5
Experimental methods

5.1. Design and construction of the cells

The initial design of the eutectic cells followed closely the original design from NPL, UK [15, 27]. The only slight modification to that design was in the internal dimensions, due to the decision not to use purified carbon cloth material (C/C sheets) between the cell crucible and sleeve to only have the graphite sacrificial sleeve [39, 40]. The C/C sheet is generally added to improve the temperature uniformity around the fixed point ingot; this is possible because of the asymmetry in the thermal conductivity of the C/C sheet. However the primary concern for these cells is robustness and according to the experience of other researchers the addition of the C/C sheet did not improve the robustness of the cell therefore for these studies it was omitted. In Figure 10, it is possible to see the main dimensions of the cell.

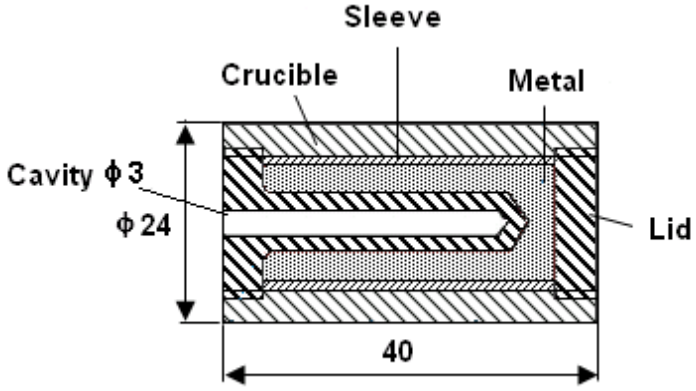

Figure 10: Blackbody cell design used in this study – dimensions in millimeters

The emissivity of the blackbody cavity, calculated using the model developed by Bedford and Ma [41] and Ballico [42], considering isothermal conditions (reasonable when the fixed point is melting or freezing) and 3 mm diameter, 28 mm length and a 120° conical end, was found to be 0,9997 ± 0,0001, with the assumption of the emissivity of the graphite 0,87. The internal volume of

the cell available to be filled was determined to be 4,5428 cm^3, obtained by filling it with pure water and measuring its weight. All the graphite parts used the same specification graphite (1940PT – 10 ppm ash content) from Mersen do Brasil, which was also responsible for the machining of the crucible components.

Before filling the crucibles, all the graphite parts were baked at Inmetro in a vertical furnace, in vacuum for at least 45 minutes at 1850 °C, in order to remove any remaining impurities introduced during the manufacture of the graphite components. The main properties [43] of this graphite grade are shown on table 8.

Table 8: Thermophysical properties of 1940PT graphite

Property	**Quantity**	**Value**
Density	g/cm^3	1,80
Porosity	% vol	12
Flexural Strength	MPa	50
Compressive Strength	MPa	100
Shore Hardness	C2	55
Coefficient of Thermal Expansion	10^{-6}/°C	3,5
Thermal Conductivity	W/m °C	90
Electrical Resistivity	μΩ cm	1500

During the purification step, besides the crucible components, all the other graphite parts of the furnace (heating element, insulations, etc.) were also purified to eliminate the possibility of cross contamination. The furnace [44] used for the purification step was manufactured by Thermal Technologies Inc., and has a cylindrical heat chamber with 150 mm internal diameter and 255 mm of height, which can operate in an inert atmosphere or vacuum in the temperature range from 400 °C up to 2200 °C. It is equipped with a programmable electronic temperature controller from Eurotherm, model 2404, using a type C thermocouple as the temperature sensor. It is also equipped with a cut-off temperature controller connected to a second type C thermocouple.

Figure 11: Parts of the eutectic cell

In figure 11 it is possible to see the graphite parts of the eutectic cell. From the right to the left there is the cap, blackbody cavity, crucible, liner and the hopper that is used during the filling step.

Figure 12: Typical eutectic cell

In figure 12, an assembled eutectic cell is shown with a scale to display its real size.

In figure 13 it is shown a view of the Thermal Technologies furnace, where the graphite parts were purified and the eutectic cells subsequently filled.

Figure 13: Thermal Technologies Furnace

5.2. Choosing of the dopant for the eutectic cell

The first cell constructed and measured was the Co-C eutectic. In order to select an adequate element to act as dopant in the Co-C alloy, the position of cobalt in the periodic table was taken as a reference. The objective was to find an element able to modify the melting temperature of the eutectic cobalt-carbon (1324 °C) without adding too much material. The first selection criterion was the neighbors to Co in the periodic table; the best candidates were elements like nickel, copper and palladium. Ni was discarded as a dopant because the melting temperatures of Co-C and Ni-C are very similar and so a lot of Ni would need to be added to the fixed point to make an appreciable change to its temperature. Other important factors considered were:

- the crystal structure of the dopant, which should match that of cobalt to avoid inducing dislocations in the lattice

- the atomic radius of the dopant was another important parameter, because a much bigger atom than cobalt would induce stress in the lattice

Both of these were important to avoid affecting negatively the melting and/or freezing plateaus.

Other factors to consider in the selection of the dopant were the availability in high purities, reasonable prices, reactivity and level of toxicity.

Taking into account all this parameters, it is possible to focus on a small number of dopant candidates and copper was considered an adequate doping element, because it is available in high purities (purity level of 99,9999%) at relatively low prices, its melting temperature is 300 °C lower than the cobalt's, which decreases the amount of material to be used and its phase diagram with carbon is simple. Rhenium could also have been chosen, but it is five times more expensive than copper.

A second family of eutectic cells was also constructed. For this, nickel was chosen as the base metal for the eutectic fixed point. This element has almost identical properties found in cobalt, even the carbon-eutectic has only a slightly higher melting temperature (1329 °C versus 1324 °C). Copper was also found to be a suitable dopant element for nickel. Another dopant, tin, was chosen for Ni-C. This was selected because it has a very different melting temperature (~232 °C) compared to Ni-C and so possibly affects the melting and freezing plateau of the Ni-C eutectic in a different way. A set of five Ni-C eutectic cells were constructed, which are a Ni-C reference cell, two Ni-C-Cu doped cells at two different dopant concentrations and two Ni-C-Sn, also doped at two different concentrations. The dopants were added in the quantity as indicated by the modeling so as to test its efficacy.

The preparation of the eutectic cells used materials in powder form, cobalt, nickel and graphite, from Alfa Aesar with a purity of, respectively, 99,998 %, 99,996% and 99,9999 %. The copper and tin used as dopants were in the form of shot, also from Alfa Aesar with a purity of 99,9999 %. The components in powder form were mixed close to the eutectic composition, but in the metal rich region of the phase diagram. This was decided in order to avoid the formation of a highly viscous alloy, which makes the casting of a high quality ingot much more difficult. This problem tends to be present when using carbon rich mixtures. The

remaining carbon required to attain the eutectic composition was taken from the crucible walls.

The dopants were added at the last filling step, which permitted a more precise determination of the molar concentration of the dopant in the metal-carbon eutectic matrix.

5.3. Melting point determination

Since the first work published by Yoshiro Yamada of NMIJ [19] in 1999, it has been common practice to use the melting point temperature of a eutectic alloy as its reference point. According to an extensive study by Lowe and Machin [45], it was demonstrated that, the determination of the point of inflection of the melting curve of the metal-carbon eutectic against time, was best effected by fitting a third degree polynomial to the measured data, differentiating it twice with respect to time and finding the temperature at the minimum.

For the determination of the point of inflection of the melting-curve of the M-C eutectic, and hence its temperature, in this thesis an automatic and specific procedure was developed using the Gnuplot graphical environment and Scilab programming language.

The method used to find the point of inflection was based on the least squares fit to the photocurrent versus time melting plot. A third-order polynomial $Y = a.x^3 + b.x^2 + c.x + d$ was fitted to the melting curve. This was differentiated twice and the resulting function evaluated at $d^2Y/dx^2 = 0$ to determine the time at $x = -b / 3.a$. This value was then substituted into the fitted polynomial to get the corresponding inflection point photocurrent. Typically, the fitted range was of the order of 20 minutes, being the point of inflection situated close to the half of the interval.

The fitted polynomial can be seen in figure 14 as a continuous line and the photocurrent of the inflection point is also indicated by the horizontal line in the graph. This exact value of time and photocurrent are enclosed in the rectangle.

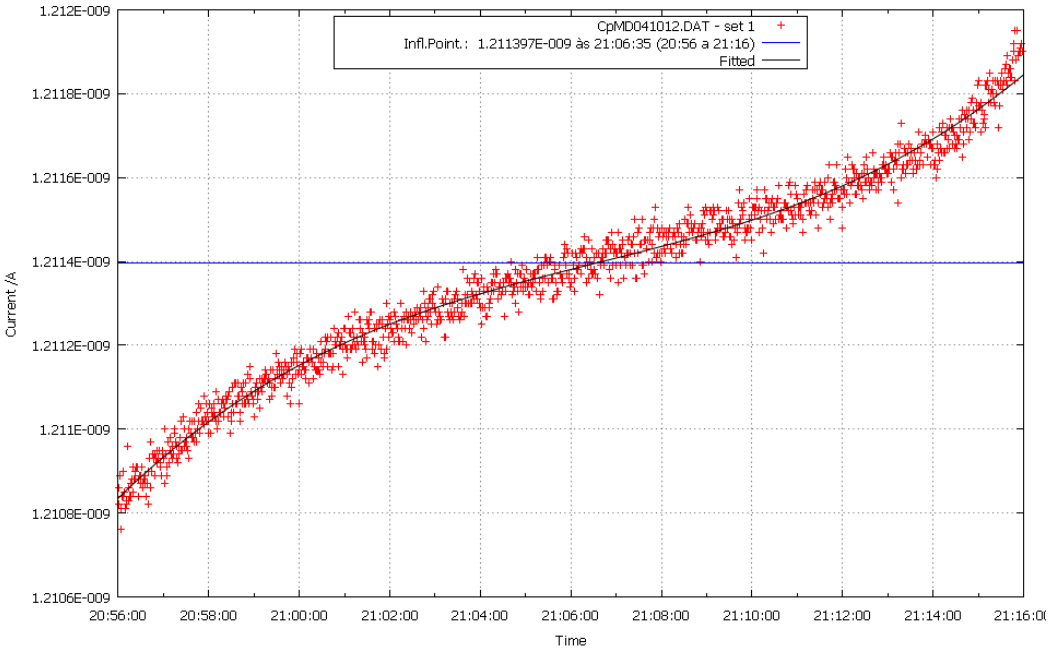

Figure 14 - Example of the determination of the point of inflection of the melting curve of the Ni-C eutectic.

5.4. Preliminary measurements with Co-C system

The first measurements made were of a cobalt-carbon eutectic cell realized in the Thermogauge HT9500 furnace [46]. This furnace is capable of reaching 3000 °C in 500 s, using a graphite tube of 38 mm diameter as a heater and 48kW of electrical power. Though very fast in heating and cooling, this furnace does not present an adequate temperature distribution along its heating tube. A second cobalt-carbon cell doped with 700 ppm was also realized in this same furnace. Both cells experienced a small number of cycles and the results [47] of these melting temperatures can be found on figure 15. During this phase a change in the melting temperature of the cells was observed in the last cycle. After checking the integrity of the blackbody cavities, it was found that both cells were broken because of the far from the ideal thermal and control conditions of the Thermogauge furnace. Both the blackbody cavities were broken and tilted, which could explain the step change in the melting temperature of the cells perceived by the monitor pyrometer the LP3, described in section 3.3.

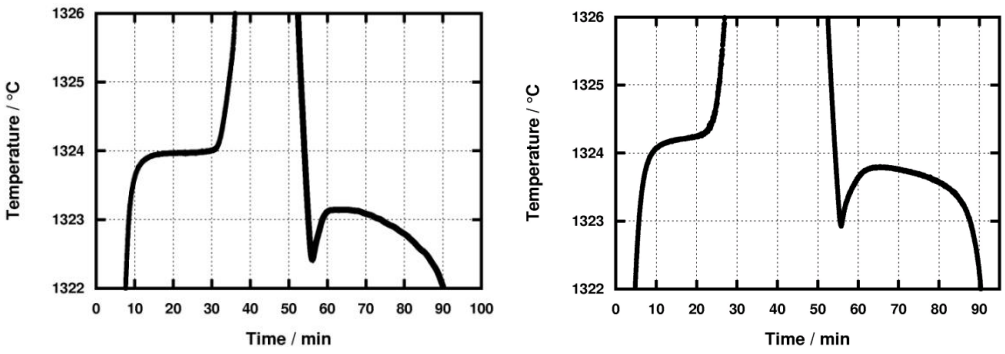

Figure 15: Realization of Co-C (left) and Co-C-Cu (right) eutectics

The Thermogauge HT9500 furnace proved to be inadequate for the realization of the eutectic fixed-points, due to its extremely fast heating and poor temperature uniformity, which it is felt contributed to the breakage of the Co-C and Co-C-Cu blackbody cavities.

During the development of this work, it was found that the original design of the cells from NPL had possibly a problem, because of the successive breakages of the blackbody cavities occurred during their realization in the Thermogauge furnace. Analyzing the design of the blackbody, it is possible to observe a huge stress concentrator caused by the right angle between the tube of the blackbody cavity and the front plate of the blackbody cavity. This characteristic can increase the stress in that area 5 to 10 times, or even more, which can break the cavity. In order to avoid this possibility of failure, a slight modification to the original design was made, where a 3 mm radius was added between the two parts. This modification should significantly reduce the stress in that area and hence improve the robustness of the cells. The old and new cavity designs are given in figure 16.

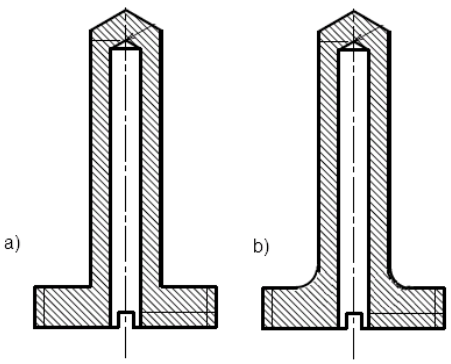

Figure 16: Blackbody cavities – (a) Old model (b) New model

The other modification to the measurement set up that was used was a different furnace. This was a three zone tube furnace, which has much better temperature uniformity and better temperature control than the Thermogauge furnace. It was also capable of performing ramps and dwells in a more controlled way. The furnace used was the Carbolite TZF 18/75/600 furnace [48], detailed in 5.7.1.

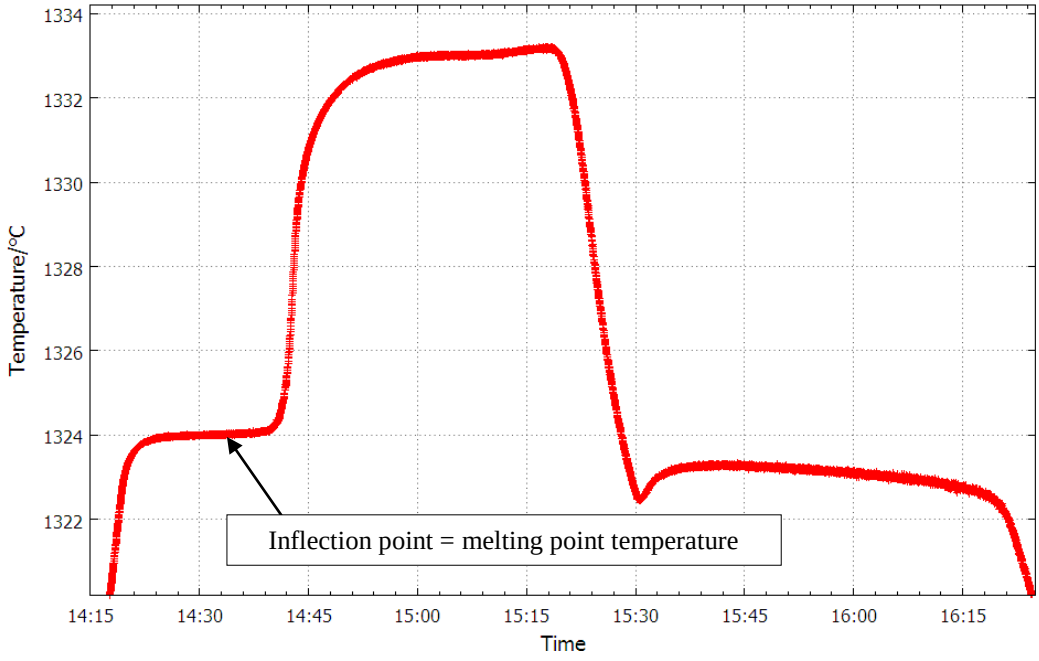

Figure 17: Typical realization curve of the Co-C eutectic in the carbolite 3-zone furnace

In figure 17 it is easy to see the melting and freezing features of the Co-C eutectic when realized in the Carbolite three-zone furnace. There is a relatively flat melting plateau, with a temperature assigned of 1323,88 °C, which is in agreement with previous results found in the literature for this alloy [49, 50]. The recalescence (supercool) is clearly seen in this plot. The first measurements performed in the Thermogauge furnace did not show any recalescence at all, due to the poor temperature uniformity along the eutectic cell.

Even with the use of the three-zone furnace, still more problems happened, causing the breakage of more two Co-C cells. In the literature it appears that cells made of Co-C eutectic are prone to breakage [40], this knowledge motivated a change in the eutectic alloy. In order to make use of the same construction and measuring process, it was decided to use nickel. This is because the metal has

basically the same characteristics as cobalt, as it can be seen in table 9, with only a slightly higher eutectic melting temperature (1329 °C instead of 1324 °C) and it is available at the similar purity and cost. It is interesting to note that the crystal structure of cobalt is hexagonal close packed (hcp) at ambient temperature, but at 421 °C there is a phase change to cubic face centered (cfc).

Table 9: Physical properties of Co, Ni and selected dopants

Element	Atomic radius (pm)	Crystal Structure	Melting temperature (°C)
Co	152	hcp cfc	<421 1495
Ni	149	cfc	1455
Cu	145	cfc	1084,62
Sn	145	tetragonal	231,928

According to the studies of Sasajima *et al.* [51], the melting plateau shape for the Ni-C system is not significantly affected by the melting rate of the previous freeze. However, the authors are clear that the furnace temperature uniformity and precise control of the furnace temperature do improve the plateau shape and the repeatability of the point of inflection. The furnace used in this study will facilitate this having very good uniformity and fine control of the set point. Measurements of the Ni-C eutectic fixed points are described in a subsequent chapter.

5.5. Definition of transition temperature in M-C fixed-points

In the vast majority of published works on eutectics, their melting temperature has been taken as the point of inflection of the temperature versus time melt plot, as this has been found to be highly reproducible (see Section 5.3). The melt temperature is preferred to the freezing point, because the freeze temperature of a eutectic alloy is known to be rate dependent – with undercooling at the solid-liquid interface resulting from the need for the homogeneous liquid to separate into two solids [7].

Metal-carbon (M-C) melt plots are typically found to be curved with melting taking place over a temperature range rather than a fixed-value. In some cases, like Fe-C, this has been shown to depend on the microstructure of the sample introduced on the previous freezing [52]. This effect becomes less significant at higher temperatures. Lowe *et al* [53] observed a lowering by 40 mK in Co-C, compared to the 200 mK found in Fe-C; no effect was detected in the Pd-C.

In the ideal case, the point of inflection of a M-C fixed-point will be the eutectic liquidus temperature. In practice, furnace gradients will cause the measured temperature to start to rise while there is still solid present [54].

It is clear from this that the measured point of inflection will be a lower limit on the eutectic liquidus temperature. The more uniform the furnace conditions are, the closer to the eutectic liquidus temperature the point of inflection will be [23].

It would be expected that poor filled fixed-points, which for example can lead to ingots with voids, would be more sensitive to non-isothermal furnace conditions. Cells with better formed ingots will tend to have a higher temperature point of inflection, flatter melt curves and show a sharper rise at the end of the melt [7].

5.6. Filling of high temperature fixed points

The filling of the eutectic fixed-points was carried out separately from any other operation, and care was taken to avoid cross-contamination between different materials. Disposable items were used where possible and all items were cleaned with ethanol. Plastic wrap was used to avoid contact with any surface.

The complete filling of the eutectic cells took four to six cycles. The hopper was attached to cell crucible and completely filled with metal-carbon powder mixture. This assembly was inserted in the Thermal Technologies furnace to melt the mixture. This cycle was repeated until the crucible was considered sufficiently full. At this point the hopper was removed and the crucible end cap was screwed into place and the fixed point cell was complete. No problems were detected with the empty of the hopper; the molten alloy always flowed to the cell crucible. Typically the furnace was set to heat the cell assembly from ambient temperature,

in an argon atmosphere, until 1300 °C at 10 °C/min heating rate. It was left at this temperature for 20 minutes to uniform the temperature and then set to 1350 °C at 5 °C/min. The complete heating and cooling program can be seen in figure 18. This same procedure was applied to every cell used in this study.

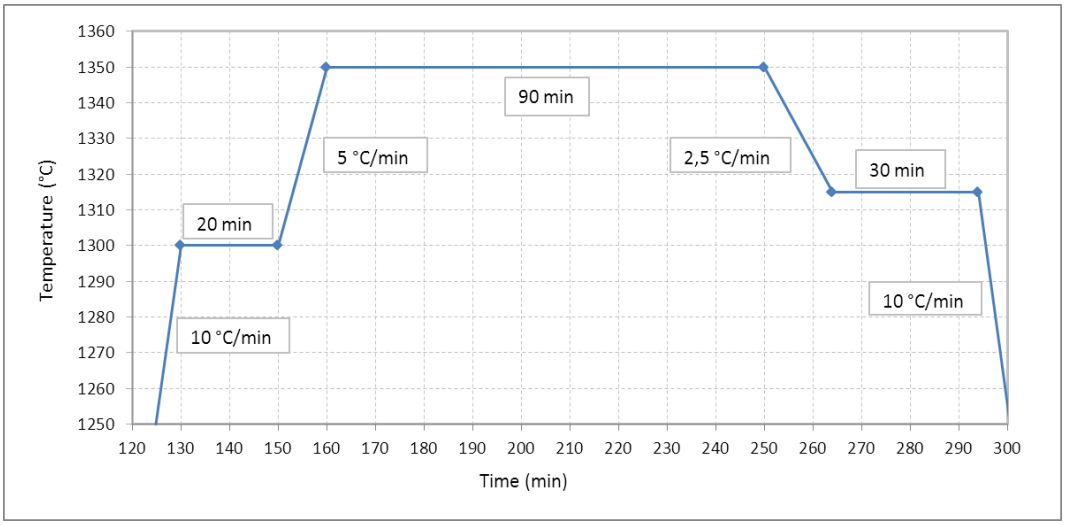

Figure 18: Heating and cooling program for Thermal Technologies furnace

Initially an undoped metal-carbon (Co-C or Ni-C) cell was made to act as a reference so as to determine the change in the realization temperature (and plateau quality) in the doped cell. To avoid confounding impurities in the base material the same lot of Co and Ni was used to construct all the fixed points.

Once the undoped cell was prepared the doped cells were then constructed. These were prepared in the same way as the undoped cell with the dopant added at the last filling step. These cells had a mass of 34,4 – 34,8 g of eutectic alloy, which corresponds to a filling of 91% of the available volume. This was a deliberate choice, leaving some free space for thermal expansion of the solid phase during heating – and particularly for expansion on melting.

5.7. High temperature equipment used for this research

All the measurements reported in this thesis were performed at the Pyrometry Laboratory of the Thermal Metrology of Inmetro. Figure 19 shows the equipment used in this research. From the left to the right the equipment is the Thermogauge furnace, the Carbolite TZF 12 furnace for the realization of the silver point, the Carbolite TZF 18 used for almost all the realizations of the eutectic points, the standard pyrometer LP3 directly in front of the TZF 18 furnace

and the rails which allow easy moving of the pyrometer in front of any of the thermal radiation sources.

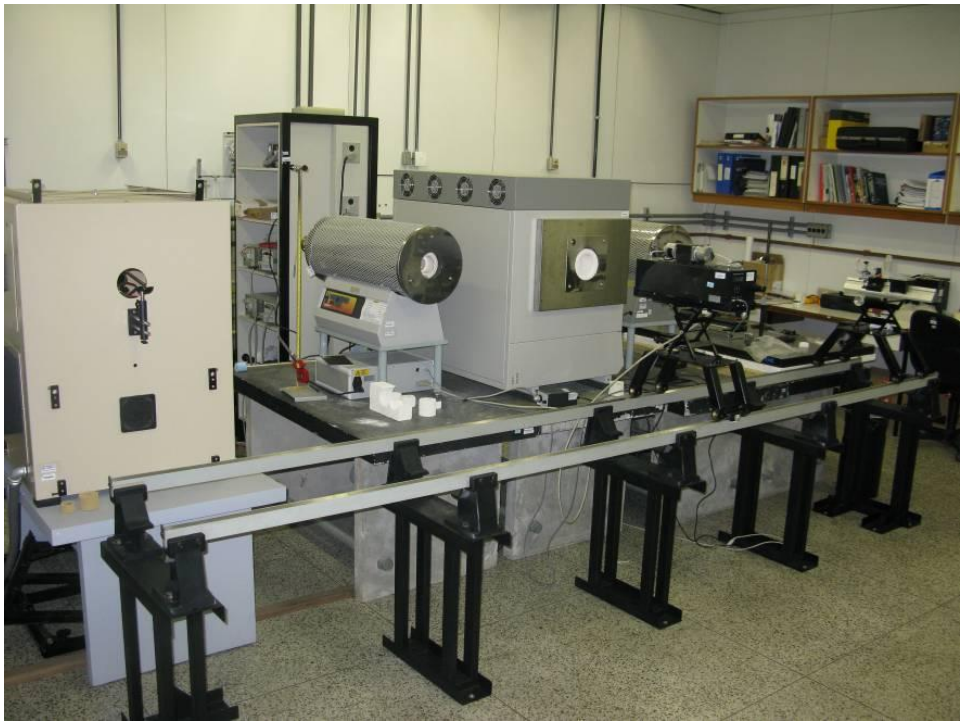

Figure 19: Measurement setup at Pyrometry Laboratory

The furnaces were positioned perpendicular to the optical axis of the pyrometer, with the target located at the same distance and at the same height, in order to define the same viewing angle. The focusing of the LP3 pyrometer was made for one cell and locked in that position for all the measurements. Eventually necessary adjustments were made by displacing the eutectic cell to the focus distance of the pyrometer. The blackbody cavities of the silver fixed point cell and the eutectic cells have the same aperture diameter, so as to be viewing the same target. This minimized any errors that could have arisen because of scattered light due to the size-of-source effect [55 – 57] of the pyrometer.

To correct for any pyrometer drifts a measurement of the reference photocurrent of the LP3 was performed at the freezing point of silver either on the previous or the following day of the measurements of the eutectic cells.

All the measurements made using the LP3 pyrometer were performed using the data acquisition software provided by the manufacturer, which reads via the digital RS232 serial interface and subsequently saves the following parameters

every second,: date, time, position of the interference filter wheel, position of the neutral density filter wheel, range, actual photocurrent, actual temperature in K, actual temperature in °C, mean value of the last ten photocurrent measurements, mean value of the last ten temperature measurements in K and actual detector internal temperature.

At every measurement made using the LP3 pyrometer, the dark current of the photodetector was measured prior to the beginning of the fixed point or eutectic temperature.

The LP3 pyrometer is kept always on, in order to avoid inevitable condensation of humidity in the electronic and optical parts of the instrument, which could happen in spite of the control of temperature and humidity in the Pyrometry Laboratory.

Below is a detailed description of the key equipment used for the construction, realization and measurement of the eutectic melting points.

5.8. Experimental setup

In this section it will be described the equipment used in the weighing of the metals used in the fixed points, the furnaces used for the reference Ag cell and the eutectic cells and the silver point cell. The linear pyrometer LP3 was already described in chapter 3.

5.8.1. Weighing balance Sartorius ME235S

In order to measure the masses of each of the components for the eutectic cells a weighing balance manufactured by Sartorius, model ME235S was used. This is shown in figure 20 it has 230 g of capacity and resolution of 0,01 mg [58]. This equipment was also used for weighing the dopants, making possible the calculation of their molar concentration.

This equipment was calibrated by the Mass Laboratory of the Mechanical Metrology Division of Inmetro, with its calibration certificate identification of DIMCI 0266/2011. The uncertainty of this balance in the range up to 100 mg was 0,01 mg and in the range from 30 g to 70 g was 0,05 mg, at 95,45% confidence level (k=2)

Figure 20: Sartorius ME235S Balance

5.8.2. Carbolite furnace model TZF 18/75/600

The furnace used for the realization of the metal-carbon eutectic cells described in this work was a three heating zone tube furnace, equipped with ten U-shaped Kanthal heaters [48]. The equipment has a programmable temperature controller, allowing an independent temperature setting for each zone, in order to achieve an optimum temperature distribution throughout the working space. It was manufactured by Carbolite (UK), model TZF 18/75/600 and its temperature range goes from 400 °C up to 1800 °C, using a 75 mm internal diameter alumina working tube. The main temperature controller was a Eurotherm model 2416, which can be programmed with up to eight steps, which can be ramps, dwells and/or whole cycles. For these measurements the heating rate was limited to 5 °C/min to protect the cells from thermal shock. This controller is connected with two additional temperature controllers, Eurotherm model 2216e, which performed the temperature control of the auxiliary zones. This strategy facilitates the realization of a central heated zone with the best possible temperature uniformity which is important for the realization of the metal-carbon eutectic cells. The three temperature sensors used in this furnace are Pt/Pt-Rh thermocouples, which are stable enough for the application. A detailed heating and cooling program of the temperature controller of the Carbolite TZF18 furnace used in the realization cycles of the (Ni-C) eutectic cells can be seen in figure 21.

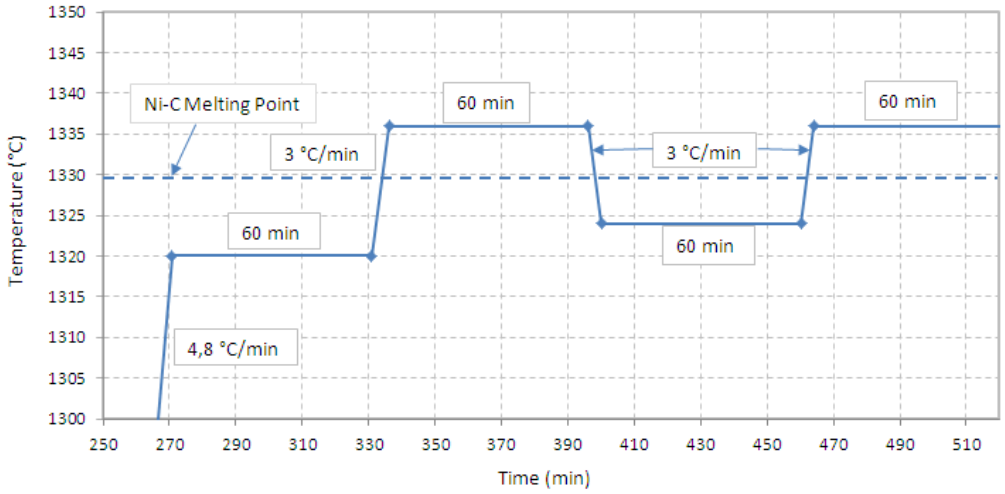

Figure 21: Heating/cooling program of the Carbolite TZF18 furnace

In figure 22 it is shown the Carbolite TZF18 tube furnace and the setup of the eutectic cell inside the furnace tube. The eutectic cell is protected by a graphite tube, surrounded by a graphite structure which allows the flow of argon around the cell. This assembly is located inside an alumina tube, which was inserted inside the furnace tube. A cylindrical refractory insulation piece with a conical opening enhances the temperature uniformity of the furnace.

Figure 22: Assembly of the eutectic cell inside Carbolite TZF 18 furnace

5.8.3. Carbolite furnace model TZF 12/75/600

This equipment is dedicated to the realization of the freezing point of silver (961,78 °C) the reference point of the ITS-90. It is a tubular furnace manufactured by Carbolite, model TZF 12/75/600, with three independent heating zones controlled by a programmable Eurotherm main temperature controller model 3216 and two Eurotherm model 2132 auxiliary temperature controllers [59]. The equipment uses type N thermocouples as temperature sensors and was fine tuned to achieve the best possible temperature distribution along the fixed point cell. The fixed point cell is installed inside an alumina tube of 53mm internal diameter supported by graphite holders, where it is purged with 99,999 % pure argon, in order to prevent any oxidation of the graphite parts. The flow of argon was adjusted to 0,2 to 0,3 l/min. The whole assembly is inserted inside the furnace

tube, which is 75 mm in internal diameter. Figure 23 shows a schematic view of the cell inside the furnace tube.

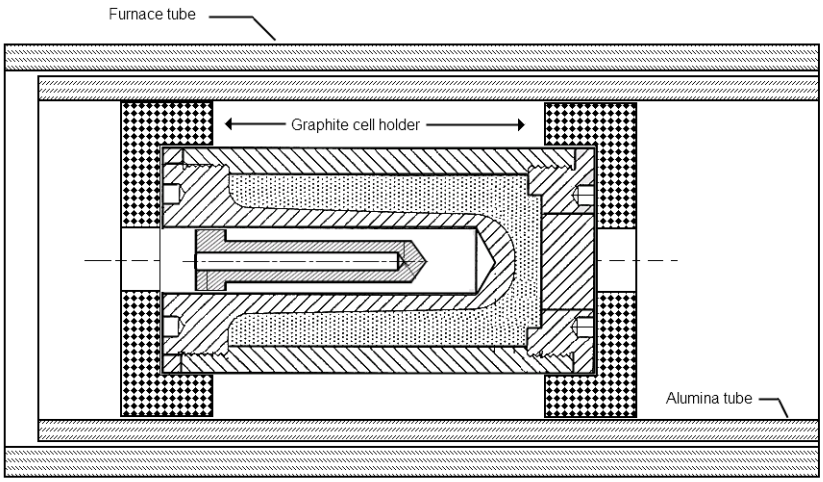

Figure 23: Assembly of the silver cell inside the furnace

5.8.4. Silver fixed point cell

The radiometric reference for all the measurements performed using the LP3 radiation thermometer was a silver fixed point blackbody cell which holds approximately 250 g of silver of 99,9999% purity. This fixed point was manufactured by Isotech, UK. This cell was calibrated by the Thermometry Laboratory of NPL (UK), with the certificate number PM06/BN99/004, with a stated uncertainty of 0,05 °C (k=1).

The original design of this cell can be seen in figure 24. The blackbody cavity of silver point blackbody has an internal diameter of 10 mm, which is much bigger than the diameter of the blackbody cavity of the eutectic cells. So, in order to minimize the effect of the size-of-source effect the silver point was modified by introducing a blackbody of the same design as the high temperature fixed point blackbody into the silver point cell, thus approximately equalizing the viewing conditions. This modification can be seen in figure 24.

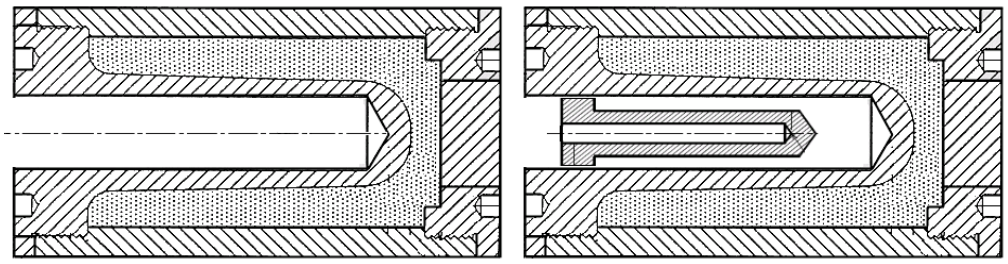

Figure 24: Original (left) and modified (right) silver blackbody cavity

5.9. Data reduction in the temperature determination

This section will describe the procedure used in the determination of the temperature from the readings of the photocurrent from the Si photodiode detector of the linear pyrometer LP3.

Firstly, with the objective lenses of the LP3 covered, the dark current was measured; this corresponds to the electronic noise of the measurement system. This value will be named $I_{ph,0}$. After the identification of the point of inflection of the photocurrent versus time curve, which corresponds to the melting temperature of the metal-carbon eutectic cells the value $I_{ph,t'}$ was obtained. This is the photoelectric current of the LP3, related to the incident thermal radiation on the detector.

$$I_{ph,t} = I_{ph,t'} - I_{ph,0} \tag{24}$$

The value in Equation (24) represents the net photocurrent of the melting temperature of the eutectic cell. In order to attribute a temperature to this value $I_{ph,t}$, it was necessary to have a ITS-90 reference point to perform the calculation, as cited in section 2.2. The reference available at the Pyrometry Laboratory was the silver freezing point (961,78 °C), which as described in a previous section was originally calibrated by NPL. Immediately after (or before) each of the cycles of measurements performed with the eutectic cells, the photocurrent of the freezing point of silver $I_{ph,Ag}$, was measured in a similar way to the measurement of the eutectic cell.

$$I_{ph,Ag} = I_{ph,Ag'} - I_{ph,Ag0} \tag{25}$$

Where $I_{ph,Ag0}$ is the dark current of the measurement performed at the silver freezing point and $I_{ph,Ag}$ is the current related to the temperature of the first fifteen minutes, in the most flat part of the freezing plateau just after the recovery from the recalescence (marked by rise out of the supercool). In figure 25 it is possible to see a typical freezing plateau of the freezing point of silver.

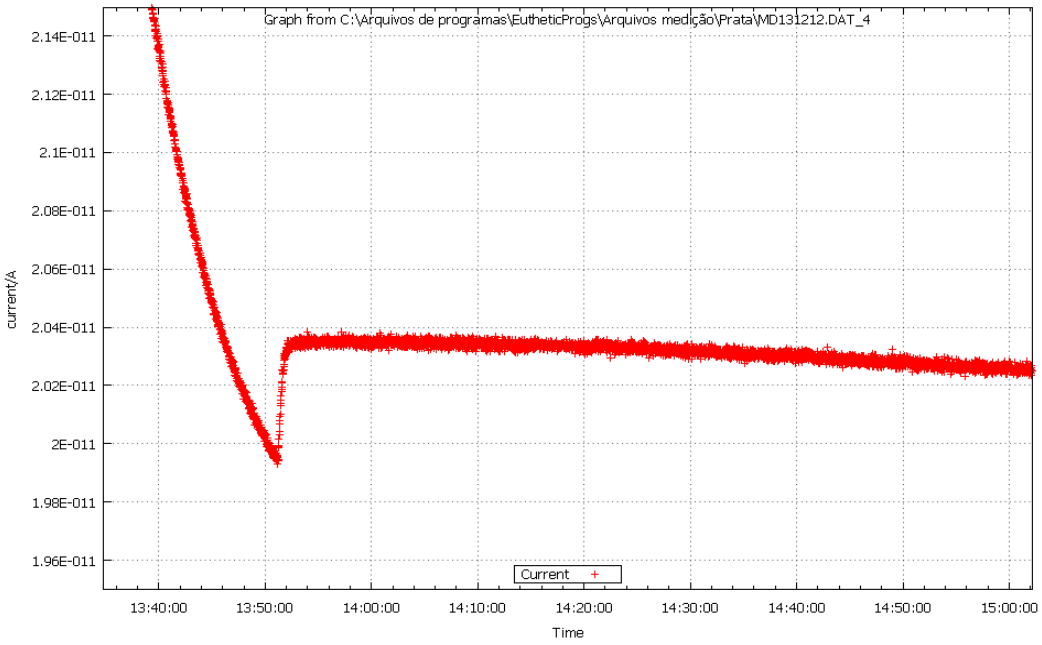

Figure 25: Typical freezing plateau of the silver fixed point cell

The software Scilab/Gnuplot developed for this application takes this data automatically and calculates its mean value and the standard deviation of the mean, which can be seen in figure 26.

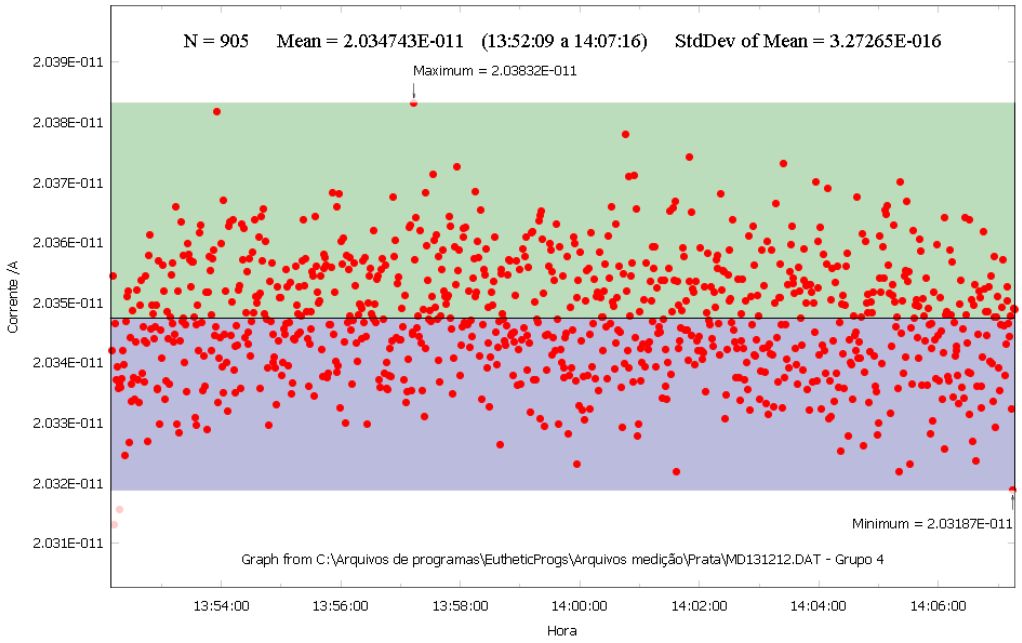

Figure 26: Measurement data as processed by the Scilab/Gnuplot software

With all these data i.e. that of the melting point of the eutectic alloy and the freezing point of silver, the signal ratio, *R* is determined:

$$R = \frac{I_{ph,t}}{I_{ph,Ag}} \tag{26}$$

Now, with ratio *R* and spectral information of the interference filter *and* radiation detector of the LP3 pyrometer, it is possible to calculate the temperature of the melting point of the eutectic alloy using the Planck radiation equation, in its integral form. The spectral information of the LP3 pyrometer was determined prior to this work and detailed information can be obtained in [29]. The temperature can then be finally obtained by solving the integral equation (27), which can be performed numerically.

$$\int_{\lambda_a}^{\lambda_b} L_\lambda(T_{90}) \cdot \tau(\lambda) \cdot V(\lambda) \cdot d\lambda = R \cdot \int_{\lambda_a}^{\lambda_b} L_\lambda(T_{Ag}) \cdot \tau(\lambda) \cdot V(\lambda) \cdot d\lambda \tag{27}$$

All variables in the equation are known with the exception of T_{90}.

5.10. Summary of the chapter

In this chapter the design and details of the construction of the high temperature fixed point eutectic cell were presented. It was also determined the criteria for choosing adequate dopants for the eutectic cells. Preliminary results of the realization temperature of Co-C and Co-C-Cu eutectic cells were also shown, which may indicate the behavior of the Ni-C based cells to be presented in the next chapters. Details of the equipment used in the filling and realization of the cells were detailed as well as the procedure to identify and calculate the melting point temperature of the eutectic cells.