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Heat Flow metric Policy for Dynamic Monitoring of Fixed-point Cells

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Topic

In this paper, an original configuration, named as cell-within-cell, is described together with a specific measurement policy. To this end, concomitant temperature and heat flow assessments are carried out so as to obtain the exhaustive energy monitoring within the thermometric fixed-point cell. Two different melting schemes are investigated so as to consider an optimized control strategy in the near future.

1. General considerations regarding thermometric fixed-point cells

The characterization of temperature transition processes associated with numerous pure materials is since a long time ensured by way of high grade Resistor Temperature Detectors, such as classical Pt-25 devices. Such a way to operate enables a well-known and reliable measurement policy as regard various fixed points defining the International Temperature Scale, especially EIT-90 [1].

Sensitivity and low-noise sensor structures are most fundamental prerequisites for best operation together with specific designs regarding the cell, so as to screen out any polluting materials. Moreover, a prominent issue is to be considered as the phase transition is most often associated with an unfortunate marked density variation of the material experimented with. Then, the dimensional parameters of the cell must meet specifications allowing to reduce any untoward temperature gradient highly susceptible to alter the quality of the measurement process.

2. The cell-within-cell configuration

Indeed, numerous issues are known to arise as regards the response time of the quite cumbersome Pt-25 device, allowing just a sole integrated accurate measurement. Hence, the zeroing of any distributed temperature gradient is mandatory prior to analyse the temperature reading. Figure 1 illustrates the original design so-called cell-within-cell.

The main concept, keystone of such a device, hinges on a generic cell inserted into another one arranged as an isothermal structure: to this end, the isothermal condition is ensured by way of the same material in its own solid-liquid changing state than that experimented with. Considering as verified the mandatory metrological measurement provisions [2], such a cell should allow better performance than the same unit that would be directly arranged within a standard controlled furnace as its medium environment. A real and effective thermal shield is allowed thanks to both transitions occurring simultaneously, with the straightforward provision met as the load experimented with in the inner cell is far lower than that inserted within its encompassing guard. Then, the measurement policy presented in this paper provides strong evidence of the efficiency of the cell-within-cell configuration.

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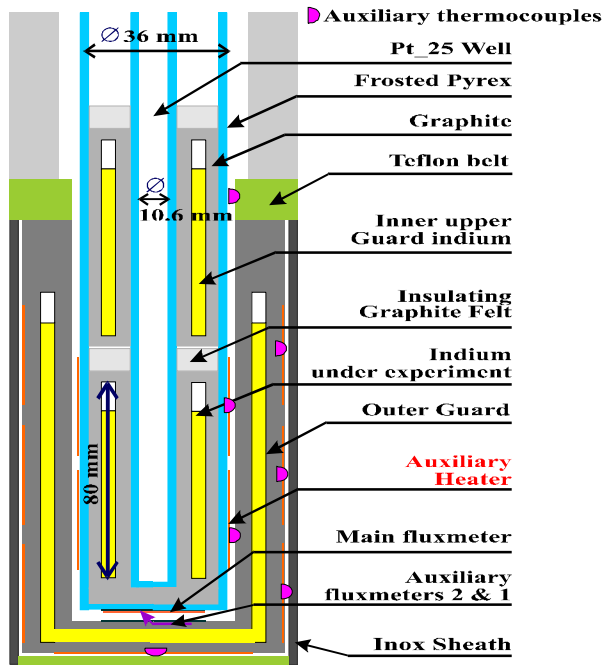


Figure 1a: Cell-within-cell configuration

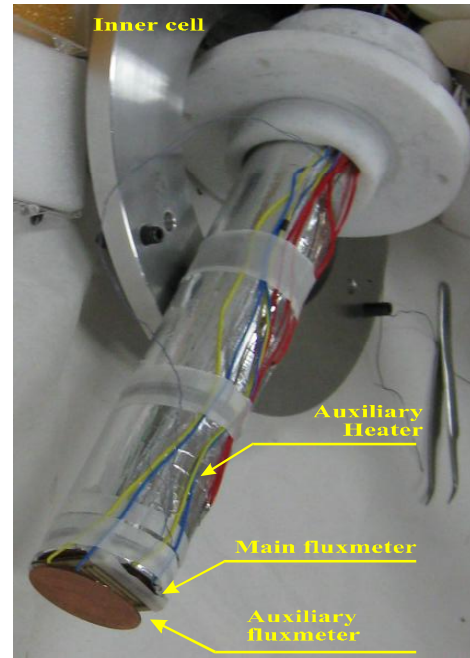


Figure 1b: Implementing heat flow meters

3. Defining a challenging measurement policy

As regard the thermal state of any system, Temperature and heat flow rate are both the state variables to be controlled [3]. In this paper, the coupling of a high-graded heat flow meter (HFM) with an effective temperature sensor (PRT 250) is considered, with a view to enhancing classical designs while keeping their proven metrological qualities. Such an approach is most valuable for taking advantage of the energetical monitoring in addition with the classical time visualization, so as to clearly demarcate the real part of the incoming energy devoted to a given state transition. To this end, a specific HFM was developed in the laboratory and implemented within two cells most different in their design and respectively associated with both indium and gallium melting points assessing. Then, the improved cell-within-cell design was experimented with indium ($t_{\text{melt}} = 156.598 \text{ } ^\circ\text{C}$) while an other investigation was conducted with the gallium ($t_{\text{melt}} = 29.764 \text{ } ^\circ\text{C}$) by way of a classical design also equipped with a HFM. The results presented in this paper address the indium, considering two different ways to control the melting plateau, referred to as steady-melting and pulse-melting schemes. Indeed, as depicted in figure 1, the auxiliary heater may be acted either with a steady supplied voltage (206.33 mW), or with pulsed signals (630.4 mW, 750 seconds in duration). Whatever the material experimented with, thermal disturbances, especially due to density differences between both solid and liquid phases, may be observed as mechanisms altering the quality of the monitored transition plateaus: The HFM implementation allows us to illustrate such phenomena, especially at the end of the processes as the so-called *run-off* key point is observed, several tens of minutes before the *melt-off* point marking a whole liquid state of the material under experiment. As a significant trouble to analyse the melting point temperature, the chaotic character of such mechanism is to be discarded. To this end the geometry of the cell has to be optimized, and a relevant heat flow rate measurement proved to be of help to identify several quality criteria associated with the transition process. Indeed, as regard energy monitoring, the observed results together with modelling [4] allowed us to consider paramount cell structural improvements to be carried out. As a result, the cell-within-cell design was developed so as to enhance the quality of the transition process together with defining optimal measurement policy and control schemes.

4. Heat flow meters (HFMs) specifications and design

As a most invaluable information, the control of the adiabatic condition is made possible by way of implementing a low-noise HFM within the guard encompassing the cell. However, the ratio signal over noise to be optimized stands as the parameter susceptible to put at stake the quality of such a control. Classical HFMs show off sensitivity values averaging $4 \mu\text{V}/(\text{W}/\text{m}^2)$, with electrical internal resistances ranging around 100Ω . Unfortunately the materials making off their structure cannot allow to operate steady measurements with temperatures over 450°C . To overcome such a drawback and try to investigate the EIT-90 materials up to the aluminium ($T_{\text{melt}} = 660.323^\circ\text{C}$), a specific unit, based on machinable ceramics and microtechnologies was developed in the laboratory with the following performances: $S = 10.5 \mu\text{V}/(\text{W}/\text{m}^2)$, $R = 22\,000 \Omega$, and $T_{\text{max}} = 800^\circ\text{C}$. Although HFMs are now classical devices commercialized for many years their use is far from being generalized in the field of control and measurement. Since they behave as self-generating sensors, with no need to supply current, the heat flow rate measurements may only be altered by the Johnson noise (and Flicker noise in a lesser insignificant part) [5]. Then, as relying on the thermoelectric Seebeck effect, the design of effective heat flow meters is based on planar thermopiles, most often fabricated by way of microtechnologies [6,7]. Stability and precision of such devices require heat aging at their operating temperature for several days [8]. Figure 1b illustrates the implementation of such sensors, arranged on the bottom of the inner cell before being inserted within the guard. Considering both ways to drive the auxiliary heater, figures 2a&b exhibit the recorded temperatures and relevant heat flow rates. It must be noted that in both cases the HFMs are arranged in such a way that their output signal is positive as heat is flowing from the cell down to the encompassing guard.

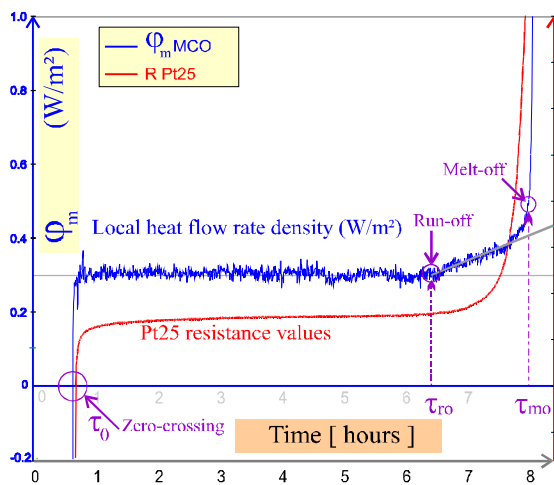


Figure 2a: Steady-melting scheme

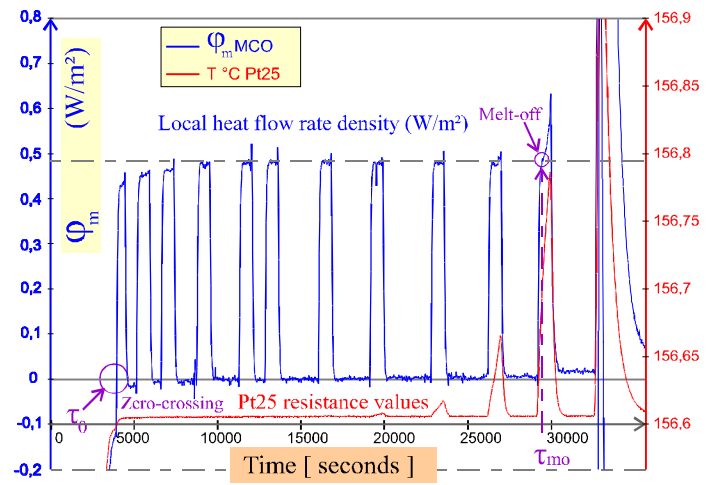


Figure 2b: Pulse-melting scheme

As a result, the problem to be considered is that of the meaning of the unusual information provided from the HFM, while taking account of its specific implementation within the cell experimented with. Moreover as the energy supplied with the auxiliary heater is precisely measured the system configuration allows us to consider a relevant *in situ* calibration.

5. Heat flow meters and *in situ* calibration

The best implementation of a given HFM should try to be made with a view to avoiding any kind of guarded hot plate [8]. The ability to relate the obtained signals with the supplied energy, together with the knowledge of the mass of indium ($M_{\text{in}} = 115.96 \text{ g}$) and its melting enthalpy ($L = 28\,576 \text{ J}/\text{Kg}$, $H_{\text{melt}} = 3313.7 \text{ J}$) yield an effective energy balance. As we observed that the HFM provides a signal related to the heat leaking to the guard without participating to the melting process, the following rationale may be considered whatever the melting scheme. Referring to f_m the heat flow rate density sensed across the HFM, and P_h the dissipated energy into the auxiliary heater we have

$$\left[? W_0 = \int_{t_0}^{t_{mo}} P_h(t) \cdot dt \right] = \left[Q_g = \sigma \cdot \int_{t_0}^{t_{mo}} \Phi_m(t) \cdot dt \right]_{t_0}^{t_{mo}} + \left[? H_{melt} = \int_{t_0}^{t_{mo}} \Phi_{in}(t) \cdot dt \right]_{t_0}^{t_{mo}} \quad (1)$$

with Q_g the known difference between the whole dissipated energy $? W_0$ and the melting enthalpy. Then, the quantity σ (m^2) stands as an apparatus constant, defined as the ratio Q_g over the estimated integral related to the prior raw calibration of the HFM. Then, as P_h is a constant, such as equation 1 yields

$$\Phi_{in}(t) = P_h - \sigma \cdot \Phi_m(t) \quad [\text{with } \Phi_{in} \text{ in } W, \sigma \text{ in } m^2] \quad (2)$$

Consequently, the energy balance may be performed either instantaneously (as in equation 2) or integrated along the melting process (according to Eq.1 with the running time instead of the end of the process [melt-off]). For instance, the incoming energy really distributed within the indium, processed according to the pulse-melting scheme (figure 2b), may be determined as depicted in figure 3.

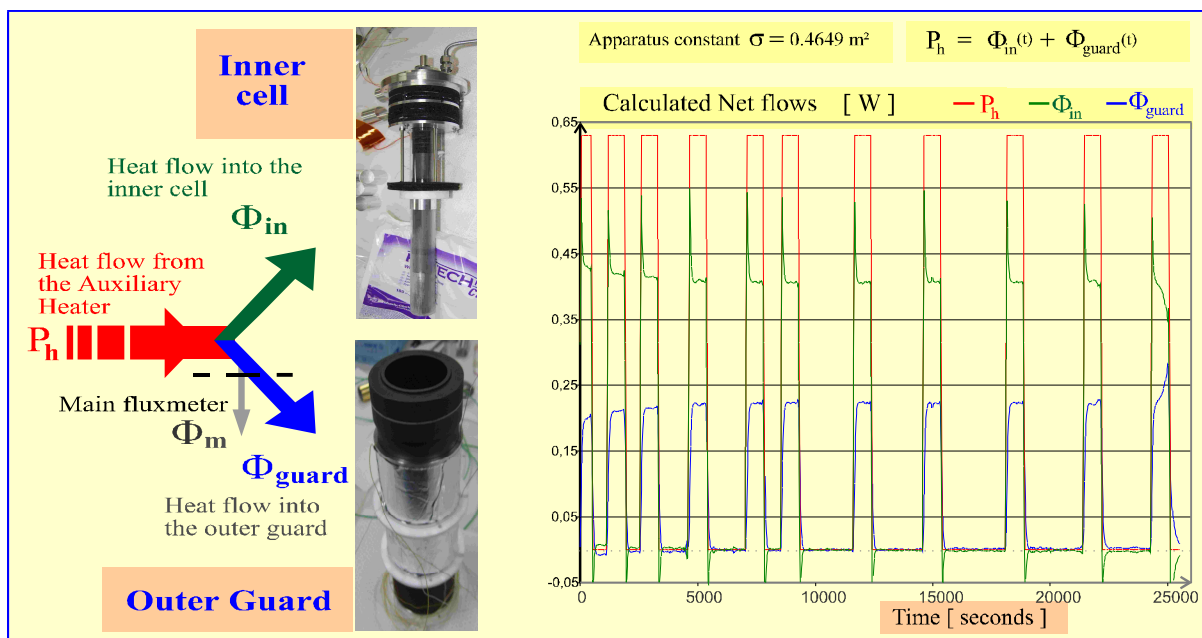


Figure 3: Pulse-melting scheme Energy balance within the indium cell

As a most important result, one can clearly see on figure 3 that the heat flow rate incoming within the indium is not an invariant value, contrary to what is commonly admitted by the scientific community as regards a given transition plateau. Indeed, both beginning and end of the process can be identified and are marked with a significant decay of the incoming flow.

6. Specifications for sharpening the adiabatic condition control

Considering a unit signal-noise ratio, the lower heat flow rate value to be considered may be easily estimated. Indeed, the aforementioned parameters yield $V_{Noise-RMS} = 20 \text{ nV} \cdot \text{Hz}^{1/2}$. On the other hand, considering an operative frequency bandwidth limited to 100 Hz, an ideal resolution close to $df_{MCO} \approx 20 \text{ mW/m}^2$ could be expected, with a tenfold lower value in case of a unity bandwidth. The output signal can be monitored with a simple electronic low-drift chopper-stabilized amplifier. A classical TLC 2652, operated with a Gain = 50, and whose residual offset was measured and compensated for by way of software, proved to allow best results.

Conclusion

Both contrasting ways (pulses or steady state) to drive the auxiliary heater yield a behaviour of the cell-within-cell such as concomitant measurements of temperature and heat flow illustrate the ability to localize the boundaries of the plateau so as to consider an effective control the transition process. Indeed, classical control rules (Feedback with T, and feedforward with f [9]) should hold, considering

one or several heat flow rate measurements judiciously arranged around the inner cell. Then, forcing back the practical limits of adiabatic control could prove to be a most powerful challenge to enhancing the thermometric measurement policy as regard the International Temperature Scale, ITS-90 [10]. Moreover, considering thermodynamic implications [11], it is expected that in the near future heat flow controlled thermometric fixed-point cells will be operational for either metrological or industrial purposes

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