

Research on Characteristic Parameter of Ta-ZrO₂ Fiber Blackbody Cavity Temperature Sensor

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Abstract

By sputtering and plasma spraying technology, a sapphire optical fiber blackbody cavity temperature sensor of tantalum (melting point is 2997°C) - zirconium oxide (melting point is 2715°C) thin film was developed. Static calibration system for the sensor to measure target temperature 1721°C is also designed by using three oxygen (produced by water electrolysis) flame guns. When its impact resistant capacity is more than 50MPa, the corresponding temperature is 2802°C. A high power and high frequency modulation CO₂ laser pulse is used as exciting source to heat it to 1500°C, and the dynamic calibration device is designed independently, thus this sensor in experimental results show a time response on the order of μs. Signal collection and transmission through fiber optic cable are proposed, which can meet the need of transient high temperature measurement in harsh environment.

Keywords: Ta-ZrO₂ thin film, dynamic characteristic, transient ultra-high surface temperature, impact resistant capacity, measurement

1. Introduction

Transient temperature measurement is characterized by high temperature, change quickly, which accompanied by high pressure or flow of high-speed airflow and do not repeat a one-time process. So the condition of the measurement is very bad and the technical difficulty is very high, especially in many occasions, the test cost is very high and has a high requirement to the reliability of the system and data capture rate. Although there are a lot of suitable temperature sensor for different temperature range and response speed, but in this field, the theory and practice still cannot meet the needs of the measurements and satisfy the large temperature range (> 2000°C) and high response speed (the time constant of tau is < 1ms) of the instantaneous ultra-high temperature testing requirements.

Sapphire fiber tantalum zirconium oxide black body temperature sensor is combined the radiation temperature measurement technology with optical fiber sensing technology organically with high sensitivity and immunity to electromagnetic interference, which can solve the problem of transient high temperature measurement under the bad environment successfully. Its high temperature stability and transient response characteristics, which the traditional thermocouple cannot unmatched, has a good application prospect in the field of transient high surface temperature measurement.

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2. The Composition of Transient High Temperature Sensor

Transient high temperature sensor is combined with sapphire fiber blackbody cavity of tantalum oxide zirconium, transmission optical fiber, optical fiber coupler, a solid state photomultiplier tube and amplifying circuit. As is shown in the Figure 1.

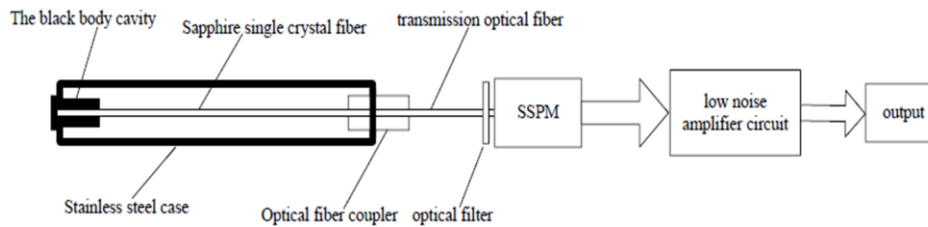


Figure 1. The Composition of the Sensor

2.1. The Formation of Tantalum Oxide and Zirconia Black Body Cavity

Black body cavity can choose low cost and high melting point metal tantalum (or molybdenum) as a black body cavity bottom film layer, due to its transparency, high melting point oxide film on tantalum film plating system instead of the expensive metal film to compose the sapphire fiber black body cavity to adapt to the instantaneous ultra-high temperature measurement.

The melting point of the tantalum is 2997°C , the boiling point is 5427°C and the density is 16.5 g/cm^3 . Tantalum has excellent chemical properties, which has extremely high corrosive. Sputtering tantalum film of sapphire optical fiber is shown in the Figure 2, tantalum film membrane layer of SEM figure is shown in the Figure 3. See from the figure that the film surface is smooth, no cracks and other defects^[1-3].



Figure 2. Sputtering Tantalum Thin Film

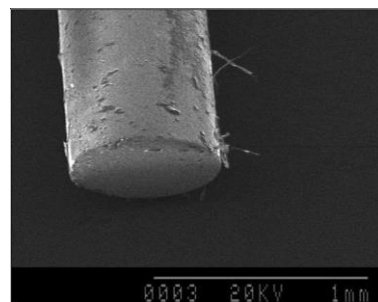


Figure 3. The SEM Figure of Tantalum Thin Film

In the air, tantalum oxidation begins from $300 \sim 325^{\circ}\text{C}$, when the temperature is higher than 550°C , oxidation rate increased significantly, generate Ta_2O_5 . The ratio of the Ta_2O_5 molar volume and the Ta molar volume is 2.50, which lead to the oxide film rupture or layered, when the temperature is higher than $500 \sim 550^{\circ}\text{C}$, with the increase of oxidation time, gradually transformed into a destructive oxidation^[4-6].

Obviously, the sapphire fiber head plating tantalum film to form a black body cavity cannot work under the environment of high temperature oxidation (Molybdenum also has similar drawbacks. Its melting point is 2630°C , boiling point is 5700°C and the relative density is 10.23 g/cm^3), so need protective film layer on the tantalum film plating system to adapt to the oxidation conditions^[7,8]. Coating zirconia films on tantalum film surface by plasma thermal spraying process^[9,10]. Zirconia melting point is 2715°C and with stable chemical properties, which can protect the tantalum film from the oxidizing environment. The imaging of zirconium oxide film layer plated on the sapphire fiber by plasma thermal spraying technology is shown in the Figure 4.

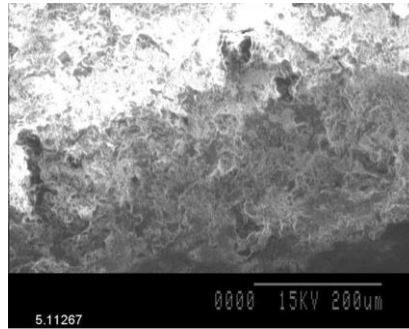


Figure 4. The SEM Figure of Ta-ZrO₂ Blackbody Cavity

From the Figure 4, zirconium oxide film layer is without stripping, crack phenomenon and combined with optical fiber closely. As is shown in the Figure 5, three oxyhydrogen flame spray gun to produce heat source to heat the Ta-ZrO₂ fiber head, using Modline3 IRCON company (M3:1000°C-3000°C) type infrared thermometer to measure the end face temperature of the Optical fiber. Oxyhydrogen flame temperature can reach 2800°C, the flame is small, the heat is concentrated. When the heating time is 15 seconds and the heating temperature is above 1500°C, Zirconium oxide film layer is not damaged. When the heating time is more than 30 seconds and the heating temperature is above 2000°C, the zirconium oxide film layer begin to melt. By analyzing, the temperature of the infrared radiation thermometer shows the average temperature in the field of view, the local temperature of the optical fiber head temperature has exceeded the melting point (2045°C) of the sapphire fiber.

This phase is called the shearing element. In the shearing element, the plastic resinous material completes further plastication and homogenization. According to the Weissenberg effect, the motion direction of the plastic resinous material is from the periphery of the cavity 14 to the center, which is similar to the motion of dish extruders. For further homogenization, the short screw 5 connected to the rotor 1 by a bolt is disposed in the center. At last, the plastic resinous material, which has been homogenized sufficiently, is extruded out through a die 17. We call this phase the homogenization element, just like the conventional extrusion process.

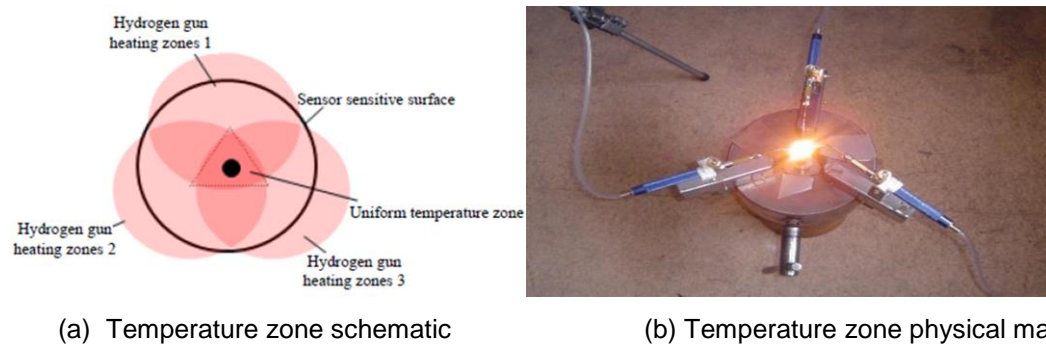


Figure 5. Constant Temperature Zone of Hydrogen and Oxygen Flame Guns

2.2. The Photoelectric Conversion and the Low Noise Amplifier-Circuit

In order to realize the miniaturization of the sensor structure, SSPM can be used as a photoelectric conversion element. The SSPM adopts the plane array and work in the Geiger mode of APD avalanche diode with a total of 556 APD infinitesimal.

Since every APD batteries work in Geiger mode and each unit can be used as a digital photon detector, so the SSPM has the very high internal gain and fast response characteristics, its gain is 4×10^5 , response time is less than 1ns and have higher spectral responsivity between 450nm to 800nm, spectral response curve is shown in the Figure 6.

In addition, the shape of the SSPM is smaller, which the cross section area was about 11mm^2 . Make it can be coupled with sapphire fiber directly, which simplify the structure design of photoelectric conversion module. It is advantageous for the sensors to be used in some narrow test environment and broaden the using range of the sensor. Low noise photoelectric amplification circuit is shown in the Figure 7.

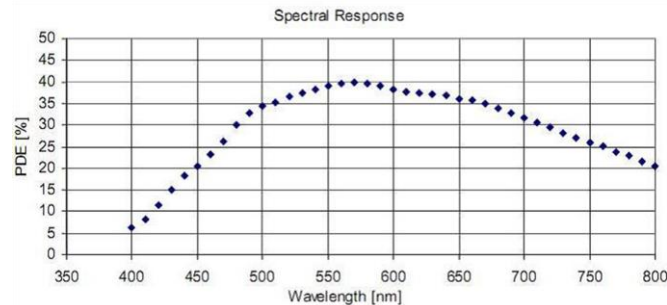


Figure 6. Spectral Response Curve of SSPM0701



Figure 7. Low Noise Photoelectric Amplifier

3. The Principle of Temperature Measurement Sensor

Put the Ta-ZrO₂ black body cavity in the temperature of T region, Monochromatic radiation flux $\Phi(\lambda, T)$ of λ is determined by the Planck blackbody radiation law, it is the single value function of the temperature T. To measure the blackbody temperature, only need to measure the blackbody at a given wavelength near the monochromatic radiation flux. As shown in the formula1, after the Radiation through the optical fiber to the solid state photomultiplier tube (SSPM) the voltage output can be expressed as

$$U(\lambda_0, T) = K \int_{\lambda_0 - \Delta\lambda/2}^{\lambda_0 + \Delta\lambda/2} \phi(\lambda, T) d\lambda = KR(T) \quad (1)$$

In the formula, λ and $\Delta\lambda$ are of the detection wavelength and bandwidth respectively, R(T) can be obtained by numerical integration, K depends on the insertion loss of the optical signal transmission in the process of various kinds of optical fiber transmission, coupling and other optical components and the sensitivity coefficient of narrowband photodetector. If ignore the loss caused by the temperature variation and emissivity change with temperature, it is a device constant that has nothing to do with the temperature, which can be obtained by static calibration. Since the K is independent of temperature that can be calibrated in a temperature only^[11].

4. Static Calibration Experiment of Sensor Sensitivity of K

4.1. High Precision Calibration of Static Sensitivity K

The sensitivity of K for the static calibration, can determine the relationship between the temperature and the optical amplifier output voltage. In the theory, K value can be calculated by the formula (1), but since the variety and complexity of the influence of the system, it is difficult to calculate accurately and depends on the experimental calibration [12].

According to Planck's law of blackbody radiation, the higher temperature of constant temperature zone, the higher the calibration precision. This paper presents a high temperature zone of high precision calibration scheme, which is shown in the Figure 8. The device is composed of an infrared thermometer, three jaw self-centering chuck and three sets of oxyhydrogen flame spray gun. The three root flame gun fixed in the hollow of three jaw self-centering chuck and adjusts the flame gun distance manually.

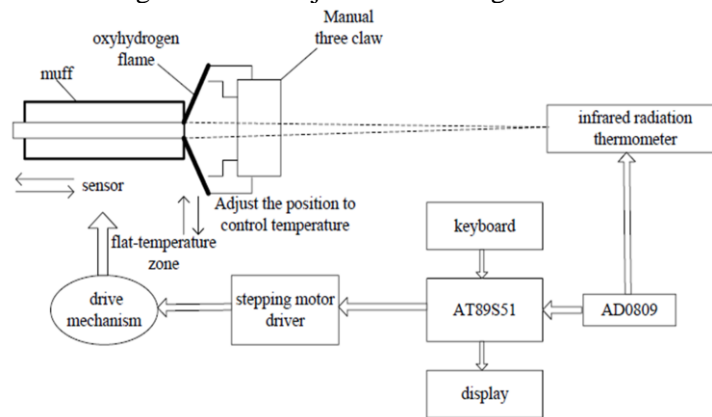


Figure 8. K-Value High Precision Calibration of a Sensor

The output analog signal of the infrared temperature instrument is acquired by AD0809 and input SCM. The SCM will be compared with the value of the set temperature and the value of the infrared thermometer measured temperature to determine whether the stepper motor drive to work. Drive sensor move back and forth and adjust the distance of oxyhydrogen flame and the sensor. Stepper motor drive mechanism of the ball screw drives mechanism. Display module using one of four static digital tube displays, which displays setting temperature value. Keyboard adopts independent buttons. The process is shown in the Figure 9.

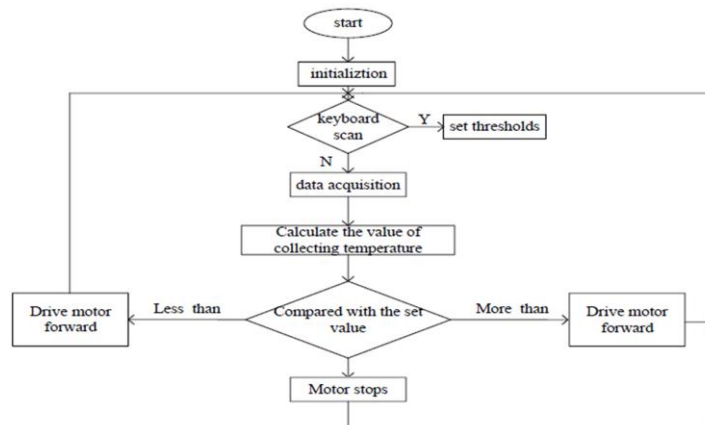


Figure 9. Program Flow Chart

Experiment chooses the zirconia is a diameter of 1.5 cm, 7cm high cylinder, suppose oxyhydrogen flame focus to the end of the zirconia circular spot and the diameter is 0.002 m, formed a diameter of 0.002 m hemispherical pool in the center position near the surface. The whole temperature field distribution after ANSYS thermal analysis is shown in Figure 10. The temperature curve of zirconia ceramic surface infrared thermal imaging face is shown in the Figure 11. As can be seen from the Figure 11, the highest temperature of the center in the end surface of the ceramic is 1791.12°C, the temperature fluctuation is small, precise control the temperature of constant temperature zone with the blackbody furnace calibrated infrared thermometer M3 and SCM, put the sapphire fiber blackbody cavity temperature sensor in the temperature region, which formed by oxyhydrogen flame, the static sensitivity coefficient K can be calculated using the formula (1).

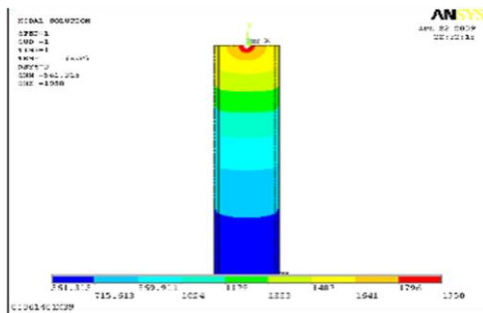


Figure 10. The Whole Temperature Field Distribution

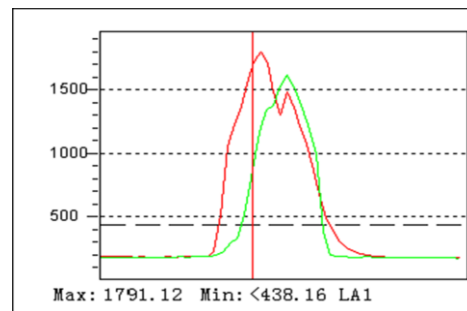


Figure 11. Temperature Curve of Transverse Plane

4.2. Voltage Withstand Test of the Transient High Temperature Sensor

Temperature sensor test device mainly comprises a pressure sensor, hammer pressure generator, the laser heating and temperature sensor, as is shown in the Figure 12. Exerting pressure on the temperature sensor probe and heating probe for temperature sensor with laser. Computer receives the output data of the pressure sensor and temperature sensor. As is shown in the Figure 13 and the Figure 14.

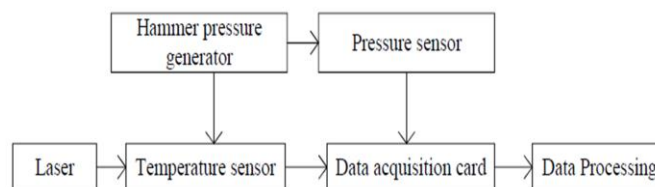


Figure 12. Pressure Resistance Test of Temperature Sensor

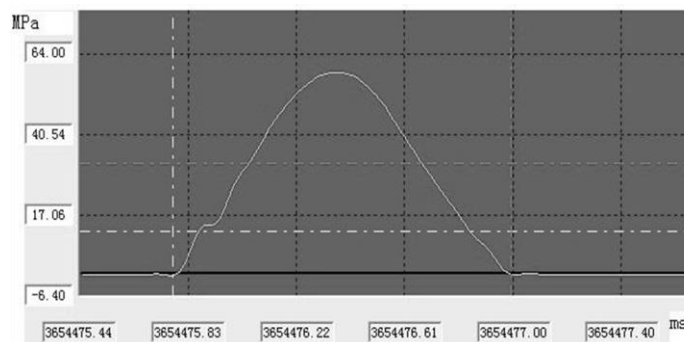


Figure 13. Pressure-Time Curve

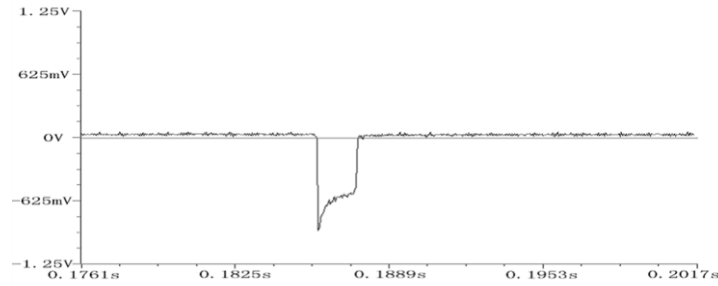


Figure 14. Voltage-Time Curve

According to the test data, when the hammer type pressure generator produces pressure is greater than 50 mpa, the maximum output voltage value of Temperature sensor is 903.8 mV, the K value of the static calibration is 65.084×10^6 ($V \cdot m^2/W$) and the corresponding temperature is $2802^\circ C$.

5. Calibration Experiment of Time Constant of the Sensor

The dynamic response of the sensor measurement is shown in the Figure 15. Adopt high power CO_2 laser beam with step upward to focus to sapphire fiber blackbody cavity temperature sensing head from the 45 degree mirror, the output of SSPM is recorded in the oscilloscope, get the output voltage temperature signal changes with the step laser signal. Since the time constant is influenced by many and complex factors, it is difficult to obtain accurate data with the theoretical calculation method, so in practical application use dynamic calibration experiment (wind tunnel or laser) method^[13]. The dynamic characteristics of the most of the temperature sensor can be approximately described by first-order system. Sensor step response curve, which the dynamic response of the sensor is represented by 63.2% of steady state time^[14]. As is shown in the Figure 16, which is about $794\mu s$.

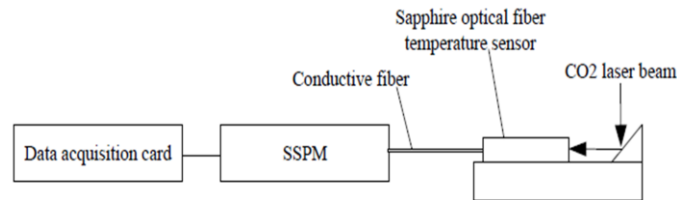


Figure 15. Response Time Measurement Principle

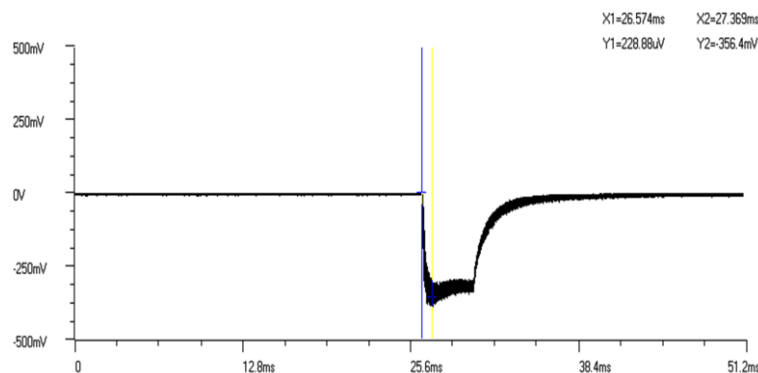


Figure 16. Dynamic Response Time of a Sensor

6. Conclusions

1) Tantalum oxide and zirconia blackbody cavity sensor sample is processed, adopt solid photomultiplier SSPM as photoelectric detector, which can be directly coupled with high gain and high response speed sapphire fiber and the small sapphire fiber blackbody cavity transient surface ultra-high temperature sensor is developed.

2) The value of the temperature, the pressure, the time constant of the sensor temperature is tested and provide a means of measurement to instantaneous ultra-high temperature (2000°C to 3000°C, the order of magnitude of response time is microseconds) under the harsh environment.

3) Design a 1721°C high temperature static calibration device, which composed of three water electrolysis oxyhydrogen flame gun, the internal temperature distribution of zirconia as the metal heat conductor is analyzed and simulated with the ANSYS, which determine the constant temperature zone and improve the calibration precision.

4) SEM test shows that tantalum zirconium oxide black body is ablated easily in ultra-high temperature, further research and explores a appropriate coating process for the blackbody cavity and prolong its service life.

Acknowledgments

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References

- [1] R. van de Krol, Y. Liang and J. Schoonman, "Solar hydrogen production with nanostructured metal oxides[J]", *Journal of Materials Chemistry*, vol. 20, no. 18, (2008), pp. 2311-2320.
- [2] X. Feng and T. J. LaTempa, "Ta₃N₅ Nanotube Arrays for Visible Light Water Photoelectrolysis[J]", *NANO Lett*, vol. 10, no. 3, (2010), pp. 948-952.
- [3] R. Abe, M. Higashi and K. Domen, "Facile Fabrication of an Efficient Oxynitride TaON Photoanode for Overall Water Splitting into H₂ and O₂ under Visible Light Irradiation[J]", *Journal of The American Chemical Society*, vol. 132, no. 34, (2010), pp. 11828-11829.
- [4] Q. Gao, C. Giordano and M. Antonietti, "Controlled Synthesis of Tantalum Oxynitride and Nitride Nanoparticles[J]", *Small*, vol. 23, no. 7, (2011), pp. 3334-3340.
- [5] J. B. Varley, A. Janotti and C.G. Van de Walle. Mechanism of Visible-Light Photocatalysis in Nitrogen-Doped TiO₂[J], "Advanced Materials", vol. 20, no. 23, (2011), pp. 2343-2347.
- [6] Fuding Lin, Shannon W. Boettcher. Adaptive semiconductor/electrocatalyst junctions in water-splitting photoanodes[J]. *Nature Materials*, 2014, 13: 81-86.
- [7] Thomas W. Hamann. Water splitting: An adaptive junction [J]. *Nature Materials*, 2014, 13: 3-4.
- [8] S.David Thlley Dr., Maurin Cornuz, et al. Light-Induced Water Splitting with Hematite: Improve Nanostructure and Iridium Oxide Catalysis [J]. *Angewandte Chemie International Edition*, 2010, 36(49):6405-6408.
- [9] Blaise A. Pinaud , Peter C. K. Vesborg and Thomas F. Jaramillo. Effect of Film Morphology Thickness on Charge Transport in Ta₃N₅/Ta Photoanodes for Solar Water Splitting[J]. *The Journal of Physical Chemistry*, 2012, 116(30): 15918-15924.
- [10] Hoang X. Dang, Nathan T. Hahn, et al. Nanostructured Ta₃N₅ Films as Visible-Light Active Photoanodes for Water Oxidation[J]. *The journal of Physical Chemistry*, 2012, 116(36): 19225-19232.
- [11] Monica Barroso, Camilo A. Mesa, et al. Dynamics of photogenerated holes in surface modified α -Fe₂O₃ photoanodes for solar water splitting[J]. *PNAS*, 2012, 109(39).
- [12] WANG Xiao-ming, HAO Xiao-jian, ZHOU Han-chang. Calibration system of sapphire-fiber temperature sensor's coefficient K[J]. *Instrument Technique and Sensor*, 2012, (2):1-27.
- [13] NICHOLAS J V, WHITE D R. Tranceable temperatures: an introduction to temperature measurement and calibration. 2nd ed. John Wiley & Sons, 2001:21-24.
- [14] MICHALSKI L, ECKERSDORF K, KUCHARSKI J, et al. Temperature Measurement. 2nd ed. John Wiley & Sons, 2001:279-33

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