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THE COMPARISON OF THE REALISATION OF THE FREEZING POINT OF INDIUM INSIDE THE THREE ZONE FURNACE AND THE FLUDISED POWDER FURNACE

Jovan Bojkovski, Valentin Batagelj, Igor Pušnik

University of Ljubljana, Faculty of Electrical Engineering
Laboratory of Metrology and Quality, Tržaška 25, 1000 Ljubljana, Slovenia

Abstract – In the paper the comparison of the realisation of the freezing point of indium inside the three-zone furnace and the fluidised powder furnace is presented. The emphasis of the paper is given to the comparison of the metrological characteristics of the realisations such as time duration of the plateau and the width of the plateau. The same fixed-point cell (closed fixed-point cell, serial number In 86, made by Isotech) was realised in both furnaces. Also the analysis of the immersion curve is performed in order to see how it agrees with the expected curve.

Keywords: temperature, freezing point of indium, comparison of realisations

1. INTRODUCTION

The International Temperature Scale ITS-90 was designed by the CCT in order that temperature values obtained on it do not deviate from the Kelvin thermodynamic temperature values by more than the uncertainties of the later values at the time the ITS-90 was adopted. The ITS-90 substitutes the IPTS-68, the International Practical Temperature Scale of 1968, Amended Edition of 1975 [IPTS-68 (75)] and the 1976 Provisional 0,5 K to 30 K Temperature Scale (EPT-76), [1].

Temperatures on the ITS-90 are defined in terms of equilibrium phase states of pure substances (defining fixed points), interpolating instruments and equations that relate the measured property of the instruments to the T_{90}. These states are explained in details in the ITS-90 definition and supplementary information.

Depending on the temperature of the phase state, different types of temperature-controlled media are used for realisation of equilibrium phase states of pure substances. These media are used for providing the uniform temperature zone over the length of the ingot. Typical values for the axial gradient needed for the proper realisation of phase state is axial gradient of less than 30 mK over the length of the ingot. The stability should be in order of at least 100 mK, [2]. In this paper we compare two different types of furnace which can be used for realisation of the freezing point of indium (156,5985 °C) together with their influence to the realisation and the value of the freezing point of indium including any additional source of the uncertainty, which can occur as a result of the poor performance of the furnace.

2. FURNACES USED

2. 1. The three zone furnace

The three-zone furnace has three heater zones (top, middle and bottom). The top zone and the bottom zone are acting as guard zones, preventing influence of the ambient temperature to the temperature of the freezing point. The middle heater zone is used for providing the uniform zone over the length of the ingot. Each heater zone can be controlled separately, thus providing optimum set-up for the purpose. This furnace can be used for the realisation of the freezing points in the range from 150 °C up to 670 °C.

2. 2. The fluidised powder furnace

This furnace was originally developed for the calibration of the thermometers by comparison as an alternative to the salt bath. The temperature working range of the furnace is from 50 °C up to 600 °C. As a medium an aluminium oxide Al_2O_3 powder is used. Compressed air under the pressure of 1 bar is passed through the aluminium oxide. As a result, the aluminium oxide powder becomes fluidised and thus its thermal characteristics are much better.

The insert made of inconel, with a larger diameter than the diameter of the fixed-point cell, is put to the fluidised aluminium powder, as shown in Fig. 1. This insert acts as a protection and prevents any contamination of the the fixed-point cell and its thermometer entrant tube. Around the bottom part of the fixed-point cell, where the actual pure metal is, the tube made of aluminium is used in order to increase thermal conduction from the fluidised powder to the fixed point cell itself. The upper part of the fixed-point cell is insulated with washed fiberfrax thus reducing any influence of the ambient temperature to the fixed-point temperature. The set-up is shown in Fig. 1.

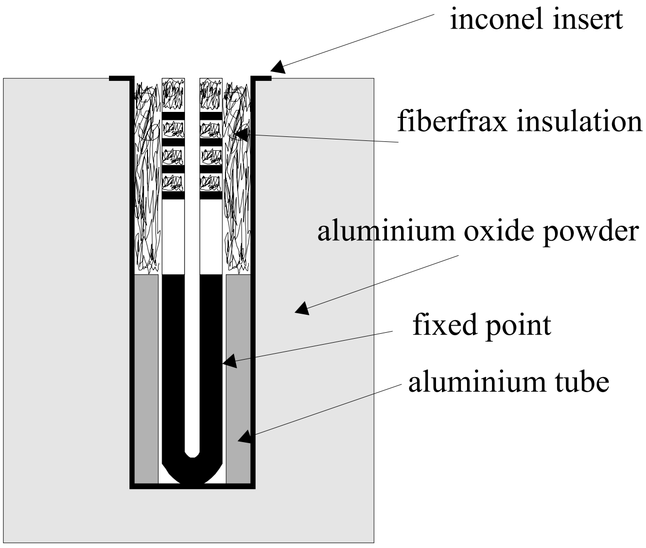

Fig. 1. Arrangement of the fixed point cell inside the fluidised powder furnace

2. 3. Measurement procedure

In order to collect as much information as possible, the measurement procedure used in this comparison consisted of three steps. All measurements were performed using the automatic DC resistance bridge Measurements International, type 6010 B, the quartz sheathed Hart Scientific SPRT (standard platinum resistance thermometer) and the 25 ohms reference resistor Tinsley Wilkins type. A special custom made software was used for data acquisition. The sample rate was 10 seconds.

In the first step, the stability and the homogeneity inside the cell was measured with indium in a solid state. The temperature of a furnace was set approximately 1 K below the melting point. The stability was determined as a double standard deviation of measurements over two hours. The homogeneity inside the cell was measured in 3 cm steps from the bottom of the cell over the length of 18 cm. After each step, in order to allow the thermometer to stabilise a new temperature, we waited for 15 minutes.

In the second step, same measurements as in the first step were performed, but this time with indium totally melted. The temperature of the furnace was set approximately 2 K above the melting point.

In the third step, the actual freezing plateau was measured. After melting it in the second step, in order to freeze indium the furnace controller is set 1 K below the actual freezing temperature. The furnace cools to the new set temperature. When the SPRT indicates that recalescence occurred, the thermometer is removed from the cell and replaced by a cold quartz rod, which has an ambient temperature. It is kept in the cell for two minutes. The furnace controller temperature is set to 0,5 K below the freezing point. After two minutes the SPRT is put back into the cell and the freezing plateau is reached typically in 30 to 45 minutes. This procedure initiates freezing of the cell from the inside and the outside wall towards the centre. If the cell is left too long in the furnace, without initiating the freeze as described above, nucleation occurs and the cell freezes from the bottom of the cell upwards. This would result in a short, imperfect plateau and would give an incorrect value of the freezing point. Also the measurement of the immersion curve is performed in order to see how it agrees with the expected curve.

2. 4. Measurement results

The measurement results for the fluidised furnace are presented in Table 1 and for the three-zone furnace in Table 2. The homogeneity results are presented as differences in K between the temperature at the bottom of the cell (0 cm) and temperatures at different distances from the bottom of the cell.

TABLE 1 Results of the measurements for the fluidised furnace

Solid		Liquid	
stability	0,004 K	stability	0,004 K
homogeneity		homogeneity	
3 cm	-0,0051 K	3 cm	-0,0019 K
6 cm	-0,0066 K	6 cm	-0,0058 K
9 cm	-0,0053 K	9 cm	-0,0034 K
12 cm	-0,0052 K	12 cm	-0,0062 K
15 cm	-0,0250 K	15 cm	-0,0190 K
18 cm	-0,2802 K	18 cm	-0,2358 K

TABLE 2 Results of the measurements for the three-zone furnace

Solid		Liquid	
stability	0,002 K	stability	0,002 K
homogeneity		homogeneity	
3 cm	-0,0042 K	3 cm	-0,0036 K
6 cm	-0,0092 K	6 cm	-0,0109 K
9 cm	-0,0160 K	9 cm	-0,0188 K
12 cm	-0,0246 K	12 cm	-0,0275 K
15 cm	-0,0447 K	15 cm	-0,0432 K
18 cm	-0,3013 K	18 cm	-0,2109 K

As one can see from the results presented in the Tables 1 and 2, the fluidised furnace showed better homogeneity, but the stability was slightly worse. On the other hand, as suggested in [2] and explained in the introduction of this paper, these values are sufficient for proper realisation of the freezing point of indium. The measurements showed no significant difference between measurements made in the solid state and the liquid state.

As previously mentioned, also the measurement of the immersion curve was performed. There was no significant difference between two furnaces. In Fig. 2. the measurements inside the three-zone furnace are presented.

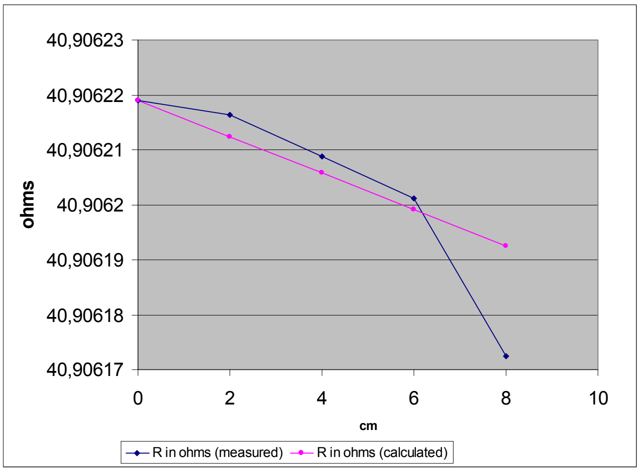

Fig. 2. Immersion curve inside the three-zone furnace

We can see that measured curve follows theoretical immersion curve up to 6 cm above the bottom of the cell. Since the typical length of the SPRT sensor is 6 cm or less, this is satisfying.

According to the measured values, the differences between two realisations are very small, but yet notable. All the measurements were taken in accordance with the procedure well described in supplementary information, [3] and [4]. In both cases the fixed point cell was melted overnight at the temperature approximately 2 K above the fixed point value. After that the controller was set to the temperature which is 1 K below the freezing point and immediately after the monitoring thermometer indicates that the temperature inside the cell is bellow the freezing point and that the recalescence occurred, the temperature of the controller is set to the temperature which is 0,5 K bellow the freezing point.

The main difference is time duration of the plateau. Due to smaller gradients inside the fluidised powder furnace, the length of the plateau tends to be larger than inside the three zone furnace. The plateau inside the fluidised powder furnace lasted easily more than 24 hours, with the standard deviation of the measurements 40 µK over 24 hours. On the other hand, inside the three-zone furnace the plateau lasted for 16 hours with the same standard deviation of 40 µK over 16 hours. The width of the plateau was the same in both realisations, 0,15 mK. The difference in absolute values of the freezing point of indium was less than 10 µK. This indicate that actually there is no difference between the realisation in a fluidised furnace or in a three-zone furnace, in terms of the absolute value.

Finally, if we compare time needed for the complete realisation, from the installation of the fixed point cell to the furnace until we can measure at the freezing point of plateau, we can see that the three-zone furnace is faster. In the case of the fluidised furnace we can measure after 20 hours (6 hours from room temperature to the melting point, 6 hours for melting itself, and 8 hours to allow complete melting 2 K above the melting point). In the case of the three-zone furnace we can measure after 16 hours (2 hours from room temperature to the melting point, all other time ranges are the same like in the fluidised furnace). This can be explained with larger heat capacity of the fluidised furnace and the time needed to heat it up.

In Fig. 3. we can see the freezing plateau of indium over the period of 16 hours.

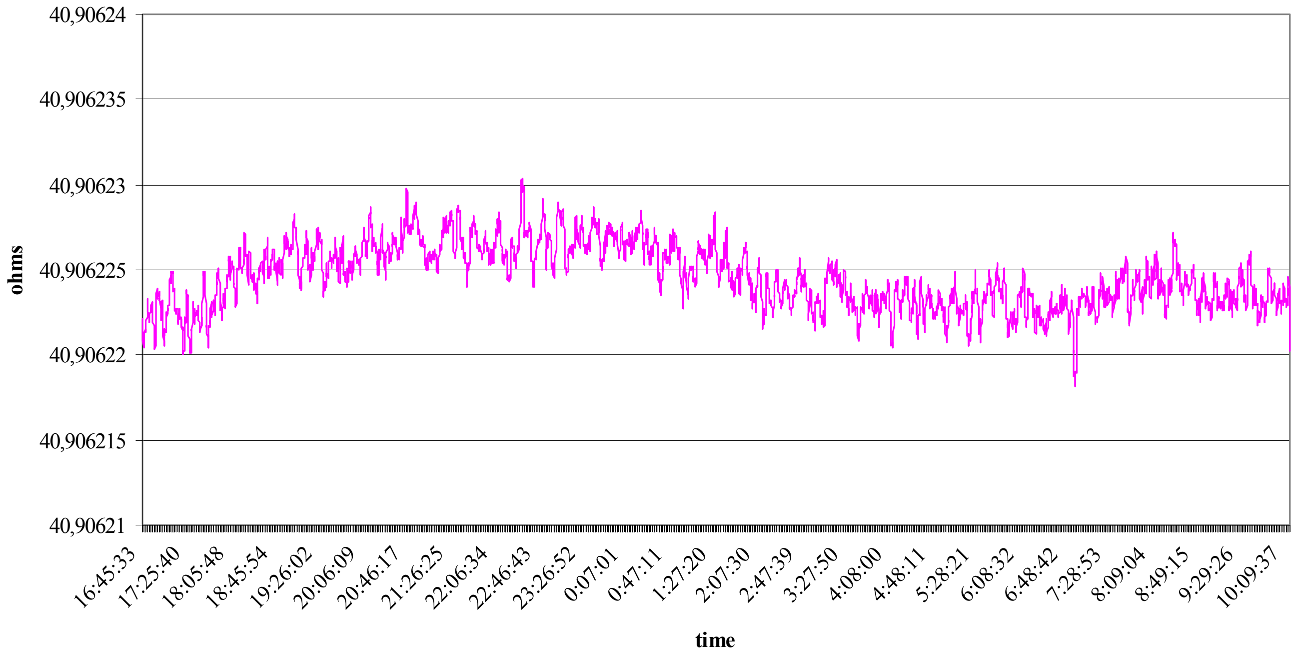

Fig. 3. The freezing plateau of the indium in the three-zone furnace

3. CONCLUSIONS

In order to achieve the long time duration of the freezing plateau the stability as well as the axial gradient of the furnace used has to be small. On the longer plateau we can calibrate more thermometers and thus decrease time needed for the calibration of the group of thermometers. This fact is particularly important when calibrating at higher temperatures i.e. freezing point of zinc or aluminium.

It has been shown that with expected duration of the plateau, there is no major differences between the realisation inside the fluidised furnace or inside the three-zone furnace. Even few time worse homogeneity inside the three-zone furnace didn't influence the actual value of the freezing point. Further investigation will be done at higher temperatures.

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dr. Jovan Bojkovski: Laboratory of Metrology and Quality, Faculty of Electrical Engineering, Tržaška 25, 1000 Ljubljana, Slovenia, Tel:+386 1 4768 224, Fax:+386 1 4264 633, E-mail: jovan.bojkovski@fe.uni-lj.si

Other authors (**Valentin Batagelj M.Sc., Igor Pušnik M.Sc.**) have the same affiliation and contacts except E-mail.