SIB 52 - THERMO Stakeholder meeting

May 16

Metrology for thermal protection materials

Good Practice Guide on performance checking and Validation of HTGHP

MESURES & RÉFÉRENCES
Clés de la COMPÉTITIVITÉ et d'un MONDE PLUS SÛR

Laboratoire national de métrologie et d'essais

Outline

- Introduction
- GHP technique (High temperature configuration)
- Difficulties to validate HTGHPs
- Uncertainty sources
- Control of the temperature gap imbalance
- Thermal contact resistance - Control of the plates (Flatness)
- Uniformity of temperatures of the plates
- Control of the temperature imbalance of the edge guard
- Correction of the electrical power (junctions correction)
- Electrical isolation of the heating loop, of the thermopile
- Emissivity of the plates
- Recommendations - Conclusions

Introduction

Recommendations and specifications for performance checking of “room temperature” GHPs are given in standards.

Performance checking of HTGHPs is more difficult than performance checking of “RT” GHPs.

Difficulties arise from the configurations of the instruments, the temperature sensors usable at high temperatures, the materials used and the high temperature difference between the samples and the room temperature.

This presentation is not a list of recommendations and procedures for performance checking. It is mainly focussed on some specific tests we have try to applied at LNE for the performance checking of a home-made HTGHP which has been upgraded during the project.

Presentation of the GHP technique

- Measurement of the thermal resistance of a sample with a uniform thickness in steady state conditions → calculation of the equivalent thermal conductivity assuming homogeneity of the material.

  The heat power crossing the “metering section” is measured directly (electrical power supplied to the metering heater).
  Measurement of the temperature gradient: usually by measurement of the temperature of the plates and measurement of the thickness of the sample.

$$\lambda = \frac{\Phi \, . \, d}{A \, . \left(T_H - T_c\right)}$$

Presentation of the GHP technique

- Typical configuration of a HTGHP (double specimen).

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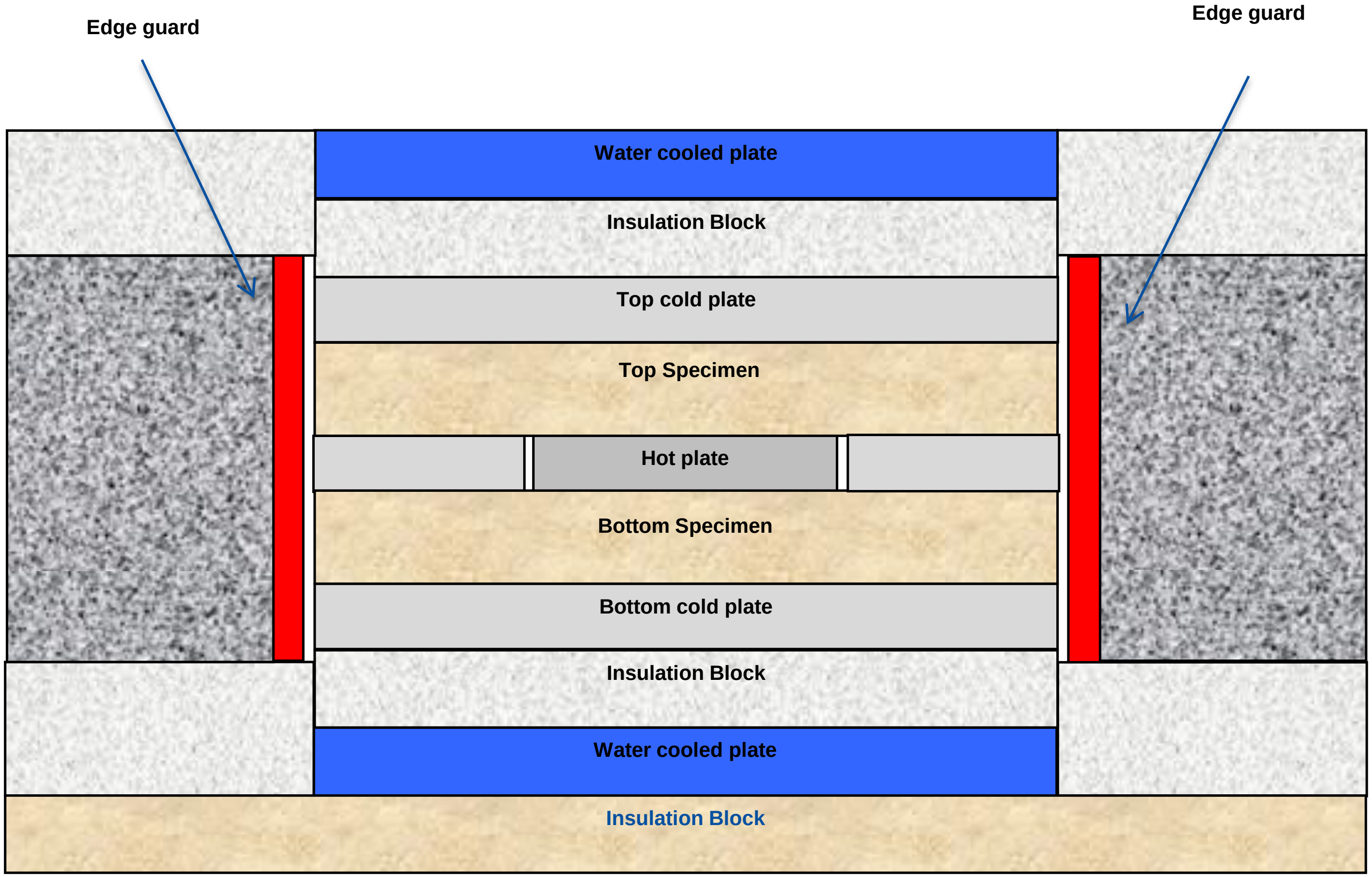

Presentation of the GHP technique

Configuration of LNE's HTGHP (double specimen).

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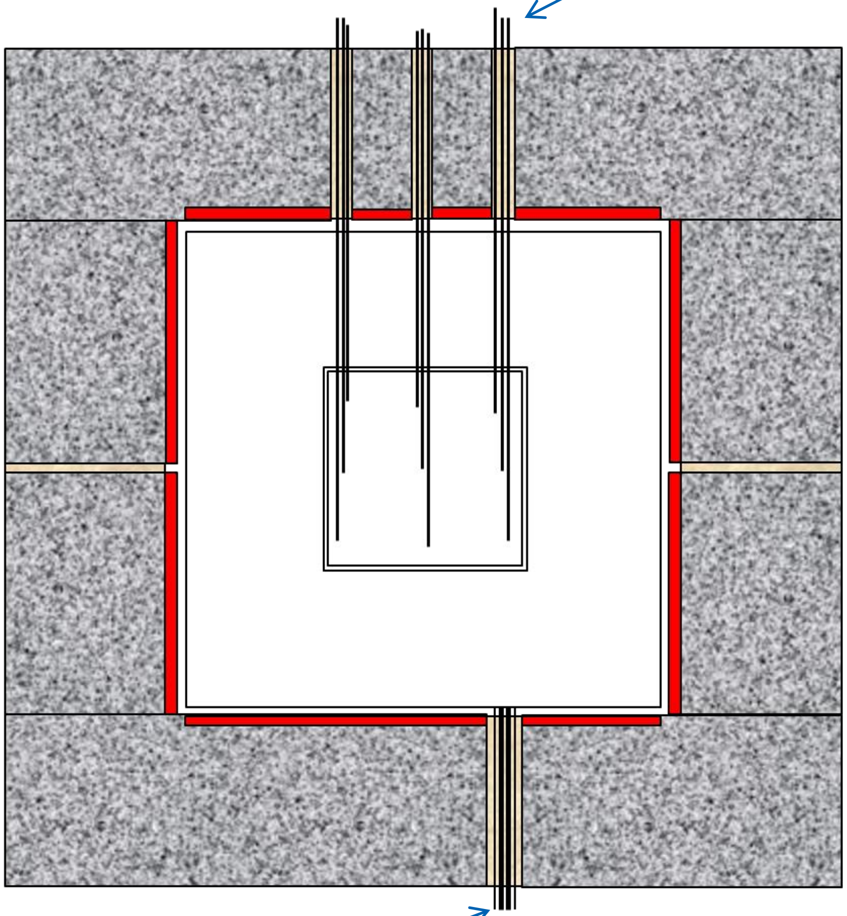


Difficulties to validate high temperature GHPs

No certified reference materials available (up to 800°C).

Reference materials are not sufficient to validate completely a GHP : reference materials should cover all the ranges of the GHP (thickness, temperature level, temperature drop, thermal resistance).

Sources of difficulties :

Configuration of the HTGHPs:

- 5 plates (3 of the plates are at high temperatures)
- Usually the size of a HTGHP is smaller that for low temperature GHPs.
- Edge guard is positioned as closer as possible to the edges of the samples and sometime the gap is filled with insulation material to avoid convection (confined geometry and poor accessibility to the hot plates and to the samples).
- The edge guard and the stack are heavily thermally insulated.
- High temperature difference between the mean temperature of the samples and the room temperature (risk to create significant heat bridges).
- Long time required for heating and cooling the instrument.

Difficulties to validate high temperature GHPs

Sources of difficulties :

Temperature sensors usable and reliable at high temperatures :

- Mineral insulated metal sheathed thermocouples (∅ 1 mm) → can be sources of heat bridges, the number of sensors usable for performance checking is limited (heat bridges).
- MIMS TCs cannot be bent at a short radius and are not very flexible.

High temperatures → specific materials to avoid degradations or reactions between materials

Main Uncertainty sources

Priorities an efforts in performance checking must be defined regarding the influence of the potential sources of errors an of uncertainties.

- Temperature gap imbalance → main source of uncertainty.
- Thermal contact resistance for rigid materials with low thermal resistance.
- Temperature drop measurement.
- Measurement of the electrical power supplied to the metering heater (correction factor).
- Edge guard temperature imbalance.
- Uniformity of temperatures over the cold and hot plates.
- Dimensions of the metering section at the temperature of measurement (hot plate)
- Stability (temperature control)
- Reproducibility

- Control of the temperature gap imbalance (mean temperature difference between the metering heater and the guard ring)

  - In the LNE's in-house made GHP, the temperature gap imbalance is measured with a 16 junctions thermopile (MIMS ∅ 1mm N type thermocouples connected in differential mode).
  - The thermocouples for the thermopile were selected rigorously (to anticipate potential problems) :

    A batch of thermocouples (same wires, same production) was heat cycled and calibrated up to 400°C. The temperature was limited at 400°C because the thermocouples of the thermopile must be bent when fitted in the hot plate. The risk of breakage of the sheath is high if bending is done after the TCs have been heated at high temperature (> 600°C).
  - The thermocouples retain for the thermopile were selected by pairs regarding the similarity of responses and of electrical resistances and the level of electrical isolation (elec resistance wires/sheath).
  - Once in place in the hot plate the difficulty is to check or calibrate the thermopile to check that the zero signal corresponds to the zero temperature gap imbalance (no offset).

- Control of the temperature gap imbalance (mean temperature difference between the metering heater and the guard ring)

Solution :

- Map the temperatures non uniformities over the hot plate : long process, not very accurate (uncertainties of temp.sensors used, heat bridges, results depend on the samples)
→ looking for mean differences of temperature of a few 1/10 Kelvin.

Solution tested by LNE.

- Using samples with very high thermal conductivity (metal samples) and having the hot plate in isothermal configuration:
  - No power supplied to the metering heater or to the guard ring of the hot plate → the hot plate is completely passive.
  - The two cold plates are set at the same temperature. The edge guard is set at the temperature of the cold plates.

→ the hot plate is almost in isothermal configuration and the zero level of the thermopile can be “calibrated” in-situ.

Control of the temperature gap imbalance (mean temperature difference between the metering heater and the guard ring)

Calibration of the thermopile in isothermal configuration : The hot plate is not heated.

- Samples : aluminum 318 mm x 318 mm x 30 mm , (Alplan material, purchased rectified)

Temperature level (°C)	Temperature of the plates in the metering section (°C)				Signal of the thermopile	Temperature difference
	Top cold plate	Bottom cold plate	Top surface of hot plate	Bottom surface of hot plate		
150 °C	147.39	147.24	147.28	147.15	-19.95 µV	0.063 K
350 °C	354.86	354.86	354.73	355.03	+ 9.1 µV	0.03 K

- The techniques seems to be appropriate for measuring in-situ the offset of the thermopile (signal at zero ΔT). It is quite easy to apply.
- More tests must be performed to confirm the technique.
- The distribution of temperatures over the hot plate can be change (/ measurement on insulation)
- For higher temperatures (up to 800 °C) → samples in Nickel 201 alloy are possible.

Limit : The “calibration” is not made with the conditions of measurement of λ . The distribution of temperatures in the hot plate can be modified by the high λ samples.

Thermal contact resistance - Control of the plates (Flatness)

- Mechanical stability of the plates : Risk of warping when heated at high temperatures (even after long time of use in correct conditions).

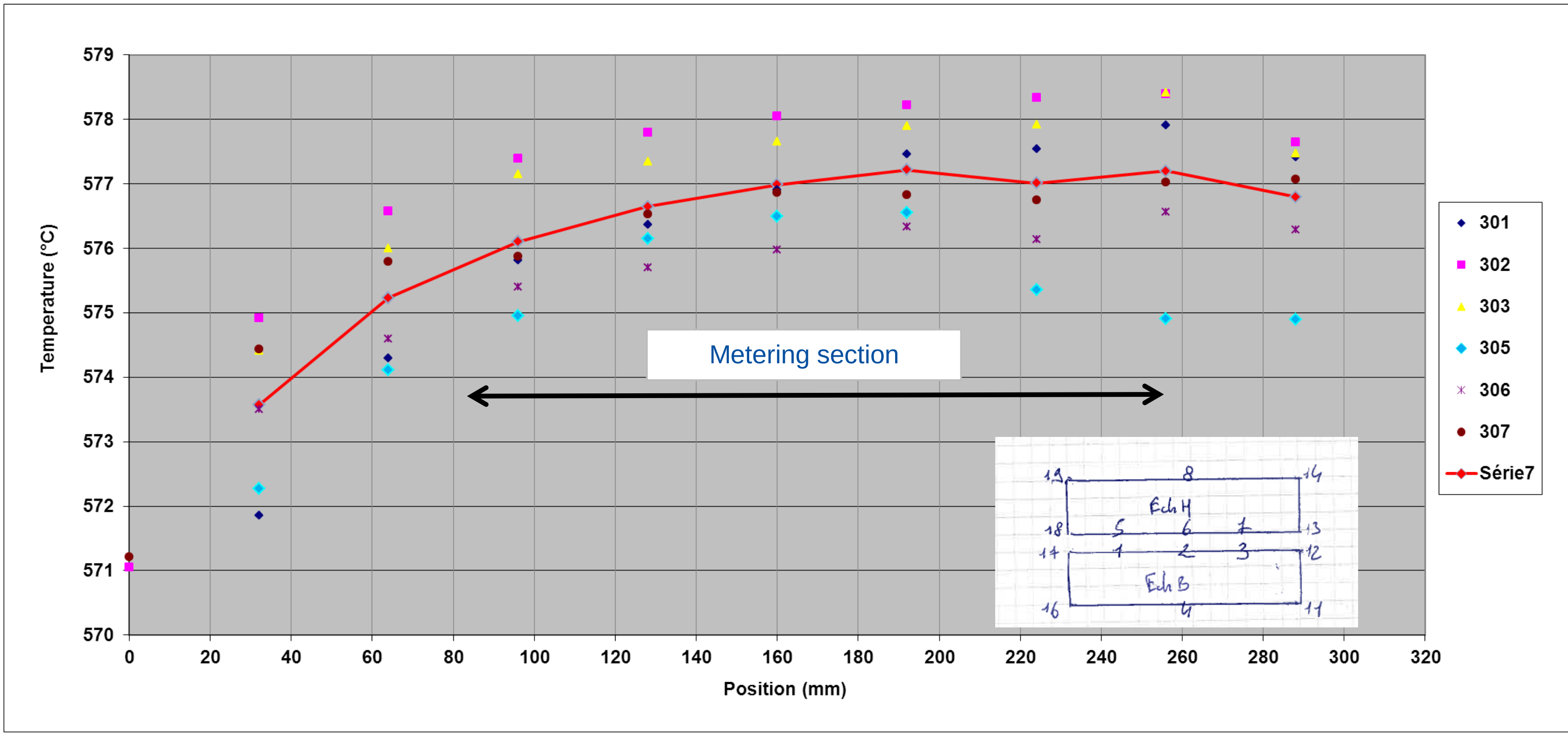
  - Checking at room temperature periodically with high frequency → quick systematic checking (with metal straightedge and thickness gages) after each use at high temperatures (> 500 or 600°C).

  - The control of warping in-situ and at high temperature is very difficult.

  → May be by measuring in-situ the thermal expansion of all the stack and quantify the warping → probably not sensitive (due to thermal expansion of all the elements in the stack, warping of samples and insulation blocks) .

Uniformity of temperatures of the plates

- LNE : measurement of distribution of temperatures by moving thermocouples in grooves at the surfaces of a HDCASi samples.



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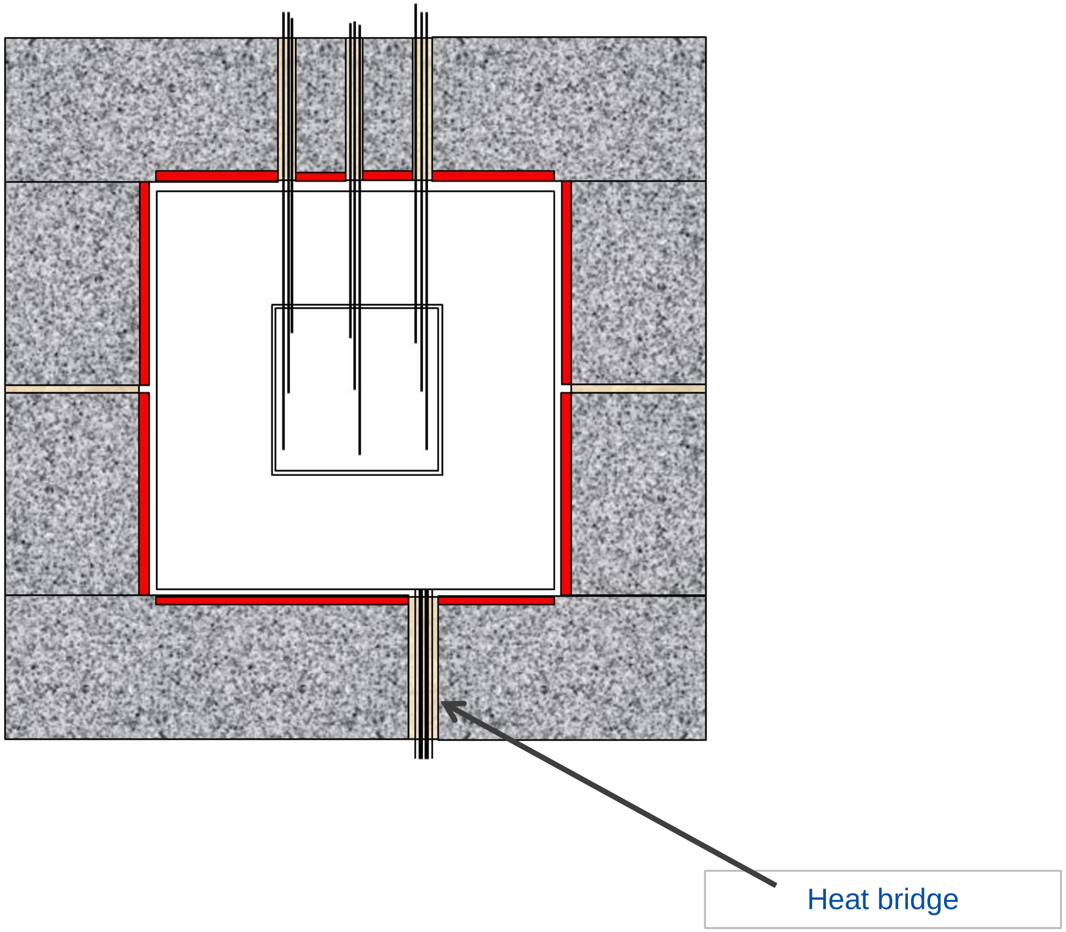


- Control of the temperature imbalance of the edge guard (mean temperature difference between the edge guard and the mean temperature of the samples)

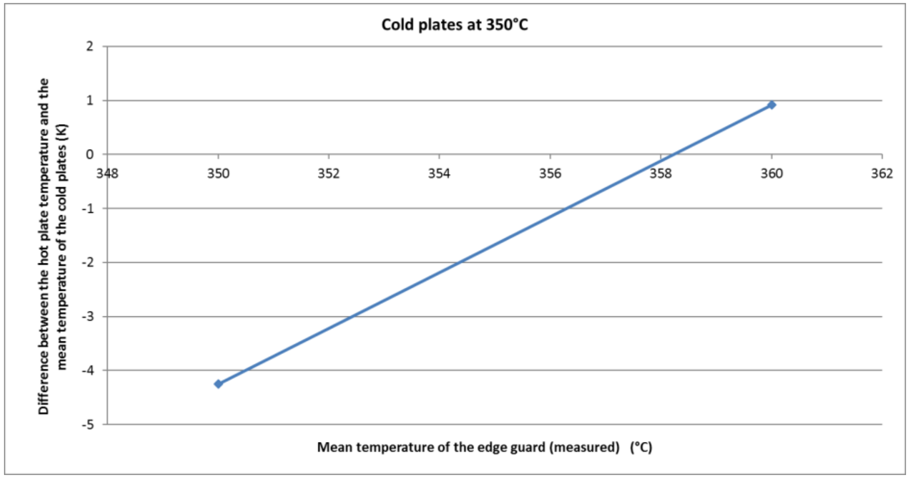
In the LNE's in-house made GHP, the mean temperature of the edge guard is measured by type thermocouples clamped to the NI metal plates of the edge guard blocks. The temperature set point for each block is the mean temperature of the samples.

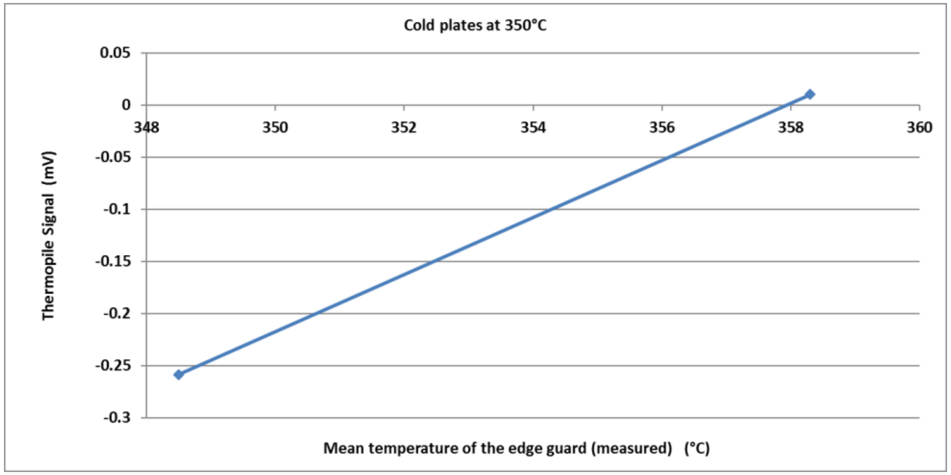
Solution tested by LNE.

- Using two samples of an insulation material (th = 48 mm) and having the hot plate in isothermal configuration:
  - No power supplied to the metering heater or to the guard ring of the hot plate → the hot plate is completely passive.
  - The two cold plates are set at the same temperature. And the edge guard set at different temperatures around the mean temperature of the cold plates.

  → the hot plate is almost in isothermal configuration.

- Control of the temperature edge guard imbalance (mean temperature difference between the edge guard and the mean temperature of the samples)

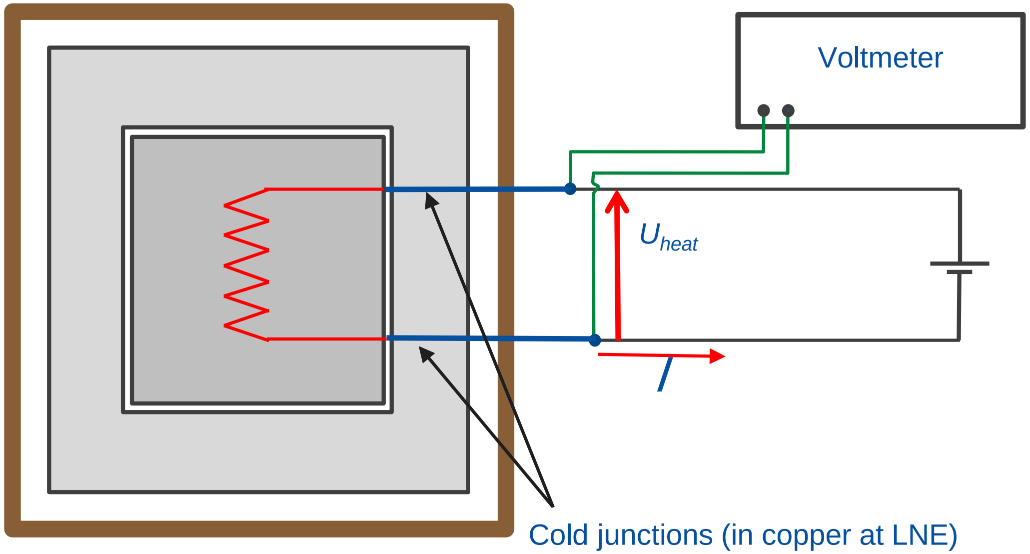




- This test is probably not an accurate way to get the ideal temperature of the edge guard, the temperature which gives a zero mean heat flux on the edges of the stack in the conditions of measurement of λ.
- It gives a least a limit value for the uncertainty of the temperature imbalance of the edge guard (about 8 °C for the case presented).
- In the case of LNE HTGHP the edge guard should be probably set a bit higher than the mean temperature of the samples (heat losses, probably air circulation in the air space around the stack).

Correction of the electrical power

- Mineral insulated metal sheathed (MIMS) heating wire (Thermocoax) used. Measurement of voltage performed outside the hot plate → a portion of the measured power dissipated outside the metering area (in the "cold junctions").



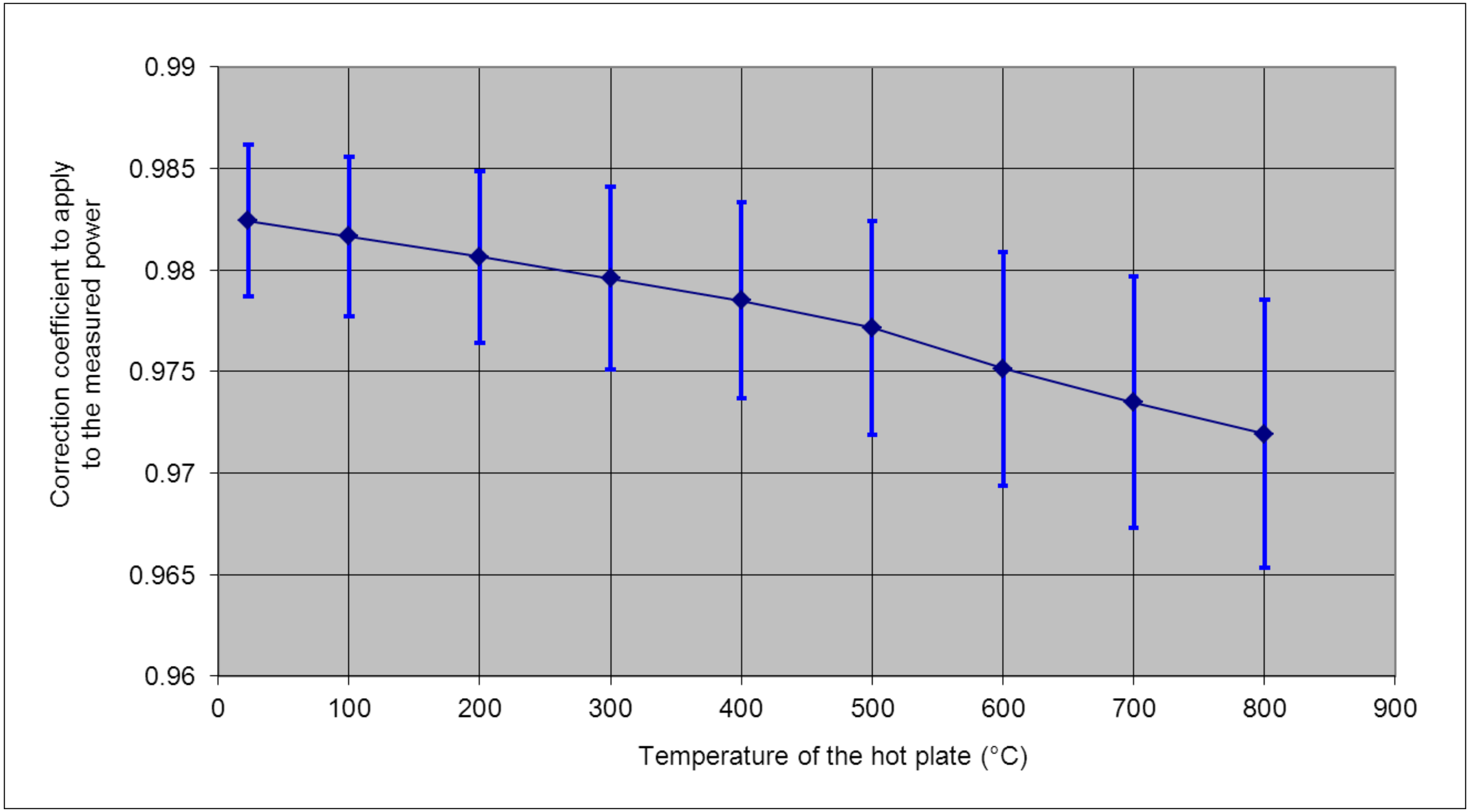
- A correction factor must be applied to the measured power.

- Electrical resistivity data for wires only available at room temperature (Thermocoax)

- Measurement of electrical resistances of the 3 type of wires constituting the loop (hot wire MIMS, cold wire MIMS, flexible wire) in function of temperature (RT to 800°C). Measurements done on samples of the 3 wires cut in a similar heating element.

Correction of the electrical power

- Calculation of the correction factor and of uncertainties using the measured electrical resistances and the temperature distributions along the loop.



- Electrical isolation of the heating loop, of the thermopile.

  - Electrical isolation of the heating wire in the metering heater (particularly mineral insulated metal sheathed) can decrease significantly at high temperature. That can be a source of current leaks.

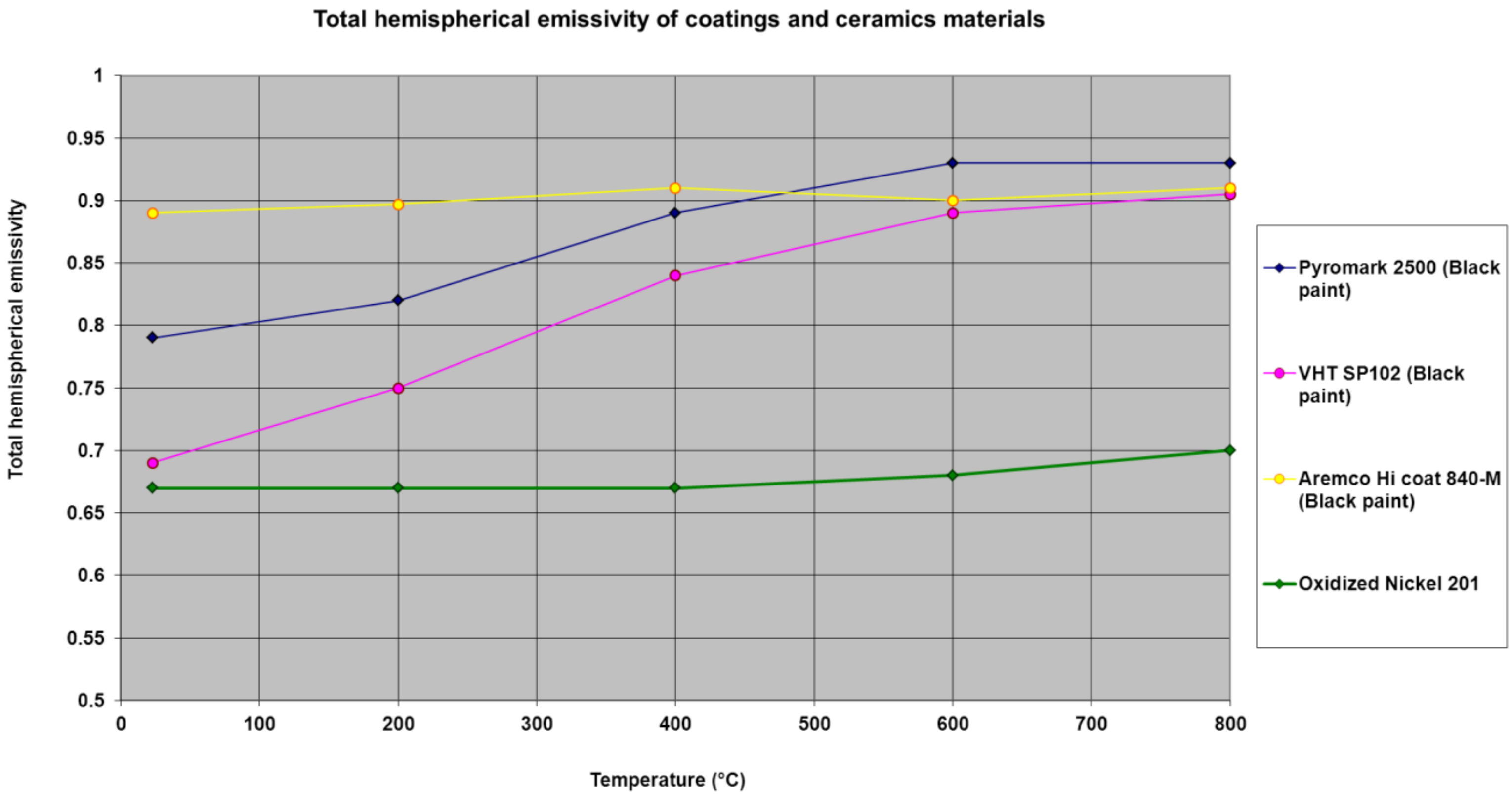
    → the electrical isolation of the heating wire must be controlled over the range of temperature.

    A limit of the electrical resistance of isolation of the heating wire should be set in the standard for HTGHP.
    A procedure for evaluating the uncertainty related to that problem should be established.

  Thermopile isolation : risk of decrease of sensitivity → must be checked.

Emissivity of the plates

- Emissivity of potential coatings measured by LNE in function of temperature.



Aremco paint Hie Coat 840-M has generated irreversible pollution of CaSi samples

Pyromark 2500 Black and VHT SP102 paints are OK.

Recommendations - Conclusions

- Think to the performance checking when designing a HTGHP
 If the instrument is very integrated and confined → difficulties.

- Temperature sensors (for the thermopile or for the temperature drop measurement)
→ selection by tests at the highest possible temperature regarding the stability, electrical isolation, homogeneity of response.

- If electrical power is not measure directly at the limit of the metering heater, try to measure the electrical resistances of the different wires in the loop in function of temperature and calculate the correction factor an the uncertainty on the factor.

- Control the electrical isolation of the heating wire and of the thermopile.

- Emissivity of plates → use a coating validated in emissivity.

- All recommendations for performance checking already existing in standards that can be applied for HHTGPs must be applied.

- A work is still to be done for improving and refining techniques for performance checking of HTGHPs → future projects ?