A NEW THERMOMETER FOR THE COPPER POINT

J. P. Tavener[1]

[1] Isothermal Technology Ltd (Isotech), Southport, United Kingdom
E-mail: info@isotech.co.uk, Tel: +44 (0) 1704 543830

Abstract

The ITS-90 has Silver (961.78°C) as the upper limited for Contact Thermometry.

During the 1980's when ITS-90 was being designed a lot of work was done by researchers such as John Evans, Dr. Nubbermier and others to develop a High Temperature Thermometer to work up to the Copper Point (1084.62°C). Their efforts failed because of unexpected contamination problems and so the scale was restricted to the Silver Point.

Up to, and including the Silver Point a ¼ ohm High Temperature Standard Platinum Resistance Thermometer (HTSPRT) exists with sub-mK reproducibility. The problem is to extend the design a further 120°C to the copper point.

The main hurdle is to find a reliable sheath material. Quartz glass has an annealing temperature of 1050°C and leaks ions which contaminate the platinum coil.

Recrystalised alumina is widely used at high temperatures, and is impervious to ions except chromium. Unfortunately alumina is not completely gas tight. A sapphire sheath would be perfect but will not accept even mild thermal shocks. Of eight sapphire sheathed HTSPRTs made by this author all eight fractured.

One solution could be to put a +Ve air pressure inside an alumina sheathed SPRT so that the very slow flux was outward through the sheath.

The slow flux of air would also keep the platinum windings under oxidizing conditions (platinum loves oxygen hates reducing atmospheres).

This article presents results from this thermometer design at the Cu point.

Keywords: Copper Point, Thermometer

Introduction

There is a significant demand for Copper Cells and Apparatus for thermocouple calibration. However the uncertainties associated with measuring the Copper Point are larger than those at the Silver Point for example. The major source of measurement uncertainty is the sensor. The preferred sensor at the millikelvin level would be a quarter ohm Platinum Resistance Thermometer.

This presentation describes the results obtained with a new design.

The design

1 The Sheath – Recrystalised alumina was chosen for its rigidity. Because alumina is slightly porous the inside of the senor was kept at a slight positive pressure compared to the outside of the sheath using oxygen rich gas. The theory being that any leakage would be outward. Alumina has a tempco of about 9ppm/°C and is thermally fragile and so the sheath is designed to be easily replaceable.

2 The Mandrel on which the Platinum sensor is wound is made of synthetic sapphire because firstly sapphire is a more nobel insulator than either quartz or alumina, and secondly its expansion and contraction closely matches that of the Platinum.

3 The Bias – The thermometer is biassed to +9vDC. The reasons are two fold. Firstly biasing the thermometer increases its insulating resistance and secondly a positive voltage acts as an electro magnet repelling the metalic ions.

4 Physically – The thermometer is 500mm long, 7.8mm diameter with a 100mm long handle and 2 metres of lead.

The rest of the Apparatus

A microK 100 was used in conjunction with a Temperature Controlled 1Ω Tinsley Resistor.

The Copper Point was realised in an Isotower described elsewhere. It is however significant to mention that the Copper Cell is housed in a metalic enclosure, including the re-entrant tube in which the thermometer was positioned during measurements.

Testing

Testing was simple, the thermometer was used repeatedly for the melts and freezes of the Copper Cell in the Isotower. Between each sequence Rtpw and Wga were measured. Part way through the testing the Copper Cell was heated to 1100°C under vacuum to remove oxygen since 1ppm of oxygen in Copper can depress its transition point by 5mK.

Results

The complete history of thermometer measurements is tabulated below, taken from the thermometers log [Table 1].

Immersion Test

Because, uniquely the Immersion Compensator's temperature can be adjusted the gradient over the bottom 100mm is 2 or 3mK. (less than 1mK over the bottom 40mm)

Table 1 - History of 108462/S

Rwtp & Wga			Rcu	
.236, 0412,2(Ω)		M	1.092,387(Ω)	↑
		F	1.092,362(Ω)	116 Hours
		M	1.092,350(Ω)	
		F	1.092,308(Ω)	↓

Rwtp & Wga			Rcu	
.236, 034,0(Ω)	1.118120	M	1.092,310(Ω)	↑
		F	1.092,302(Ω)	47 Hours
		M	1.092,300(Ω)	
		F	1.092,299(Ω)	↓

Rwtp & Wga			Rcu	
.236, 029,7(Ω)	1.118175	M	1.092,298(Ω)	↑
		F	1.092,294(Ω)	↓53 Hours

Rwtp & Wga			Rcu	
.236, 040,0(Ω)	1.118129	M	1.092,293(Ω)	↑
		F	1.092,287(Ω)	↓68 Hours

Rwtp & Wga			Rcu	
.236, 040,8(Ω)	1.1181244	M*	1.092,287(Ω)	↑
		F*	1.092,283(Ω)	↓25 Hours

Rwtp & Wga				
.236, 041,0(Ω)	1.118133			

Rwtp & Wga			Rcu	
5 Cycles		7 x M & F	-27(mK)	310 Hours

All measurements use 10(mA)

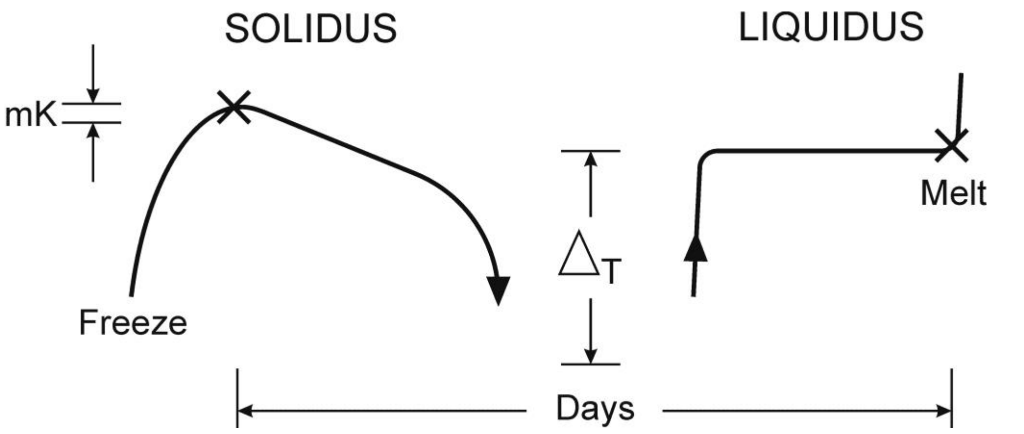
*After Copper Cell deoxidised.

Discussion

Considering Table 1

The resistances marked M and F are the liquidus and solidus resistances.

Table 2 – Thermometer Drift over a Freeze/Melt



Date	**F to M**	**ΔT(°C)**	**F/M (mK)**
3-8 Jan 2010	5 days	1085	+2
8-9 Jan 2010	1 day	5	-2
9-29 Jan 2010	20 days	1085	-1
1-2 Feb 2010	3 days	1085	-1
3-6 Feb 2010	4 days	1085	0

Graphs 1 & 2 illustrate how they were obtained.

Graph 3 charts Rwtp and Wga and shows that over the test period of 5 weeks these values remained stable having no significant drift or contamination. This is amazing considering the thermometer was being repeatedly put into and removed from a metalic re-entrant tube at 1084.62°C. The outside of the thermometer's sheath was blackened by the end of the tests.

Graph 4 charts Rcu during the tests. After the intital sequence where the thermometer was stabilising; over a period of 300 hours at or above 1085°C the total downward shift is 30mK or around 0.1mK/hour. Inspection of this graph shows that the majority (25mK) of the drift occured while the thermometer was above the liquidus temperature. In fact it occured during the cold rod process where the thermometer exits the cell at 1084°C and is quenched in air for a minute while the cold rod nucleates the Copper along its re-entrant tube – the thermometer reaches 400°C before it is replaced in the Copper Cell. What is still a puzzle is why, when Rcu is slowly decreasing Rtpw and Wga remain stable.

However at 0.1mK/hour the numbers are relatively small compared to alternative sensors.

Thermometer drift during a melt or freeze.

The change in Rcu has been identified as mainly happening during the cold rod process.

To discover the drift during a melt or freeze it is necessary to consider the changes in Rcu during a freeze followed by a melt (F→M). Table 2 shows 5 such cycles that occured during testing.

In 4 of the 5 cycles the thermometer was cooled over 3 hours to 480°C and then Rwtp and Wga were measured [see Table 2].

To summarise the table, independent of the number of days between freeze and melt or whether the thermometer's temperature excursion is 5°C or 1085°C the solidus/liquidus temperature change was between 0 and 2mK.

So the thermometer drift is between 0 and 1 mK for a melt or freeze [also see Table 1].

Conclusion

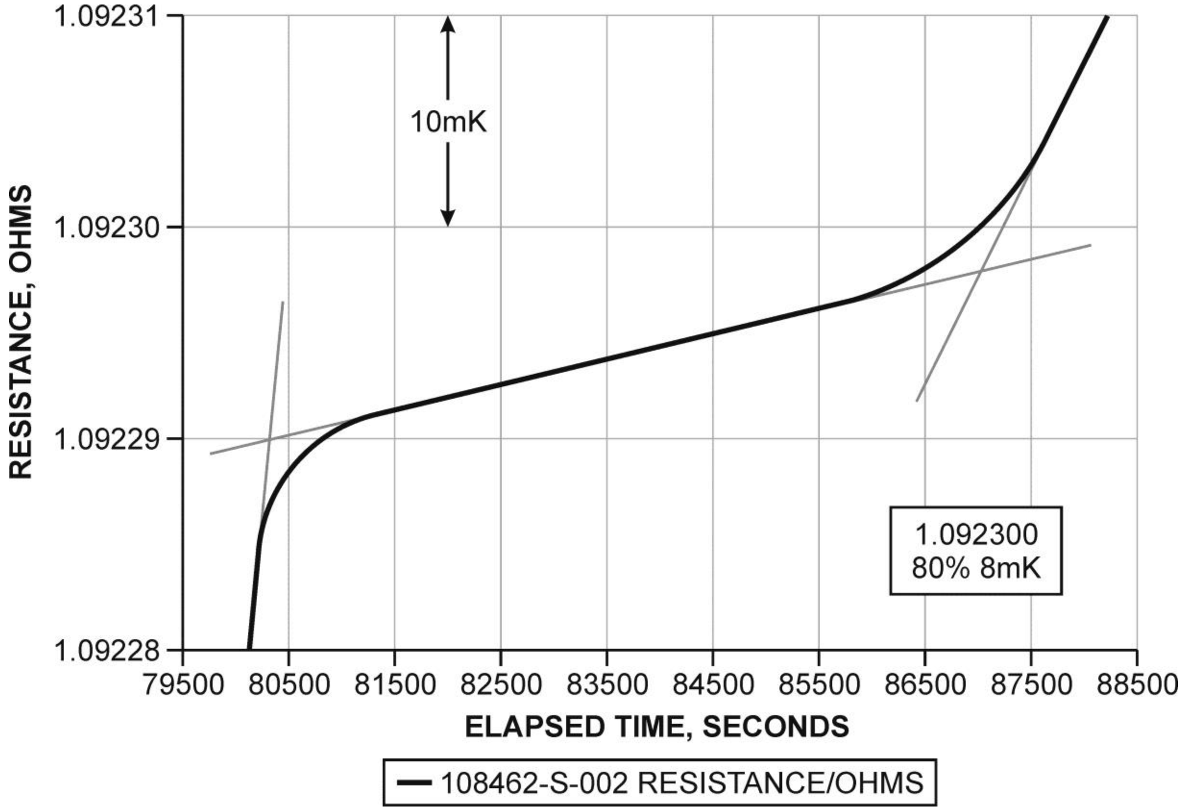
The new thermometer works up to 1085°C with little drift (0.1mK/hour) and no contamination (stable Rwtp and Wga). It is therefore suitable for the intercomparison and characterisation of Copper Cells at uncertainties smaller than previously achieved.

Further work

A second thermometer, made to the same design is available and will be tested in a similar way to the first. Both thermometers will be used in our UKAS laboratory at the Silver and Copper Point.

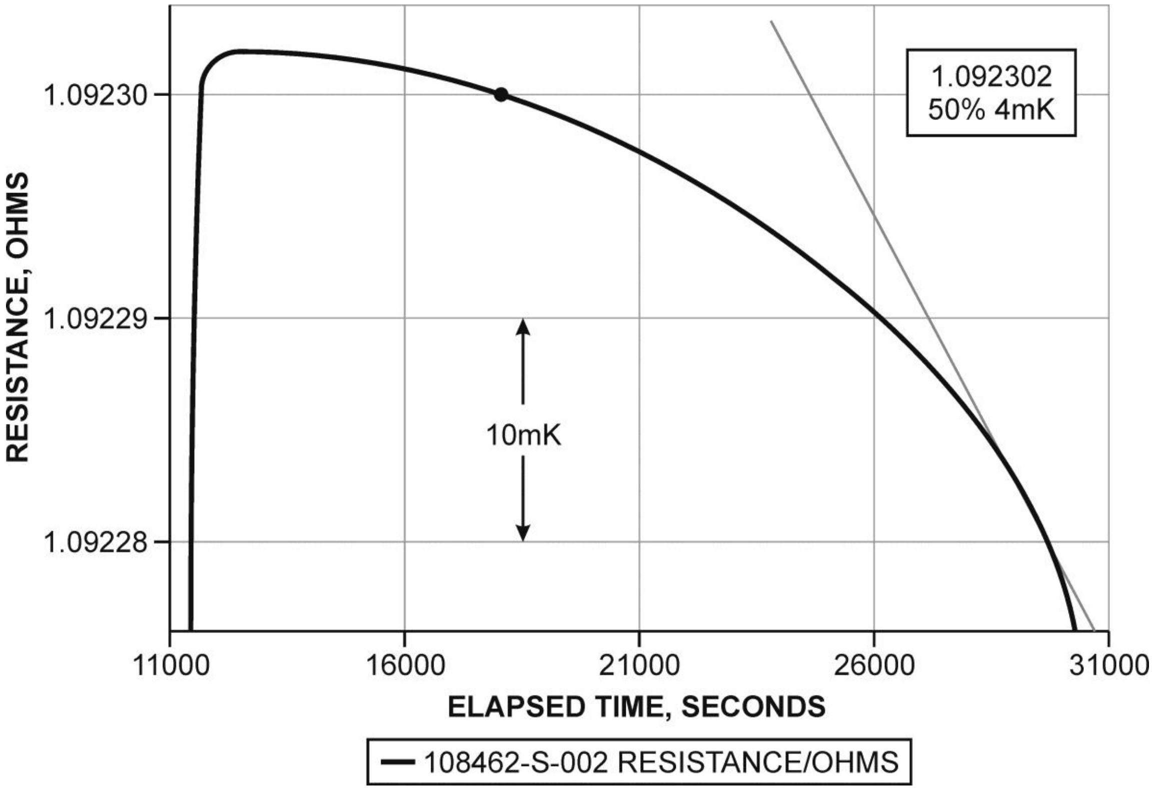
GRAPH 1 - Syphonic Copper Cell – Melt Plateau

9th January 2010

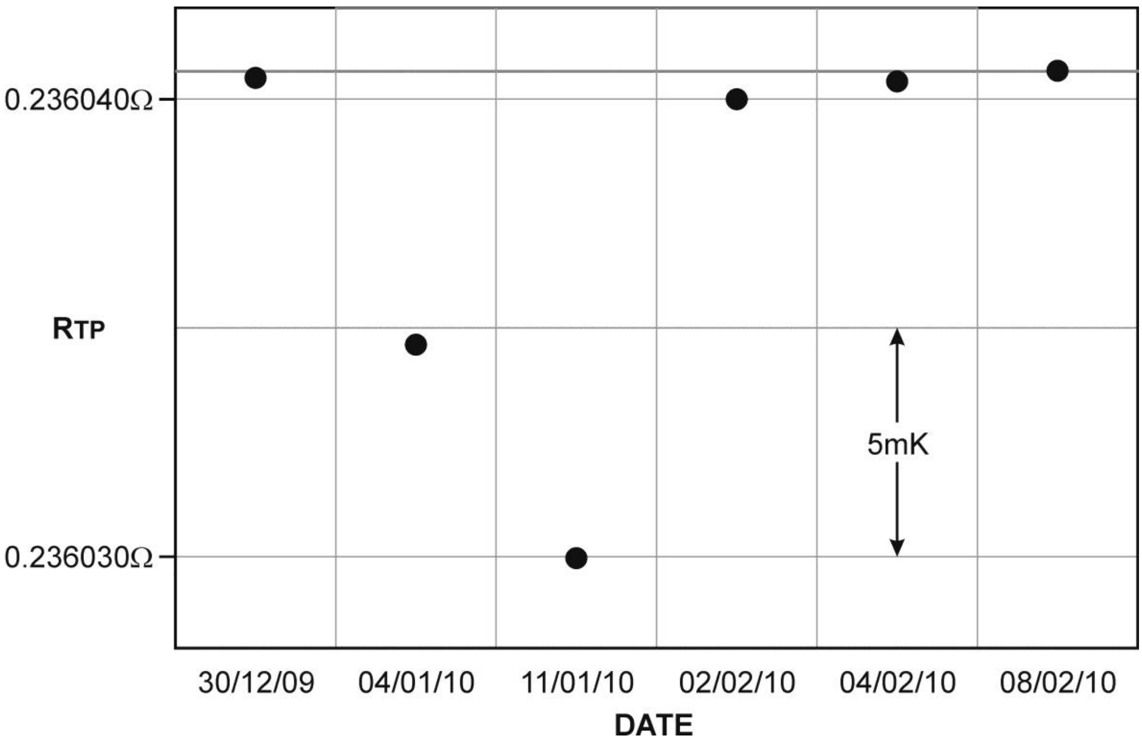


GRAPH 2 - Syphonic Copper Cell – Freeze Plateau

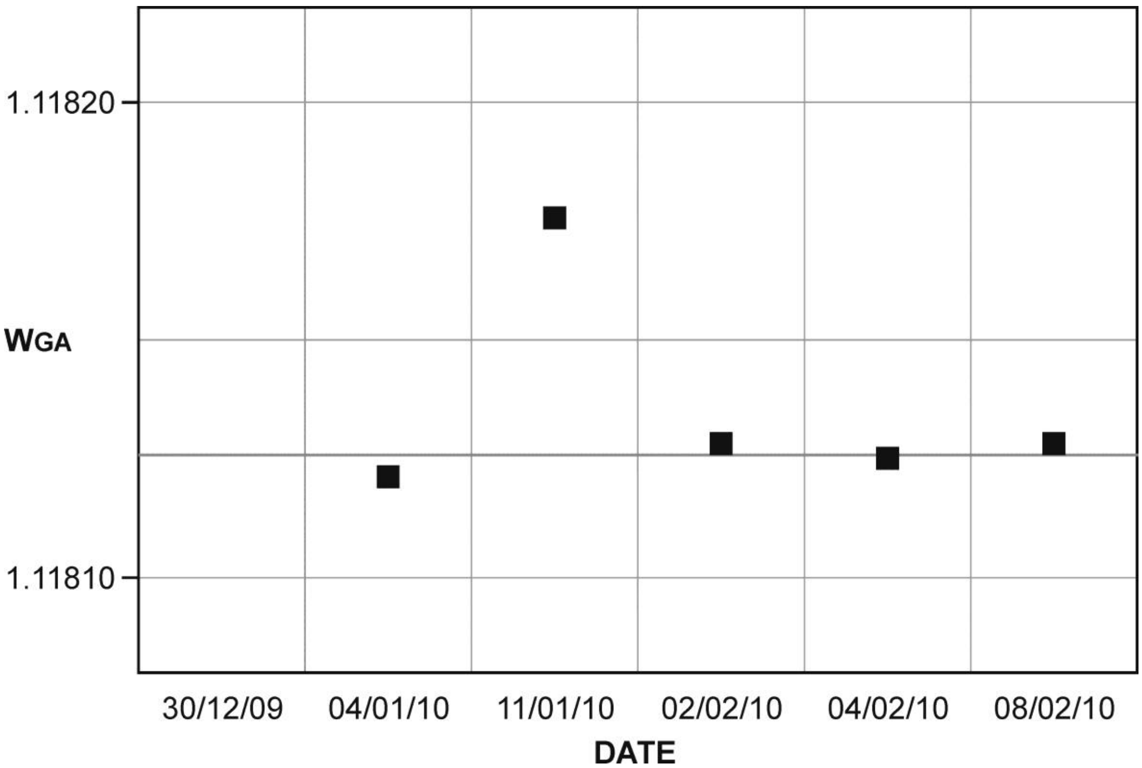
8th January 2010



GRAPH 3a – History 108462/S



GRAPH 3b – History 108462/S



GRAPH 4 – Changes in Rcu