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Improved High-Temperature Standard Platinum Resistance Thermometer

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Abstract To prevent short circuits, to improve stability, and to raise the upper temperature limit to the freezing point of copper (1084.62 °C), the high-temperature standard platinum resistance thermometer (HTSPRT) was redesigned. The most important change was an improvement in the structure of the sensor support. The strip support was replaced by a new specially designed cross support. The structure and design of the new HTSPRT are briefly described in this article. The test results of a group of thermometers are presented. The test included long-term drifts of the thermometers at the triple point of water and freezing point of silver during a period of a few hundred hours operation at 1085 °C, the short-term stability of R (tpw) and W (Ag) in a period of 5 days, and thermal cycles between 22 °C and 1085 °C. The test results show that the thermometer performance is improved, and the new HTSPRT can operate up to the freezing point of copper.

Keywords Fixed-point cell · High-temperature standard platinum resistance thermometer · ITS-90

1 Introduction

High-temperature standard platinum resistance thermometers (HTSPRTs) are used to interpolate temperature in the range from 661.323 °C to 961.78 °C on the International Temperature Scale of 1990 (ITS-90). Over the past 20 years, the reliability and performance of HTSPRTs have been troubled by instability and short circuits in the sensor coils. During the European Association of National Metrology Institutes (EUROMET) regional key comparison, EUROMET.T-K4 found that the selected HTSPRTs were

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less stable than desired, and one of the Bureau International des Poids et Mesures (BIPM) HTSPRTs failed due to a short circuit in the sensor [1]. Similar problems were reported in other laboratories such as the National Metrology Institute of Japan (NMIJ). There has been discussion about the possibility of replacing the HTSPRT with the Au/Pt thermocouple as the ITS interpolating instrument above the freezing point of aluminum. To continue using the HTSPRT as the ITS interpolating instrument, it is critical that its performance be improved.

In response to requests from national measurement institutes (NMIs), Fluke-Hart Scientific began a project to redesign its HTSPRT three years ago. The objective is to prevent short circuits, improve stability, and raise the upper temperature limit to the freezing point of copper (1084.62 °C). To reach this objective, the structure of the sensor support was redesigned and the manufacturing process improved.

To evaluate the performance of the new HTSPRT, a series of experiments were undertaken. Thorough testing showed that the HTSPRT can operate well at temperatures as high as 1085 °C. Results from tests of a group of thermometers are presented in this article. The tests included long-term resistance at the triple point of water [*R* (tpw)], drift during a period of over 1000 h at 1085 °C, short-term stability of *R* (tpw), and the resistance ratio at the freezing point of silver [*W* (Ag)] over a period of 5 days, and thermal cycle tests from room temperature to 1085 °C.

2 Improvements to Thermometer Structure and Manufacturing

In 1982, Xumo Li successfully developed a 0.25 Ω HTSPRT using a strip-shaped fused-silica support with notches [2]. The design with the strip fused-silica support was chosen because of its excellent stability during thermal cycling. The benefit of using a strip support is that the platinum sensing wire can expand and contract freely with temperature changes since the loops of the coil are supported at only two points. In the past 30 years, most of the best-performing HTSPRTs have used the strip fused-silica support. However, there have been occasional short circuits in the sensor coils after long-term operation at high temperatures, as shown in Fig. 1. Upon investigation, it was discovered that the sensor wire expanded during annealing and long-term exposure at high temperature and touched the wall of the sheath, which caused loops in the coil to bend and contact adjacent loops. Even if a short circuit does not occur, the stability of the thermometer could be significantly affected if coils contact the inner surface of the thermometer sheath during operation.

To prevent short circuits and improve stability, the strip fused-silica support was replaced by a new specially designed fused-silica cross support. Every loop of the wire now has four support points as the platinum sensing wire sits in the grooves of the cross support. The sensor wire never contacts the sheath, even as the wire expands. To allow the sensing wire to expand and contract freely with temperature changes, the angle of the groove, the diameter of the sensor coil and support, and the gap between the sensing wire and groove are carefully calculated and designed. A comparison of the construction of HTSPRT sensors with the strip support and cross support is shown in Fig. 2.

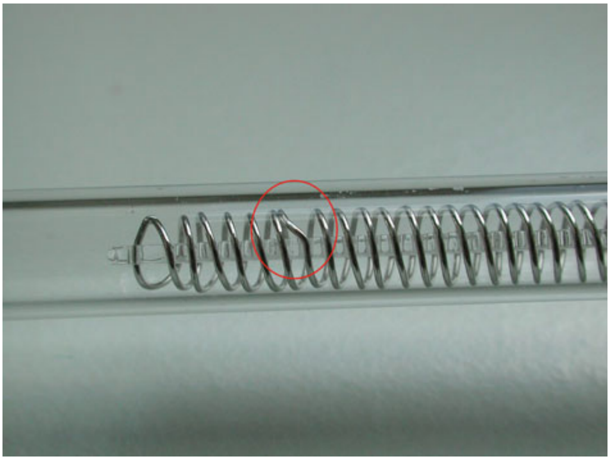

Fig. 1 Short circuit of HTSPRT sensor with strip support

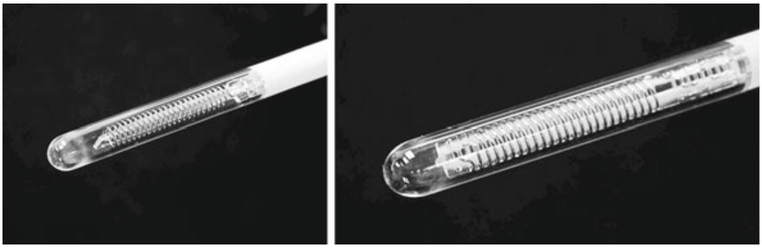

Fig. 2 Sensors with strip support and cross support

Since the new HTSPRT was developed 3 years ago, no short circuits in the sensor coil have been reported. Furthermore, after a few hundred hours of exposure to high temperatures, the coils around the fused-silica cross supports appear more uniform than those on strip supports. From experiments it was found that the R (tpw) stability of a group of thermometers with cross support was less than 0.4 mK during thermal cycling.

3 Experiment and Results

3.1 Measurement Apparatus

The resistance of the thermometers was measured using a Guildline 6675B automatic bridge with an external reference resistor, a Leeds & Northrup 1 Ω DC standard resistor. The triple point of water with a maintenance bath and the freezing point of silver with a sodium heat pipe furnace were used as temperature references. A furnace containing a non-contaminating block, with a range from 50 °C to 1100 °C, was used for heating and annealing the HTSPRTs. The measurement uncertainties ($k = 2$) at the triple point of water and freezing point of silver are 0.1 mK and 3.5 mK, respectively.

3.2 Long-Term Stability

After manufacturing, to improve stabilization, all the HTSPRTs were annealed in a non-contaminating annealing furnace. Before inserting the HTSPRT into the annealing furnace, the thermometer sheath was soaked in 50 % nitric acid for 1 min, and then rinsed by pure water for a few minutes. This is a very critical cleaning process to avoid sensor contamination and devitrification of the fused-silica sheath at high temperature. For most of the HTSPRTs, the initial stabilization period to pass the stability criterion was about 500 h. The stability criterion was that the *R* (tpw) drift must be less than 1.5 mK after 100 h of annealing at 1085 °C.

The long-term stability of the thermometers was tested at the triple point of water and the freezing point of silver (961.78 °C) after long-term exposure to 1085 °C. The *R* (tpw) and *W* (Ag) drifts for three representative thermometers are shown in Figs. 3 and 4. The thermometers were measured at the freezing point of silver after every 100 h exposure to 1085 °C, and the *R* (tpw) of the thermometers was measured before and after measurements at the freezing point of silver. The thermometers were annealed at 962 °C for 1 h after measurement at the freezing point of silver. After annealing, the

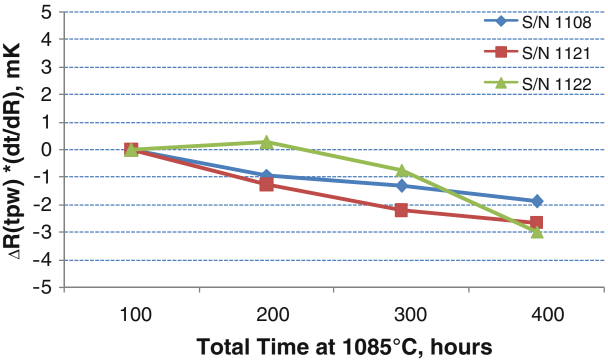

Fig. 3 Long-term stability at the triple point of water

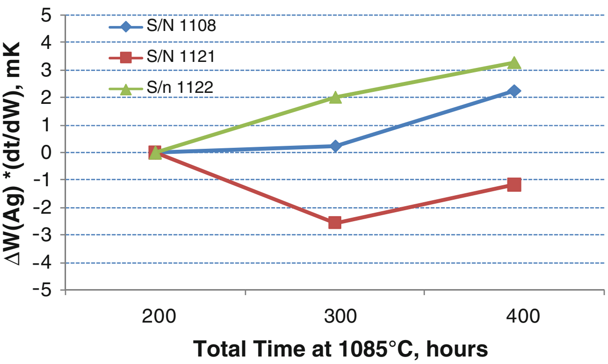

Fig. 4 Long-term stability at the freezing point of silver

furnace containing the thermometers was cooled to 480 °C at a rate of 1.8 °C · min^{-1}. The thermometers were then removed from the furnace and cooled to room temperature. The R (tpw) was measured once again.

The drifts of R (tpw) and W (Ag) were equivalent to less than 1.5 mK and 2.5 mK, respectively, after 100 h of operation at 1085 °C.

3.3 Short-Term Stability

To verify the short-term stability, the thermometers were measured at the freezing point of silver every day for one work week. The thermometers were measured at the triple point of water before the measurement at the freezing point of silver. After measurement at the silver point, the thermometers were annealed at 962 °C for 1 h, and then the furnace containing the thermometers was cooled to 480 °C at a rate of 1.8 °C · min^{-1}. The thermometers were then removed from the furnace and cooled to room temperature. The thermometers were measured at the triple point of water one more time. As an example, the test results of the three thermometers are shown in Figs. 5 and 6. The test results show that the short-term stabilities at R (tpw) are better than ±1.0 mK, and those of W (Ag) are better than ±2.5 mK.

3.4 Thermal Cycling

Cooling a thermometer rapidly from high temperatures to room temperature can quench lattice defects in the platinum crystal structure, causing the R (tpw) of the thermometer to increase [2]. For HTSPRTs used at high temperatures, the thermometers should be annealed for 2 h at the maximum operating temperature [3]. The thermal cycle performance of an HTPRT is very important, since the R (tpw) drift caused by thermal cycling may not be fully recovered after annealing. A thermometer with good design should have very low thermal cycling effects.

Five HTPRTs with the new fused-silica cross support were tested to investigate the effects of thermal cycling from room temperature to 1085 °C. Before the test, the

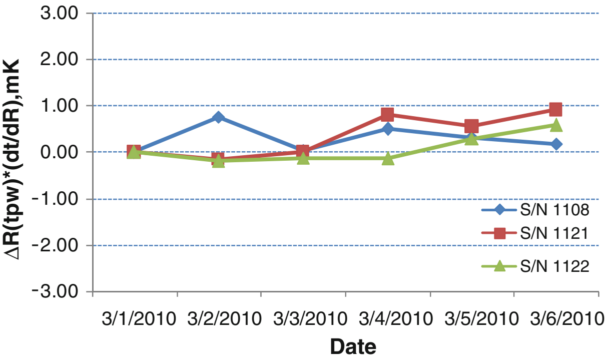

Fig. 5 Short-term stability at the triple point of water

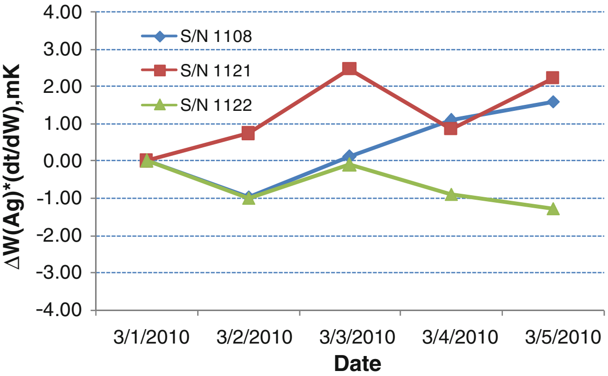

Fig. 6 Short-term stability at the freezing point of silver

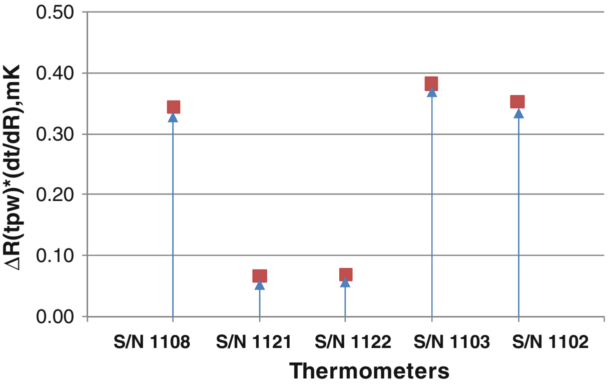

Fig. 7 *R* (tpw) changes after five thermal cycles from room temperature to 1085 °C

R (tpw) of the thermometer was measured. The HTPRT was exposed to 1085 °C for 30 min, and then the thermometer was removed from the furnace directly and cooled to room temperature. This test was repeated five times. During the fifth and final cycle, the thermometer was left in the furnace for 1 h of annealing and then cooled to 480 °C with a cooling rate of 1.8 °C $\cdot$ min^{-1}. After the temperature reached 480 °C, the thermometer was removed from the furnace to room temperature. The *R* (tpw) of the thermometer was measured again. The *R* (tpw) drift of five thermometers before and after thermal cycling was less than 0.4 mK, as shown in Fig. 7. The thermal cycle testing indicates that the newly designed HTPRT has an excellent thermal cycling performance.

4 Conclusions

To prevent the short circuits found with the HTSPRT designed with a fused-silica strip support, a new HTSPRT with a specially designed fused-silica cross support was developed. Thorough testing, including the long-term stability, short-term stability, and thermal cycling, showed that the new thermometer has excellent performance in the temperature range from the triple point of water to the freezing point of silver. The long-term *R* (tpw) stability was improved from the original specification of 3.0 mK per 100 h at 1070 °C to less than 1.5 mK per 100 h at 1085 °C. The long-term stability and thermal cycle testing support an extended temperature range of up to 1085 °C, around the temperature of the freezing point of copper. Since the new HTSPRT was developed 3 years ago, no sensor-coil short circuits have been reported. Based on these test results, the authors conclude that the performance of the newly developed HTSPRTs is better than that of a gold–platinum thermocouple, and that the HTSPRT should be kept as the interpolation instrument of the International Temperature Scale.

5 Future Research

Some limitations to further improvement of HTSPRT stability are the quality of the platinum wire and optimization of the filling gas. To improve the stability further, it may be necessary to cooperate with sensor wire manufacturers to improve the reference grade platinum wire, since it was found that the quality of platinum wire varied. Improvements in the filling gas mixture may also lead to better stability by properly balancing oxidation and contamination effects.

With the new design, as with previous designs, there might be some risk of lead wires shorting together after many years of use. This can be further investigated and possible improvements devised.

A significant factor in the performance of HTSPRTs at high temperatures is insulation resistance, especially at the freezing points of silver and copper. The insulation resistances of the HTSPRTs with strip-shaped quartz support developed by Xumo Li were examined in the 1980s [2]. Even though all other parts of the HTSPRT are the same as in the original design, since the support design has been changed, the insulation resistance should be tested again. This could be done using a specially constructed HTSPRT in which the sensor wire is cut at its mid-point. This provides a reliable method to measure the insulation resistance between the two pairs of current-potential leads.

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