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Supercooling Effect in Miniature Gallium Phase Transition Cell

Giuseppina Lopardo,* Fabio Bertiglia, Giuseppe Braccialarghe, Michael Florio, Ferruccio Girard, Domenico Giraudi, and Federico Santoro

Miniature gallium fixed-point cells are widely used as temperature standards and, because of their near-ambient transition temperature (29.7646 °C), are used in a variety of applications for the accurate in situ calibration of thermometers traceable to international system of units. A peculiarity of gallium, compared to other metal fixed-points, is its high degree of supercooling which can limit its possible applications. However, in literature, different authors observed the suppression of supercooling in case of small gallium cells. The reason for this is not completely clear. In this article, the supercooling effect was investigated in the specific case of a miniature gallium cells designed for space applications. The results show that the degree of supercooling is function of the thermal history of the cell. As the temperature of the metal, in the liquid state, increased, a linear dependence between the overheating of the sample and the supercooling effect was observed. This relationship is particularly important when the cell must be used to perform subsequent calibration cycles. An understanding of the factors that govern the supercooling, as proposed in this study, could help to develop ways to control it, in the design of calibration systems, and procedures to be used in different applications.

1. Introduction

Fixed-point cells with reduced dimensions, compared with the larger cells employed in primary calibration laboratories, are currently used as temperature references in different application fields. They satisfy the requirements for traceability to International Temperature Scale of 1990 (ITS-90)^[1] and high-accuracy calibrations for onsite applications particularly where access restrictions exist. Actually, many temperature sensors are calibrated before setting them up and any drift or malfunctions, while in use, are difficult to detect. Miniature fixed-point

cells allow to recalibrate industrial temperature sensors^[2,3] and thermocouples^[4] directly on-site without the need of removing sensors.

Miniature gallium (Ga) phase transition cells are extensively studied for space applications.^[5] For example, they are being embedded in blackbody cavities as temperature references for satellite-borne radiometers.^[6–8] This thanks to a set of favorable advantages, like: 1) melting point close to ambient temperature ($T_{mp} = 29.7646$ °C); 2) reduced dimensions and weight that allow them to be easily accommodated on-board;^[8] 3) SI traceability throughout all the lifetime of satellite;^[7] and 4) high-accuracy measurements for long-term climate change study.^[9]

In this study, we focus on a peculiar property of gallium: its supercooling. It is often mentioned in the literature^[10–12] that Ga has the tendency for deep freezing far below melting point. This means that it

does not solidify even when cooled well below its melting point over dozens of degrees Celsius. The degree of supercooling or simply—supercooling—is defined as $\Delta T_{SC} = T_{mp} - T_{nuc}$, i.e., the temperature difference between gallium melting point (T_{mp}) and its nucleation temperature (T_{nuc}).

The ability to keep Ga in the liquid state is technologically interesting because of its use in many emerging applications, such as microfluidics, stretchable electronics, and catalysis.^[13] However, it may be undesirable for other applications such as variable stiffness elastomers,^[14] shape memory systems^[15] or automatic calibration of sensors on autonomous platforms.^[6]


Deep supercooling of high-purity Ga is very evident in primary standard fixed-point cells used for the realization of ITS-90 and this is the most obvious reason why the gallium is the only metal-based fixed point on the temperature scale that is realized as a melting rather than freezing point.^[16] However, different authors^[6,8] in their experiment with Ga in small cells measured a supercooling of only a few degrees below freezing temperature. Pearce et al.^[8] report that the cell was placed into dry ice only once after the filling process to ensure that the gallium is initially solid.

The reason for the suppression of the supercooling effect in Ga small cells is not completely clear.^[6]

Speculation in the early literature that the supercooling behavior of gallium is related to the high purity of the material has not

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been confirmed. Freezing without deep supercooling has been observed with 6 N samples.^[6,17]

Some authors assumed, tentatively, that the suppression of supercooling, found in their experiments with miniature cells, depends by material of the Ga container. For example, Sostman and Manley^[17] didn't see the effect when using Nylon for the thermometric well. However, Burdakin et al.^[6] using Polytetrafluoroethylene (PTFE), which is not wetted by liquid Ga, in contrast to Nylon, reported that supercooling never exceed 1 °C. Therefore, they concluded that the size of the cell seems to be the most probable factor causing this phenomenon.

It is known that sample size is a factor influencing the degree of supercooling. Kumar et al.^[18] examined the thermal behavior of gallium particles and found that the supercooling effect was more pronounced for micron and submicron particles compared with bulk gallium. Parravicini et al.^[19] give evidence that pure Ga (99.999%) nanoparticles in the range between 3 nm and 15 nm range can be undercooled at least down to -183.15 °C without crystallization. Miyazawa and Pound^[20] observed supercooling (ΔT_{SC}) in the range from 95 to 102 °C with extremely high-purity (99.99999%) gallium droplets (between 2 and 36 μm , $\approx 10^{-7}$ g) suspended in silicon oil that was cooled down at a rate of 0.1 °C min^{-1} .

Turnbull^[11] interpreted the nucleation phenomena in liquid metals based on the critical size and interfacial energy concepts. In large continuous masses, nucleation is almost always catalyzed by extraneous interfaces. However, in very small droplets, the probability that a catalytic inclusion is present is low.

On this base, additives have also been considered to either mitigate or promote the supercooling of gallium.^[21–23] Joshipura et al.^[12] demonstrated that the native oxide formed on bulk gallium enhances supercooling. Finally, some studies have been carried out to reduce supercooling by applying external forces such as impact^[24] or electrical field.^[25]

A dependence of supercooling on the thermal history of the melted Ga was reported by different authors.^[26,27]

Burdakin et al.^[6] for example observed a 5 °C supercooling in a small cell for space-borne radiometers application only after intentional overheating between 50 and 60 °C. He explained that the dependence of the degree of supercooling upon the previous overheating means that, in the vicinity of the phase-transition temperature, the liquid Ga has a short-range ordered micro-heterogeneous (clustered) structure.

Elston and Ervin^[27] studied the effect of thermal history and samples mass on solidification of gallium bulk, with differential scanning calorimetry and rapid thermal cycling techniques. They excluded a dependence of supercooling on sample mass for values ranging from 84 to 10 g (99.99% gallium purity). Instead, they found a linear dependence between supercooling and overheating ($\Delta T_{OH} = T_{max} - T_{MP}$), for T_{max} (maximum temperature at which the sample is heated) below 60 °C (critical temperature), above this temperature, the supercooling is referred to be independent and constant.

A similar trend was observed by Wolny et al.^[24] performing electrical resistivity measurements on Ga droplets (6 N purity) at cooling rate between 10 and 15 °C min^{-1} , and by Aleksandrov et al.^[28] with critical temperature equal to 54 °C, on a 0.5 g gallium sample (no purity information) using cyclic thermographic analysis. Zhang et al.^[23] found that, for a gallium sample of 40 mg, the critical temperature is dependent on cooling rate in the range from 55 to 70 °C. In their experiment, the cooling rate is scanned in the range between 5 and 20 °C min^{-1} .

Turnbull^[26] attributed the thermal history effect to surface tension. Some crystal embryos can be retained with the help of surface tension when the heating temperature is not greatly exceeding the melting point. Those retained embryos serve as a nucleation centers reducing the supercooling of the liquid.

In this article, we analyze the dependence of supercooling on overheating and sample cooling rate of a high-purity Ga with a sample mass of 8.6 g used for the realization of a miniature phase-change cell for space applications. The peculiarity of this work is in the measurement of the degree of supercooling in real

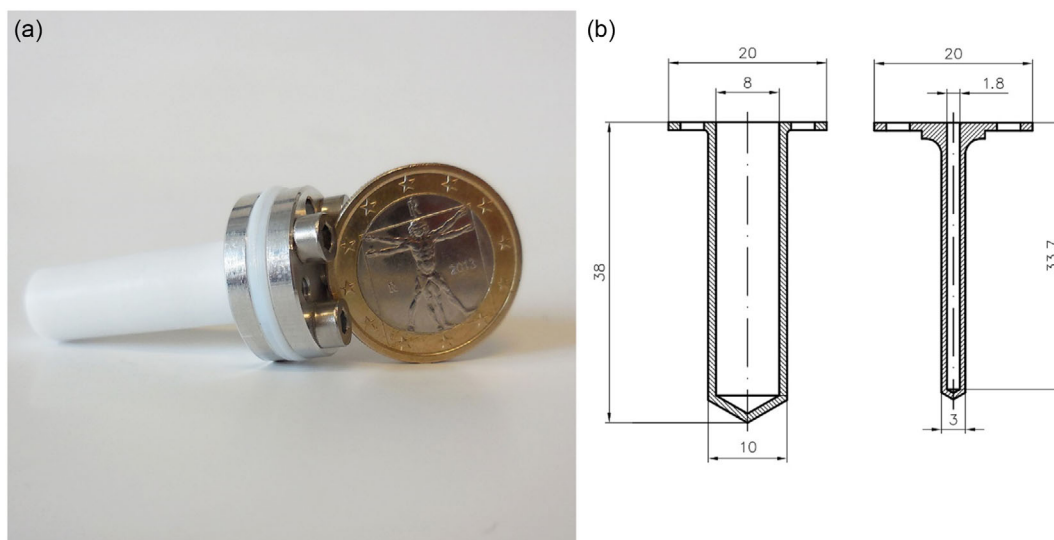


Figure 1. a) Picture of miniaturized Ga phase-change cell; b) technical drawing of miniaturized Ga phase-change cell, the dimensions are in millimeters.

working conditions to define the limitations of the device when used to calibrate temperature sensors in accordance with ITS-90. In fact, miniature size cells mounted on board of satellite may be subjected to unintentional overheating during flights.^[29] It is important to know the relation between overheating and Ga nucleation temperature to successfully repeat calibration cycles. In fact, in case of large overheating, the cell is no more able to repeat melting and freezing cycles following the thermal cycle routine established before the overheating event. In addition, in the phase of thermal management design, a dedicated cooling source should be planned to bring the gallium back to its original solid state.

However, this argument can be generalized for any other application where a Ga phase-change cell is submitted to heating well over its melting point.

2. Experimental Section

A miniaturized gallium fixed-point cell was realized in which all the parts such as the container, the lid, and the thermometric well were made of PTFE (Figure 1a). The thermometric well allowed inserting the thermometer completely surrounded by the Ga ingot. The PTFE also offered the advantage to be inert

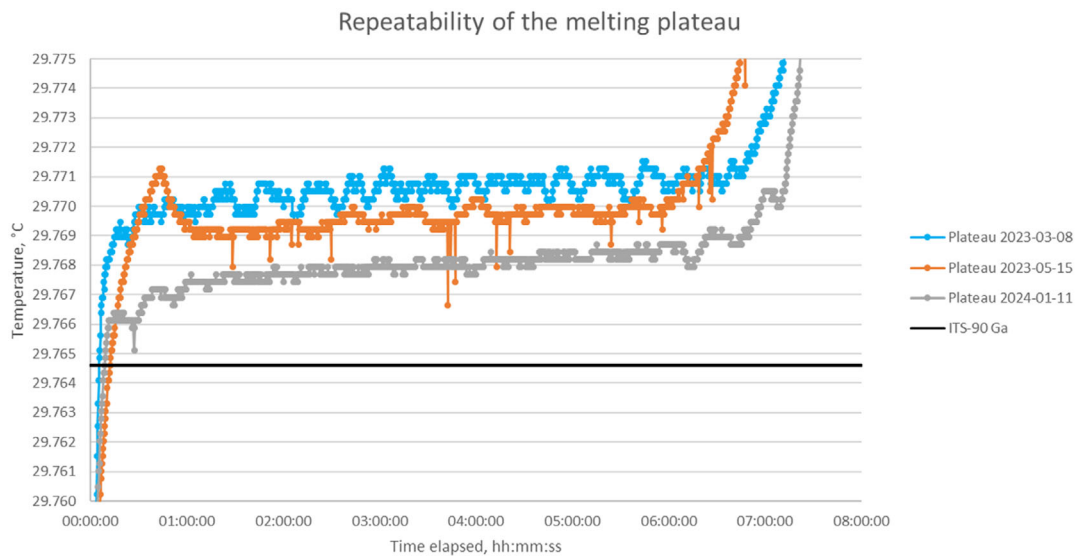


Figure 2. Repeatability of the melting plateau of the miniature Ga cell.

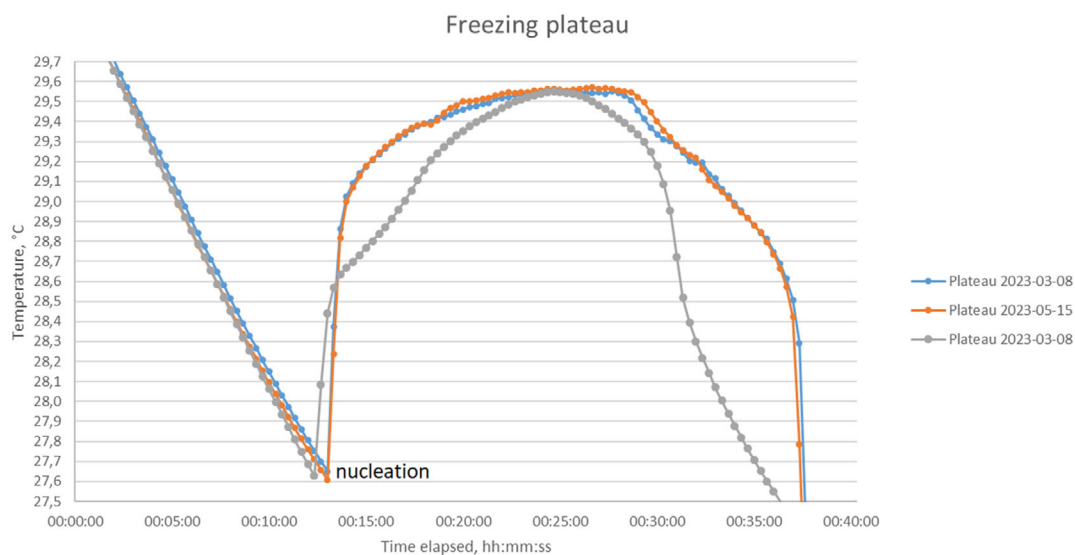


Figure 3. Solidification process of Ga after melting plateau.

with respect of Ga corrosion and able to compensate for its expansion during solidification (4% increase in volume). The selected cell configuration, for materials and geometry, was very similar to conventional ITS-90 primary standard fixed-point cells.

The cell (Figure 1b) had length of 38 mm, outer diameter of 10 mm, and internal diameter of 8 mm, respectively. The thermometric well was 33.7 mm long with an inner diameter of 1.8 mm. The sealing of the cell was guaranteed by a PTFE cap clamped between two aluminum flanges. All container parts in direct contact with Ga were made of PTFE.

The cell contained 8.6 g of high-purity Ga (99.99999%) manufactured by Goodfellow.

The temperature within the cell was measured by using an industrial-type platinum resistance thermometer (Pt100 with diameter 1.5 mm and length 15 mm) and recorded with a 1586 A Fluke Super-DAQ data acquisition system along with a

2588 Multiplexer. Resistance data from Pt100 were logged to file with a sampling time of 20 s.

The thermometric system was calibrated by comparison against the primary gallium Italian national standard. The calibration uncertainty was estimated to be 0.01 °C ($k = 2$). A typical industrial temperature sensor was considered to be more representative to real working condition of the cell, even if at the cost of an increase in uncertainty.

The small cell was accommodated into a cylindrical aluminum block to simulate the thermal mass of a cell's body container. The aluminum block was then placed into a climatic chamber (Kambic model KK-190 CHLT) for thermal performance assessment of the cell. The climatic chamber has a thermal stability of ± 0.08 °C and a thermal uniformity of ± 0.4 °C. The air temperature in the chamber was monitored using a second Pt100.

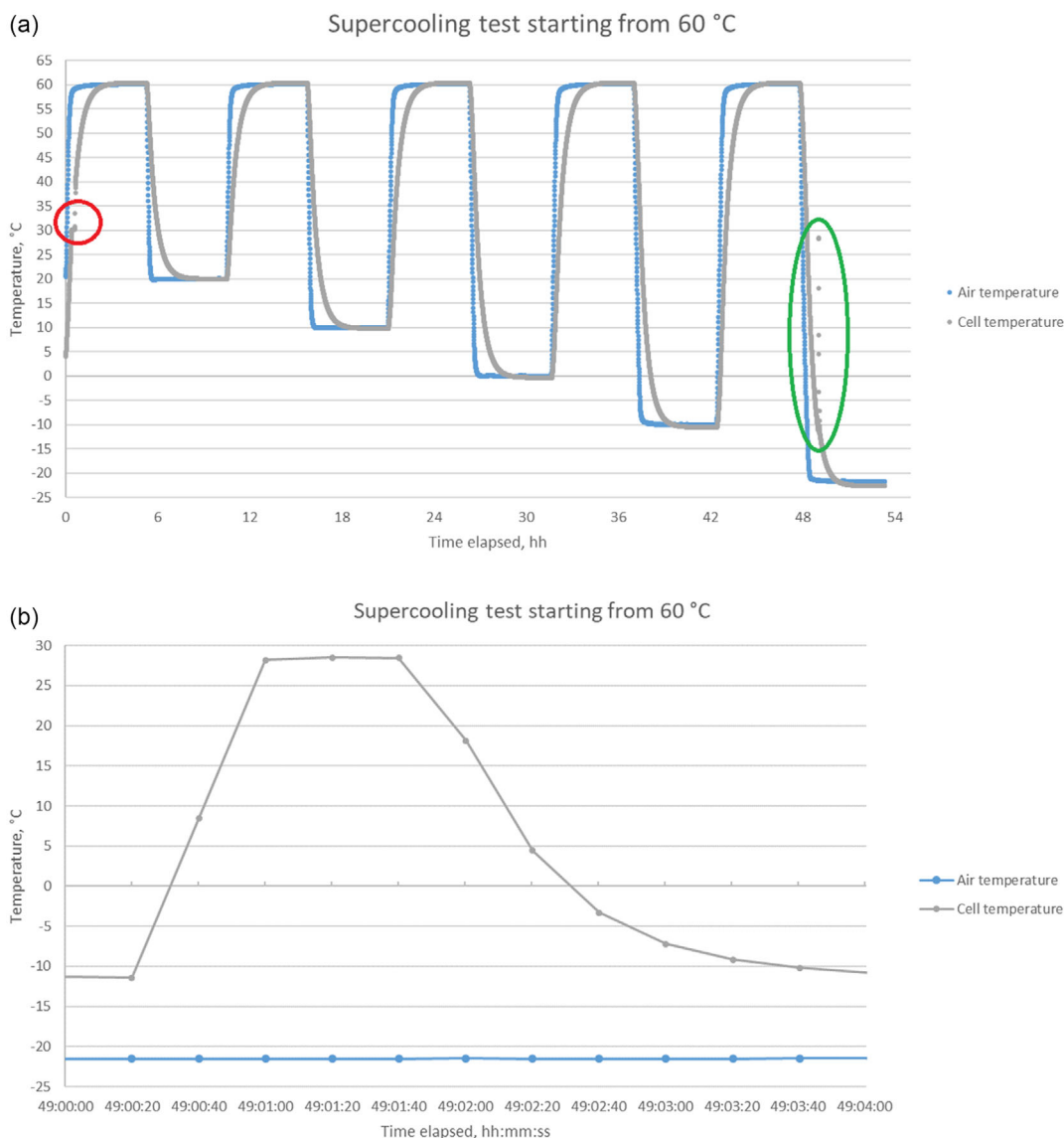


Figure 4. a) Test of solidification of Ga sample after heating at 60 °C. The gallium remains solid until -11.75 °C. b) A zoom of freezing process.

Before starting the experiments, to ensure that the gallium in the cell was in solid state, the cell was cooled down to -40°C for 3 h inside the climatic chamber.

3. Results

3.1. Melting and Freezing Plateaus

Typical melting plateaus of the gallium, recorded with thermometer inside the cell, are shown in **Figure 2**. They were achieved setting the chamber air temperature at 29.9°C and the cooling and heating rate of the climatic chamber equals to $250^{\circ}\text{C h}^{-1}$ ($4.2^{\circ}\text{C min}^{-1}$).

The plateaus are about 6 h long, the temperature stability is better than 1 mK (in agreement with the resolution of the sensor)

Table 1. Summary of the overheating tests. In the first column is reported the maximum temperature at which the gallium cell was heated repeatedly, in the second column the temperature of nucleation, and in the third column the calculated degree of supercooling.

T_{\max} [$^{\circ}\text{C}$]	T_{nuc} [$^{\circ}\text{C}$]	ΔT_{SC} [$^{\circ}\text{C}$]
30	27.51	2.25
35	23.15	6.61
40	17.22	12.54
45	9.44	20.32
50	2.15	27.61
55	-5.20	34.96
60	-11.71	41.47
65	-19.79	49.55
70	-20.44	50.2
75	-20.19	49.95
80	-20.06	49.82

and the offset from the ITS-90 temperature of gallium is less than 6 mK. Finally, the reproducibility is within 2 mK. After a period of 10 months, effects due to contamination of the metal are not evident because they are less than Pt100 calibration uncertainty.

After each plateau, subsequent freezes were achieved a few degrees below the melting point. **Figure 3** reports the temperature recorded by Pt100 during the cooling. The temperature of liquid gallium sample follows the chamber air temperature until nucleation. An upward peak, the *recalescence*, corresponds to the start of the nucleation process. During the transition from liquid to solid the temperature is approximately stable and equals the melting temperature. Finally, completed the freezing plateau, the sample is solid and continues to follow the trend of the chamber air temperature.

It can thus be concluded that, following a temperature program to realize the plateau (maximum climatic chamber air temperature equals to 29.9°C and then cooling down to 22°C), a minimum degree of supercooling, equals to $\Delta T_{\text{SC}} = 2.2^{\circ}\text{C}$, is observed. These results are in accordance with those of other authors.^[6,8] They didn't find any significant supercooling effects in miniaturized gallium cells.

The ability of miniature phase-change cells to reliably perform melt and freeze cycles without the need to reduce the temperature significantly for freezing makes them suitable for use in remote environments such as space.

3.2. Thermal History Effect

The effect of overheating temperature on supercooling was studied performing a series of thermal cycles on a gallium cell inserted in an aluminum block. The temperature of gallium was measured inside the thermometric well of the cell and the heating and cooling process was obtained by changing the climatic chamber set point, as done during phase transition

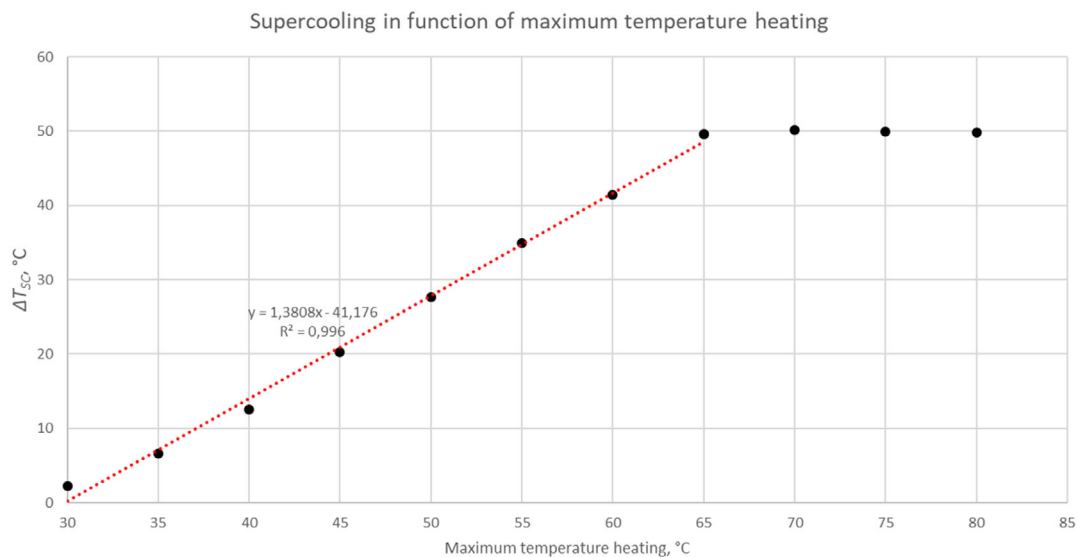


Figure 5. Supercooling (ΔT_{SC}) in function of maximum temperature of Ga cell heating (T_{\max}).

measurements. The cooling and heating rate of the chamber was maintained at $250\text{ }^{\circ}\text{C h}^{-1}$ ($4.2\text{ }^{\circ}\text{C min}^{-1}$).

We intentionally heated the cell to $T_{\text{max}} = 60\text{ }^{\circ}\text{C}$ and then looked for the successive freezing. The test is reported in **Figure 4**, where both the chamber air temperature (blue line) and the temperature in the gallium cell (gray line) are reported. The cell was heated to $60\text{ }^{\circ}\text{C}$ for at least 5 h and then cooled down to ever lower temperatures until the supercooling nucleation occurred. The red circle in **Figure 4a** shows the melting plateau at constant temperature. The plateau is very short in duration because the temperature raises faster compared with the test reported in **Figure 2**. Instead, the green circle represents the exothermic solidification process (nucleation). The supercooled nucleation occurred at $-11.75\text{ }^{\circ}\text{C}$, and this value is used to calculate the amount of supercooling $\Delta T_{\text{SC}} = 41.5\text{ }^{\circ}\text{C}$. Immediately after the nucleation (**Figure 4b**), the temperature raises to the gallium melting point (among $29\text{ }^{\circ}\text{C}$) and, on

conclusion of solidification, the cell temperature drops, following once more the trend of the chamber air temperature.

To study extensively the supercooling effect as a function of sample heating, the climatic chamber was programmed to follow a complete thermal cycle in the range between $35\text{ }^{\circ}\text{C}$ and $80\text{ }^{\circ}\text{C}$ with a step of $5\text{ }^{\circ}\text{C}$ to scan a set of possible heating temperatures. The settling time for each temperature equals 5 h as in the previous tests. After each heating step, the chamber was cooled down to $-25\text{ }^{\circ}\text{C}$, to ensure that gallium was solid. The cooling and heating rate of chamber was settled to $250\text{ }^{\circ}\text{C h}^{-1}$ ($4.2\text{ }^{\circ}\text{C min}^{-1}$). In **Table 1**, the nucleation temperatures and the degree of supercooling calculated at each heating step are reported.

Figure 5 shows a plot of ΔT_{SC} versus maximum gallium temperature as reported in **Table 1**. For $T_{\text{max}} < 65\text{ }^{\circ}\text{C}$, a linear relationship between overheating and supercooling appears. However for $T_{\text{max}} \geq 65\text{ }^{\circ}\text{C}$, the degree of supercooling is

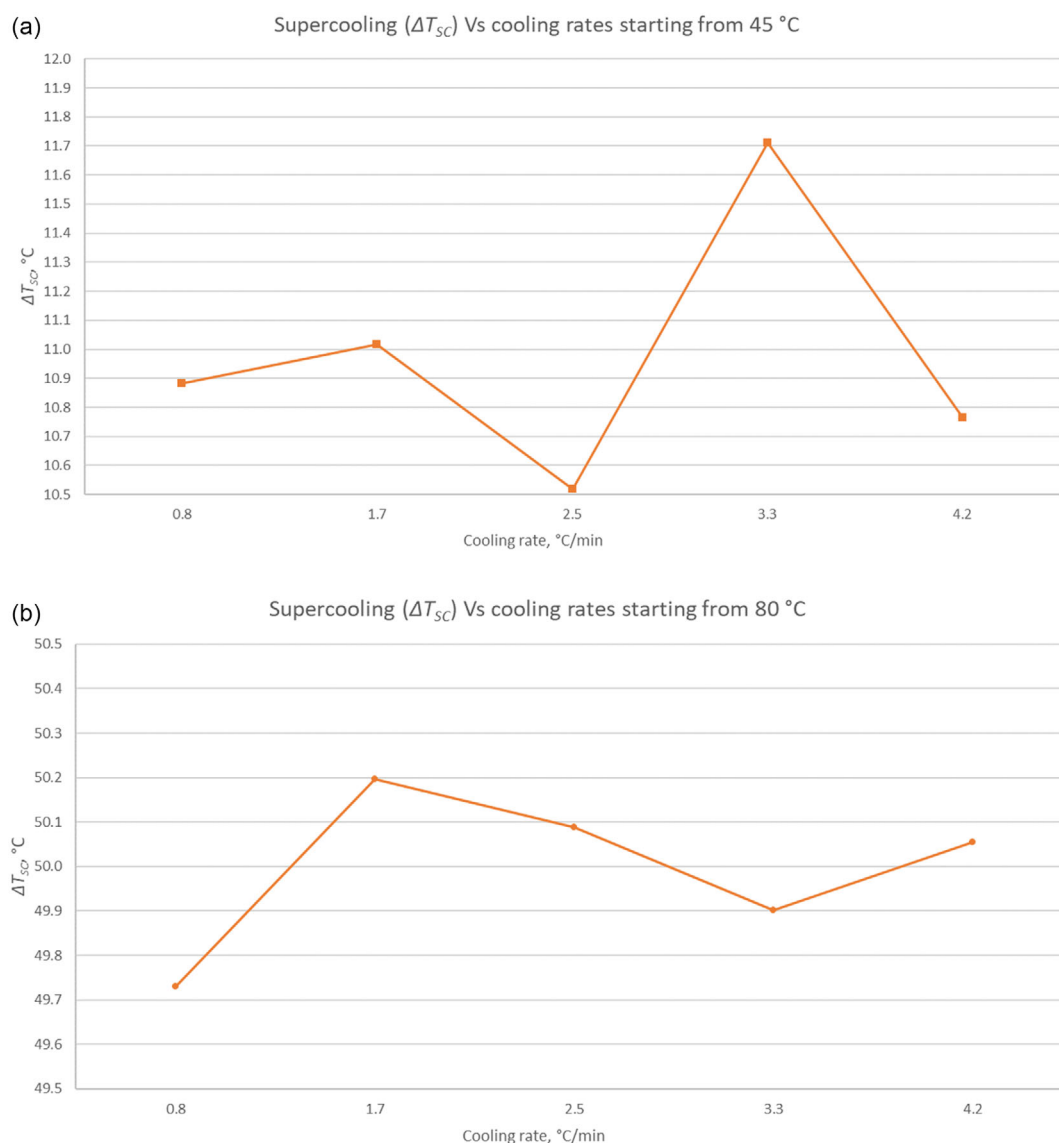


Figure 6. Supercooling (ΔT_{SC}) measured at different cooling rates, starting from two different maximum temperatures: a) $45\text{ }^{\circ}\text{C}$ and b) $80\text{ }^{\circ}\text{C}$.

independent from the thermal history of the cell and constant at roughly 50 °C. The results are in accordance with those of Elston et al.^[27] Aleksandrov and Frolova,^[28] and Zhang et al.^[23] but obtained with different techniques and gallium quantities.

As shown in Figure 5, below 35 °C, the supercooling degree is relatively small, confirming as, working in normal condition of phase transition, the gallium can turn back to the solid state just by decreasing the temperature of only few degrees below its melting point.

3.3. Cooling Rate Effect

To evaluate whether the cooling rate of the climatic chamber affects the supercooling and the thermal history dependence, the previous measurements were repeated with different climatic chamber cooling rates of: 50 °C h⁻¹ (0.8 °C min⁻¹), 100 °C h⁻¹ (1.7 °C min⁻¹), 150 °C h⁻¹ (2.5 °C min⁻¹), 200 °C h⁻¹ (3.3 °C min⁻¹), and 250 °C h⁻¹ (4.2 °C min⁻¹), respectively. It is worth noting that the last rate is the maximum value achievable with the climatic chamber in use.

Figure 6 reports the measured degree of supercooling as a function of cooling rate after setting two different maximum temperatures: 45 °C and 80 °C. The gallium cell was heated at 45 °C (respectively at 80 °C) and after 5 h cooled down at -25 °C at different rates.

The graphs in Figure 6 allow to conclude that, at these overheating values, there is not a clear dependence between supercooling degree and cooling rate.

In contrast, Zhang and co-authors^[23] found dependence between supercooling and cooling rate for a gallium sample of 40 mg, but at different scanning rates (between 5 and 20 °C min⁻¹).

4. Conclusions

In this work, a Ga miniature phase-change cell was realized and characterized for in situ accurate calibration of temperature sensors directly traceable to ITS-90. The reduced dimension of the cell and the transition temperature of the gallium point (29.7646 °C), located near ambient, make this cell newsworthy for different fields of application.

The obtained melting plateaus show long duration and high degree of repeatability, characteristics that make it suitable for a routine calibration process. Importantly, freezing of the gallium has been obtained, after complete melting of the sample, with only minimal undercool of few degrees Celsius. All measurements reported in this article were obtained with the temperature sensor located inside a thermometric well in the Ga sample.

The suppression of supercooling was extensively examined to establish the reason for the different behavior if compared to large cells used for ITS-90 realization.

The degree of supercooling was found to be strongly dependent on sample overheating and an experimental function of its effect was derived. The supercooling increased linearly with the maximum temperature at which the sample was heated above the melting point. However, above a critical temperature value

(about $T_{max} = 65$ °C), the supercooling was independent from overheating and essentially constant ($\Delta T_{SC} = 50$ °C).

This relationship agreed well with literature but, for the first time, it was demonstrated for a bulk Ga sample characterized under real-use conditions.

Finally, the effects of the climatic chamber cooling rate were investigated to evaluate the influence of real working conditions on the thermal history dependence of supercooling. In the range of cooling rates between 0.8 and 4.2 °C min⁻¹, no relationship was found.

The results presented in this work allowed to overcome the common concern surrounding phase-change materials, such as gallium, that generally has a very large supercooling and it is difficult to solidify without an externally induced large thermal gradient. This work demonstrates as a well-done characterization can provide a methodology to mitigate the occurrence of large supercooling process or activities to control it.

Conflict of Interest

The authors declare no conflict of interest.

Author Contributions

Giuseppina Lopardo: investigation (equal); methodology (equal); and writing—original draft (lead). **Fabio Bertiglia:** data curation (lead); investigation (equal); and methodology (equal). **Giuseppe Braccialarghe:** methodology (equal). **Michael Florio:** methodology (equal). **Ferruccio Girard:** investigation (equal) and methodology (equal). **Domenico Giraudi:** investigation (equal). **Federico Santoro:** investigation (supporting).

Data Availability Statement

The data that support the findings of this study are available from the corresponding author upon reasonable request.

Keywords

galliums, miniaturized cells, overheating, phase change cells, supercoolings

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