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Realization of Copper Melting Point for Thermocouple Calibrations

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Abstract: Although the temperature stability and uncertainty of the freezing plateau is better than that of the melting plateau in most of the thermometry fixed points, but realization of melting plateaus are easier than that of freezing plateaus for metal fixed points.

It will be convenient if the melting points can be used instead of the freezing points in calibration of standard noble metal thermocouples because of easier realization and longer plateau duration of melting plateaus.

In this work a comparison between the melting and freezing points of copper (Cu) was carried out using standard noble metal thermocouples. Platinum - platinum 10 % rhodium (type S), platinum – 30 % rhodium / platinum 6 % rhodium (type B) and platinum - palladium (Pt/Pd) thermocouples are used in this study.

Uncertainty budget analysis of the melting points and freezing points is presented. The experimental results show that it is possible to replace the freezing point with the melting point of copper cell in the calibration of standard noble metal thermocouples in secondary-level laboratories if the optimal methods of realization of melting points are used. *Copyright © 2011 IFSA.*

Keywords: Copper point, Freezing/melting plateau and Thermocouples.

1. Introduction

The freezing plateaus are usually used in the most defined metal fixed points according to the International Temperature Scale of 1990 (ITS-90), because of the best stability and reproducibility compared to their melting plateaus. The freezing point of Cu is defines in ITS-90, to be one of the reference point in the calibration of radiation thermometers [1], also it is important for the calibration of noble metal thermocouples as a contact thermometer. Cu point is an important calibration point for of noble metal thermocouples calibration in the range 1000 °C to 1600 °C. [2].

The melting plateau of Cu is longer than that of the freezing plateau, also the freezing plateau performance is influenced by the purity of fixed point metal and realization procedure. However the realization of the melting plateau is easier than that of freezing plateaus, because of the supercooling and induction of nucleation during the realization of the freezing plateau.

In the previous studies, the comparison between melting and freezing points of indium, tin, zinc, aluminum and silver has been studied [3-4]. In this study, comparisons of the melting and freezing plateaus of copper were carried out using type S, type B and Pt/Pd thermocouples.

The possibility of using the melting plateau instead of the freezing plateau for the noble thermocouples calibration in secondary-level laboratories is discussed.

2. Equipment

2.1. Copper Point Cell

The crucible containing the high purity copper is a sealed cell supplied by HART SCIENTIFIC - USA. The crucible assembly is shown in Fig. 1. The graphite crucible containing copper of purity 99.9999 % is completely enclosed into a silica envelope, which contains the argon atmosphere at a pressure of one atmosphere at the freezing temperature of Cu i.e. 1.013×10^{5} Pa at 1084.62 °C. The distance between the bottom of the thermometer graphite well and the surface level of the metal is 17.5 ± 0.5 cm. For the measurements, the cell is supplied with an ultra pure alumina holder and several insulating bricks in order to assemble the sealed cell for measurements.

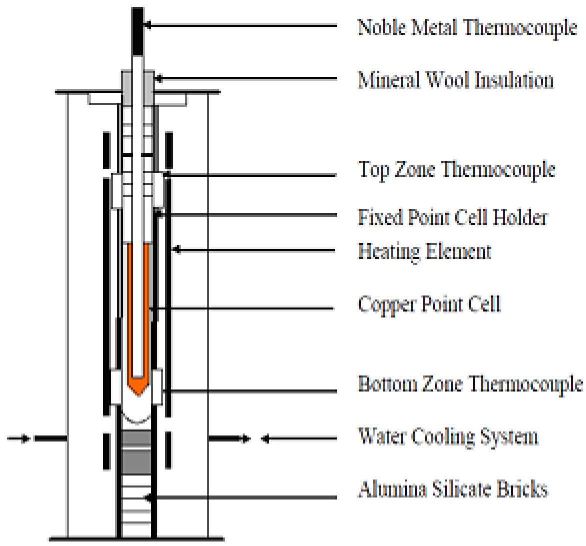


Fig. 1. The schematic diagram of the Cu point cell and three zone furnace.

2.2. The Three Zone High Temperature Furnace

The temperature of the three zone furnace used in this work can be increased up to 1200 °C and it has a separate temperature controller with 0.1 °C resolution, displayed by the control unit. In addition, the furnace is provided with two controllers for the upper and lower heater to compensate for any temperature gradient along the furnace.

The furnace is provided with a water cooling system to maintain the outer surface of the furnace at near ambient temperature. The cell holder of the furnace is made of ultra pure Alumina. In order to get long and stable melting and freezing plateaus, the temperature gradient of the furnace for maintaining copper cell must be adjusted carefully. Experiments performed in the range from 1000 °C to 1150 °C indicated a reproducibility and uniformity of furnace temperature better than 0.5 °C. In this study, the largest difference between the top and the bottom of the working zone was 0.3 °C at 1100 °C for the furnace to maintain copper cell. Fig. 1 shows a schematic view of our three zone furnace.

2.3. Thermocouples Used

Type S, type B and Pt/Pd thermocouples were made from reference grade wires supplied by Johnson Matthy. All wires with 0.5 mm diameter and 160 cm length. Both thermocouples have high purity alumina sheath of length of 600 mm, with closed end at the hot junction.

The thermocouples were prepared at NIS-Egypt [5] according to the recommendations by Mclaren and Murdock. The thermocouples reference junction were maintained at 0 °C in an ice bath consisting of a mixture of finely divided pure ice and distilled water contained in a dewar vessel. For measurement of the emf produced by the thermocouples a digital nanovoltmeter (DVM) with internal resistance higher than 10^9 Ω, model Keithley-182 having a resolution of 0.00l µV was used.

3. Experimental Work and Results

3.1. Melting Procedure

The melting temperature of pure metals is much more sensitive to impurity. In the present work the melt started as soon as possible after a freeze to reduce the diffusion of impurity through the solid. In practice it takes an hour to reheat the furnace, which falls several degrees below the freezing temperature. The furnace rise rapidly several degrees above the ingot temperature until the heat absorbed by the melting ingot equals the excess heat provided to the furnace. The melt is arranged to last 3 to 4 hours. After measurements of the melting plateau were completed, the furnace temperature was raised to 4 °C above the freezing point in preparation for testing the freezing plateau the next day.

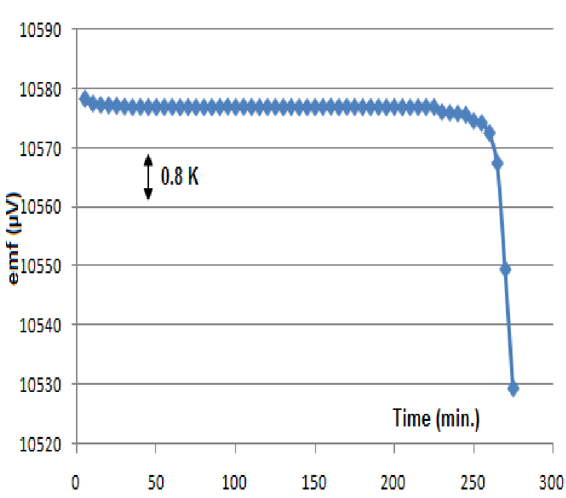
3.2. Freezing Procedure

The freezing of the copper sample is obtained by means of the induced-freeze technique [3, 4], so as to produce a constant temperature plateau. The induced freeze technique proceeds as follows. After the metal is melted to 3 K above the melting point, the temperature of the furnace is decreased by changing the power in the furnace so that its temperature becomes 1 K below the freezing point. When the nucleation becomes evident, by an initial arrest on the cooling curve, the thermocouple is withdrawn from the well and inserted in the annealing furnace. A cold inducing alumina rod is inserted for about 1 minute into the thermometer well of the copper sample. This method is repeated twice. The inducing rod is withdrawn from the well and replaced by the thermometer for the measurements.

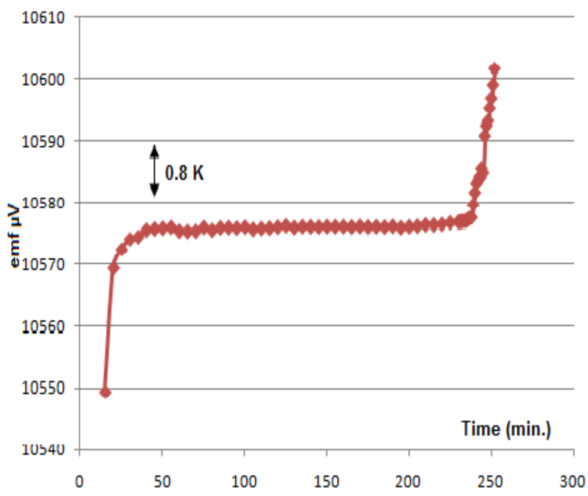
About 15 minutes are required after the insertion of the thermocouple to be certain that it has come to an equilibrium temperature. The freezing temperature is recorded every minute. The plateau continued for about 6 hours. This gives about 4 hours of stable plateau between the end of recalescence and the temperature depression at the end of the freeze. The technique used for realizing the freezing plateau can be found in other articles [6].

3.3. Comparison of Melting and Freezing Plateaus of Copper

The measurement of the freezing plateau started one hour later after induction of nucleation. The same thermocouples (S, B and Pt/Pd) were used in the same way to measure the freezing plateau as during the melting plateau test. After testing, the furnace temperature was reduced below the freezing point for the next round of measurements of the melting plateau. This procedure was repeated several times, alternately measuring the melting plateau and the freezing plateau. Fig. 2 shows one complete melting plateau (a) and one freezing plateau (b) for the copper cell.



(a)



(b)

Fig. 2. Melting and freezing plateaus of the copper cell using type S thermocouple.

Table 1 shows comparison results of several groups of measurements of the melting plateau and the freezing plateau of the copper cell. For each group of measurements, same thermocouples were used in the same sequence.

It can be seen that the freezing plateau is more stable than the melting plateau. The amplitude of the freezing curve fluctuation can reach over 45 mK, but only 30 mK for the melting plateau. The table shows also that the largest difference among the melting groups is 370 mK, and the smallest difference is 167 mK. The reason for this difference is that the normal repeatability of noble metal thermocouples.

4. Uncertainty Estimation

The measurements of the freezing point were repeatable to better than ± 83 mK for type S, ± 141 mK for type B and ± 44 mK °C for Pt/Pd thermocouple. Also The measurements of the melting point were repeatable to better than ± 154 mK for type S, ± 185 mK for type B and ± 30 mK °C for Pt/Pd thermocouple. The uncertainty budgets were calculated according to the recommendations of GUM [7] for each thermocouple incorporating the various contributory factors are shown in the Table 2 for type S, B and Pt/Pd thermocouples.

Table 1. Comparison results of several groups of measurements of the melting plateau and the freezing plateau of the copper cell using type S, B and Pt/Pd thermocouples.

Thermocouple type/ Group No.	**Freezing point µV**	**Melting point µV**	**Difference between freezing and melting values**	
			µV	**mK**
Type S/ Group 1	10575.6	10575.7	**0.4**	**30**
Type S/ Group 2	10576.6	10575.5		
Type S/ Group 3	10578.0	10578.9		
Type S/ Group 4	10577.0	10578.7		
Average of Type S results	**10576.8**	**10577.2**		
Type B/ Group 1	5638.7	5635.6	**0.2**	**20**
Type B/ Group 2	5637.0	5637.3		
Type B/ Group 3	5635.9	5639.3		
Average of Type B results	**5637.2**	**5637.4**		
Type Pt/Pd/ Group 1	13274.9	13278.5	**0.5**	**20**
Type Pt/Pd/ Group 2	13276.5	13275.0		
Type Pt/Pd/ Group 3	13276.5	13276.0		
Average of Type Pt/Pd results	**13276.0**	**13276.5**		

Table 2. Uncertainty budget for type S, type B and Pt/Pd thermocouples, at Cu freezing point.

Uncertainty Sources	**Uncertainty contribution °C**		
	Type S	**Type B**	**Pt/Pd**
Type A			
Repeatability	0.083	0.141	0.044
Plateau fluctuation	0.010	0.010	0.010
Type B			
Cu ingot purity	0.015	0.015	0.015
Nanovoltmeter calibration	0.001	0.001	0.001
Inhomogeneity of thermocouple	0.109	0.202	0.050
Drift of thermocouple	0.095	0.172	0.040
Uncertainty due to cold junction	0.003	0.003	0.003
Combined standard uncertainty	0.17	0.30	0.08
Expanded uncertainty, (k=2)	± 0.34	± 0.60	± 0.16

The major contribution to the uncertainty is attributed by the inhomogeneity of the thermocouple, at Cu freezing point and the drift of the thermocouples. The uncertainty due to inhomogeneity of thermocouple has been estimated from the immersion profile at the Cu point observed during the measurement [8]. The drift caused in each thermocouple is another component of uncertainty, which was estimated from the earlier calibration certificates and included in the uncertainty budget. The uncertainty due to purity of Cu was evaluated from the manufacturer's certificate to be ± 0.015 °C for 9.9999 % purity of Cu.

Also, Table 3 shows the uncertainty budget for copper melting point using thermocouples type S, B and Pt/Pd thermocouples. The overall uncertainty of measurement for all thermocouples is evaluated at a confidence level of 95 % approximately with a coverage factor, k=2.

Table 3. Uncertainty budget for type S, type B and Pt/Pd thermocouples, at Cu melting point.

Uncertainty Sources	Uncertainty contribution, °C		
	Type S	**Type B**	**Pt/Pd**
Type A			
Repeatability	0.154	0.185	0.030
Plateau fluctuation	0.030	0.030	0.030
Type B			
Cu ingot purity	0.015	0.015	0.015
Nanovoltmeter calibration	0.001	0.001	0.001
Inhomogeneity of thermocouple	0.109	0.202	0.050
Drift of thermocouple	0.095	0.172	0.040
Uncertainty due to cold junction	0.003	0.003	0.003
Combined standard uncertainty	0.21	0.33	0.08
Expanded uncertainty, (k=2)	± 0.42	± 0.66	± 0.16

The freezing and melting plateau duration and stability results and the uncertainty estimations for copper point show that; although the melting plateau is not as stable as its freezing plateau, it could be used for the calibration of noble metal thermocouples in the secondary-level calibration laboratories, particularly for high temperature above l000 °C.

4. Conclusion

The experiment results of the melting and freezing plateaus of copper point show that the duration of the melting plateau of it is longer than that of the freezing plateau, but the stability of the melting plateaus is worse compared to its freezing plateaus. The differences between the melting and freezing points were up to 30 mK using noble metal thermocouples. The estimated uncertainties of the freezing and melting plateaus of copper are 340 mK and 420 mK respectively using type S thermocouple. Both the experiment and uncertainty estimation results show that although the melting plateau of Cu is not as stable as the freezing plateau, it should be good enough for the calibration of noble metal thermocouples in secondary-level laboratories while offering greater convenience.

References

[1]. Comite Consultatif de Thermometrie, Supplementary Information for the ITS-90, *Bureau International des Poids et Mesures (BIPM)*, 1990.

[2]. Y. P. Singh, S. K. Nijhawan and Anjula, Realization of Copper Fixed Point in Sealed Cell for Calibration of Noble Metal Thermocouples on ITS-90, MAPAN, *J. of Metrology Society of India*, Vol. 22, No. 4, 2007.

[3]. X. M. Li, and M. Hirst, The comparison between the freezing point and melting point of tin, *Proc. of the 6th International Symposium on Temperature and Thermal Measurements in Industry and Science (TEMPMEKO '96)*, Torino, Italy, 1996.

[4]. R. Ding, M. J. Zhao, D. Cabana, D. M. Chen, Comparison between melting and freezing points of indium and zinc, *NCSL International Workshop and Symposium*, St Paul, Minnesota, 2007.

[5]. M. Halawa, F. Megahed, Y. A. Abdelaziz and M. M. Ammar, Performance, Thermoelectric Homogeneity

and Stability of Au/Pt and Pt-10%Rh/Pt Thermocouples, in *Proc. of the 8th International Temperature Symposium*, USA, 2002.

[6]. R. Ding, M. J. Zhao, D. Cabana, D. M. Chen, and R. Walker, Study of the Influence of Inducing Nucleation on the Performance of the Freezing Temperature Plateau of Metal Fixed-Point Cells, *NCSL International Workshop and Symposium*, Nashville, TN, 2006.

[7]. ISO Guide to the Expression of Uncertainty in Measurement, *International Organization for Standardization*, Geneva, 1995.

[8]. Yasser A. Abdelaziz and F. Edler, A Method for Evaluation of the Inhomogeneity of Thermoelements, *Meas. Sci. Technol*., Vol. 20, 2009.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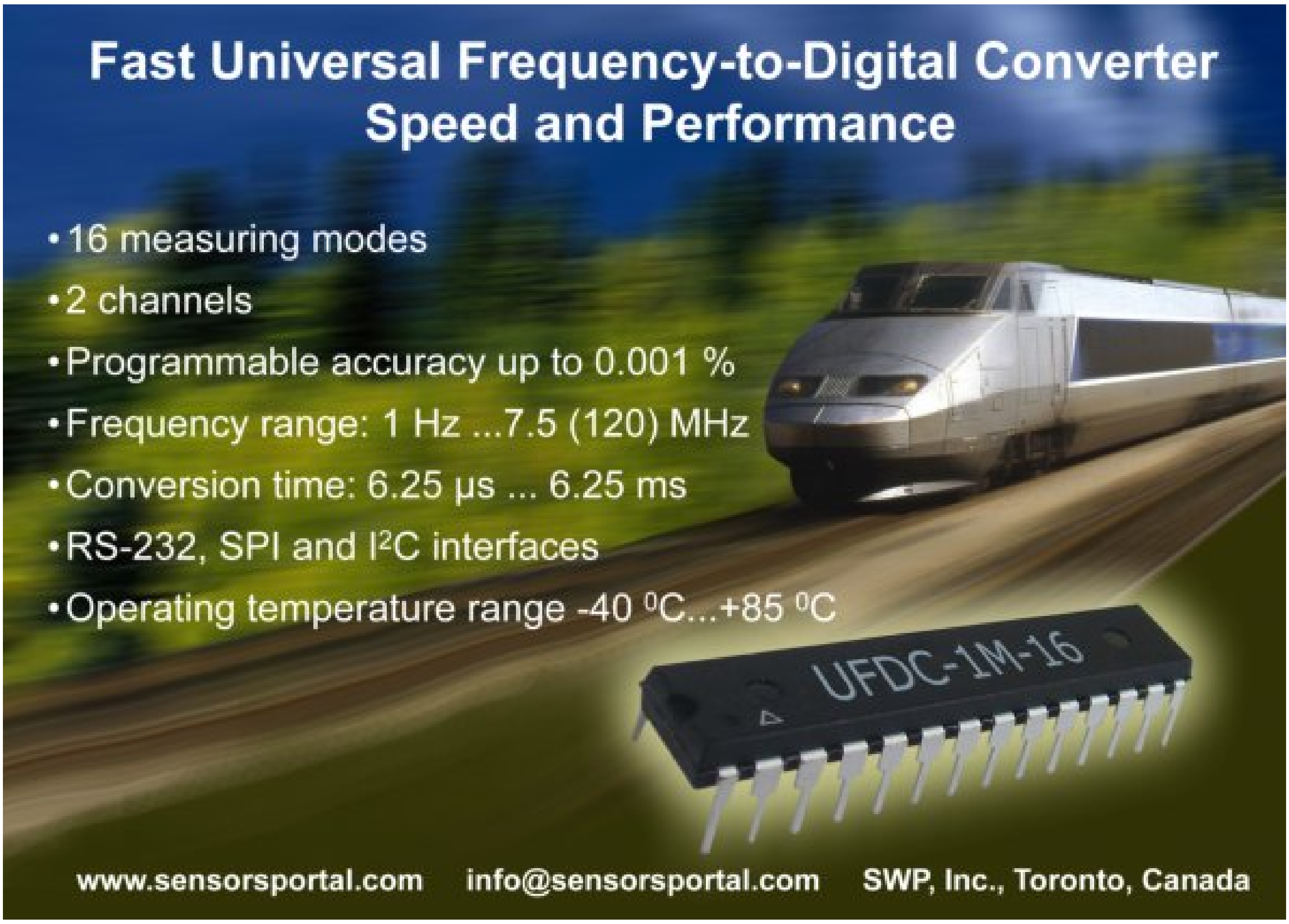
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