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THE DEVELOPMENT OF PRIMARY CELLS AND STUDY OF THE MELTING POINT OF GALLIUM

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ABSTRACT: In the SMU we performed the research aimed at reduction of the uncertainties when realizing the defining fixed point of gallium in accordance to ITS-90. We divided research into two stages: 1. development, production and study of the primary cells, which will be used as a part of national temperature standard and 2. research of influence of impurities on gallium phase transition. Material of crucible is Teflon of very high purity. Crucible was filled up with the gallium of purity 99.99999 %. We examined the effect of thermal condition of melting process on the temperature and shape of melting plateau. In this first step we created only outer liquid-solid interface. In the second step we also created the inner liquid-solid interface. In the following step of research we plan to observe the influence of two specified impurities on melting and freezing of gallium.

KEY WORDS: gallium, defining fixed point, cell

1. INTRODUCTION

Temperature scale is realized in accordance with the document ITS-90, which is internationally accepted document allowing the realization of temperature scale ranging from -270.15 up to 1084.62 °C. Gallium with about a part in 10^{-7} total impurity content is commercially available, so for realization of gallium melting point we used gallium with 99.99999 % purity [1]. A sample of this purity allows one to achieve an exceptionally stable melting point. This paper presents the construction design of the new gallium cells and the results of performed experiments.

2. EXPERIMENT AND RESULTS

Gallium cells were filled up with high pure gallium (99.99999 %). Since gallium has a large expansion at solidification (3.1 %), it makes it desirable to use a slightly flexible construction of the cell, therefore is the crucible made of Teflon. Fig.1 represents the design of gallium cell. Teflon is highly plastic, it does not interact with metal and it ensures the cell tightness. The thermometer well is made together with the cap as a single piece. In the cap assembly, a provision was made for an outlet, in order to evacuate and fill the cell with gas at a present pressure as described in [2]. To avoid the contamination of the gallium, all parts of the cell which are in direct contact with gallium were carefully cleaned before the cell filling. Description of cleaning steps is shown in Tab. 1.

Fig. 1: Photo of gallium cell

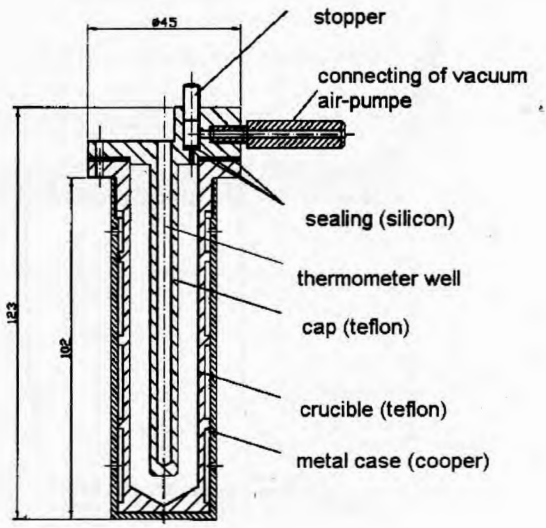


Fig. 2: Miniature gallium cell for adding the impurities

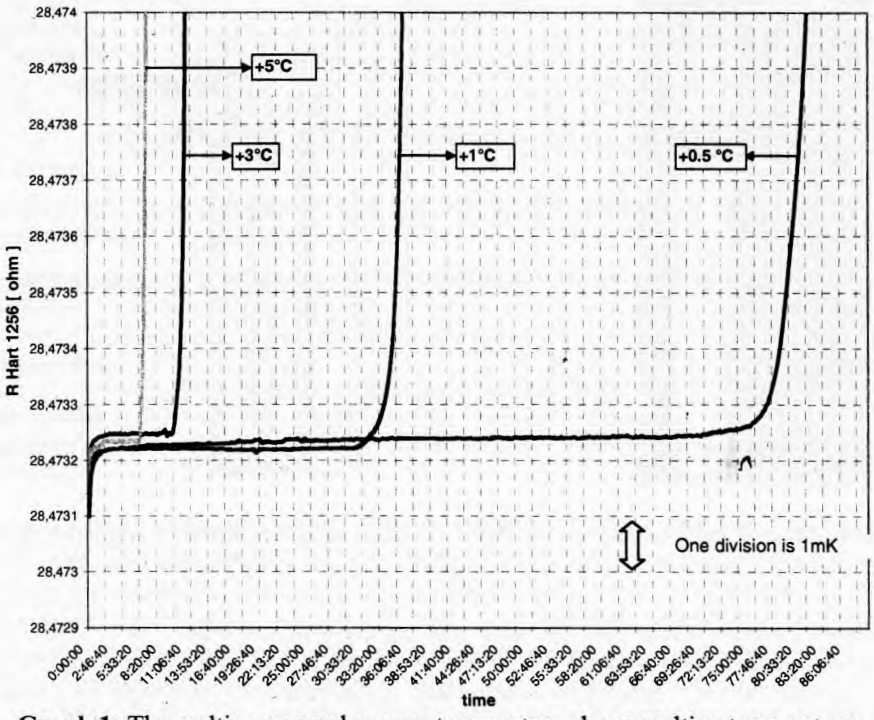
Cells were filled in glove boxes under protecting argon atmosphere.

We realized the melting point of gallium in stirred liquid bath. Temperature of the bath was initially set to 28.7 °C. After the stabilization, temperature of the bath was increased to the temperature of 5 °C above the melting temperature and plateau was observed. Similar experiments were performed when bath temperature was 3 °C, 1 °C, 0.5 °C above the melting. Plateau was monitored by standard platinum resistance thermometer (SPRT) with the nominal resistance of 25 Ω. The resistance of the SPRT was measured by means of AC Resistance Bridge. Stability of thermometer was checked by its measurement at the triple point of water.

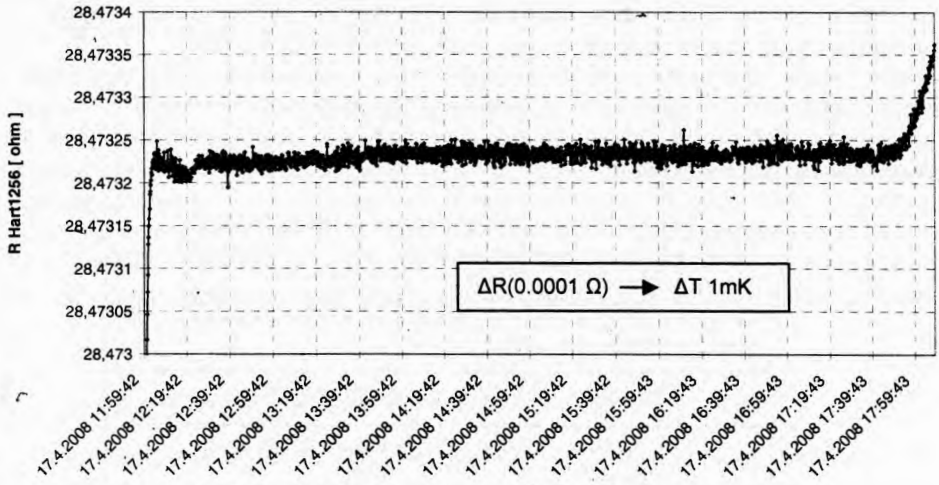
Graph 1 shows that melting curve obtained various temperatures of the liquid bath. From this graph it is evident that the temperature of thermostat has influence on the length of the plateau, just as well as on its position. Graph 2 shows melting curves when the temperature of the bath was 5 °C and 3 °C above the melting temperature.

Tab. 1: Steps of the process of cleaning the cells

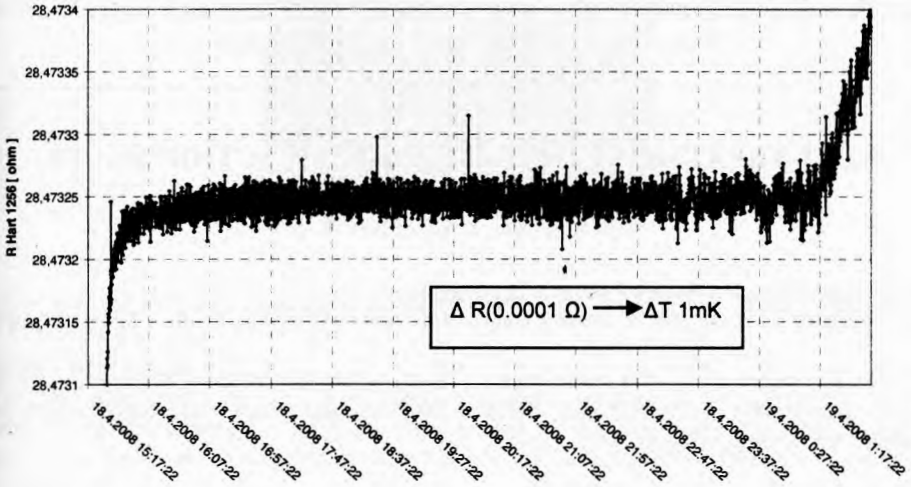
Step 1	Removing the residue of impurities by detergent after the mechanical cultivation of the cells
Step 2	Eluting the cells by warm water
Step 3	Eluting and leaching out the cells by distill water
Step 4	Chemical cleaning by the mixture of the acids $HCl+HNO_3$ in ratio 1:3, 24 hour
Step 5	Eluting by distill water
Step 6	Eluting by repeated distill water
Step 7	Leaching out in distill water 5 days
Step 8	Vaporize of the cells by repeated distill water



Graph 1: The melting curves by some temperature above melting temperature



Graph 2: The melting curve by 5 °C above the melting temperature



Graph 3: The melting curve by 3 °C above the melting temperature

3. DISCUSSION

Performed experiments were specialized at the observation of influence of the thermal condition on temperature and the phase transition of gallium. Duration of the plateau at various temperatures of the bath is presented in Tab. 2. Experiments oriented on study of influence of impurities will be performed by means of miniature cells (Fig. 2), which will be filled with gallium which contains known quantity of specified impurities.

Tab. 2: Duration of the plateau at various temperatures

Temperature of the bath	Duration of the plateau (within 0.3 mK)
5 °C	6 hours
3 °C	9 hours
1 °C	30 hours
0.5 °C	70 hours

4. SUMMARY

Two primary gallium fixed point cells have been developed and built at the SMU. Few experiments have been performed by now. Experiments were aimed at study of the effect of thermal conditions of realization on the phase transition temperature and the shape of the melting plateau. On the basis of our results it seems, that thermal conditions have effect not only on the shape of the plateau, but also on its position. To verify this, it is necessary to perform a lot of additional experiments.

5. REFERENCES

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