How Does Temperature Non-uniformity of an Annealing Furnace Affect SPRT Stability?

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Abstract

A series of investigations to determine how temperature non-uniformity of the annealing furnace affects SPRT stability was recently performed. Three stable SPRTs were annealed in furnaces with different temperature uniformity profiles: ±1°C, ±3°C, and ±8°C. Resistances of the SPRTs at the triple point of water (Rtpw) were measured and compared before and after annealing. In this paper, the method of investigation and test equipment used will be presented, and the test results will be reported. Based on the test results, the influence of the annealing furnace temperature non-uniformity on SPRT stability will be discussed.

1. Introduction

To achieve the highest level of performance, a standard platinum resistance thermometer (SPRT) must be handled and maintained correctly. With proper care and treatment, an SPRT can provide years of accurate, stable measurements. Without proper handling, an SPRT can be damaged beyond repair in a short period of time. Investigations show that SPRTs outside tolerance limits have been inadvertently used as standards in calibration work [1]. In one case, careless day-to-day handling of an SPRT over a one-year period increased its resistance at the triple point of water (Rtpw) by an amount equivalent to 0.1 K. It is inevitable that an SPRT will receive at least a small amount of mechanical or thermal shock during operation. Oxidation of the platinum sensor wire can also cause the resistance increase.

Proper annealing is necessary to mitigate the effects of mechanical and thermal shock and oxidation and maintain SPRT accuracy and stability. An SPRT must be annealed after transportation, mechanical shock, or operation above 500°C. It is usually annealed in an annealing furnace that has a non-contaminating equilibrium block [5] with the temperature tightly controlled. The recommendation from national metrology institutes (NMIs) and published literature is that the temperature uniformity should be within ±1°C.

In recent years, more temperature calibration laboratories have begun using SPRTs as their reference standards to improve calibration uncertainties. However, many laboratories cannot afford a proper annealing furnace to anneal their SPRTs. Some laboratories use alternative equipment such as dry-well calibrators. Temperature uniformity sometimes does not meet the criterion set by NMIs. Therefore, it is important to understand how temperature non-uniformity affects SPRT stability and what equipment can be used for SPRT annealing.

In order to determine how temperature non-uniformity of the annealing furnace affects SPRT stability, a series of investigations was recently performed at Fluke-Hart Scientific. Three-zone annealing furnaces that allow the vertical temperature gradient to be adjusted were used. Three stable SPRTs were annealed in the furnaces with different temperature profiles. Resistances of the SPRTs at the triple point of water (Rtpw) were measured and compared before and after annealing.

In this paper, the method of investigation and test equipment used will be presented, and the test results will be reported. Based on the test results, the influence of annealing furnace temperature non-uniformity on SPRT stability will be discussed.

2. Annealing of a Standard Platinum Resistance Thermometer

During daily operation and handling, an SPRT is subjected to mechanical shock, which induces strain in the sensor wire, resulting in a change in resistance. Mechanical shock can be incurred by slight taps while inserting or removing the SPRT from an instrument or by disturbance in a stirred temperature bath. Vibration and jolts during transport are other sources of mechanical shock. Even with great care, mechanical shock can cause significant changes in the SPRT resistance. Annealing the SPRT at 660°C for one hour can eliminate most of the strain caused by minor shocks and restore the resistance close to its original value. Figure 1 is a good example showing that necessity of SPRT annealing during the calibration interval. The Rtpw of the SPRT continued to drift upward until the SPRT was annealed in November, 1994, at which time the Rtpw shifted back to the original resistance.

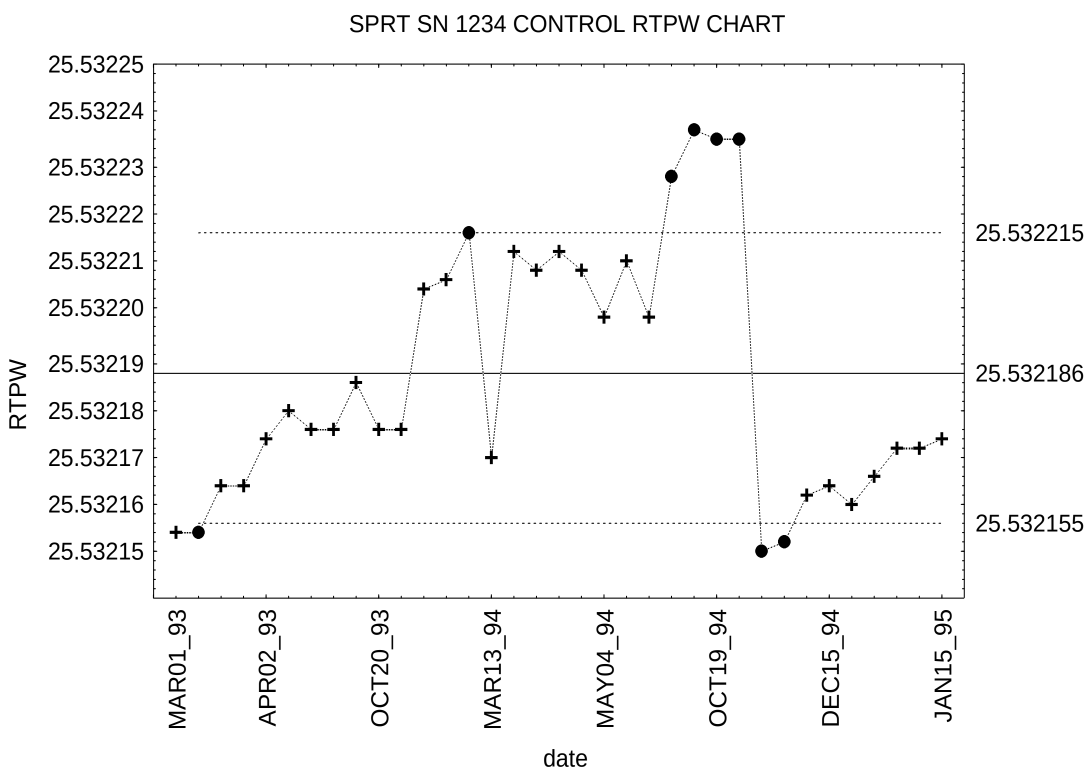

Figure 1. SPRT control chart with annealing.

All solids inherently contain defects. A “crystalline defect” is defined as a lattice irregularity having one or more of its dimensions on the order of an angstrom. Point defect, a type of crystalline defect, is associated with one or two atomic positions in the crystalline structure. The simplest and most common point defect is a vacancy or vacant lattice site, one normally occupied but from which an atom is missing. Vacancies are formed during solidification and as a result of atomic vibrations. The concentration of the point defects is dependent upon the temperature. The equilibrium concentration of vacancies (N_v) in the pure platinum wire of an SPRT increases exponentially to an increase in temperature as expressed by

$$N_v = N \exp\left(-\frac{Q_v}{kT}\right)$$

where N is the total number of atomic sites, Q_v is the vibration energy required for the formation of a vacancy, T is the absolute temperature in Kelvins, and k is Boltzmann’s constant.

For most metals, the fraction of vacancies (N_v/N) just below the melting point is on the order of 10^{-4} or one lattice site out of every 10,000 [2]. Removing an SPRT from high temperature and rapidly cooling it to room temperature traps this high concentration of point defects in the crystalline structure, causing an increase in resistance. This increase can be as high as 30 mK equivalent. Annealing the SPRT at 700°C for two hours then gradually cooling it can significantly reduce the increase due to the trapped point defects. The SPRT should be cooled to at least 500°C at a rate of roughly 100°C per hour. Once the SPRT has reached 500°C, it may be removed immediately to room temperature without harm.

According to the supplement to the ITS-90, “after using a high-temperature resistance thermometer at temperatures above about 700°C, the thermometer should be annealed before making measurements at lower temperatures, in particular before making the measurements at R_{TPW}” [3]. The National Institute of Standards and Technology (NIST) has also provided an annealing procedure for SPRT calibration [4].

3. Experiments and Discussion

3.1. Measuring equipment

A model 6675A DC Bridge is used as the measuring equipment in the study. The nonlinearities of the bridge are better than 0.02 ppm according to the manufacturer’s specifications. The reference resistors used with the bridge were maintained in baths at 25°C ± 0.01°C. The stabilities of their resistances were better than 2 ppm per year. The triple point of water was used throughout the investigation. The expanded uncertainties (k=2) for the triple point of water is 0.1 mK. The resistance of the SPRT at the triple point of water (Rtpw) is used as the main indicator to monitor SPRT stability.

3.2. Annealing furnaces with different temperature uniformity profiles

Three non-contaminating annealing furnaces with different vertical temperature gradients were used in the experiments. The three furnaces had the same design and construction. The thermal equilibrium blocks inside the furnaces were made from 99.8% pure alumina and had five holes for SPRTs. They were held within high-purity, specially cleaned fused-quartz containers. Five fused-quartz tubes with one end closed were inserted into the alumina block. Several alumina disks and fiber ceramic disks were placed on the top of the alumina block alternately for insulation. Previous research confirms that a fused-quartz sheath SPRT is not contaminated by an alumina block. The three annealing furnaces were adjusted to have different vertical temperature profiles through three-zone control, as shown in Figure 2.

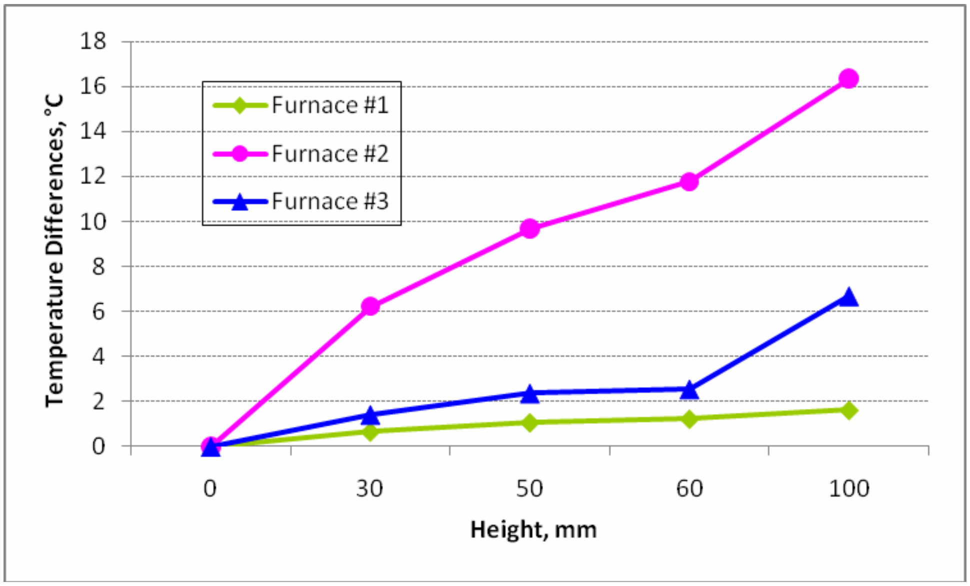

Figure 2. Temperature profiles of the annealing furnaces.

3.3. Standard platinum resistance thermometers

The SPRTs were specially designed and assembled for this study. Their sensing elements were longer than normal, 55 mm compared to the usual 50 mm. It was assumed that the longer element would be affected more by temperature non-uniformity. The three SPRTs were stabilized through an annealing process after manufacturing. The long-term stabilities of all three SPRT at the triple point of water were better than 1 mK after operation at 675°C for a few hundred hours.

3.4. Testing procedure

Before the experiments were started, the three SPRTs were annealed at 675°C for 4 hours, then they were cooled down to 480°C at a rate of 1.9 °C/min, at which point the SPRTs were removed from the furnace and their Rtpw measured. During the investigation, the SPRTs were annealed and measured in the following sequence:

(a) Anneal the SPRTs at 675°C for 15 hours in Furnace #1, cool down to 480°C at a scan rate of 1.9°C/min, remove from the furnace, and measure Rtpw.
(b) Repeat (a) four times.
(c) Anneal the SPRTs at 675°C for 15 hours in Furnace #2, cool down to 480°C at a scan rate of 1.9°C/min, remove from the furnace, and measure Rtpw.

(d) Repeat (c) four times.
(e) Anneal the SPRTs at 675°C for 15 hours in Furnace #1, cool down to 480°C at a scan rate of 1.9°C/min, remove from the furnace, and measure Rtpw.
(f) Repeat (e) four times.
(g) Anneal the SPRTs at 675°C for 15 hours in Furnace #3, cool down to 480°C at a scan rate of 1.9°C/min, remove from the furnace, and measure Rtpw.
(h) Repeat (g) four times.
(i) Anneal the SPRTs at 675°C for 15 hours in Furnace #1, cool down to 480°C at a scan rate of 1.9°C/min, remove from the furnace, and measure Rtpw.
(j) Repeat (i) four times.

3.5. Effect of annealing temperature non-uniformity on SPRT stability

By observing Rtpw drift as the SPRTs were annealed in furnaces with different temperature profiles, the authors attempted to determine how annealing temperature non-uniformity affected SPRT stability. The results show that there was no apparent degradation of SPRT stability when the annealing furnace had up to 16°C (±8°C) of temperature non-uniformity. The measurements are plotted in Figures 3 and 4. The stabilities of the SPRTs are similar for all three furnaces. One of the three SPRTs, S/N 94002, was not stable during the experiment, but this appears to be caused by a defect rather than temperature non-uniformity. The faulty SPRT was removed from the experiment.

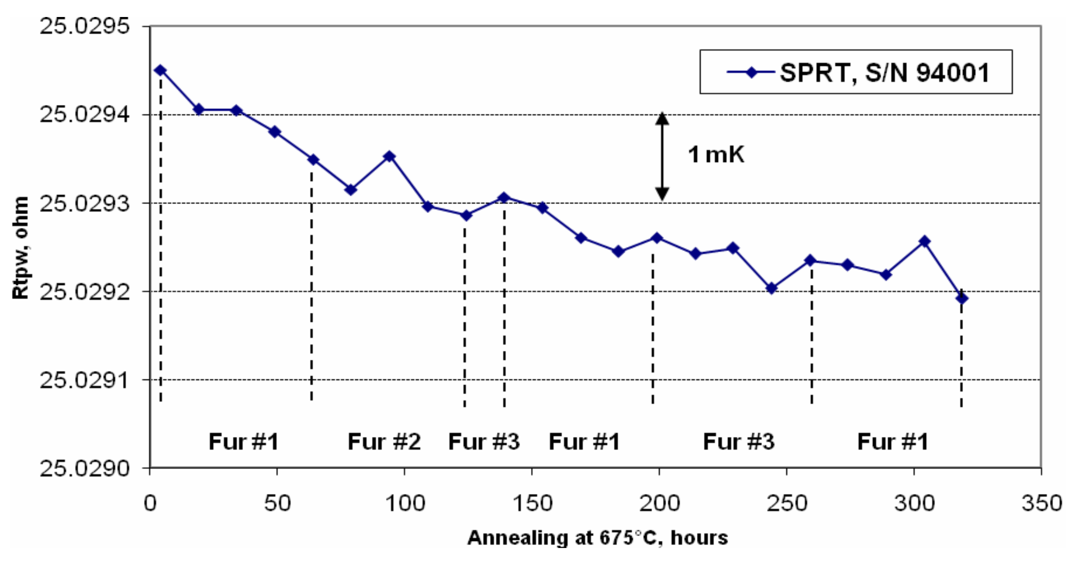

Figure 3. Stability of SPRT S/N 94001 annealed in different furnaces.

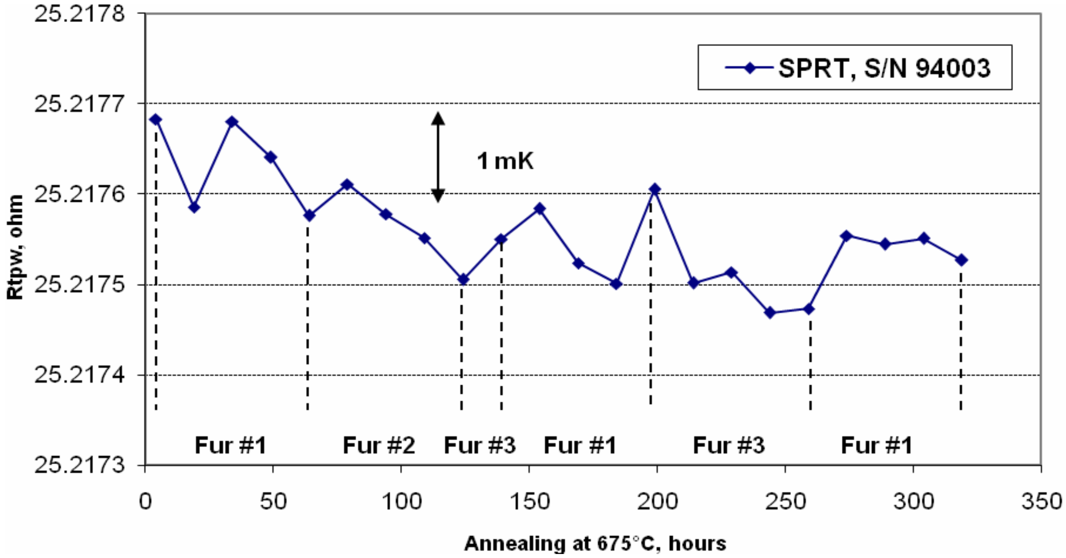

Figure 4. Stability of SPRT S/N 94003 annealed in different furnaces.

4. Conclusions

Vibration, shock, and temperature cycling during handling and use cause strain in an SPRT, which changes its resistance. Proper annealing removes strain and oxidation effects and is required to maintain accuracy and stability. Having an annealing furnace is not an option but a necessity for temperature calibration laboratories that own SPRTs. But there is the question of what type of furnace is needed and how well temperature uniformity must be controlled.

Based on the investigation, temperature non-uniformity of the annealing furnace does not seem to affect SPRT stability significantly. But it is too early to conclude that an annealing furnace with large temperature non-uniformity is adequate for a primary standard laboratory or a national laboratory. But perhaps a lower cost furnace, even a dry-well calibrator, is sufficient for a secondary temperature calibration laboratory. In any case, to avoid contamination, a fused-quartz sheath SPRT or HTSPRT should not be annealed in a metal block furnace above 660°C [5]. It also remains to be seen whether SPRTs that have drifted can be annealed just as well in a furnace that has large temperature non-uniformity. Further research is needed.

Acknowledgments

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