SIB 52 - THERMO Stakeholder meeting

Nov 14

Metrology for thermal protection materials

Perturbations of thermal conductivity measurements with a GHP due to thin samples, emissivity effects and transparency of materials

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

Laboratoire national de métrologie et d'essais

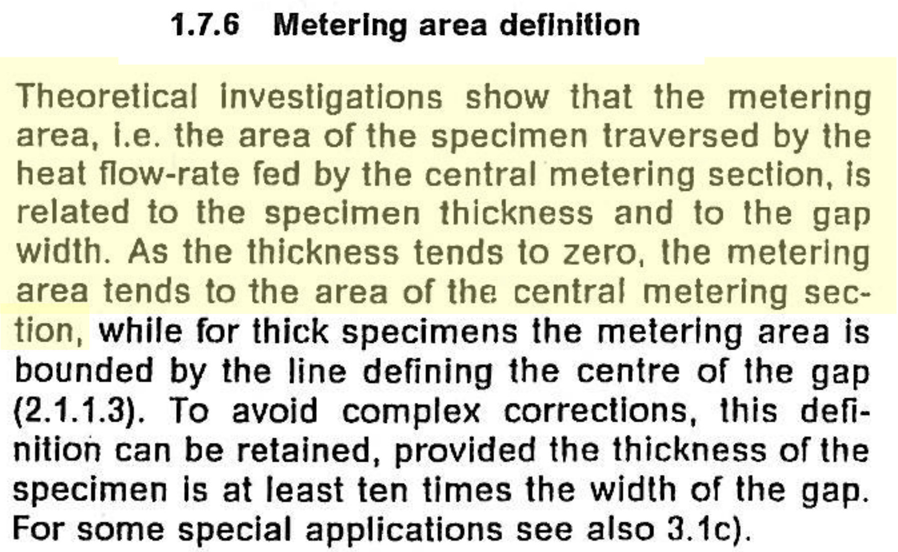
Outline

- Definition of the metering area for thin samples
- Measurements of radiative properties (emissivity, transparency) of heater plates materials and of insulation products.
- Influence of an error on the hot and cold plates emissivity on the measured λ

- Definition of the metering area for thin samples when measuring λ with a GHP

The definition at the mid-gap is a source of error for thin samples

Iso 8302 :



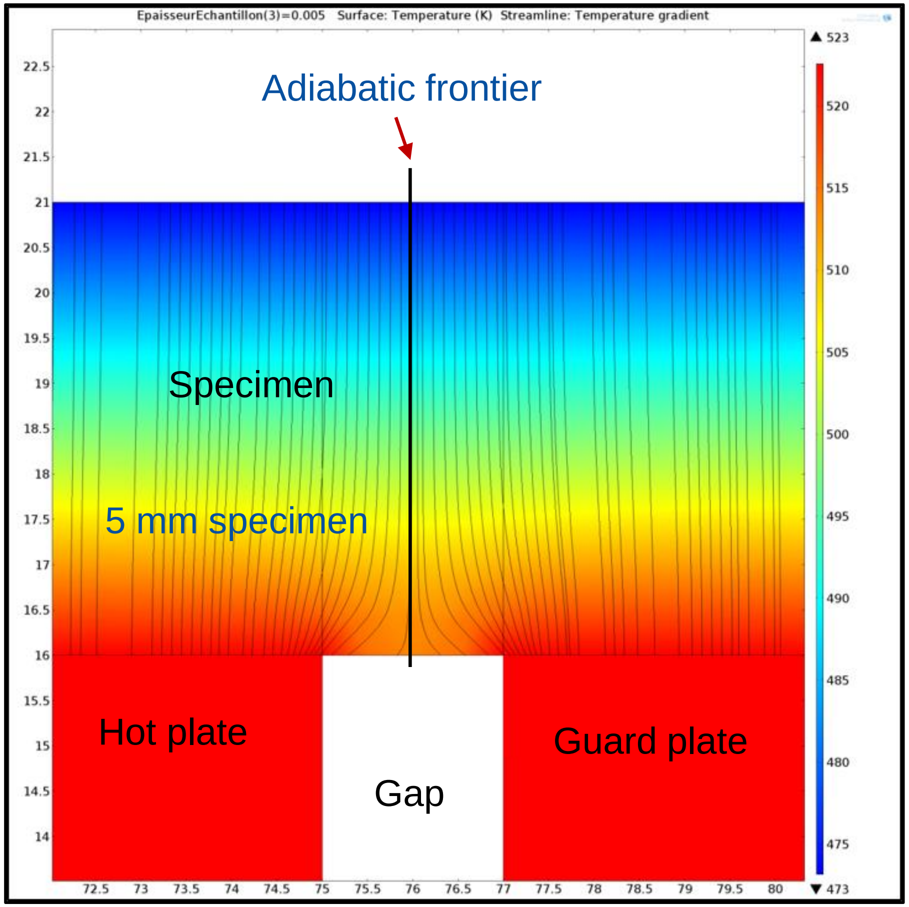
1.7.6 Metering area definition

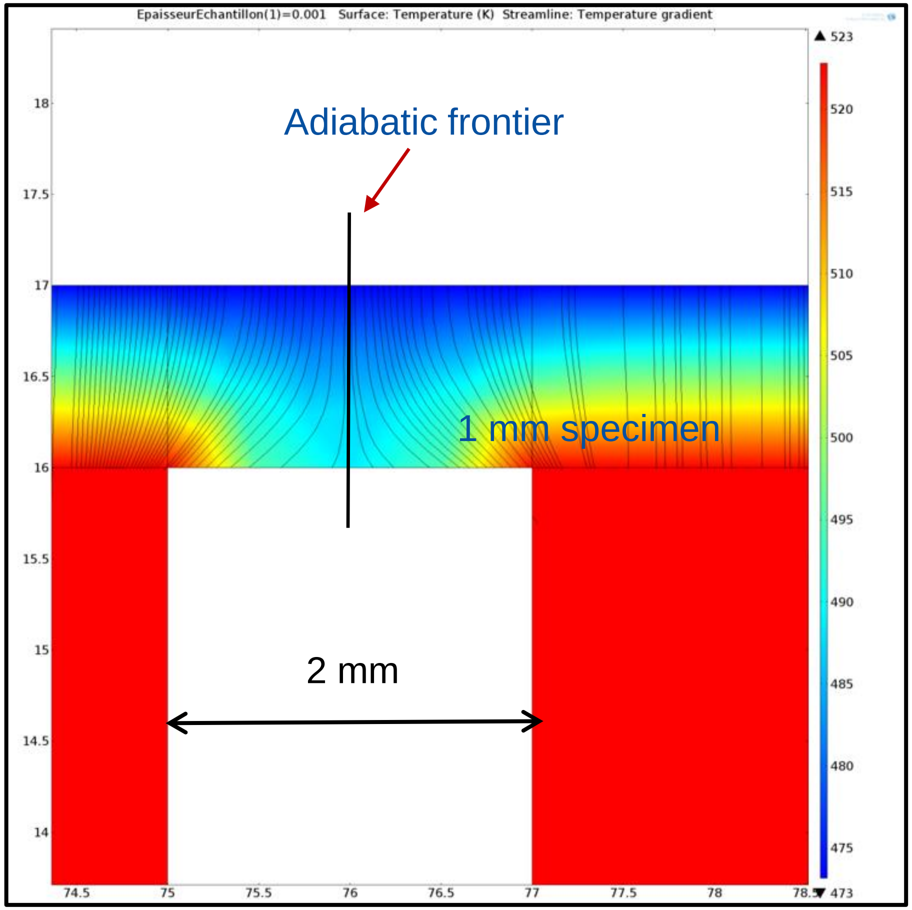
Theoretical investigations show that the metering area, i.e. the area of the specimen traversed by the heat flow-rate fed by the central metering section, is related to the specimen thickness and to the gap width. As the thickness tends to zero, the metering area tends to the area of the central metering section, while for thick specimens the metering area is bounded by the line defining the centre of the gap (2.1.1.3). To avoid complex corrections, this definition can be retained, provided the thickness of the specimen is at least ten times the width of the gap. For some special applications see also 3.1c).

⇒ The interpretation of theory is false : For good conditions of measurement (no imbalance, uniformity of thermal contacts), the area of the specimen traversed by the heat flow-rate fed by the central metering section is not related to the specimen thickness.
But it is true that for thin materials (compared to the gap width), an error is made if the metering section is defined at mid-gap as it is for thick specimens.

The source of the error is in fact a problem of “non unidirectionnal” heat flow through the sample in the gap region when the specimen is thin.

Definition of the metering area for thin samples when measuring λ with a GHP



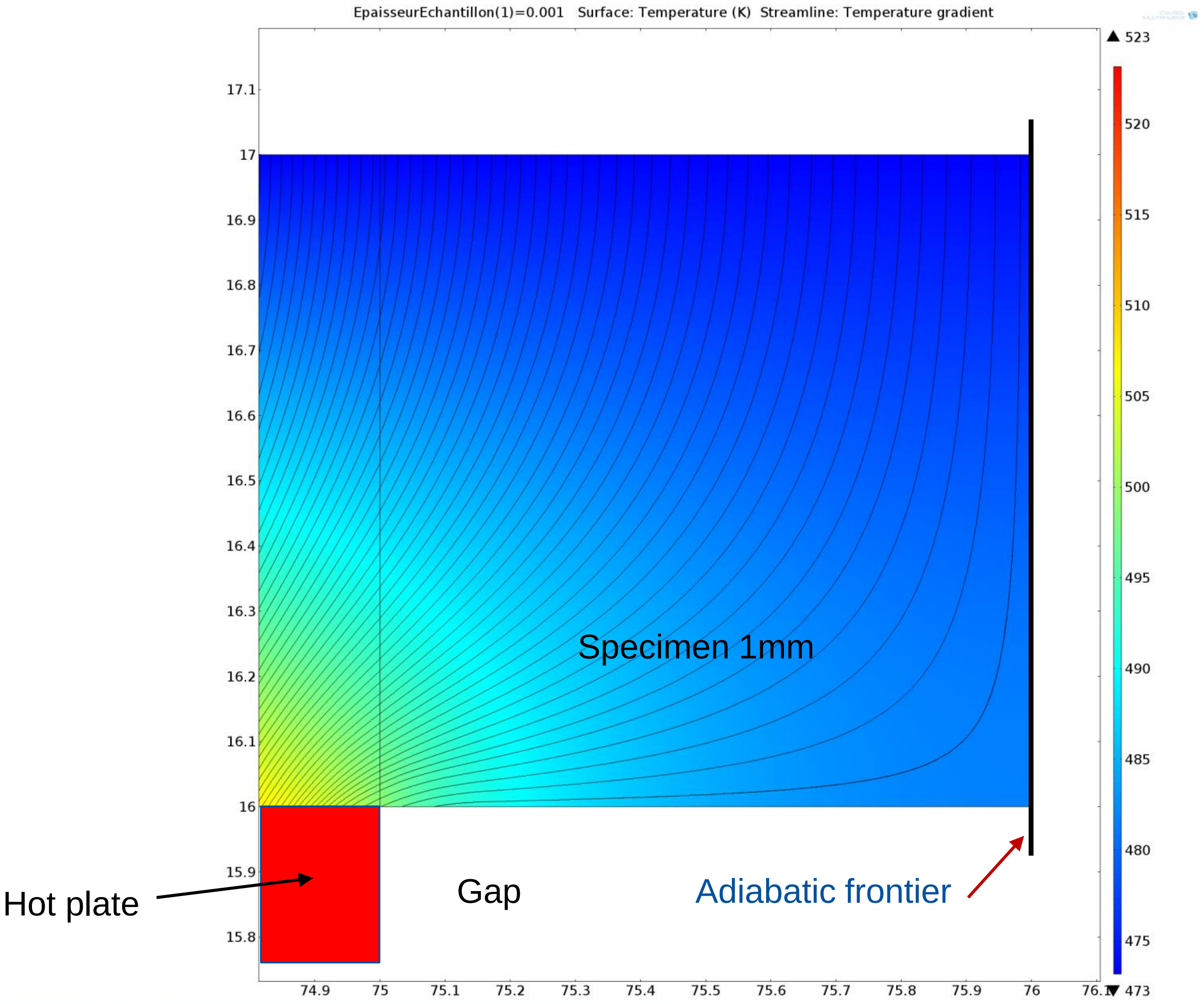


In the gap region the heat flow is not unidirectionnal, the heat must travel a longer path. → This is the source of the error on thermal conductivity. The thermal condcutivity is underestimated if the metering section is defined at mid-gap.

For thick specimens, the relative increase of the thermal path is small → “no error”.

For thin specimens, the relative increase of the thermal path is important → error.

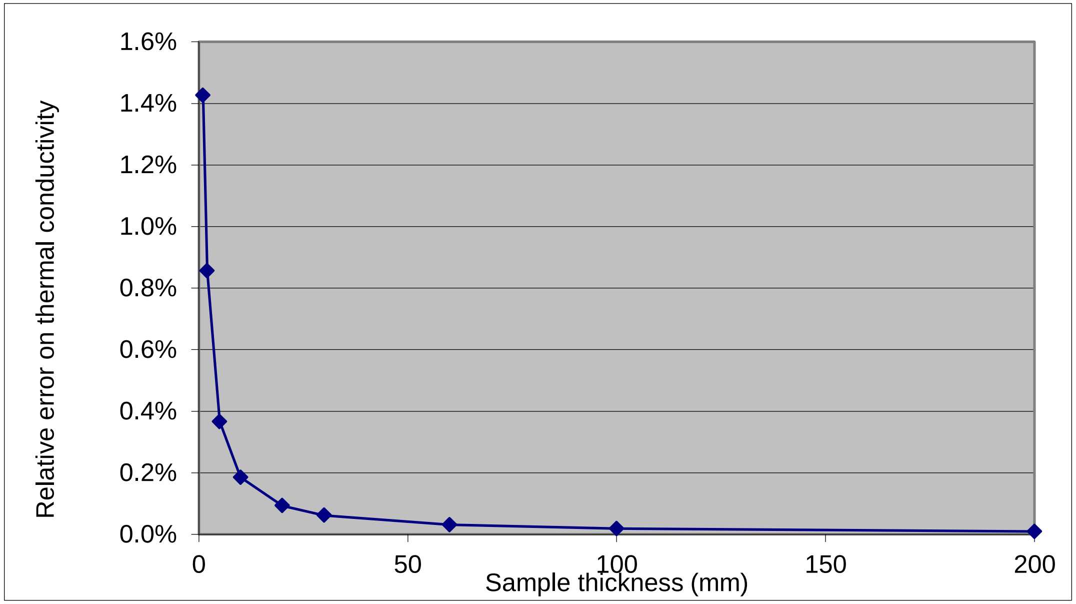
- Definition of the metering area for thin samples when measuring λ with a GHP



- Definition of the metering area for thin samples when measuring λ with a GHP
  - Numerical errors if the metering section is defined at mid-gap

Thermal conductivity : 0.1 W m^{-1} K^{-1}
Material : isotrope
Temperature drop : 50°C
Metering area : square, 150 x 150 mm
Gap : 2 mm, adiabatic
Thermal guard : 75 mm wide
Metering area/ guard imbalance = 0 °C
Edge : adiabatic

Sample thickness (mm)	Ratio : sample thickness / gap width	lambdaX	Relative error on thermal conductivity
1	0.5	0.1	1.43%
2	1	0.1	0.86%
5	2.5	0.1	0.37%
10	5	0.1	0.185%
20	**10**	0.1	**0.093%**
30	15	0.1	0.062%
60	30	0.1	0.031%
100	50	0.1	0.019%
200	100	0.1	0.009%



- Definition of the metering area for thin samples when measuring λ with a GHP

Main parameters of influence on the error

- Ratio : sample thickness / gap width
- Ratio : In plane thermal conductivity / through thickness thermal conductivity

  If ″in plane λ ″ > ″ through thickness λ ″ ⇒ the error is lower.
- Ratio : area of the apparent surface of the gap to metering area.

Conclusions :

- An error is made on λ for thin materials if metering area is defined at mid-gap.
- For the same gap width, the error decreases with the metering area.
- If the gap width is limited at 2 mm, the relative error remains at about 1.5% for a 1 mm thick isotrope material with a 150 x 150 mm metering area.
- For thin materials, that error is very probably low compared to “thermal contact resistance errors”.

Problems related to transparency and emissivities of materials

Heater plate materials

One objective of THERMO : try to find new materials with high conductivity, resistance to high temperatures in air, high mechanical properties at high temperatures → to replace metals usually used to build heater plates in HTGHPs.

A good heater plate material should be opaque : no direct radiation from heating wires to the sample faces.

The emissivity of the heater plates should be known with a known uncertainty in order to compare results

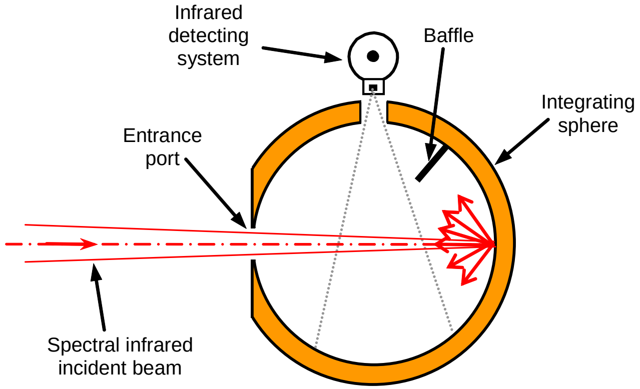
Material of the sample to be tested

- Problems of thermal conductivity measurements related to transparency.
  - Result very dependent on :
    - thermal gradient
    - temperature drop
    - thickness (for the same material)
    - emissivities of heater plates (bad reproducibility between instruments)
  - Results can be very dependent on the conditions of measurement

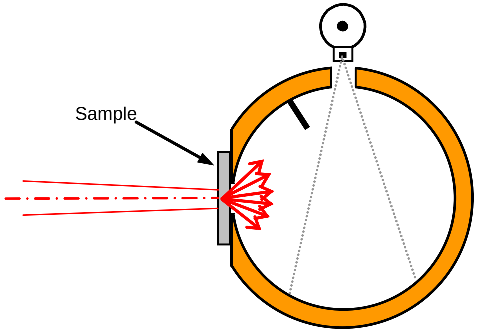
- One of the objective of the project → reference materials for high temperature
  - Ideally a reference material is opaque (no influence of transparency when measuring thermal conductivity)
  - ⇒ We need information about transparency of candidate materials

- Thin insulation materials
  - Transparency information needed to anticipate problems of measurement of thermal conductivity
  - Define critical thickness of materials to compare instruments (no problem of reproducibility related to transparency)
  - Define critical conditions of measurement for “transparent” materials (thickness, temperature range, thermal gradient)

- Problems related to transparency and emissivities of materials
  - Techniques of measurement of radiative properties
    - Measurement of spectral transparency of **diffusing** materials at LNE



Configuration : Measurement of reference signal

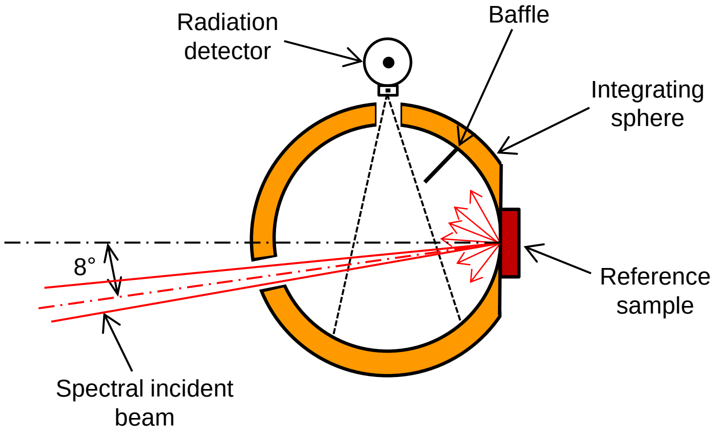


Configuration : Measurement of sample signal

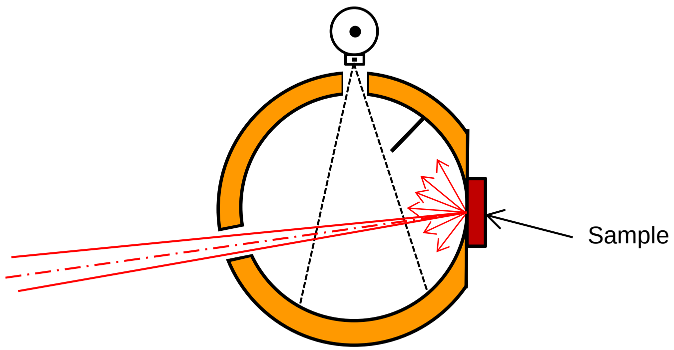
$$\boxed{\text{Normal spectral transmittance} = \frac{\text{Sample signal}}{\text{Reference signal}}}$$

Measurements are performed on samples **at room temperature**

- Problems related to transparency and emissivities of materials
  - Techniques of measurement of radiative properties
    - Measurement of near-normal hemispherical spectral reflectance at LNE



Configuration : Measurement of reference signal



Configuration : Measurement of sample signal

$$\text{Near - normal hemispherical spectral reflectance} = \frac{\text{Sample signal}}{\text{Reference signal}} \times \text{Reference sample spectral reflectance}$$

Measurements are performed on samples **at room temperature**

Problems related to transparency and emissivities of materials

Techniques of measurement of radiative properties

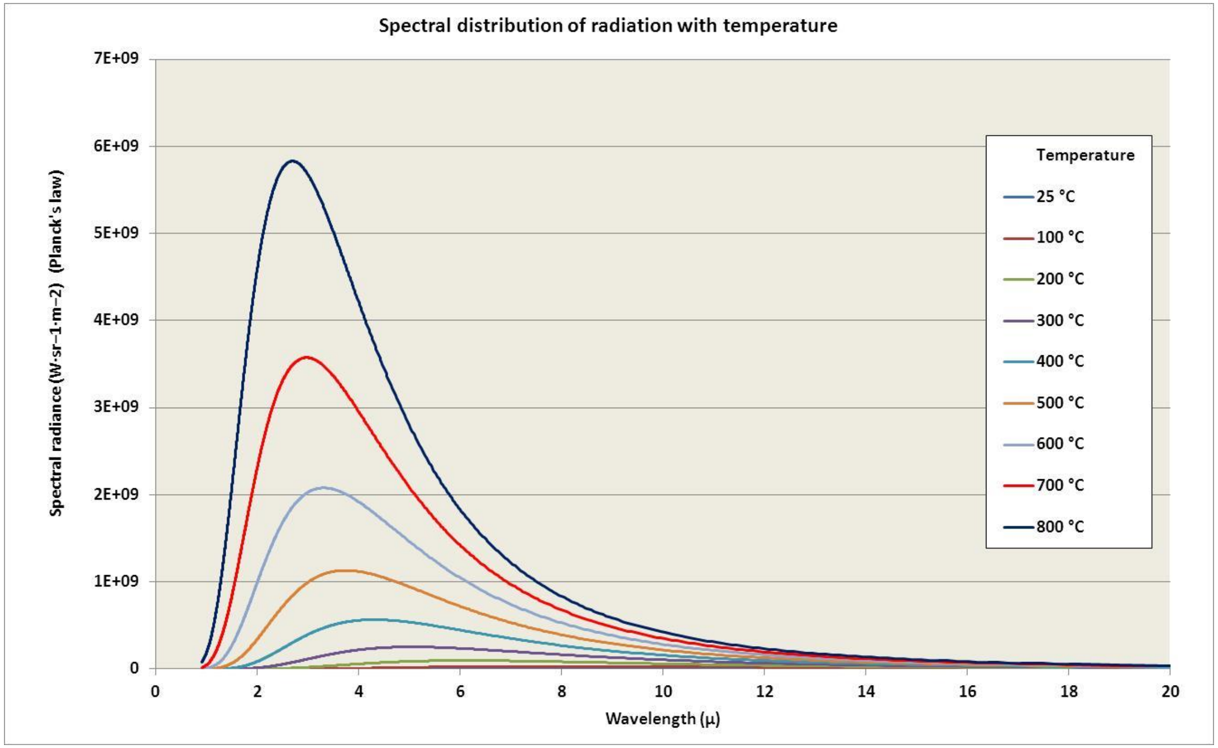
- Other measurements performed at LNE
    - Near normal spectral emissivity (1 to 16 µm – room temp to 800°C) → emissivity of heater plates coatings

- Problems related to limitation of measurement at room temperature
    - Radiative properties of insulation materials are mainly related to the nature of materials and to the structure of insulation materials
    - For most solid materials the influence of temperature is quite low as long as they are no modifications of the insulation materials and of the structure
    - If the material evolves with temperature it is better to thermal cycle it before measuring radiative properties

Problems related to transparency and emissivities of materials

Techniques of measurement of radiative properties

- Spectral range covered
  - At LNE, measurement of spectral radiative properties from **0.8 to 16 µm**

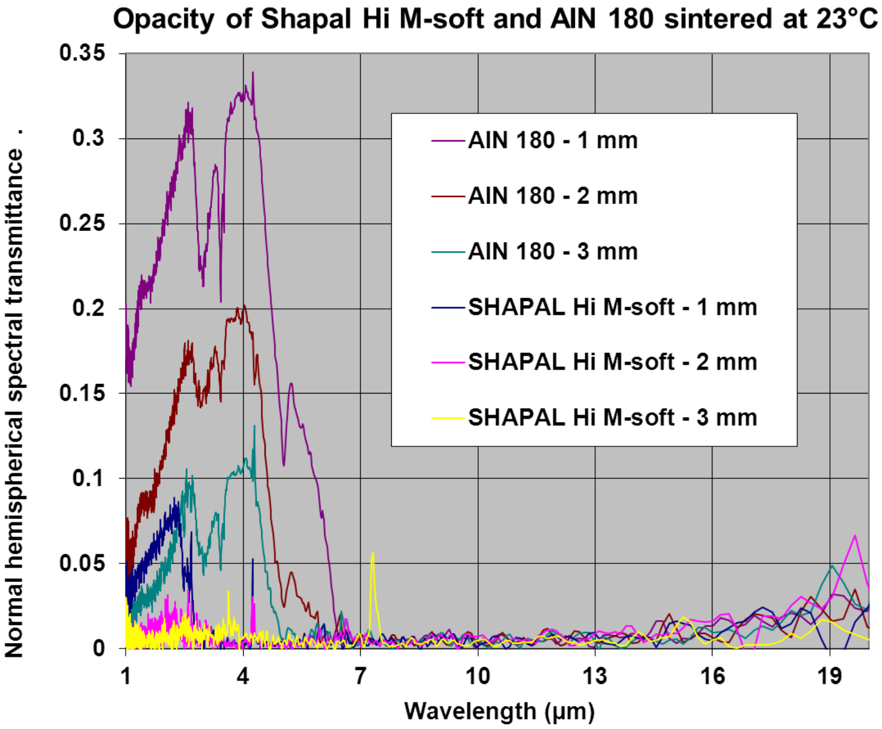


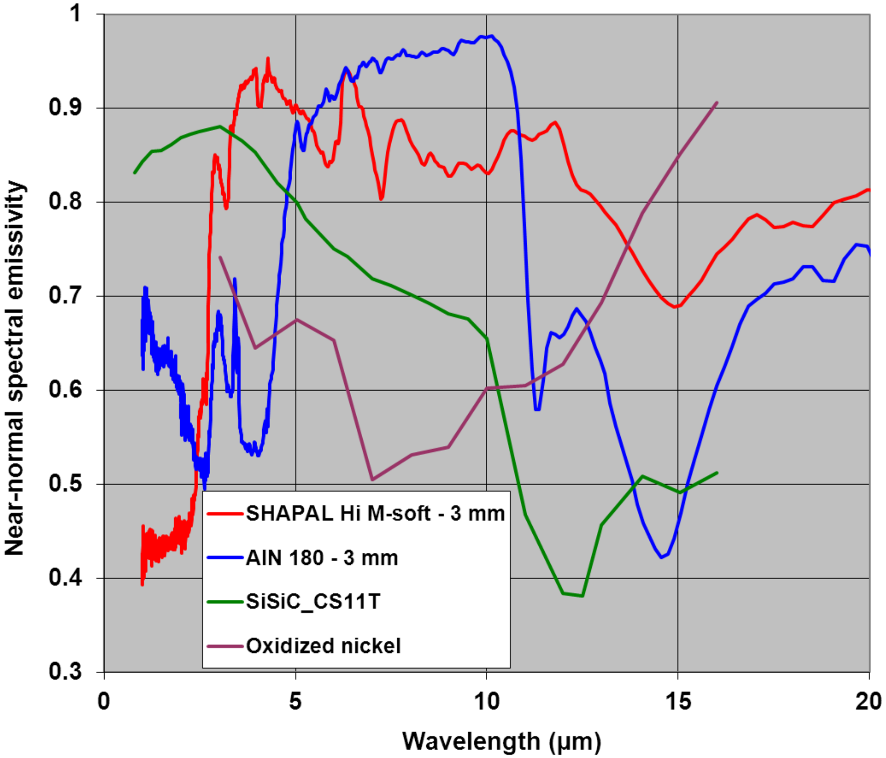
Temperature (°C)	Proportion of the radiation emitted by a black body in the 1 to 16 µm spectral band
25	61%
50	66%
100	74%
200	84%
300	90%
400	93%
500	95%
600	96%
700	97%
800	98%

Problems related to transparency and emissivities of materials

Results on candidate ceramic heater plate materials

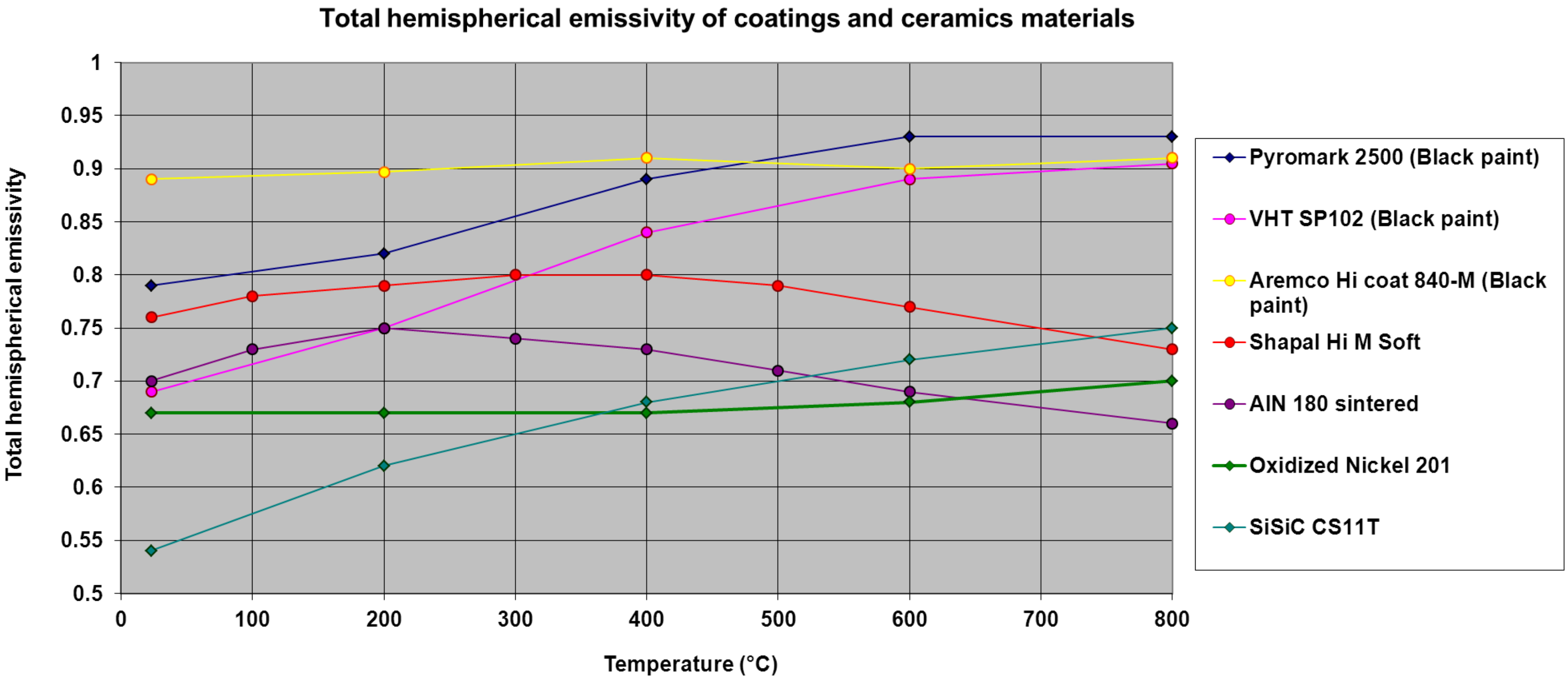
- Candidate new materials identified : AlN 180 (sintered aluminum nitride from FINAL Advanced Materials, France), SHAPAL Hi M-soft (machinable aluminum nitride ceramic from Tokuyama Corp., Japan), SiSiC CS11T (silicium infiltrate SiC structure ceramic from Ceramdis GmbH, Switzerland)



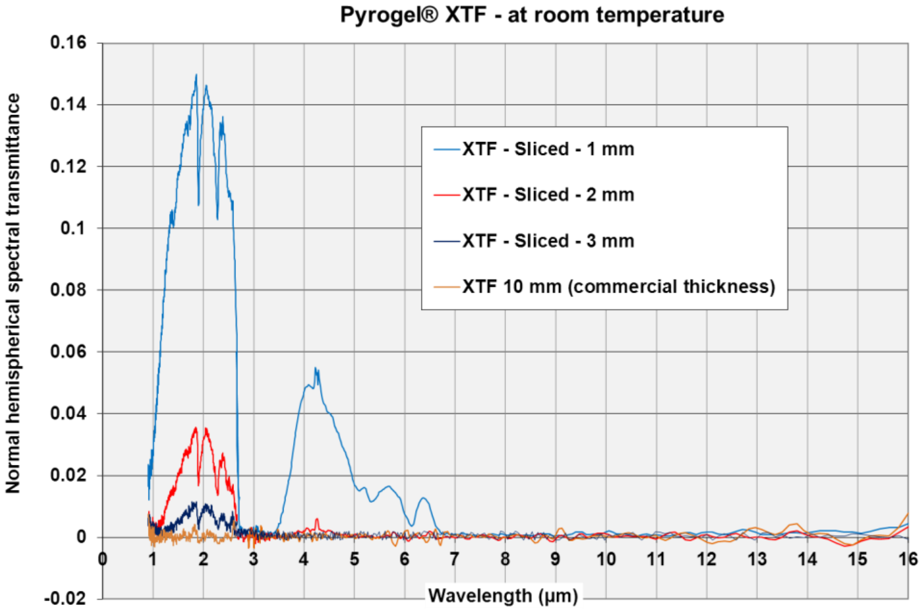


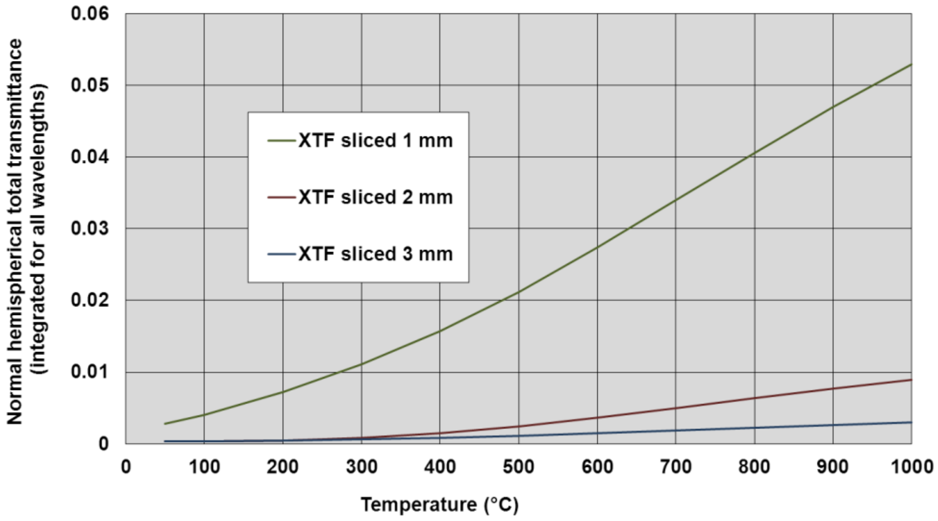
SiSiC CS11T is opaque

- Problems related to transparency and emissivities of materials
  - Thin insulation materials
    - Total hemispherical emissivity of HT coatings and candidate ceramic materials



- Problems related to transparency and emissivities of materials
  - Thin insulation materials
    - Materials : Pyrogel XTF from Aspen Aerogels Inc.

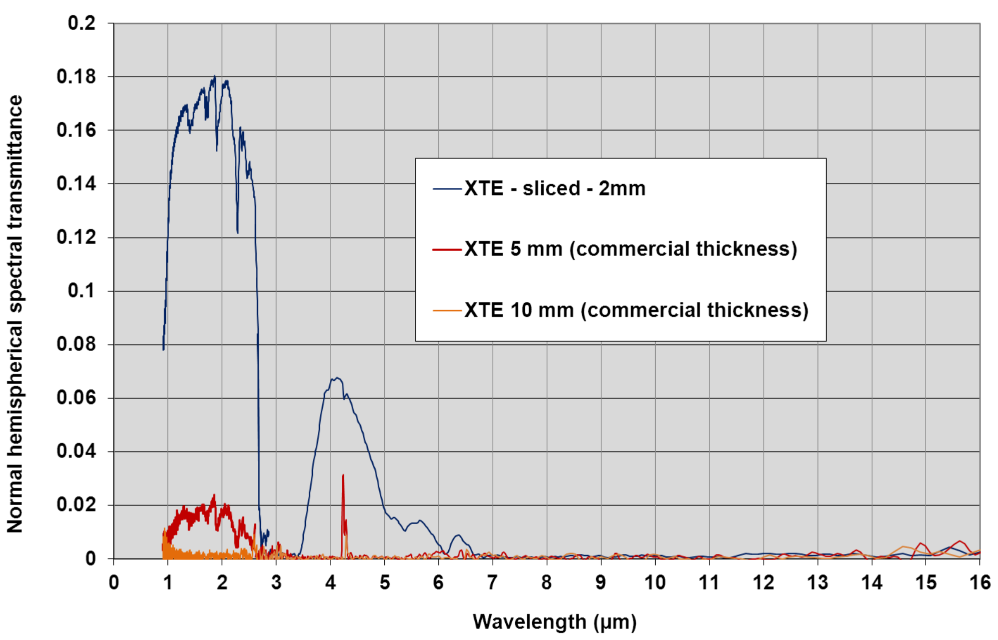


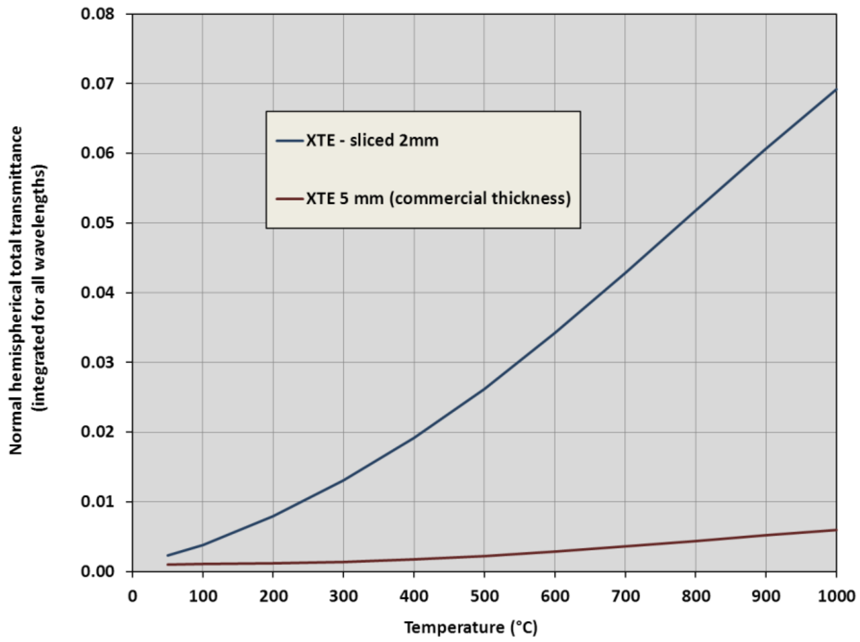


Problems related to transparency and emissivities of materials

Thin insulation materials

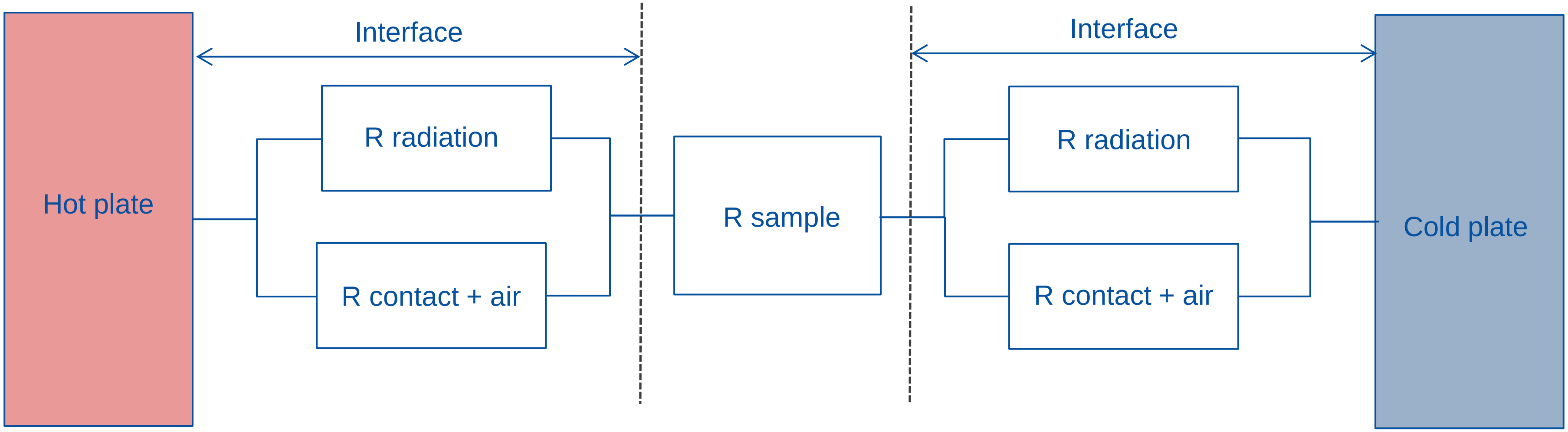
- Materials : Pyrogel XTE from Aspen Aerogels Inc.





- Conclusions
  - We can measure radiative properties of insulation materials at room temperature in the spectral range of interest
  - Transparency must be measured on thin layers to get relevant information on transparency
  - We are able to determine the “thickness of opacity” and the emissivity (for thick isothermal layers)
  - Spectral data is useful to predict evolution of wavelengths integrated parameters with temperature

- Problems related to transparency and emissivities of materials
  - Influence of hot and cold plates emissivity on the measured λ



$$R_{tot} = \frac{1}{\frac{1}{R_{rad\ cold}} + \frac{1}{R_{cont\ cold}}} + R_{sample} + \frac{1}{\frac{1}{R_{rad\ hot}} + \frac{1}{R_{cont\ hot}}}$$

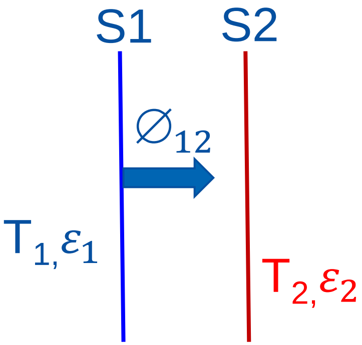
$$\lambda_{measured} = \frac{thickness}{R_{tot}} < \lambda_{sample} = \frac{thickness}{R_{sample}}$$

- Problems related to transparency and emissivities of materials
  - Influence of hot and cold plates emissivity

$$\frac{|\Delta\lambda|}{\lambda} \approx \frac{|\Delta R_{Tot}|}{R_{Tot}} = \frac{\dfrac{1}{\dfrac{1}{R_{rad\ cold}}+\dfrac{1}{R_{cont\ cold}}} + \dfrac{1}{\dfrac{1}{R_{rad\ hot}}+\dfrac{1}{R_{cont\ hot}}}}{R_{sample}}$$

The thermal resistance for “contact heat transfers” and the thermal resistance for “radiation heat transfers” must be known to calculate the error on λ.

$R_{rad\ cold}$ can be calculated from emissivities and temperatures of the plates and of the sample.



$$\varnothing_{12} = \frac{\varepsilon_1\ \varepsilon_2\ *\ \sigma\ \left(T_1{}^4 - T_2{}^4\right)}{1\ -\ (1-\varepsilon_1)\,(1-\varepsilon_2)}$$

$$R_{rad\ 12} = \frac{\Delta T}{\varnothing_{12}} = \frac{(T_1 - T_2)\,[1\ -\ (1-\varepsilon_1)\,(1-\varepsilon_2)]}{\varepsilon_1\ \varepsilon_2\ *\ \sigma\ \left(T_1{}^4 - T_2{}^4\right)}$$

- Problems related to transparency and emissivities of materials
  - Influence of hot and cold plates emissivity
    - Numerical application

Temperature (°C)	Relative variation of measured λ due to a variation of 0.05 of the emissivities of plates (plates emissivity = 0.85)					
	Sample thickness (mm)					
	20	25	30	40	50	60
50	0.0017%	0.0013%	0.0011%	0.0008%	0.0007%	0.0006%
75	0.0018%	0.0014%	0.0012%	0.0009%	0.0007%	0.0006%
100	0.0019%	0.0015%	0.0013%	0.0010%	0.0008%	0.0006%
150	0.0022%	0.0018%	0.0015%	0.0011%	0.0009%	0.0007%
200	0.0025%	0.0020%	0.0017%	0.0013%	0.0010%	0.0008%
250	0.0028%	0.0022%	0.0019%	0.0014%	0.0011%	0.0009%
300	0.0031%	0.0025%	0.0021%	0.0015%	0.0012%	0.0010%
350	0.0034%	0.0027%	0.0022%	0.0017%	0.0013%	0.0011%
400	0.0036%	0.0029%	0.0024%	0.0018%	0.0015%	0.0012%
450	0.0039%	0.0031%	0.0026%	0.0020%	0.0016%	0.0013%
500	0.0042%	0.0033%	0.0028%	0.0021%	0.0017%	0.0014%
600	0.0047%	0.0037%	0.0031%	0.0023%	0.0019%	0.0016%
700	0.0051%	0.0041%	0.0034%	0.0026%	0.0021%	0.0017%
800	0.0055%	0.0044%	0.0037%	0.0028%	0.0022%	0.0018%
900	0.0058%	0.0047%	0.0039%	0.0029%	0.0023%	0.0019%

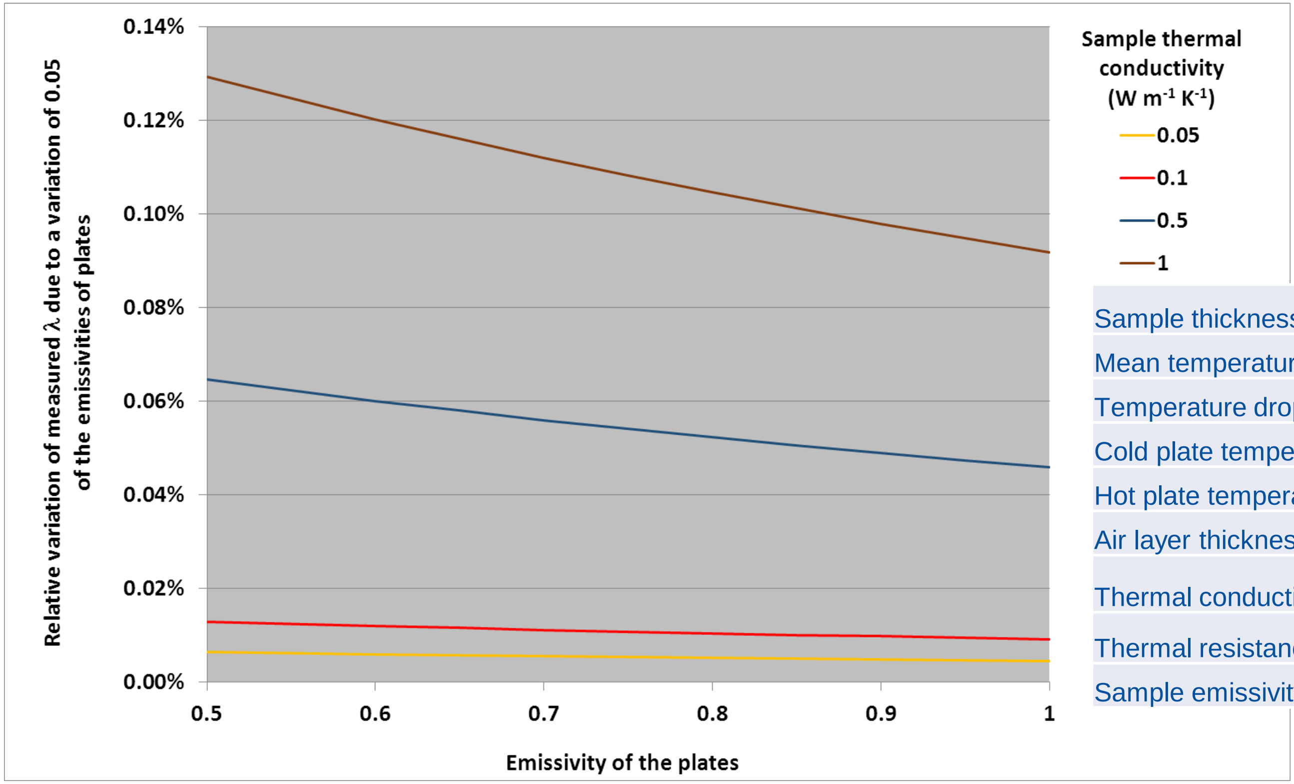
Sample thermal conductivity = 0.05 $Wm^{-1}K^{-1}$

Temperature drop = 50°C

Sample faces emissivity = 0.7

Thickness of the air layer at each interface (plate/sample) = 0.1 mm

- Problems related to transparency and emissivities of materials
  - Influence of hot and cold plates emissivity
    - Numerical application



Sample thickness (m)	0.02
Mean temperature (°C)	500
Temperature drop (°C)	50
Cold plate temperature (°C)	475
Hot plate temperature(°C)	525
Air layer thickness (m)	1.00E-04
Thermal conductivity of air (Wm^{-1}K^{-1})	5.36E-02
Thermal resistance of the air layer (m^2 K W^{-1})	1.86E-03
Sample emissivity	0.8

Problems related to transparency and emissivities of materials

Influence of hot and cold plates emissivity

Conclusions

- The order of magnitude of the uncertainty on λ due to the uncertainty on the emissivities of the plates can be evaluated.
- The level of emissivity of the sample faces and the “thermal contact resistances” must be known for the numerical evaluation.
- The uncertainty is higher for higher temperatures (higher radiation heat flux) and when thermal contact is poor