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THE CONSTRUCTION AND CHARACTERIZATION OF PLATINUM-BASED THERMOCOUPLES AT UME

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Abstract – High accuracy measurement of temperature up to 1200°C is an important concept for science, technology and for the industry. For high temperature measurements, thermocouples, which depend on Seebeck principle, are widely used. Platinum based thermocouples are preferred due to their high purity and quality for metrological use. In this paper, the construction of type S and type R thermocouples at UME will be described. After construction, thermocouples were calibrated at the tin, zinc, aluminium and silver freezing points by fixed-point method and by wire-bridge method at gold point.

Keywords: high temperature measurements, platinum based thermocouples, fixed-point calibration

1. INTRODUCTION

Measurements are essential activity in the majority of scientific, industrial and commercial concerns, and the achievement of appropriate accuracy of measurement is necessary for obtaining confidence in science and industry.

In temperature metrology the physical experiment at the top of the traceability chain is the melting, freezing and triple point plateau temperature of pre-determined chemical elements. All these temperatures together makes up a temperature scale from 13,8033 K to 1357,77 K, which is called ITS-90 (International Temperature Scale, 1990) [1]. In order to realize the scale between these temperature intervals, various interpolation instruments are used. One of the common secondary level interpolation instruments for the contact thermometry is the noble metal thermocouple.

A thermocouple directly produces a voltage that can be used as a measure of temperature. That terminal voltage used in thermometry results only from the Seebeck effect.

A realistic circuit for a thermocouple thermometer can be examined in figure 1. The reference and measurement junctions are each in an isothermal environment, each at a different temperature(T_M and T_R). The open-circuit voltage across the reference junction is the so-called Seebeck voltage and increases as the temperature difference between the junctions increases. The thermocouple, which operates on the Seebeck effect, is different from the most other temperature sensors that the output is not related to temperature directly but depends on the temperature gradient.

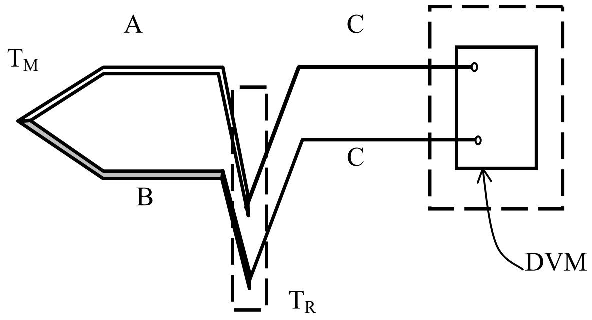

Fig. 1. A circuit to measure Seebeck potential

The increase in the Seebeck voltage, dE, along the short length of wire is proportional to the Seebeck coefficient of the wire, S (T), and to increase in temperature, dT, along the short length of wire

$$dE = S(T)\,dT \qquad (1)$$

This is the basic equation with which to analyse a thermocouple circuit. To emphasize the fact that dT arises from a temperature gradient and not a small change in overall temperature, the above equation can be rewritten as:

$$dE = S(T,x)\,(dT/dx)dx \qquad (2)$$

where dT/dx is the temperature gradient along the wire and dx is a small length along the wire. S(T,x) depends on the position along the wire, which is not uniform everywhere.

2. EXPERIMENTAL WORK

Thermocouples employing platinum in combination with platinum-rhodium alloys have been found to be the most reproducible of all the various types. The best-known members of this group are platinum-10% rhodium alloy versus platinum (or type S) and platinum-13% rhodium alloy versus platinum (or type R) thermocouples [3].

The thermocouple wires must be as pure as possible while constructing thermocouples in order to avoid inhomogeneity. There are a wide variety of causes, which can make the wire inhomogeneous such as a change in metallurgical structure due to work hardening, a change in metallurgical composition due to migration of atoms, corrosion of the wire, and a change in the wire diameter [4].

99.999% purity of wires were used which is the highest commercially available. The steps followed during construction were cleaning, electrical annealing, mounting and construction of reference junction.

Platinum and platinum-rhodium wires were cleaned using ethyl alcohol- distilled water mixture then were annealed electrically in air in order to protect them from drafts which can cause temperature variation along the wires and to make the structure homogeneous throughout the wires. The annealing system used in the laboratory was three-sided enclosure (90 cm width, 40 cm depth and 150 cm height) and has electrical leads coming through the top to two spring-loaded copper clips that hold the thermocouple wire. The front of the system was left open for easy access and to permit the temperature of the thermocouple wire to be determined with the optical pyrometer during the annealing. The thermocouple wire was suspended from two spring-loaded copper clips in the electrical annealing system. The wire was heated by passing a 50 Hz alternating current through it. The annealing current was regulated with an adjustable transformer. The annealing procedure is to heat the wire at about 1300°C for two hours and then allow it to cool quickly to about 750°C. It is held at 750°C for 30 minutes and then cooled to room temperature in a time of a few minutes. The temperature of the wire was determined with an optical pyrometer. It is necessary to add a correction to the observed apparent temperature to obtain the true temperature. The electrical current required to bring the wire to 1300 ^{o}C depends upon the type and the diameter of the wire. For 0.35 mm diameter thermoelements of type S, and R thermocouples, a current of roughly 6 to 7 amperes was required for annealing at 1300 ^{o}C.

The electrically annealed thermo-elements was inserted carefully into high purity two-bore alumina insulating tubes, one for each wire, with bore diameters of 0.6 mm approximately twice of the 0.35 mm wire diameter in order to eliminate the stress on the wires. The tubes were baked for 16 hours at 1100°C in order to remove the dirt and contamination on them.

The thermocouple junction was formed by oxygen-gas torch welding together the pure platinum leg and the platinum-rhodium alloy. In order to minimize contamination of the platinum and platinum-rhodium thermocouple wires, a laboratory workbench and special tools were reserved for preparation and assembly of the thermocouples. Gloves were used in order not to contaminate the wires by oil, dirt in our hands while handling the wires.

Copper connecting wires (0.4 mm diameter) was joined electrically to the thermocouple wires to form the reference cold junctions. The pure and isolated wires were chosen for that purpose.

After construction, the thermocouple was inserted into a high temperature furnace to be annealed at 1100°C for 24 hours.

3. MEASUREMENT and RESULTS

One from both type R and type S thermocouple was prepared for the measurements by following the procedure mentioned above. The measurements were held with a high precision nanovoltmeter.

Measurements were carried out at silver, aluminium, zinc, and tin point respectively. Each thermocouple was tested in at least three freezing plateaus in each of the metal freezing-point cells, which were prepared depending on melting-freezing approach. Thermocouples were tested for inhomogeneity and instability, which depends on repeated measurements at the freezing point of Ag before and after annealing. The uncertainties evaluated from these measurements can be found in table 4.

Immersion test was also held at each fixed-point and results related to zinc freezing point with type S thermocouple can be seen in figure 2.

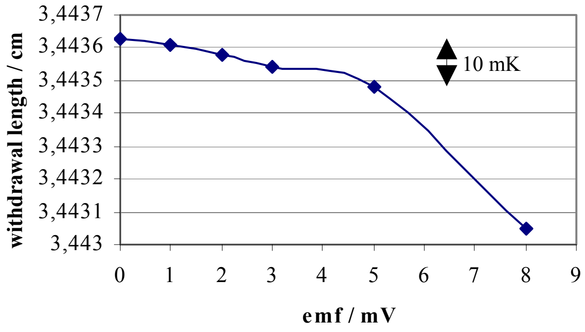

Fig. 2.Immersion characteristics obtained at Zn.

The average values obtained at each fixed-point for both type of thermocouples can be seen in table1.

TABLE I. The average values at fixed-points

Fixed points	Type R Average(µV)	Type S Average(µV)
Ag (961.78°C)	10008,23	9152,79
Al (660.323°C)	6276,45	5858,95
Zn (419.527°C)	3608,86	3443,63
Sn (231.928°)	1753,93	1712,08

Those results can also be evaluated as the deviation from the reference table values point of view either in µV or in °C in table 2.

For gold point measurement, a relatively new method, i.e. wire-melting method, has been operated for. In order to employ this technique a small piece of gold wire fastened to the thermocouple junction and the melting temperature of this wire is measured. The furnace with this assembly inside was maintained several degrees below the melting point of the fixed-point material. When the equilibrium was reached, the furnace power was increased by a constant heating rate.

TABLE II. The deviation of both thermocouples in μV and °C

Fixed points	**Type R**		**Type S**	
	Deviation (μV)	**Deviation (°C)**	**Deviation (μV)**	**Deviation (°C)**
Ag (961.78°C)	-4,80	-0,37	-4,41	-0,39
Al (660.323°C)	0,64	0,05	1,18	0,11
Zn (419.527°C)	2,44	0,23	3,26	0,34
Sn (231.928°)	2,30	0,25	2,92	0,34

As the temperature of the furnace rises through the melting point of the wire, the emf (electromotive force) of the thermocouple is recorded and the plateau is observed as it can be seen in figure 2.

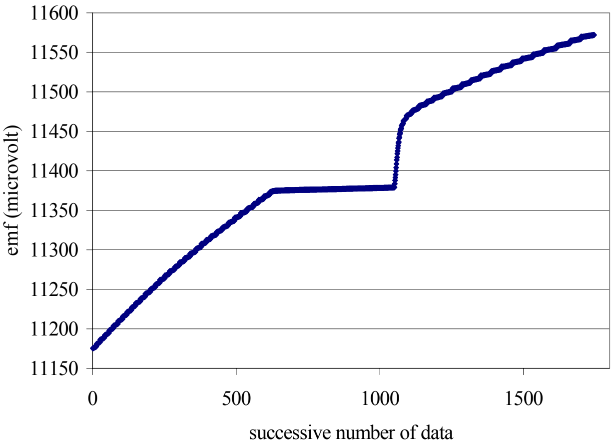

Fig.2 The gold point plateau obtained by wire-melting method.

The results obtained with type R thermocouple with this method can be summarized in table 3.

TABLE III. The gold-point results with type R thermocouple

Fixed point	**Average (μV)**	**Deviation (μV)**	**Deviation (°C)**	**Uncertainty (°C)**
Gold (1064.18°C)	11376.96	13.22	0.98	1.5

The uncertainties associated with the measurements can be divided into four main categories.

a) The uncertainties arising from the fixed-point
b) The uncertainties arising from electrical measurement
c) The uncertainty arising from reference junction and ambient conditions
d) The uncertainties arising directly from wires of thermocouple being under investigation

The uncertainty budget related to silver freezing point measurement, that yielded the largest uncertainty with 0.3°C, can be found in table 4.

TABLE IV Uncertainty components and contributions

Uncertainty Components	**Standard Uncertainty /°C**
	Ag
Type A : Statistical standard uncertainty	0.03
Type B	
a) Freezing point realization	
ingot impurity	0.02
plateau irregularity	0.005
b) Emf measurements	
Voltmeter calibration uncertainty	0.03
Voltmeter resolution	0.01
Voltmeter drift	0.03
c) Reference temperature	
Ice-point variation	0.02
Influence of ambient parameters	0.04
d) Wire characteristics	
Instability	0.07
Inhomogeneity	0.09
Combined standard uncertainty/°C	0,14
Expanded standard uncertainty (coverage factor of 2) / °C	**0,30**

4. CONCLUSIONS

The results clearly showed that the manufacture of reference thermocouples in UME with high accuracy was achieved. The deviation from the reference tables was obtained to be small and no significant difference in fixed-point values was calculated between the UME-made thermocouples and the former NPL-made ones.

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