AIP Conference Proceedings

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Citation: AIP Conf. Proc. **1552**, 445 (2013); doi: 10.1063/1.4819582
View online: http://dx.doi.org/10.1063/1.4819582
View Table of Contents: http://proceedings.aip.org/dbt/dbt.jsp?KEY=APCPCS&Volume=1552&Issue=1
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Hysteresis of thin film IPRTs in the Range 100 °C to 600 °C

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Abstract. As opposed to SPRTs, the IPRTs succumb to hysteresis when submitted to change of temperature. This uncertainty component, although acknowledged as omnipresent at many other types of sensors (pressure, electrical, magnetic, humidity, etc.) has often been disregarded in their calibration certificates' uncertainty budgets in the past, its determination being costly, time-consuming and not appreciated by customers and manufacturers. In general, hysteresis is a phenomenon that results in a difference in an item's behavior when approached from a different path. Thermal hysteresis results in a difference in resistance at a given temperature based on the thermal history to which the PRTs were exposed. The most prominent factor that contributes to the hysteresis error in an IPRT is a strain within the sensing element caused by the thermal expansion and contraction. The strains that cause hysteresis error are closely related to the strains that cause repeatability error. Therefore, it is typical that PRTs that exhibit small hysteresis also exhibit small repeatability error, and PRTs that exhibit large hysteresis have poor repeatability. Aim of this paper is to provide hysteresis characterization of a batch of IPRTs using the same type of thin-film sensor, encapsulated by same procedure and same company and to estimate to what extent the thermal hysteresis obtained by testing one single thermometer (or few thermometers) can serve as representative of other thermometers of the same type and manufacturer. This investigation should also indicate the range of hysteresis departure between IPRTs of the same type. Hysteresis was determined by cycling IPRTs temperature from 100 °C through intermediate points up to 600 °C and subsequently back to 100 °C. Within that range several typical sub-ranges are investigated: 100 °C to 400 °C, 100 °C to 500 °C, 100 °C to 600 °C, 300 °C to 500 °C and 300 °C to 600 °C . The hysteresis was determined at various temperatures by comparison calibration with SPRT. The results of investigation are presented in a graphical form for all IPRTs, ranges and calibration points.

Keywords: Industrial platinum resistance thermometers; Thermal hysteresis; Thin-film PRT; R-T characteristics; Uncertainty factors.

INTRODUCTION

Resistance thermometers (RTDs) are temperature sensors that exploit the change in electrical resistance of some materials, such as nickel, copper and platinum, with changing temperature. Among mentioned, platinum is most commonly used material for resistance thermometers (PRTs). This is because of its favorable characteristics such as best accuracy, stability and widest temperature range among the common RTD materials, limited susceptibility to contamination, fairly linear resistance versus temperature curve (R-T), high resistivity, stable physical properties and sufficient mechanical strength.

Platinum resistance thermometers are used for many different applications in temperature range roughly between -260 °C and 1000 °C [1]. For highest possible accuracy, temperature sensing element is made from high-purity platinum wire, which is wound in configuration that is as strain-free as possible (standard platinum resistance thermometers SPRTs). This makes SPRTs very sensitive instruments, which are not applicable in harsh industrial environments. On the other hand, industrial platinum resistance thermometers (IPRTs) are designed to be resistant to mechanical shocks, vibrations, electromagnetic fields. This is accomplished by providing a mechanical support to the platinum sensing element.

There are two main designs of IPRT sensors, glass or ceramic-encapsulated sensors and thin-film sensors. In glass-encapsulated and ceramic-encapsulated sensors, sensing element is a thin platinum wire, bonded to or embedded in glass or a ceramic body, whereas in the thin-film sensors, sensing element is made of very thin platinum film, deposited on the alumina substrate. In both cases, platinum is not longer able to expand and contract freely with temperature, which inevitably causes strain of platinum sensing element. The most prominent factor that contributes to the hysteresis error in an IPRT is a strain within the sensing element caused by thermal expansion and contraction [2, 3]. Thermal hysteresis represents one of the main uncertainty factors in uncertainty budgets of calibrations by comparison [4]. The strains that cause hysteresis error are closely related to the strains that cause repeatability error. Therefore, it is typical for

Temperature: Its Measurement and Control in Science and Industry, Volume 8
AIP Conf. Proc. 1552, 445-450 (2013); doi: 10.1063/1.4819582

PRTs that exhibit small hysteresis to exhibit also small repeatability error, and PRTs which exhibit large hysteresis to exhibit poor repeatability.

Aim of this paper is to provide hysteresis characterization for a batch of IPRTs using the same type of thin-film sensor, encapsulated by same procedure and by same company and to estimate to what extent the thermal hysteresis obtained by testing one single thermometer (or few thermometers) can serve as representative of other thermometers of the same type and manufacturer. This investigation should indicate the range of hysteresis departure between IPRTs of the same type. Hysteresis was determined by cycling IPRTs temperature from 100 °C through intermediate points up to 600 °C and subsequently back to 100 °C. Within that range several typical sub-ranges are investigated: 100 °C to 400 °C, 100 °C to 500 °C, 100 °C to 600 °C, 300 °C to 500 °C and 300 °C to 600 °C. The hysteresis is determined at various temperatures by comparison calibration with SPRT. The results of investigation will be presented in a graphical form for all IPRTs, ranges and calibration points.

MEASUREMENTS

Tested IPRTs

Group of eight PT100, class B thermometers, assembled by Austrian company was used in this study. Thin film sensors, used in thermometers were produced by the same company. Design of thin film sensors is shown in Figure 1. The platinum layer is applied to a ceramic body in a sputter process and subsequently given a meander-structure in a lithographic process. Precise resistance adjustment is then carried out in a laser trimming process. To protect the sensor against external influences and for insulation purposes, the platinum meander is coated with a glass layer. The electrical connection is made by connection wires welded onto the contact surfaces. An additional glass layer applied to the contact surface fixes the connection wires and also serves as a tension relief. The connection wires are made of pure palladium. Dimensions of a sensor are 2 mm x 10 mm x 1.2 mm and the application temperature ranges between -50 ... +600 °C.

The thin-film platinum sensors are fitted into stainless steel sheaths with a diameter of 6 mm and length of 450 mm. For precise resistance measurements, four low resistance copper wires were welded to a sensor. The sheaths were filled with aluminum oxide in order to insulate electrically lead wires and the sheath.

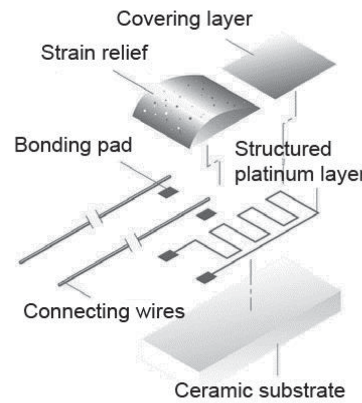

FIGURE 1. Design of a thin film sensor.

Measurement Equipment

Temperature range selected for hysteresis measurement was from 100 °C to 600 °C. In order to avoid transferring the standard and the test thermometers form one isothermal zone to another, dry-well calibrator was used for hysteresis testing. In this way, possible change of thermometers R-T characteristics due to thermal and/or mechanical shock was minimized. Calibrator was interfaced to a PC using RS-232 communication port and temperatures were automatically cycled by custom-made LabVIEW software. Although the measurement temperatures were controlled by the built in calibrator sensors, actual temperatures and their stability were determined using the calibrated metal sheathed standard platinum resistance thermometer (SPRT) with measurement uncertainty of 2 mK. The thermometers resistances were measured with an ASL F700B AC Resistance Bridge, which when used with a 100 Ω standard resistor, has the resolution of 1 mK for a SPRT and 0.3 mK for PT100. The bridge was connected to a 10-channel scanner, and all the data were collected through an IEEE-488 connection to a computer. The average balance time for the bridge was 18 to 20 seconds, giving approximately 100 seconds for the one measurement cycle with five thermometers. The standard resistor used was Wilkins-type 100 Ω, and it was kept immersed in thermostated oil bath kept at temperature of 23 °C.

Measurement Procedure and Analysis

The thermal hysteresis of eight identical industrial PRT thermometers was investigated. The aim of the measurements performed was to determine:

1. Hysteresis dissipation among all tested IPRTs after exposure to thermal cycling between 100 °C and 600 °C
2. Influence of temperature span on hysteresis
3. Influence of thermal cycling on hysteresis
4. Influence of temperature change rate on hysteresis

Four IPRTs were tested in each hysteresis cycle. The SPRT and the test thermometers were inserted inside 200 mm deep borings of a calibrator equalizing block. In order to get precise resistance measurements, all thermometers were connected to the bridge in four-wire configuration. The calibrator temperature was then cycled between 100 °C to 600 °C with steps of 100 °C. In order to protect both the SPRT and tested PRTs, the temperature change rate was limited to 3.3 °C per minute in the range between 100 and 400 °C and to 1.6 °C per minute in the range between 400 and 600 °C. Resistance measurements of the SPRT and the thin-film PRTs were collected automatically after a predefined period of time, which was determined separately for each temperature step, based on time needed for stabilization in previous cycles. Although majority of temperature measurements were performed with temperature stability being within boundaries of 10 mK, there were a few readings taken while stability was as poor as 20 mK. Those readings were, however, not rejected as hysteresis results were consistent with those obtained by rest of the measurements. Time required for performing one temperature loop in the temperature range from 100 °C to 600 °C, with the temperature steps of 100 °C was approximately 24 hours with stabilization time of 2.5 to 3.5 hours between steps.

Hysteresis was calculated for each tested IPRT as a difference between resistance readings taken on same temperature in both rising and falling direction. Conversion of IPRTs resistance readings to its temperature equivalent was performed by use of quadratic temperature/resistance equation:

$$R(t) = R_0\left(1 + At + Bt^2\right);\ t > 0\ ^\circ\mathrm{C} \qquad (1)$$

where t is temperature, R_0 is the resistance at $t = 0\ ^\circ\mathrm{C}$, $R(t)$ is the resistance at temperature t, and A and B are constants. In order to be able to represent hysteresis data obtained from the several temperature cycles or of the several IPRTs on the same chart, values of R_0, A and B were recalculated for each IPRT in each separate temperature cycle by averaging the resistances taken at same temperatures for increasing and decreasing temperatures.

After completing hysteresis tests, calibrator was subjected to stability and uniformity investigations to determine its uncertainty contribution to the hysteresis measurements. It is reasonable to assume that calibrator uniformity didn't affect the hysteresis measurement uncertainty as thermometers remained at same position, during the tests. Any deviations in IPRTs readings caused by temperature gradients inside equalizing block should be present in the approximately same amount at the same temperature point, and cancel out after subtracting the IPRTS's readings at the particular temperature.

Two SPRTs were used to obtain uncertainty contribution due to calibrator uniformity. Rest of the calibrator borings were filled by IPRTs, assuring these way conditions similar to those present during hysteresis tests. Calibrator was cycled between 100 °C and 600 °C, and uniformity was calculated based on the change of the difference between two SPRT's readings at s same temperature point. Results of the calibrator investigations are presented in table 1.

TABLE 1. Calibrator stability and uniformity

Contribution	Uncert. at 100 °C (k=2), mK	Uncert. at 300 °C (k=2), mK	Uncert. at 600 °C (k=2), mK
Stability, 30min	3	4	9
Uniformity	1	2	2

PERFORMED TESTS AND RESULTS

Actual measurements started with temperature cycling of first group of four thermometers in the temperature span between 100 °C and 600 °C. Results obtained in the fifth loop were similar to those obtained in the previous one and thermometers were considered to reach stability in regard to hysteresis. To save some testing time, second group of four thermometers was exposed to thermal cycling prior to taking any measurements. As first group revealed repeating of similar hysteresis results after conducting five temperature loops, second group of thermometers was also cycled for five times in temperature range between 200 °C and 600 °C, using a furnace with maximum rate of 2.5 °C/min. No measurements were taken during first five cycles. Thermometers were then placed into the calibrator equalizing block and connected to the resistance bridge in a same way as it was done with thermometers from group one. Thermometers used in group two were exactly the same as ones used in group one (same producer, same manufacturing procedure, same production batch, same type of thin-film platinum sensor).

Hysteresis curves of all tested IPRTs, after exposure to thermal cycling, are shown in Figure 2. The chart on the left side represents the differences between the ascending and the descending temperature readings of IPRTs while the chart on the right side shows the differences between the SPRT and the IPRTs readings taken at same temperatures. In the both charts solid line represents a mean value of readings taken from all tested IPRTs, while dotted lines represent the maximum and the minimum value of the same readings. The hysteresis data were calculated from the readings obtained in the fifth cycle for the first group and from eighth cycle for the second group of IPRTs. The thermal hysteresis was distributed from 84 mK to 116 mK, with all sensors showing maximal values of hysteresis at the temperature of 400 °C. The readings on rising temperatures were lower than those obtained when temperature was decreasing. Summary of the hysteresis results for all eight IPRTs is given in table 2.

TABLE 2. Hysteresis results for eight IPRTs tested.

Sensor mark	Hyst. at 200 °C, mK	Hyst. at 300 °C, mK	Hyst. at 400 °C, mK	Hyst. at 500 °C, mK
T1	43	83	106	85
T2	45	80	106	78
T3	39	74	99	75
T4	59	100	115	89
T5	34	55	84	82
T6	40	62	90	85
T7	56	83	99	90
T8	40	60	87	85

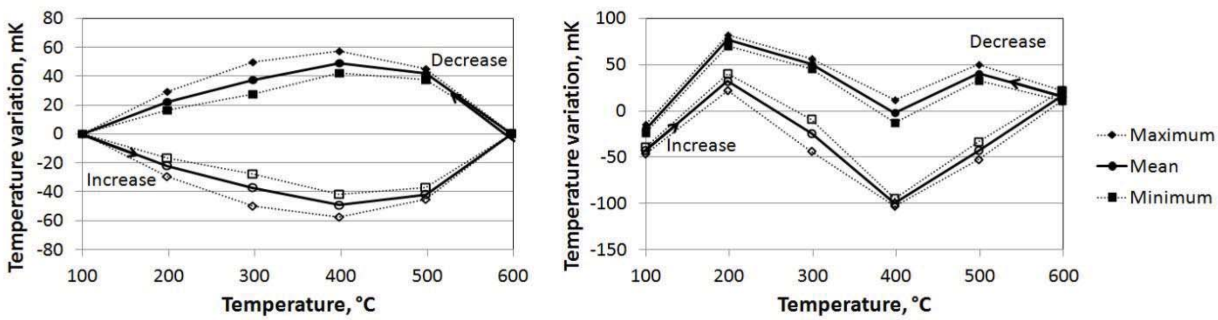

FIGURE 2. Hysteresis curves of tested IPRTs obtained after exposure to thermal cycling in the range from 100 °C to 600 °C.

Figure 3 shows change of hysteresis of the first group of thermometers, depending on the number of cycles performed, starting from the first measurement cycle, immediately after thermometers were delivered from producer. Mean values of all thermometers are used for presentation.

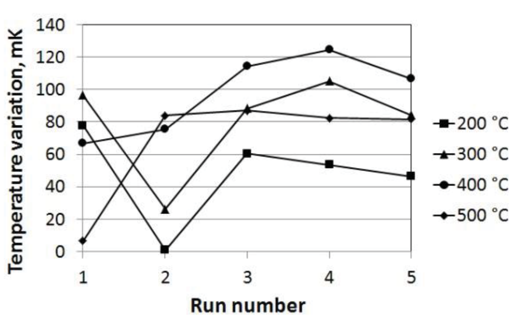

FIGURE 3. Variations of the hysteresis values of first group of IPRTs tested over first six measurement cycles.

Despite the fact that second group of thermometers was cycled for five times in a furnace prior to performing measurements, readings taken in the sixth cycle gave increased value of hysteresis in comparison to the mean hysteresis of all thermometers, obtained in a way described above. The hysteresis difference was 80 mK (mean hysteresis of all PRTs was 98 mK and hysteresis measured in the sixth cycle of the second group PRTs was 178 mK). This indicates that hysteresis results depend not only on a number of cycles done within the same temperature limits but also on the way cycles are conducted, (meaning here on the rate of temperature change and on the number of temperature steps). Hysteresis increase of the second group of IPRTs was also observed in the twelfth loop, which was performed after measurements in the narrower temperature spans, regardless of the fact that IPRTs were already cycled for eight times. Figure 4 shows the hysteresis curves obtained in the sixth, eighth and twelfth cycle of a second group of IPRTs. For simplicity of presentation, mean values of four thermometers were used.

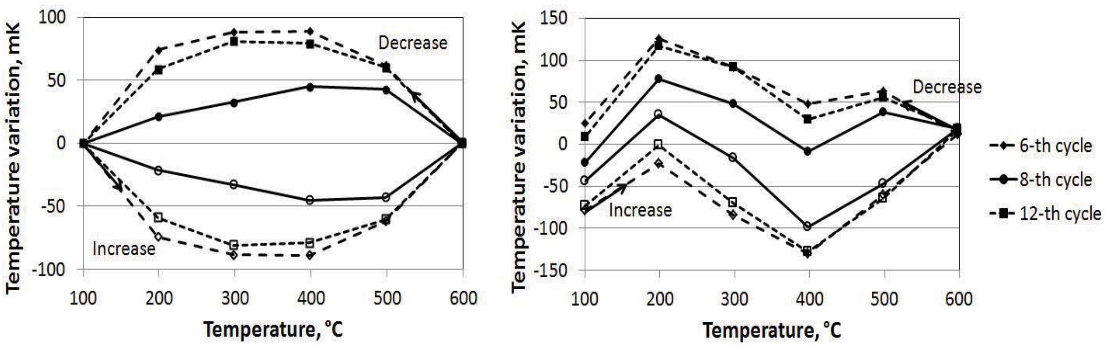

FIGURE 4. Hysteresis curves in the sixth, eighth and eleventh measurement cycle for the second group of IPRTs.

In order to determine the influence of temperature span to the hysteresis, both groups of thermometers were subjected to three additional measurement cycles, all starting at temperature of 100 °C and having steps of 100 °C. Aim of such assessment was to determine if it is possible to predict the thermal hysteresis in the wider temperature span on the basis of data obtained for the limited range.

Second group of thermometers was additionally cycled between 300 °C and 600 °C. Obtained results indicate that hysteresis of the tested IPRTs decreased drastically with narrowing temperature span only when starting at temperature of 100 °C. In case of cycling between 300 °C and 600 °C, hysteresis remained almost at the same level as when cycling between 100 and 600 °C. Graphical representation of results is given in Figures 5 and 6.

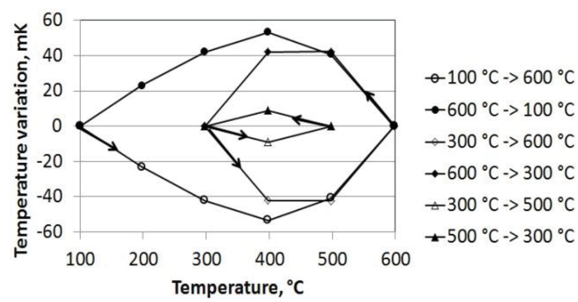

FIGURE 5. Hysteresis curves of second group of IPRTs obtained for a different temperature spans.

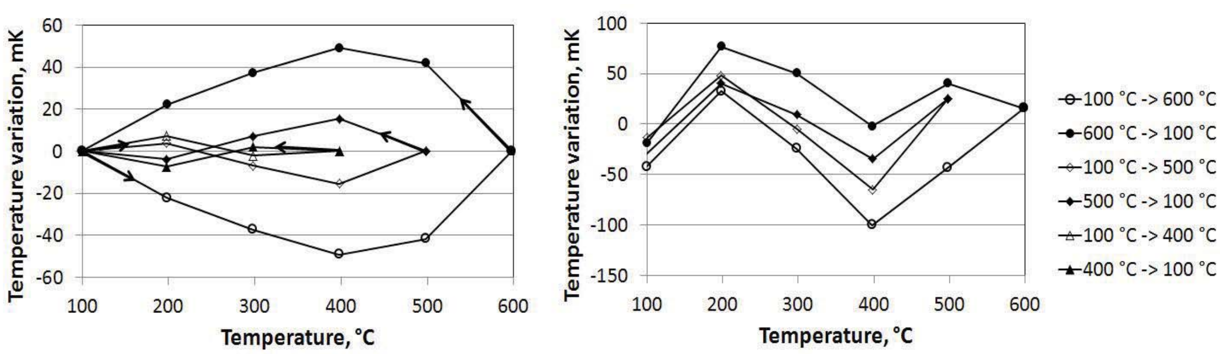

FIGURE 6. Hysteresis curves of all tested IPRTs, obtained for a different temperature spans.

Determination of the influence of temperature change rate on hysteresis was performed over the first group of IPRTs. For the purpose, IPRTs were cycled with three different rates: 1.6 °C/min, 3.0 °C/min and 2.3 °C/min. Measurements were performed in a following sequence: 100 °C – 400 °C – 600 °C – 400 °C – 100 °C. Intermediate temperature of 400 °C, was chosen based on results acquired during previous tests, as all sensors revealed maximum hysteresis on this temperature. Although it would be sufficient for hysteresis assessment to take resistance readings only at 400 °C, resistances were also recorded at 100 °C and 600 °C to allow the calculation of the coefficients required for presentation of results in temperature equivalents. Reason for analyzing hysteresis at just one intermediate temperature point, instead of many, was to leave enough time for temperature change (increase or decrease) to stabilize and advance at a desired rate. Results of test are given in Table 3.

TABLE 3. Hysteresis at different rates of temperature change.

Sensor mark	Hyst. at 1.6 °C/min, mK	Hyst. at 2.3 °C/min, mK	Hyst. at 3.0 °C/min, mK
T1	158	84	130
T2	162	90	139
T3	158	85	132
T4	165	102	142

Obtained results indicate that magnitude of hysteresis depends on speed of temperature change.

CONCLUSIONS

In order to investigate hysteresis in a batch of same IPRTs, various tests were performed using a dry-well calibrator in the range between 100 °C and 600 °C. The thermal hysteresis of eight tested IPRTs was distributed within 84 mK and 116 mK after five thermal cycles, which represent dissipation of ±15 % around the mean value of 100 mK. Obtained results confirmed the complexity of the hysteresis phenomenon as it showed dependence on virtually every aspect of change in thermal environment where IPRTs were tested. Results of tests performed during this investigation indicate that in our particular case, thermal hysteresis obtained by testing of few thermometers (or even one single thermometer) can serve as representative of other thermometers of the same type but only to a certain degree. Furthermore, this conclusion applies only to hysteresis results obtained by testing with the same procedure, under same conditions and with a sufficient number of thermal cycles performed. Nevertheless, if similar test data were provided by the manufacturer covering the full nominal temperature range, they would give good indication to the user what to expect regarding hysteresis for thermometers of the same type and manufacturer.

REFERENCES

1. Michalski, L., Eckersdorf, K., and McGhee, J., *Temperature Measurement,* Chicester: Wiley, 1991, pp. 14-16, 100-111
2. Curtis, D.J., *Thermal Hysteresis and Stress Effects in Platinum Resistance Thermometers, Temperature, Its Measurement and Control in Science and Industry, vol. 5*, New York: American Institute Of Physics, 1982, pp. 803-812
3. Zwak, J., *Error Sources That Effect Platinum Resistance Thermometer Accuracy Part 5 – Hysteresis*, Minnetonka: Burns Engineering, pp. 1-4
4. Hashemian, H.M., and Petersen, K.M., *Themperature, Its Measurement and Control in Science and Industry, vol.6*, New York: American Institute Of Physics, 1992, pp. 427-432