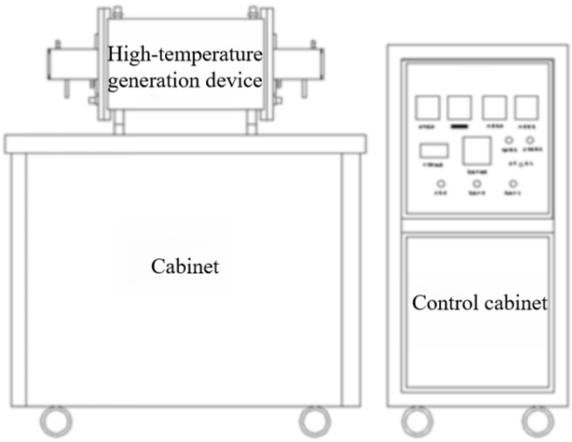

Figure 4. Calibration system architecture diagram

3.2 System uniformity test

When the central temperature field is uneven, it can cause the crucible at the eutectic point to be heated unevenly, and the metal-carbon eutectic reaction will occur in different areas. As the eutectic reaction may cause different thermal stresses on the crucible, there is a risk of crucible cracking, so the

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uniformity of the temperature field is tested. The nominal temperature of the eutectic reaction is 2474℃, so the temperature field uniformity is tested at 2470℃. Since the axial length of the eutectic point crucible is 50mm, the temperature field along the central axis of the high-temperature apparatus over a distance of 50mm is measured, with one test surface every 10mm, dividing the axial temperature field into six test surfaces, as shown in the figure 5.

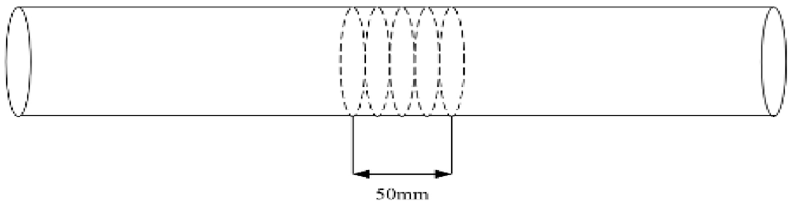

Figure 5. Illustration of test face position

The curved base of the movable target surface is used as the temperature testing surface. Each testing surface measures five positions: top, middle, bottom, left, and right. The maximum axial temperature difference is required to be less than 10℃, and the uniformity of the measured surface should be less than 5℃.

The test results show Table 1-Table 2, that the maximum axial temperature difference of the high-temperature generation device is 8.1℃, better than 10℃, and the uniformity of each test surface is better than 5℃, meeting the requirements for the calibration of the metal-carbon eutectic point.

Table 1. Axial uniformity test results

Location	0	1	2	3	4	5
Temperature/℃	2470.8	2469.6	2468.4	2467.0	2465.2	2462.7

Table 2. Test surface uniformity

Location	0	1	2	3	4	5
Uniformity/℃	1.4	1.9	1.8	2.7	2.9	2.8

4. Calibration test

4.1 Experimental design

The calibration test process includes installing the crucible and grating, aligning and focusing, setting the temperature control curve, cleaning the inner cavity, heating, and collecting data.

The designed temperature curve is shown in the figure 6:

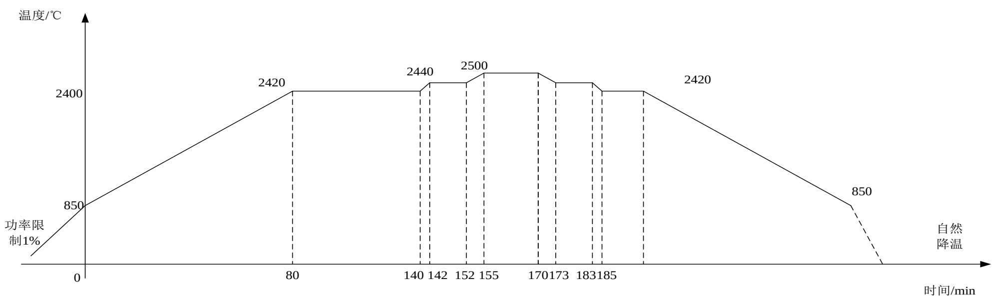

Figure 6. Temperature curve design

① Raise the high-temperature generation device from room temperature to 850℃;② Increase the temperature to 2420℃ at a rate of less than 20℃/min;③ Maintain at 2420℃;④ Increase the temperature to 2440℃ at a rate of less than 20℃/min;⑤ Maintain at 2440℃;⑥ Raise the temperature from 2440℃ to 2500℃ within 3 minutes;⑦ Maintain at 2500℃;⑧ Decrease the temperature to 2440℃ at a rate of less than 20℃/min;⑨ Maintain at 2440℃;⑩ Decrease the temperature to 2420℃ at a rate of less than 20℃/min;⑪Maintain at 2420℃;⑫Decrease the temperature to 850℃ at a rate of less than 20℃/min;⑬Let cool naturally to room temperature

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4.2 Data processing methods

High-frequency acquisition, forming data tables and storing them, with data information including acquisition time (absolute time or relative time with the start of acquisition as zero) and the output voltage of the photothermal pyrometer. A single heating process is extracted for data processing and analysis.

The time-output value function is fitted using the least squares method:

The least squares method fits discrete data as:

$$f_m(x)=\sum_{j=0}^{m} a_j x^j \tag{2}$$

Set the deviation of $f_m(x_i)$ at x_i to:

$$\Delta R=f_m(x)-y_i \tag{3}$$

Then the sum of the squares of the deviation ΔR_i of the fitting function $f_m(x_i)$ for all data is:

$$S=\sum_{I=0}^{n} \Delta R_I^2=\sum_{i=0}^{n} [f_m(x_i)-y_i]^2 \tag{4}$$

To obtain the minimum value of S, partial derivatives of the above formula with respect to a_0, $a_1 \cdots a_m$ must be taken, that is, a_0, $a_1 \cdots a_m$ must satisfy the conditions.

$$\begin{cases} a_0P_0+a_1P_1+\cdots a_mP_m=Q_0 \\ a_0P_1+a_1P_2+\cdots a_mP_{m+1}=Q_1 \\ \cdots \\ a_0P_M+a_1P_{m+1}+\cdots a_mP_{2m}=Q_m \end{cases} \tag{5}$$

Finally, substitute the solved a_0, $a_1 \cdots a_m$ into the fitting function

Find the second derivative of the fitting function to obtain the inflection point:

Perform the second derivative of $f_m(x)=\sum_{j=0}^{m} a_j x^j$ using the polynomial differentiation method, set the second derivative to zero, and solve for the two inflection points.

Mark the starting and ending points on the time-output curve and calculate the voltage difference between the two points: if the voltage difference between the start and end points is less than or equal to the differential voltage value of the calibrated radiation thermometer, it can be proven that during the temperature ramp-up process, the temperature drift is within an acceptable range; if the voltage difference between the start and end points is greater than the differential voltage value of the photodiode high-temperature meter, the temperature range for calculation should be reduced, the selected data segment should be moved as much as possible towards the middle, and the starting and ending points should be re-marked on the time-output curve. The operator repeat the operation until the voltage difference between the start and end points meets the requirement of not exceeding the differential voltage value of the photoelectric pyrometer. Then, calculate the average voltage of the selected interval and derive the corresponding temperature in accordance with the instruction manual of the radiation thermometer.

4.3 Experimental verification results

The experimental verification involved four heating and cooling cycles, producing a total of four sets of melting and solidification platforms, as shown in the figure 7.

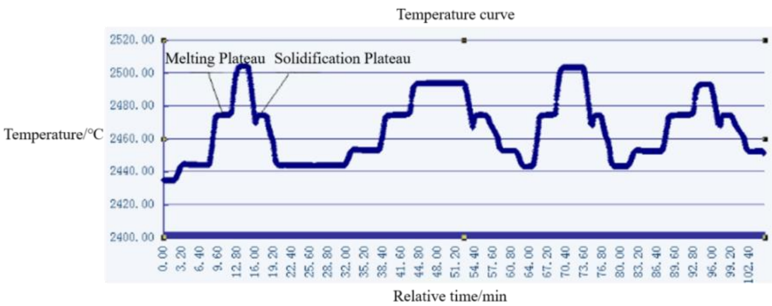

Figure 7. Temperature curves of four experiments

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The left side of the temperature peak represents the melting curve platform, the right side represents the solidification curve platform. The curves of four melting platforms were extracted, as shown in the figure 8.

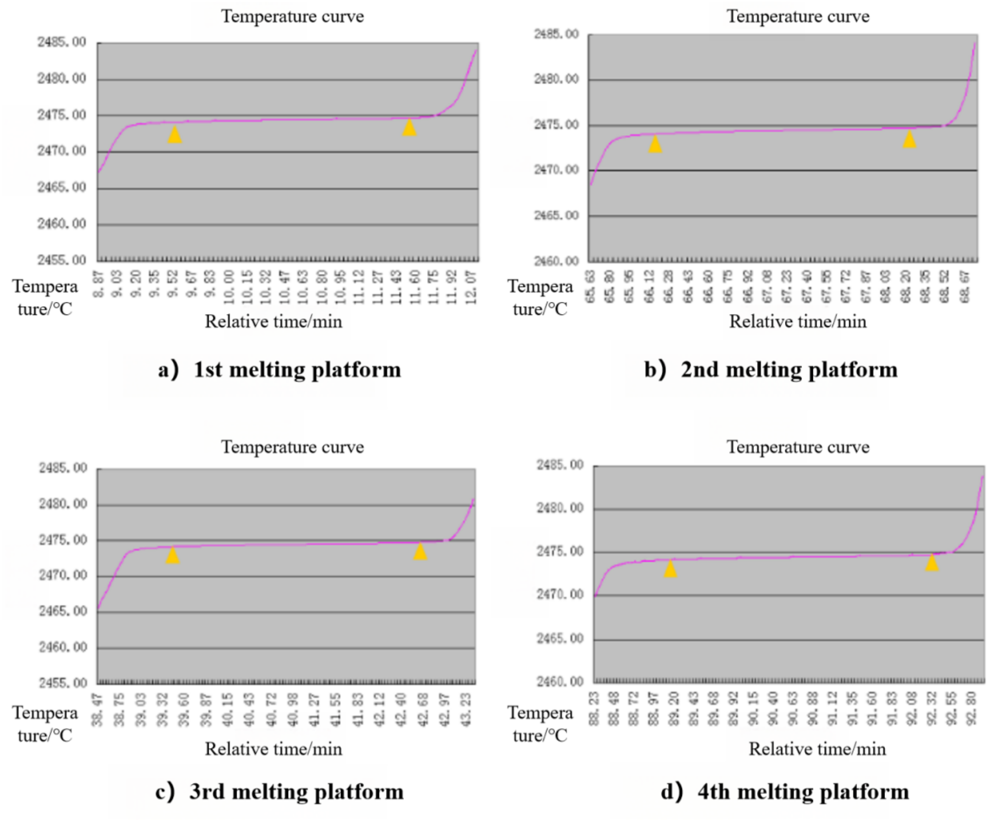

Figure 8. Four melting platform decomposition curve

The position marked by the triangular symbol indicates the starting point of melting, and the temperature calibration results calculated are shown in Table 3:

Table 3. Test results

Test Run	1	2	3	4
Temperature (℃)	2474.56	2474.60	2474.60	2474.79

Conduct four repeated measurements, using the range method to characterise the experimental standard deviation, with $1/d_n$ being 0.486, then $s = \frac{R}{d_n}$ =0.11℃.

The calibration result of the photoelectric high-temperature thermometer is taken as the average of four test results, T=2474.64℃.

5. Evaluation of measurement uncertainty

Evaluation of measurement uncertainty in the calibration of the metal-carbon eutectic point by the GUM method:

① Standard uncertainty introduced by repeated measurements, calculated using the same method as repeatability experiments, u_1=0.11℃;

② Standard uncertainty introduced by the deviation of the crucible temperature at the eutectic point, evaluated by the B-type method, treated as a uniform distribution, deviation 0.5℃, standard uncertainty u_2=0.28℃;

③ Standard uncertainty introduced by short-term repeatability of the crucible at the eutectic point, evaluated by the B-type method, treated as a uniform distribution, deviation 0.08℃, standard uncertainty u_3=0.05℃;

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④ Standard uncertainty introduced by internal temperature gradient of the crucible at the eutectic point, evaluated by the B-type method, conservatively estimated not to exceed 1.0℃, treated as a uniform distribution, standard uncertainty u_4=0.58℃;

⑤ Standard uncertainty introduced by size effects, evaluated by the B-type method, conservatively estimated not to exceed 0.2℃, treated as a uniform distribution, standard uncertainty u_5=0.12℃;

⑥ Standard uncertainty introduced by microstructure effects due to fluctuations in the high-temperature device and heating rate, evaluated by the B-type method, conservatively estimated fluctuations of 0.5℃, treated as a uniform distribution, standard uncertainty u_6=0.29℃;

⑦ Standard uncertainty introduced by electrical measurement equipment, evaluated by the B-type method, conservatively estimated not to exceed 0.1℃, treated as a uniform distribution, estimated standard uncertainty u_7=0.06℃;

The above factors are independent of each other; therefore, the combined uncertainty is:

$$u_c = \sqrt{u_1^2 + u_2^2 + u_3^2 + u_4^2 + u_5^2 + u_6^2 + u_7^2} = 0.8℃;$$

The expanded uncertainty is:

$$U = 2 \times u_c = 1.6℃ (k=2).$$

6. Conclusion

Calibration method and equipment for absolute radiation thermometers based on the rhenium-carbon eutectic point. This method utilises the physical phenomenon of constant temperature in the eutectic state of metal rhenium and carbon to calibrate absolute radiation thermometers, photoelectric high-temperature thermometers, infrared thermometers and other radiation thermometers. High precision and reproducibility can be achieved at the metal-carbon eutectic point, with a measurement uncertainty of up to U=1.6℃ (k=2), greatly improving the level of measurement uncertainty.

This method is applied to calibrate radiation thermometers, providing reliable data support for product testing and design verification.

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