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Conclusions and suggestions for future work

Artefacts for interlaboratory comparisons using doped metal-carbon eutectic cells were developed. Eutectic cells based on the Ni-C eutectic alloy doped with two different elements at two different concentrations were constructed and measured. Most of the constructed cells accumulated more than 50 hours in excess of 1300 °C, during more than 15 cycles melting-freezing. The cells demonstrated good stability and repeatability. These properties demonstrate that such an approach to developing key comparison artefacts is feasible.

Two of the cells constructed for this thesis were used in a blind comparison with the Thermometry Laboratory of NPL (UK) that used their own methods and equipment to realize and determine the melting temperature of the cells. The cells were sent to UK by normal courier service, and in no moment prior to the measurements did this laboratory have any information about the melting temperature of the cells. Despite of that, the comparison demonstrated that both Inmetro and NPL have similar temperature scales, at the melting temperature of the artefacts, with differences of less than 0,1 °C, well within their realization uncertainty.

It is planned to use such artefacts in a wider comparison, with a large number of participant laboratories. The result of this future comparison will supply valuable information to CCT of BIPM in the planning of a future key comparison using eventually doped high temperature fixed points.

During the development of this thesis, three publications were submitted and, are or will be published, in International Symposia such as “ITS9 – 9th Temperature: Its Measure and Control in Science and Industry” and “Tempmeko 2013” (to be finally published in the International Journal of Thermophysics).

As part of this study, it was shown that heating of the interference filters causes drift in the pyrometer. A simple way to avoid this problem was developed which proved to be effective.

With regards the simulation of doping of metal-carbon eutectics, the modeling showed that in general terms simulation of the addition of tin to Ni-C eutectic is verified by the experimental work. However, for the other dopant element, copper, the modeling and the results were poorly matched. Specifically the effect of the addition of this element to the Ni-C was to increase its melting temperature in the simulation, but in the experimental work, lower temperatures than the pure eutectic were found. This result was also achieved by NPL during the interlaboratory comparison, which adds confidence to the measurement data. It is suggested for future studies, the investigation in the simulation data of the copper and/or copper-carbon binary in a search for systematic errors and/or uncertainty of these data. It is also envisaged the metallographic study of the broken cell Ni-C-Cu #7, in order to understand the behavior of the alloy and to propose an adequate model to be incorporated in the database needed for thermochemical simulation. In addition, additional doping studies are required with other elements than Ni in conjunction with simulation to confirm whether simulation is actually a useful tool for selecting the dopant to adjust the temperature. This is particularly important for higher temperature fixed points where the materials are expensive and the experiments difficult.

During the measurements of the Ni-C-Cu #7 in the Thermogauge furnace, it was found that the blackbody cavity was broken. This may have occurred due to the extreme thermal gradients found in that furnace. Two changes are suggested in future measurements using this kind of furnace: an improvement in the temperature control, which can decrease the actual speed of heating and also an improvement of the temperature uniformity by adequately shaping the heating tube or other means, such as in the work reported by Wang *et al*. [66], to be published in the near future.

In order to expand the temperature range in which doped high temperature fixed point cells can be used, it is suggested for future studies an investigation of the doping of metal carbon eutectic cells of higher temperatures, such as palladium-carbon (1492 °C), platinum-carbon (1738 °C) or even rhenium-carbon (2474 °C) be performed. In principle, these are the best candidates, due to their relatively simple phase diagrams they form with carbon. For the metal-carbide-carbon eutectics or metal-carbide-carbon peritectics, it may be more difficult to find adequate dopants without adding too much complexity to their phase diagrams.

In conclusion, this study has shown that empirically there are no barriers to producing doped HTFPs that are robust and stable and that have unknown temperatures. That in turn means that they are suitable candidates for scale comparisons between NMIs. The results with the thermochemical modeling were inconclusive and further investigation needs to be performed to demonstrate whether at these low solute levels modeling is a useful tool in the design of key comparison artefacts.