Industrial Resistance Thermometry up to 700°C without Drift, or Contamination

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Abstract

For many years Platinum Resistance Thermometers for industry (Pt100) have been limited to 450°C or so, because of drift in the Pt100 Resistor and contamination from the protecting stainless steel sheath.

Recent developments in glass technology and new high temperature alloys have enabled Pt100's to be developed for stable use up to 700°C and sheaths which will not contaminate the Pt100 Resistors. Thus the prospect arises that, up to and slightly beyond the Aluminium point of ITS-90 (660.323°C). Simple construction now exists for industrial usage offering long and stable results.

Introduction

A key temperature for glass is what is known as the lyttleton point, simply it is that temperature where the glass characteristics begin to change from those of a solid, to those of a liquid.

A glass was found with lyttleton point well above 700°C and other characteristics compatible, both with Platinum and Alumina (the other constituents of the Pt100).

Results

In the first tests the Pt100's were assembled with 4 Platinum lead wires, in ultra clean quartz sheaths thus eliminating the possibility of contamination from the sheath.

A number of elements were tested by sequentially cycling them to higher and higher temperatures, measuring the Triple Point of Water and Gallium Point resistances after each cycle.

A typical set of results is presented in Figures 1 and 2.

As will be seen from Figure 1 a small amount of annealing occurred during the first 3 or 4 cycles up to 400°C, thereafter the results remained very stable up to 700°C. Ro then slowly increased until around 900°C and then decreased fairly rapidly reaching a minimum at around 1150°C.

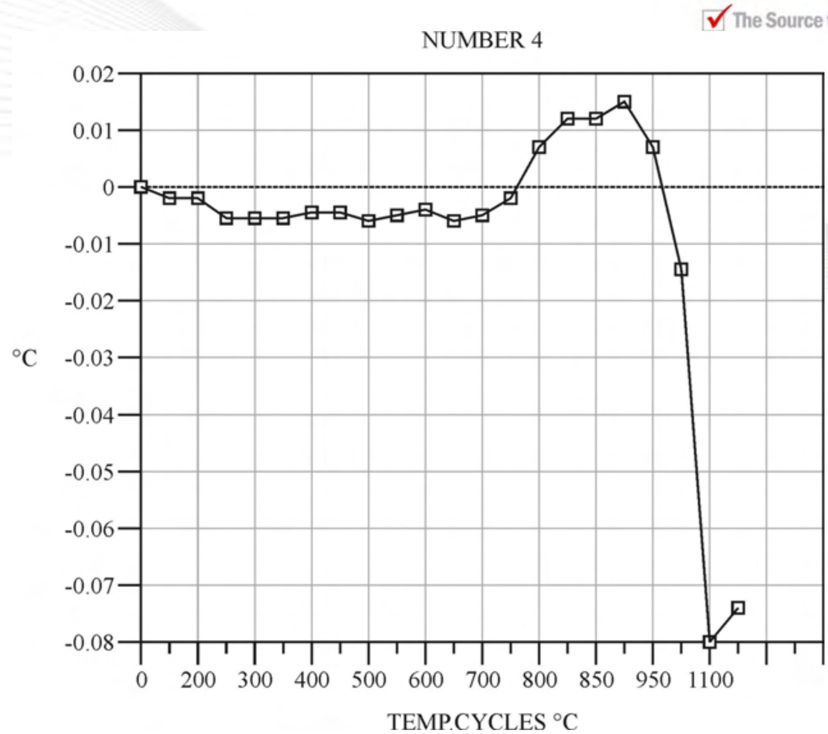


Figure 1. Change in °C at the Triple Point of Water

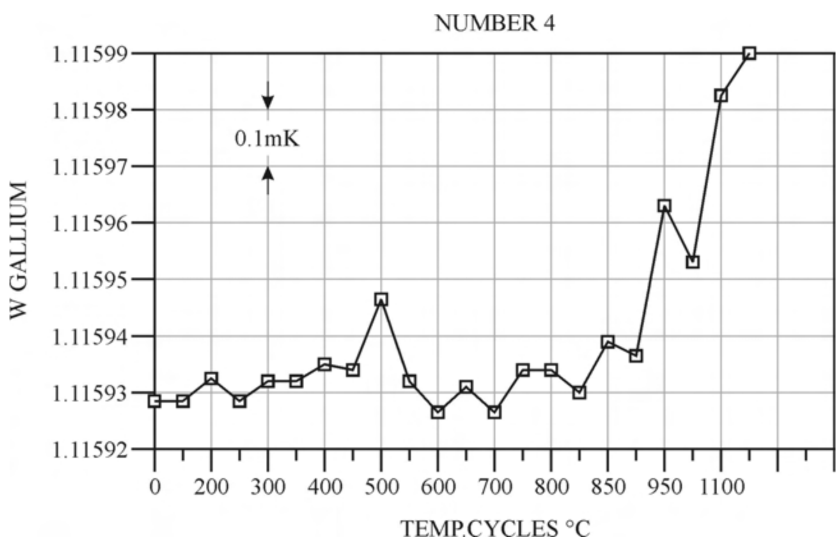


Figure 2. Change in W Gallium

Figure 2 is also interesting. W Gallium (a measurement indicating the Platinum materials state and purity) remained constant until 900°C and then increased. These results are considerably different than those expected of a Standard Platinum Resistance Thermometer. What I believe is happening is that the constituents added to the pure Platinum to reduce its slope from 3925 to 3850 begin to volatilise at 900°C. However this is irrelevant to the performance at 700°C.

The results of a number of trials indicate that a Platinum Resistor stable up to 700 or 750°C can be consistently produced at an economic price. Above 700 or 750°C a small positive shift occurs in the Ro of the Platinum Resistor due to induced strain as the element goes through the lyttleton point. Wga is not affected.

Only after 900 to 950°C is reached do changes occur in the composition of the Platinum Alloy used to make the Platinum Resistors.

In handling Standard Platinum Resistance Thermometers (SPRT's) special annealing procedures have to be adopted to avoid vacancy quenching. In this series of tests with the industrial thermometers no special precautions were taken and no vacancy quenching effects were noted – presumably this is because there are no vacancies in the alloy. This could explain the results of some researchers who have obtained more stable results with high temperature SPRT's if a lower purity is used.

Having successfully developed a Platinum Resistor with stable results up to 700°C and above, our attention focused on finding a metallic sheath 6mm diameter in which to house the Resistor.

The basic problems of encapsulation revolve around two key temperatures, the temperature at which the stearates used in drawing the tubing to size (a mixture of lanolin and soap) carbonise, their organic vapours are death to the Platinum Resistor and can contaminate it by tens of degrees equivalent in a few hours, at temperatures as low as 200°C.

The solution to this is quite simple; the cleanliness of the tubing, welding and assembly procedures can ensure that this is not a problem.

More intractable is that most metals give off metallic vapours well before they melt, for example stainless steel forms iron vapour at temperatures above 450°C. Platinum becomes contaminated rapidly by these vapours under reducing conditions, such as those found inside sheaths.

Special non-oxidising materials such as high temperature inconels have been successfully used after special preparation for high temperature use.

Unfortunately no articles or information has been published about the preparation of the sheath materials, specifically for this type of application.

We tried a number of commercially available alloys, using pre-ageing techniques in either air or vacuum, according to manufacturer's instructions.

Assemblies were made up using Silver lead out wires (the original test used Platinum lead outs) and as in the original tests the assemblies were cycled to higher and higher temperatures. We limited the maximum to 700°C since this was our goal.

A set of typical results are given below in Table 1.

Table 1 – R_{TPW} after Temperature Cycling

As received / cycled to	RTPW
	99.9682
400°C	99.9633
500°C	99.9636
550°C	99.9641
600°C	99.9638
650°C	99.9631
700°C	99.9619
700°C	99.9628
700°C	99.9628

As can be seen, the results mirror those of the quartz sheathed tests. At least 3 materials and a number of the manufacturers recommended ageing procedures worked in producing a contamination-free assembly up to 700°C.

Having settled on a material and construction, we made 3 production batches to test the results in a normal commercial situation. A typical procedure and set of results is presented in Table 2.

The model reference of our high temperature 100Ω Semi-Standard is 935-14-95.

Standard test procedure is to place the assemblies into a furnace set to 670°C with a 24 hour timer set 4 hours on / 4 hours off.

Table 2 - R_{TPW} after Temperature Cycling

SERIAL NO.	**1**	**2**	**3**	**4**	**5**
Ro after 6 cycles	100.0070	100.0038	100.0050	100.0079	100.0016
After a further 3 cycles	100.0060	100.0024	100.0037	100.0073	100.0002
A further 3 cycles	100.0068	100.0027	100.0036	100.0065	100.0009
And 3 more cycles	100.0062	100.0013	100.0031	100.0058	100.0002

Conclusion

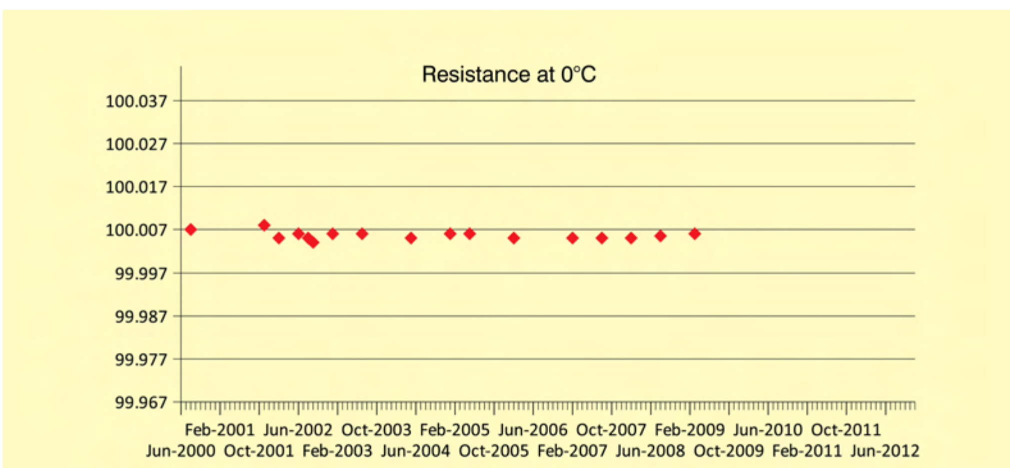
A new and exciting product has been developed giving stable and reproducible results up to 700°C. The techniques and materials used make the 6mm diameter unit commercially attractive and the assembly can be produced in any quantity at an economic price.

Part 2 – The Real World

Since the development of the 935-14-95 (Part 1) many hundreds have been produced and in constant use throughout the world. The experience of one user who gave us permission to use his 12 year results illustrates the real world performance of the product.

ISOTECH

" I thought that you might like to see the calibration history of one of our probes from the past 12 years – 935-14-95.
You may recall that we purchased this probe to use as a laboratory standard when our company was still young. This particular probe is still in daily use and is regularly taken to 600°C in our dry block calibrator. While we handle it with care, being in daily use for 12 year it has take the occasional mild knock and accidental abuse - I believe that it was even taken to close to 700°C once, although I wasn't involved so I can't testify to the temperature reached.
You'll see from the attached history of the calibration by our NMI that the probe has remained stable and accurate, and bearing in mind the daily variations in temperature that it has undergone, these results are testimony to the high quality of this product.
John, you are to be congratulated on developing and producing such a fine measuring instrument, and feel free to use us as a product reference any time
Kind regards,
Guy Snelling
Temperature Metrologist
InterCal (South Africa) "



The Platinum Resistors developed for 700°C can be obtained from Thermal Developments International Ltd – info@t-d-i.co.uk and have the suffix H.T.P.F.