Determination of Freezing Point of Silver usinga Detector Based PhotoelectricLinear Pyrometer

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Abstract
The freezing point of silver (1234.93K) is defined as the junction temperature in the current international temperature scale, ITS-90 to measure it using a contact method by a high temperature platinum resistance thermometer (HTPRT) in the fixed-point cell and using a non-contact method by a radiation pyrometer with a fixed-pointcavity to ascertain the linearity, uniformity and continuity of the scale above 1234 K. In the present work, the temperature of freezing silver was determined using a recently procured and calibrated spectral photoelectric linear pyrometer, LP4. The pyrometer has the traceable calibration from PTB Institute Germany and used as a transfer standard in the measurement process. A silver metal-in-graphite blackbody cavity containing a known quantity of pure silver with a cavity aperture of 10mmwas used in the measurement. The melting and freezing plateaus during phase transitions of silver metal were measured and are shown. The freezing temperature of silver was determined to be 1234.86 K(±0.367 K at k=2),differing by 0.07 K lower than the assigned value in the International Temperature Scale of 1990 (ITS-90) respectively. The fixed point of Ag would serve a unique standard of temperature for calibration of variable sources of transfer standards like tungsten strip filament lamps and spectral and optical pyrometers in the range from 1000 K to 2500 K.

Keywords:*Fixed point, photoelectric pyrometer, blackbody cavity, ITS-90, uncertainty.*

I.INTRODUCTION

International Temperature scale of 1990 (ITS-90) assigns agreed temperatures to the phase transition (such as melting or freezing) of pure materials, which can then be used to calibrate standard thermometer or pyrometer. The freezing point is depressed slightly by presence of impurities, so the use of materials with sub-ppm impurity levels is essential. Fixed point set-ups are used in calibration laboratories for temperature to realize the melting or freezing temperature of fixed point materials. These set-ups consist of a furnace and standard fixed-point blackbody cavity with a several cubic centimeters of highly pure metal. A well-known phase transition temperature arising inside the cavity during melting and freezing is used to calibrate standard tungsten lamps with variable intensity of radiation by estimating the true value of temperature. Above the freezing point silver, the temperature on the International Temperature Scale of 1990 (ITS-90) is defined in terms of the radiance ratio of source of interest to that of silver, gold or copper freezing point [1-4]. In practice, the fixed-point blackbody radiator suffers from practical limitations. Realization of fixed points for radiation thermometry is a complex time-consuming process and the graphite parts are to be replaced often, even though the cavities are heated in an inert gas environment using argon gas over pressure.In addition, the cavity apertures are relatively small in order to realize a high effective emissivity. Consequently, an excellent size-of-source characteristics and relatively small measurement spot is required for a primary radiation pyrometer. In order to overcome these practical limitations, the authors used a sodium heat-pipe blackbody source to realize the silver point. For most defined metal fixed points in the international Temperature Scale of 1990 (ITS-90), the freezing plateaus are used because of the best stability and reproducibility compared to their melting plateaus[5-8]. Although the temperature uncertainty of melting plateau is higher than that of freezing plateau but operation and realization of melting are easier than freezing, primarily because of super-cooling and induction of nucleation during realization of the freezing point. Realization of melting plateaus avoids these problems and the duration is also longer than that of the freezing plateau. In the freezing process, the performance of plateau is influenced by the purity of metal and current operation procedure but not prior freezing and melting history while in contrast, the performance of melting plateau is influenced by the prior freezing history. Asa consequence, the melting plateaus are not used in the ITS-90 for most metal fixed points [9-13]. In this paper comparison of melting and freezing plateaus of silver point were carried out and discussed. The fixed point of silver was measured and uncertainty has been evaluated considering the most affecting components which are contributing towards the accuracy in determining the fixed point.

II. MATHEMATICAL EQUATIONS

The thermodynamic temperature of silver blackbody is found from radiation responsively and Planck's radiation law by use of

$i_c = \int S_L.L(\lambda, T)d\lambda$ ……..(1)

Where i_c is the calculated photocurrent, S_L is the absolute radiance responsively and $L(\lambda,T)$ is the spectral radiance given by Planck's radiation law.

$L(\lambda,T) = (c_1/n^2\lambda^5)[\exp(c_2/n\lambda T)-1]^{-1}$…….(2)

Where c_1 and c_2 are first and second radiation constants, T is thermodynamic temperature of Ag, λ is the wavelength of radiation in air and n is the refractive index. For these calculations, the CODATA values [14]for the radiation constants and the refractive index of air n=1.00029 were utilized. Any unknown temperature can be evaluated if photocurrents (I_{ph}) are determined using the equation (2) above. Thus, taking the ratio

$L(\lambda,T)/L(\lambda,T_{Ag})= [\exp (c_2/\lambda T_{Ag})-1]/[\exp(c_2/\lambda T)-1]$

$=I_{ph}(T)/I_{ph}(T_{Ag})$ ……. (3)

Using Wien's approximation, the equation is written as following in order to determine the unknown temperature in respect of silver point temperature from the measured values of photocurrents at silver point and that of unknown temperature.

$[e^{(c2/\lambda TAg)}]/[e^{(c2/\lambda T)}]= I_{ph}(T)/I_{ph}(T_{Ag})$ …....(4)

Using equation (4), the one can evaluate temperature of any unknown radiating source by using a LP4 pyrometer which is calibrated to measure silver point in terms of photocurrent signal, $I_{ph}(Ag)$. Now for a serious of unknown temperatures of a radiating source like tungsten strip lamps, the photocurrent signals,$I_{ph}(X)$ could be generated and thus the unknown temperatures are calculated by putting the values of photocurrent and known temperature of Ag to be 1234.93 K in the equation (4), all unknown temperature could be determined. In the present, an effort has been made to determine Ag freezing point in terms of photocurrent and its deviation from the assigned value in ITS-90 using a radiation pyrometer. In other words, silver freezing point is used to calibrate the tungsten strip lamps and the radiation pyrometer for accurate and precise measurement of high temperature by non-contact measurement technique.

III. EQUIPMENT DESCRIPTION

A. Ag Fixed-point Blackbody Cavity

Fig.1 shows the blackbody cell structure made from high purity (99.999%) graphite, the stainless steel cylindrical enclosure and the front sighting insulating cone for directingradiation to reach the detector of the pyrometer.

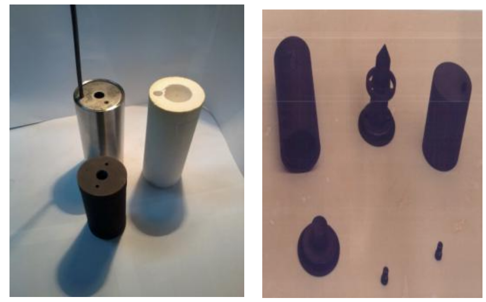


Fig. 1: Structures of silver point blackbody cavity

The graphite crucible is held in a stainless-steel tube with an argon purge at the front. The stainless-steel tube is placed in a sodium heatpipe cylindrical chamber constructed from Inconel 601 for temperature uniformity and is sealed except for front opening. The cell having 120 mm long and 45mm in outer diameter, having centralized cavity aperture of 10 mm and a length of 50 mm immersed in the metal. In the present work, we have used silver-in-graphite blackbody cavity commercially procured. The blackbody comprises of high purity silver of 750 g filled in the graphite cell.The assembly is inserted inside the blackbody heating source exactly at the middle position.

B. High Temperature Blackbody Source

A horizontal blackbody heating furnace having a cylindrical sodium heat pipe lining assembly, automatically controlled with EUROTHERM make built-in-controller was used to provide stable temperature source covering a range from 450 ºC to 1200 ºC. The source stability was measured by a Type-S thermocouple at a temperature near 962 ºC to use it for measurement of silver point. The stability of the source was evaluated to be of the order of ±0.06 K over a period of about an hour in the above range which makes it suitable to use for realization of the fixed points by radiation technique.**Fig. 2**shows the study of stability of blackbody source at temperature of melting point of silver (962 ºC), monitored and measured by using a platinum/rhodium (Type-S) thermocouple**.**

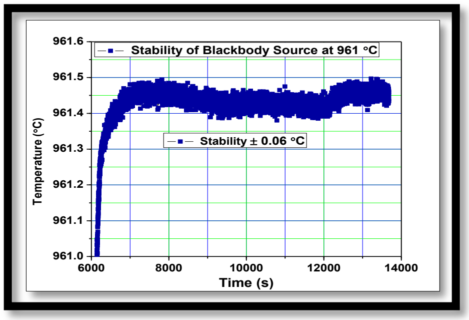
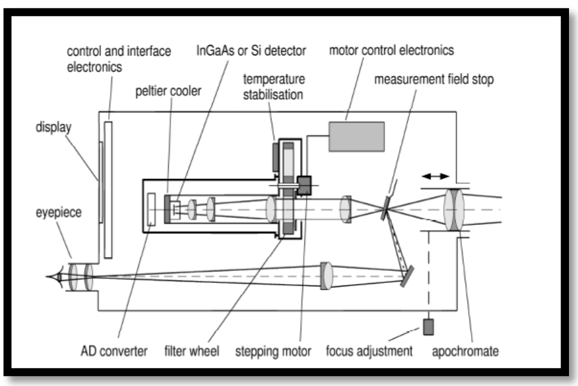


Fig.2: Stability study of blackbody Source at 962ºC

C. Description of Photoelectric Spectral Pyrometer

A schematic of internal structure of photoelectric spectral linear pyrometer, LP4 is shown in **Fig.3.** The pyrometer has been procured from M/s K.E. Technologies, Germany and calibrated from the national metrology institute (PTB), Germany was used in this realization of silver point. This pyrometer is a unique instrumentto be used as a transfer standard formeasurement of radiation temperature of a blackbody source like metal-in-graphite cavity. The photoelectric pyrometer has specific feature of linear temperature relation with the photocurrent generated when radiation fall on the detector having significant



spectral response.

Fig. 3: Internal structure of Photoelectric Spectral Linear Pyrometer, LP4

A high precision silicon photocell detector is used for measuring photocurrent. The instrument has an especial vacuum photocell with a guard ring and a homogenous cathode as receiver in connection with high standard electronics and isequipped with interference filters of suitable wavelength (say, λ=650 nm). The instrument has a nominal spot size of 0.25 mm at a focal distance of 600 mm. The image of aperture isreflected over the plane mirror placed at an angle of 45º in parallel to view it by an eyepiece using optical alignment. The image of aperture (as a black spot) of the pyrometer is focused over the image on the exact Centre of the cavity aperture of a metal-in-blackbody cavity. This confirms the radiation exactly falling over the detector surface of the pyrometer after making all required alignments between the radiating source and the pyrometer [15]. The optical system along with detector is a vacuum sealed chamber whose temperature is controlled and stabilized during measurement of photocurrent. The pyrometer has its capability of measuring photocurrent in the range from $1x10^{-12}$A to $1x10^{-8}$A and temperature in the range from 500 K to 3500 K. The chamber temperature, photocurrent and the temperature of the source are displayed in ºC/K on the front panel of the pyrometer. The pyrometer can be hooked to a computer through RS232 cable so that the temperature of the source, the photocurrent and the time variation curve of temperature can be displayed and recorded simultaneously.

D. Argon Gas Flow System

The fixed-point cell of silver is a pure metal-in-graphite cavity fabricated from high purity graphite. When graphite is exposed to high temperature, it starts oxidizing in the presence of air. Therefore,it is required to make arrangement of pure dry argon gas to be purged all around the cell in order to avoid oxidation of the graphite material. The pressure from the gas cylinder is so adjusted by using a flow meter to maintain the flow rate of argon gas to in-circulate it all around the cavity and at the same time does not affect the melting or freezing plateaus of the silver. The outer cylinder of stainless steel which contains the graphite cell has a cover plate with a tube that is connected to argon gas cylinder through a calibrated flow meter.

IV. MEASUREMENT TECHNIQUES

The silver point was realized by measuring melting and freezing plateaus on heating the metal-in-graphite blackbody cavity in the heat-pipe blackbody source.The heating source has an automatically controlled sodium heat pipe cylindrical tube contained in it. The blackbody cell containing silver is inserted in the heating source and placed at the middle position for uniform heating. The cell is set to heat initially at 600 ºC and the pyrometer, LP4 is set to focus the middle point of the cell and centrally align it by using laser lamp. The source temperature is then increased at every 100 ºC till the temperature reaches to 20 ºC higher than the melting temperature of silver i.e. at 982 ºC. The melting of sample starts and the temperature is stabilized. The pyrometer is hooked to computer and the display of temperature, corresponding photocurrent generated due to radiation from the cell and the plot between time and with temperature can be shown in the computer screen. The data on time variation of temperature in both scales (ºC, K), and with photocurrent can be stored in a file in order to analyze later. The radiation temperature from the bottom of fixed point cell is measured at different locations axially in a direction away from the source in order to study the uniformity and stability from the temperature profiles during freezing state.The measurement on melting and freezing of silver can be repeated at least three times to confirm the Ag point temperature.**Fig. 4** shows the experimental set-up for realizing the silver point temperature along with all necessary arrangements.

Black body source

Pyrometer, LP4

Objective lens

Fig. 4: Experimental set-up for realization of Ag-point using blackbody

V. MELTING & FREEZING PLATEAUS OF AG-POINT TEMPERATURE

In the measurement of silver point temperature,the complete equipment set-up comprises of a radiation pyrometer, LP4, high stability heat-pipe black body source, an 8-1/2-digitdigital voltmeter (DVM), a Type-S thermocouple to monitor the cell temperature and the argon flow arrangement to inflow required for purging of argon gas inside the metal cavity all around the metal-in-graphite cell in order to avoid oxidation of graphite and silver metal. The melting and freezing plateausof Ag have been measured. Freezing temperature is more preferred because of natural control of temperature for better precision achieved during the measurement. In this work a commercially made crucible was used to measure the melting and freezing plateaus and the results were obtained. The temperature profile of the source was measured at varied distance of pyrometer detector from the blackbody source in order to know the influence of temperature on the performance of melting and freezing plateau. The temperature difference between melting point and freezing point was investigated and the precision of stabilization was investigated as shown in the curves**Figs.5a** and**Figs. 5b**. The precision of the melting and freezing plateaus are shown in **Figs. 6a** and **Figs. 6b** which clearly show the level of stabilization with long time duration for each physical state of the metal.It is found that the melting and freezing points of silver were measured with a precision of ±0.060 ºC and ±0.090 ºC. This stability was possible because of sodium heatpipe cylindrical liner along with the metal in graphite cavity providing uniform temperature. The graphite crucible was placed inside the stainless steel cylindrical cavity and is purged with high purity argon gas with a controlled flow to avoid oxidation of graphite at 400 ºC and above.

VI. RESULTS & DISCUSSION

We have made efforts to measure the melting and freezing states of Ag in graphite blackbody cavity using high precision spectral radiation pyrometer in terms of stabilized temperature and photocurrentunder the condition of monochromatic radiation of wavelength 650nm in vacuum. **Fig.5a** and**Fig.5b.**

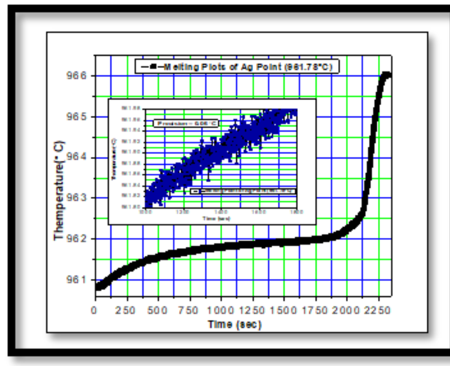


Fig. 5a: Melting plateaus of Ag-point

show the melting and freezing plateaus of silver point, both in time variation of temperature.

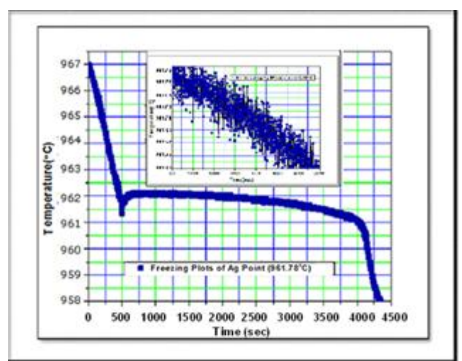


Fig. 5b: Freezing plateaus of Ag-point

Fig.6a shows the melting state along with precision of best curve measured for the melting point of silver while **Fig.6b** shows the freezing state with precision of best curve measured for the freezing point. A precision of better than ±0.06ºC was achieved for the best of repeated measurement plots at the melting and ±0.08 ºC at the freezing state of silver. In order to ascertain the best stability, data was collected on temperature variation due to locational change of the pyrometer detector from the bottom of the cavity.

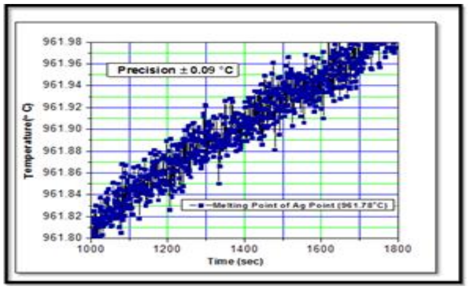


Fig. 6a: Melting plateau of Ag-point with its Precision of realization

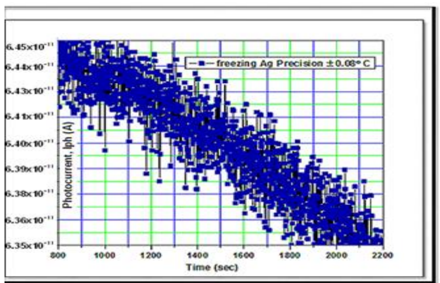


Fig. 6a: Freezing plateau of Ag-point with its Precision of realization

Fig.7 shows the temperature profiles of the blackbody source at silver point i.e. a locational variation of temperature shown by the pyrometer with respect to the radiation source during the freezing of silver metal. A stability of ±0.06 ºC has been achieved along a distance of 120 mm in the profile which is a significant stability under the experimental limitation. The mean value of photocurrent measured at the silver point realization for melting state is determined to be $6.450x10^{-11}$A, while the same as determined at freezing state realization is to be $6.395x10^{-11}$A. The I_{ph} at freezing is measured to be slightly lower than at melting state because of a number of contributory factors like the trace impurity contents in the metal, size of source effect, stability of detector response and condition of metallic sample which lowers the state under practical condition of realization.The melting and freezing states are affected by the impurity

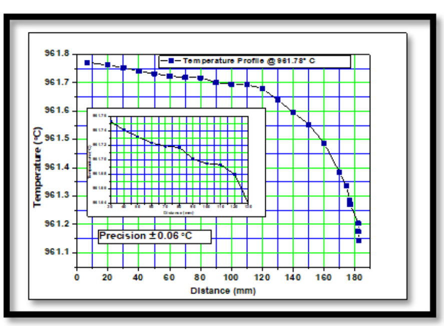


Fig. 7: Temperature profiles of the blackbody source at silver point.

contentspresent in the metal, though these may be present at the ppm level. Due to the impurities in the metaland comparatively highercontribution due to size of source effect a significant differencewas observed between the melting and freezing points. In the present work the melting point was measured to be 1235.04 K (961.89 ºC) which is 0.11 K higher than the assigned value in ITS-90 (1234.93K) while the freezing point was measured to be 1234.86 K (961.71°C), which is 0.07 K lower than the assigned value of ITS-90. The results although, show a significant difference between the melting and freezing point of the silver sample signifying another reason that the metal purity would have degraded to a greater extent. With all these practical implications in the present work, it was found that the silver point was measuredwith an uncertainty not better than ±0.367 K.

VII. EVALUATION OF UNCERTAINTY IN THE MEASUREMENT

The uncertainty in the measurement of each radiance temperature determined at freezing of silver source was estimated by using two different methods namely the Type-A, where the uncertainty component has been evaluated using statistical analysis of a series of repeated observations taken at stable freezing temperature and the other by Type-B estimation, where the uncertainty components were determined arising from different sources contributing to the measurement [16-22] i.e. those associated with the blackbody crucible, spectral responsivity, size of source effect, immersion effect, the non-linearity of output signal of the pyrometer detector. The mathematical model for uncertainty evaluation is defined by the following equations.

$T_m = T_F \pm U(\Delta T)$…….(5)

$(\Delta T) = k.\, u_c$ ……..(6)

$u_c = \sqrt{[\sum u^2_i(\delta T)]}$ ..…….(7)

Where T_m = Temperature to be measured, T_F = Fixed point temperature, U (ΔT) = Expanded uncertainty, *k* is the coverage factor and $\mathbf{u_c}$ is the combined uncertainty evaluated from various components of uncertainty affecting the measurement. The uncertainty components designated $\mathbf{u_i}$(δT), estimated from $\mathbf{u_1}$ to $\mathbf{u_9}$by Type-A or Type-B methods depending upon the source of uncertainty. Where,$\mathbf{u_1}$,is the uncertainty component estimated due to impurity content in the sample of silver used in the measurement, $\mathbf{u_2}$, the uncertainty estimated due to emissivity variation at the silver point blackbody, $\mathbf{u_3}$, the uncertainty estimated from the identification of the best plateau measured, $\mathbf{u_4}$, the uncertainty evaluated due to immersion effect over the cell, $\mathbf{u_5}$, the uncertainty estimated due to repeatability of data measured out of three sets, $\mathbf{u_6}$, the uncertainty due to stability of spectral pyrometerestimated from the available data in the instruction manual of the pyrometer, $\mathbf{u_7}$, the uncertainty evaluated due to size of source effect, a variation of temperature in the axial direction, $\mathbf{u_8}$, the uncertainty component estimated due to drift (the overall fluctuations) of signal during the measurement and $\mathbf{u_9}$, the uncertainty evaluated due to non-linearity of temperature estimated with respect to received signal from the blackbody source (effect of angular deviation). It is observed that uncertainty components due to stability of pyrometer and the size of source effect are the major components of the measurement.

The complete uncertainty budget is as shown in the **Table-1**, describing the significant contributions of uncertaintyin the measurement of the silver point as described above. The combined standard uncertainty and finally the expanded uncertainty of measurement was evaluated and reported to be ± 0.367 K at a coverage factor, k=2, for a level of confidence approximately 95%.

VIII. CONCLUSION

On realizing and measuring the freezing point of Ag in the laboratory using the spectral photoelectric pyrometer, the radiation temperature scale is established as per ITS-90. The fixed point of Ag is the primary standard of temperature to measure and calibrate high temperature radiation pyrometers and blackbody or near blackbody thermal sources. The experimental results show that the sodium heat pipe radiation source provides a stable and uniform temperature for attaining freezing plateau of longer duration minimizing the gradients caused along the fixed-point cavity. The results in this study leads to the conclusion that although, the silver point was measured to be 0.07 K lower than the defined value of ITS-90, it can be very effectively used for calibration of spectral and infrared radiation pyrometers, optical pyrometers and tungsten strip lamps which are employed as transfer standard devices and the sources for radiation temperature in the range from 1000 K to 2500K by measuring photocurrent at unknown temperatures and application of Planck's equations (3, 4).

Table 1: Uncertainty budget for Ag-point Measurement using Silver blackbody Source

S. No. Source of uncertainty at Ag-freezing point/K	Type	Standard uncertainty
1. Uncertainty estimated from Impuritiesin the sample of Ag used in the measurement	B	0.008
2. Uncertainty estimated due to emissivity variation	B	0.005
3. Uncertainty estimated due to stability of data measured (Plateau identification)	B	0.080
4. Uncertainty estimated due to immersion of the sample in the black body cavity	B	0.025
5. Uncertainty estimated due to repeatability of the Measurement at Ag point	A	0.030
6. Uncertainty due to pyrometer stability	B	0.100
7. Uncertainty estimated due to size of source effect, a variation of temperaturein the axial direction	B	0.120
8. Drift of signal in the measurement	B	0.026
9. Uncertainty due to non-linearity of temperature estimated with respect to received signal from the Blackbody source (effect of angular deviation)	B	0.022
Combined uncertainty at k=1		0.1833
Expanded uncertainty of measurement at k=2		**0.367**

Fixed point	Measured	Deviation	Photocurrent	Uncertainty
Freezing Ag	1234.93 K	-0.07 K	6.395×10^{-11} A	±0.367 K

IX. ACKNOWLEDGEMENT

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