Measurement of the thermal conductivity at cryogenic temperature

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Measurement of the thermal conductivity at cryogenic temperature

For storing and transporting liquid gas, it is useful to know the thermos-physical properties of materials.

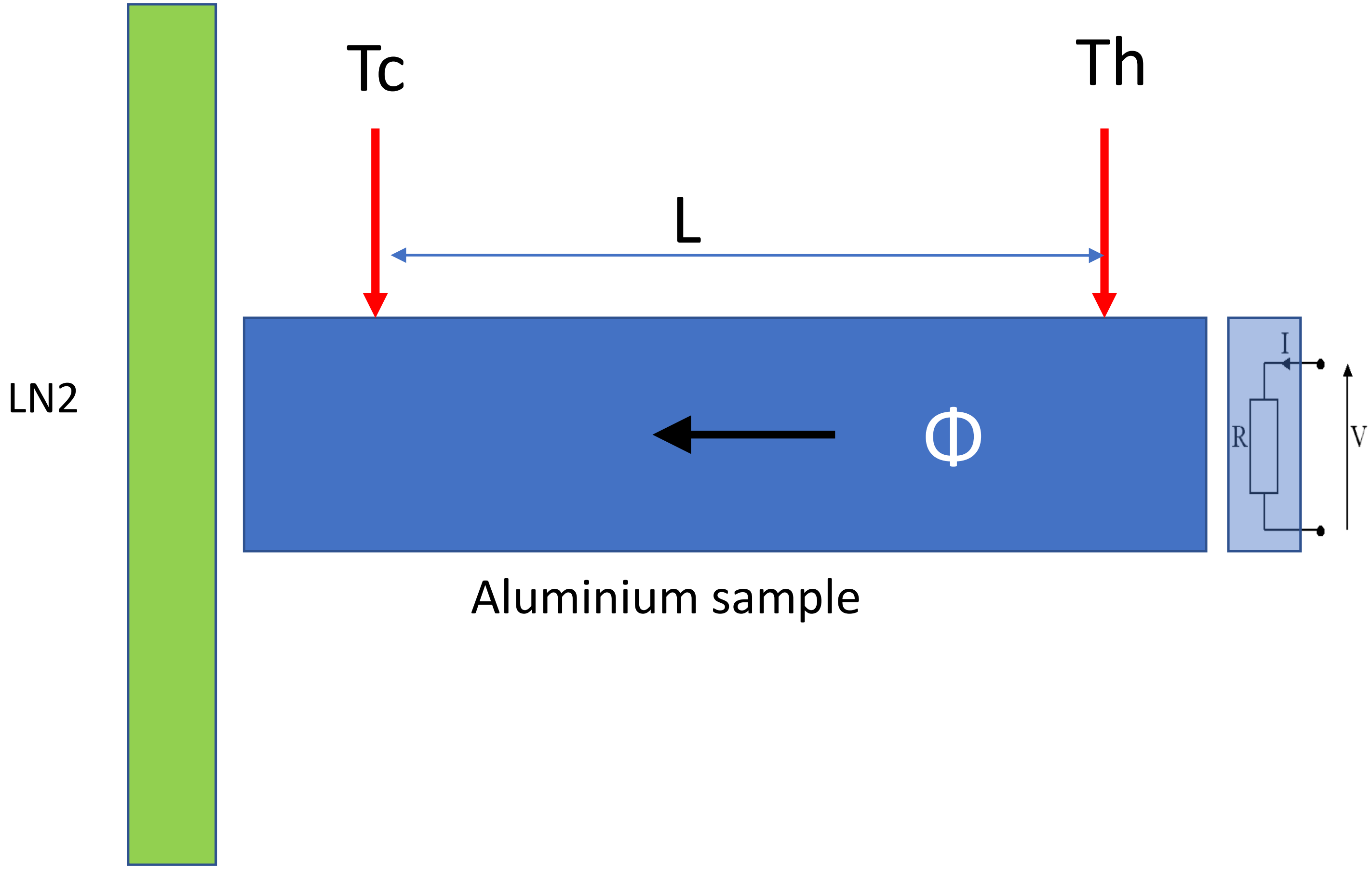
Liquid hydrogen is now an important subject of development.

This study concerns the development of a device for characterizing thermal conductivity at low temperature

Measurement of the thermal conductivity at cryogenic temperature

- Measuring principle
- Measurements on metals
- Measurements on conductive films
- Measurement of thermal conductivity and heat capacity on composites

Measuring principle



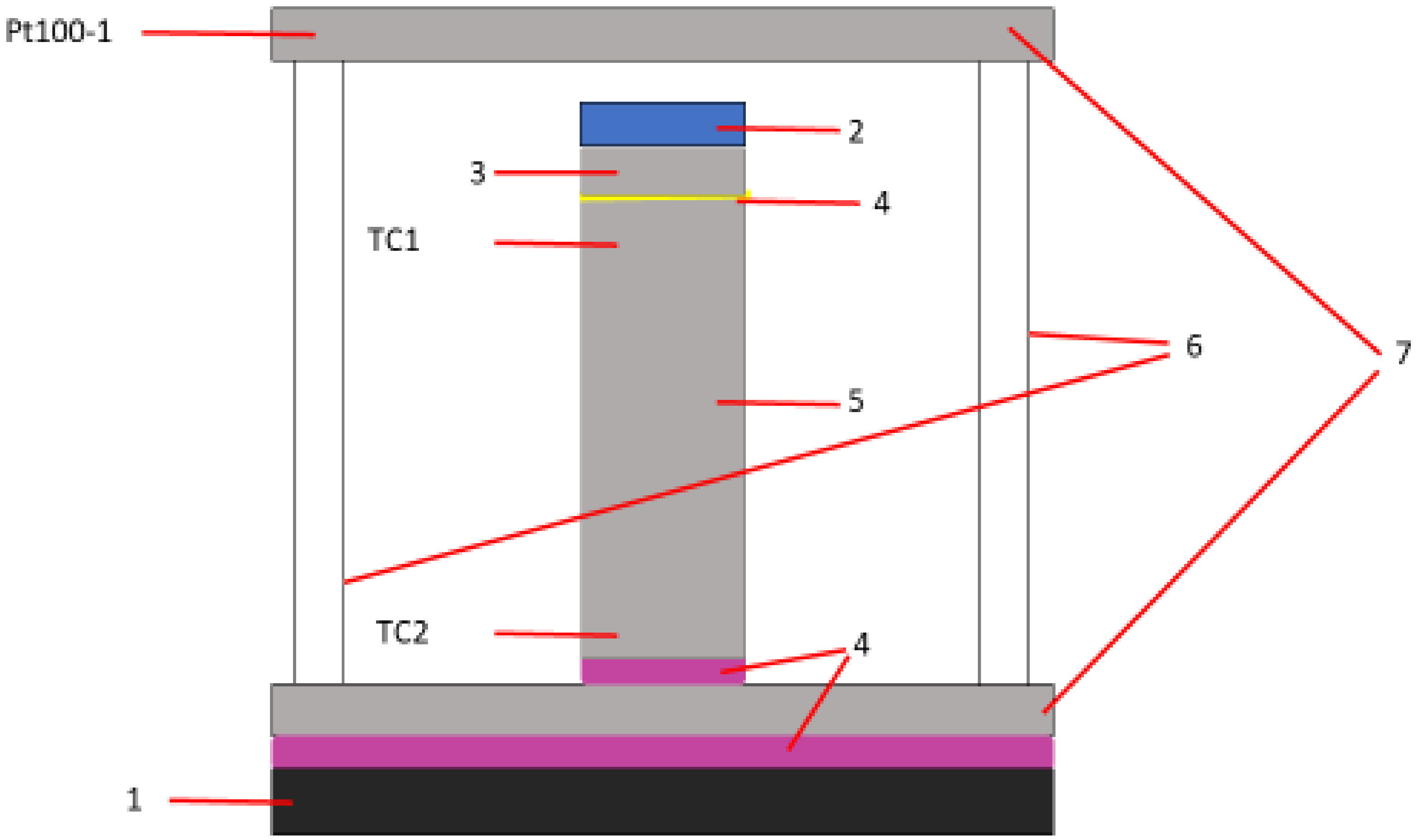
The principle consists of heating an aluminium rod at one end with a constant power setting and measuring the thermal gradient along the rod. The expression for the thermal conductivity λ is as follows:

$$\lambda = \frac{L * P}{S * \Delta T}$$

Where P=V.I and ΔT=Th-Tc

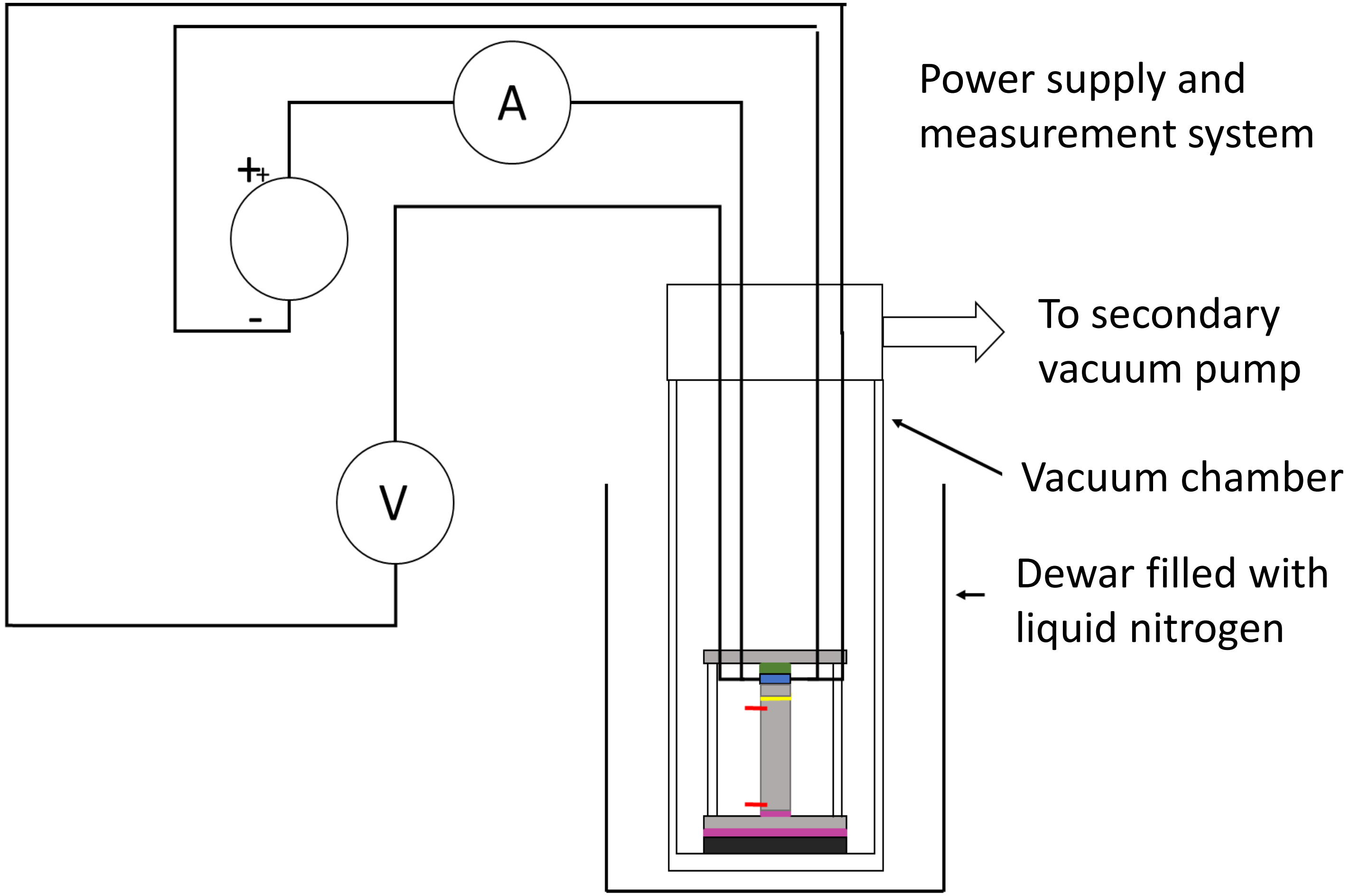
Where L is the measurement length, S is the cross-sectional area of the rod, P is the power reading and ΔT is the temperature difference at the rod ends.

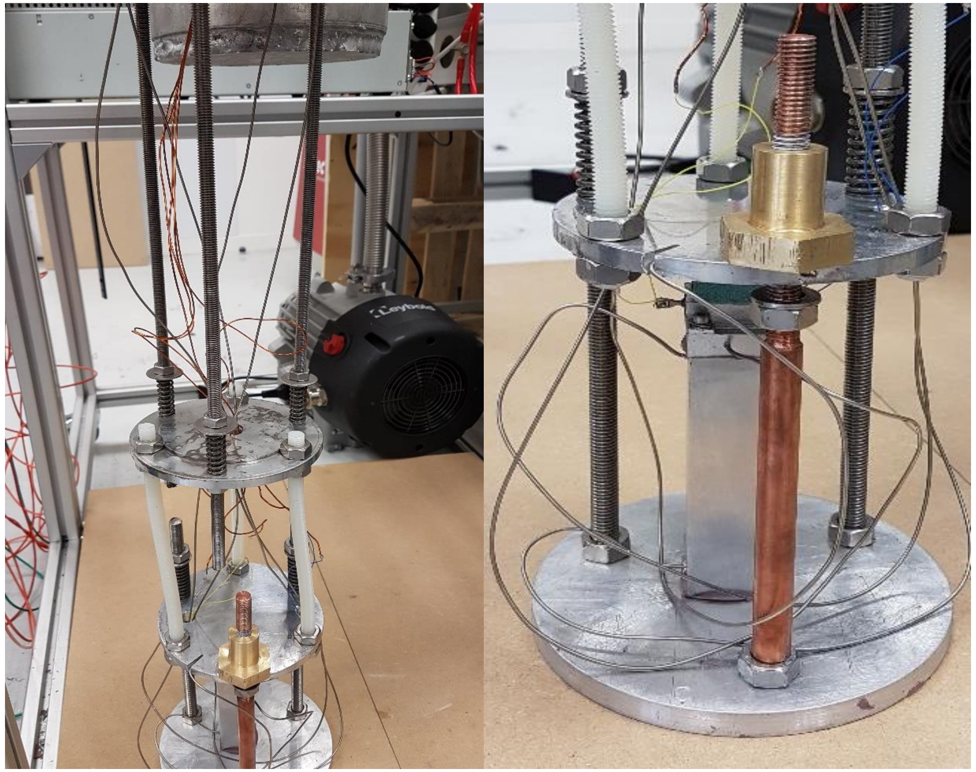
Measuring principle



TC1	Measurement of the hot rod temperature
TC2	Measurement of the cold bar temperature
Pt100-1	Temperature control measurement of the thermal guard
1	Aluminium vacuum tank bottom
2	Heating resistor
3	Aluminium block ensures good thermal contact with the sample
4	Thermal paste (0.5mm thick, λ=6 $W.m^{-1}.K^{-1}$)
5	SAMPLE
6	Threaded rods
7	Aluminium plates

Measuring principle

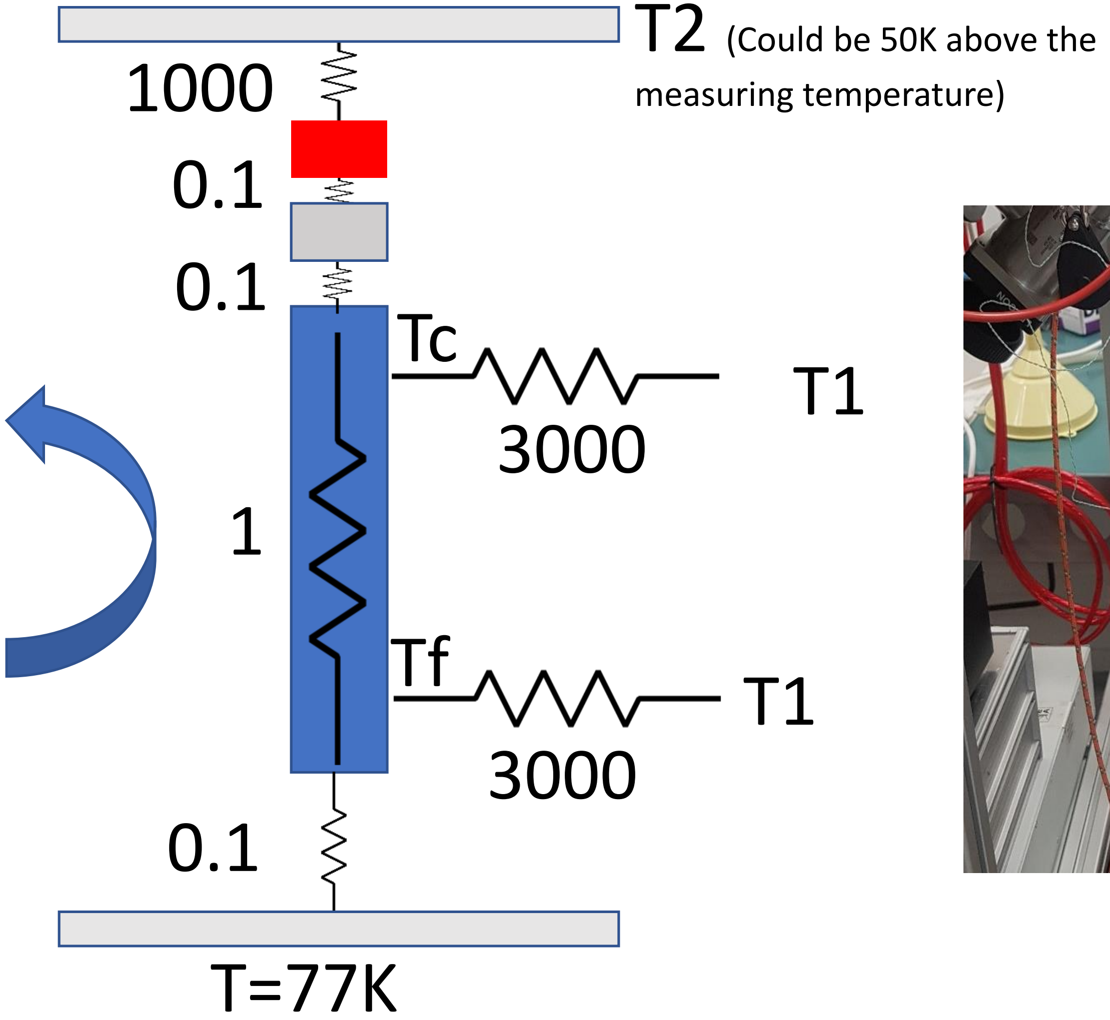




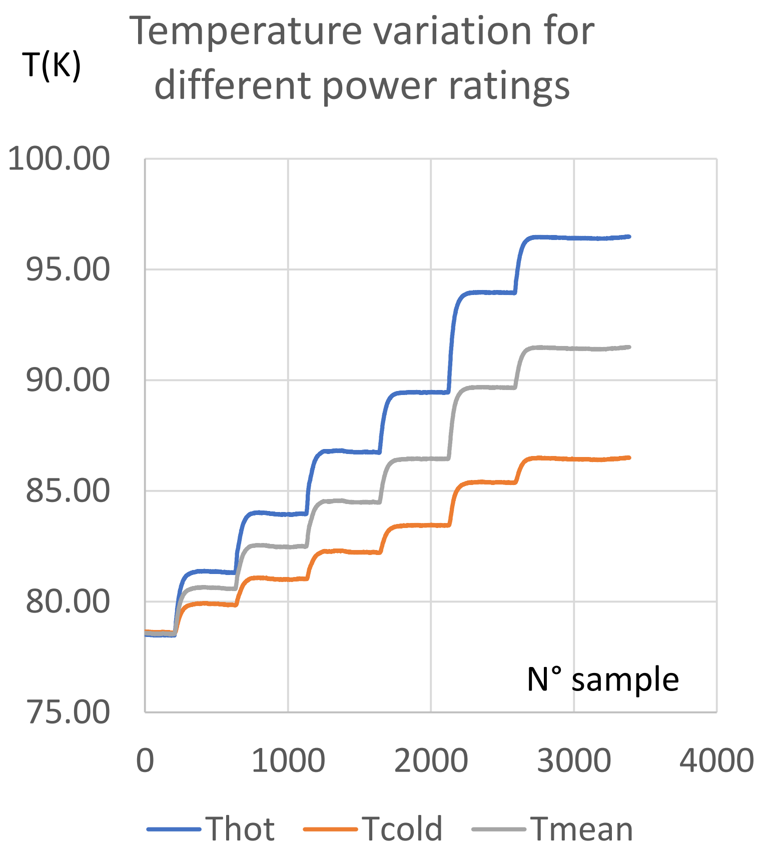
Measuring principle

Relative thermal resistances

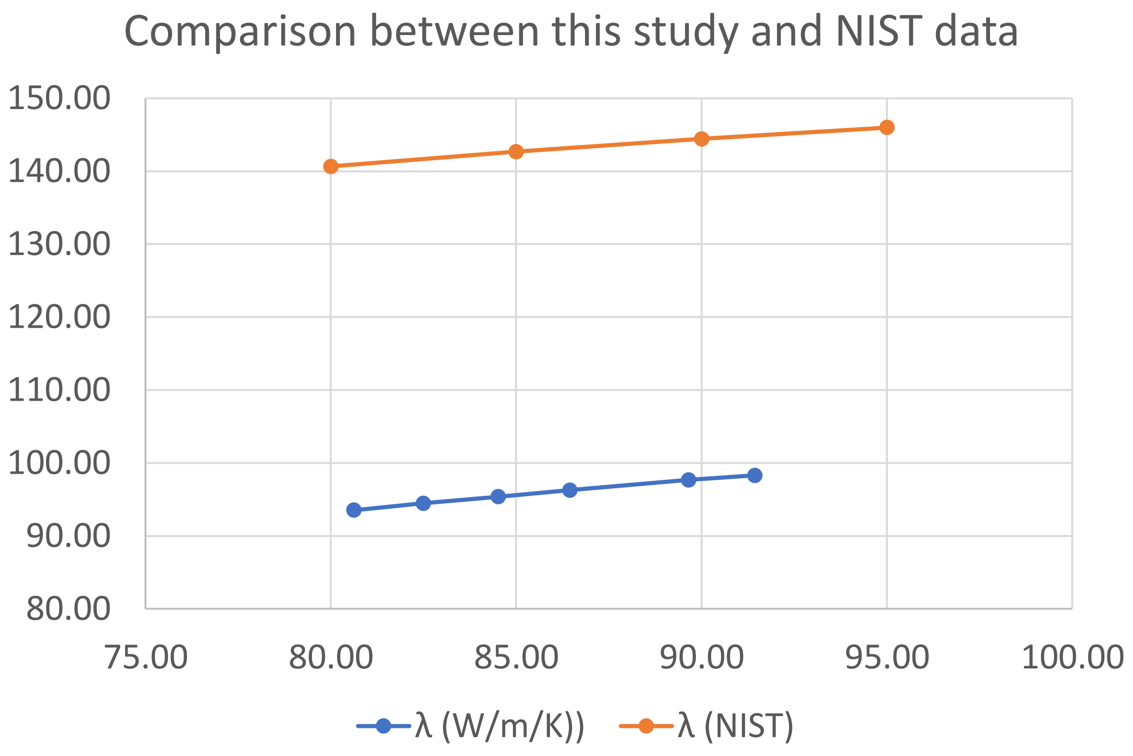
Convection= h. ΔT.S



Measurement on an aluminium grade

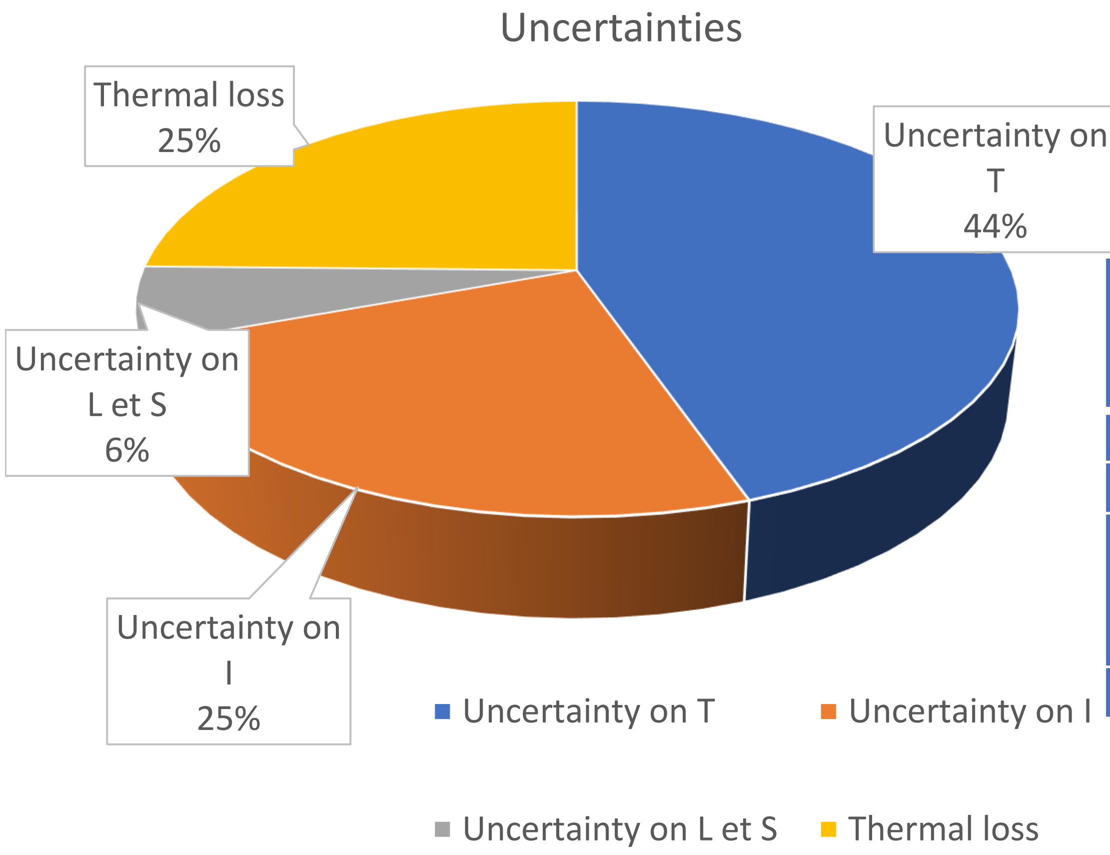


T(K)	λ (W/m/K))
80.62	93.53
82.50	94.46
84.51	95.37
86.45	96.27
89.65	97.67
91.43	98.28



Measurement on an aluminium grade

Uncertainties



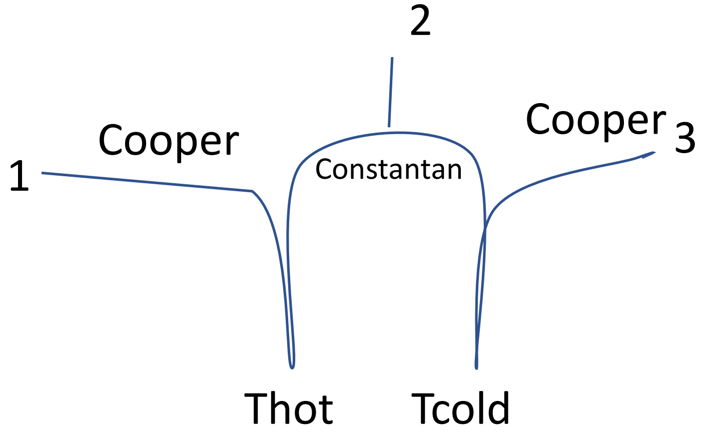
λ (W/m/K)	uncertainty ΔT ($W.m^{-1}.K^{-1}$)	uncertainty I (W.m-1.K-1)	uncertainty L and S (W.m-1.K-1)	heat loss (W.m-1.K-1)
119.10	2.16	1.19	0.29	1.19
	Uncertainty total (W.m-1.K-1)	Total uncertainty (%)		
	2.8	2.3		

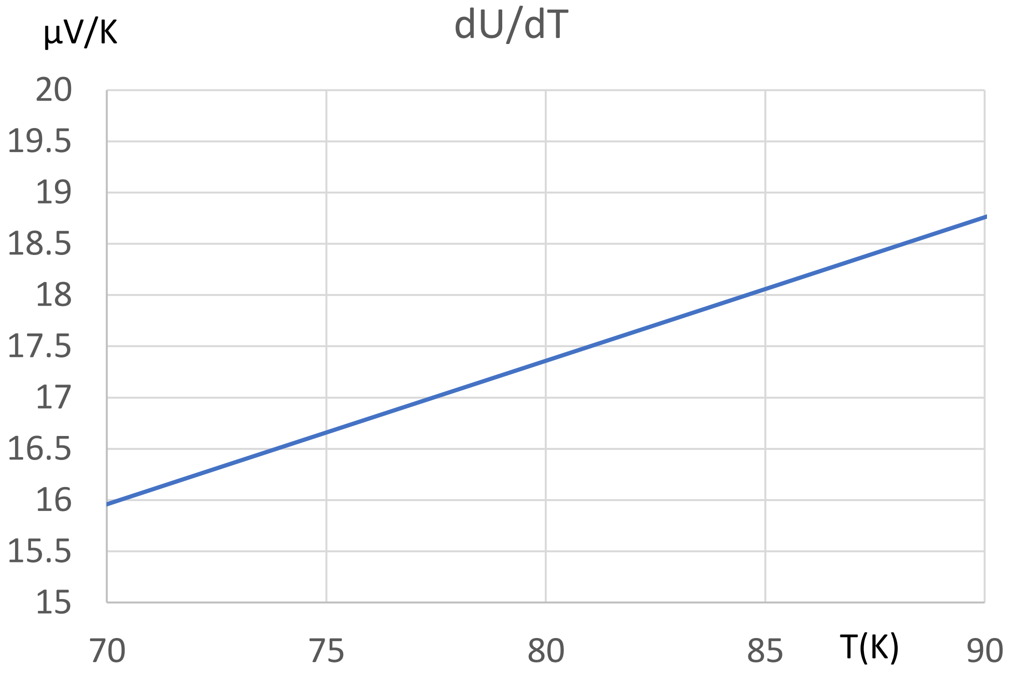
Measurement on an aluminium grade

Thermocouple type T

Uncertainties

T(K)	U(T) (µV)
83.15	-5439
93.15	-5261
103.15	-5069
113.15	-4865
123.15	-4648
133.15	-4419
143.15	-4177





We need to measure a temperature difference but we need to know the conversion coefficient for the temperature difference

U12->Thot
U23-> Tcold
U13->Thot-Tcold

Thot-Tcold=U13/dU/dT)

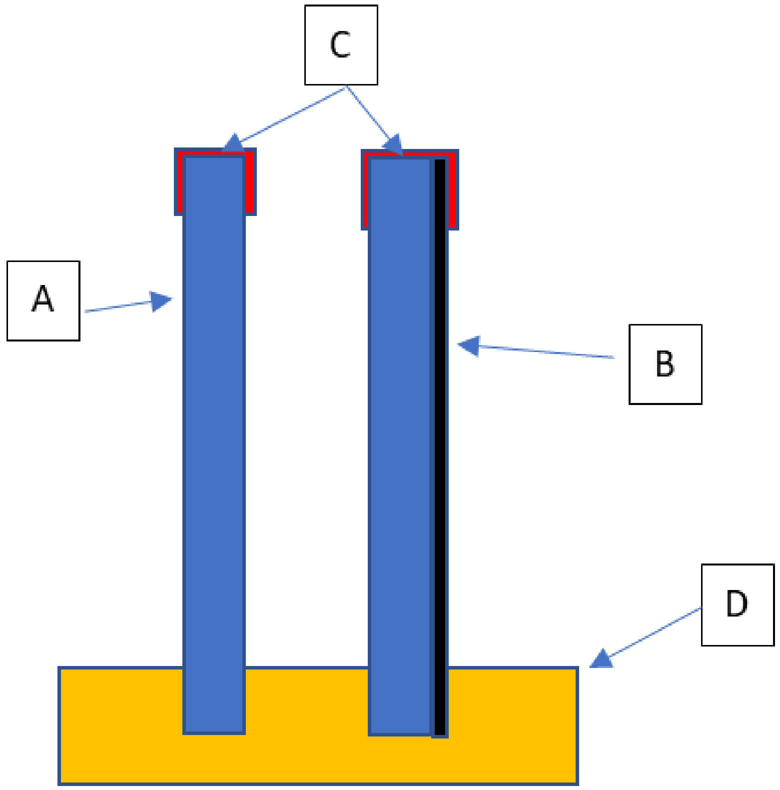
1K error in absolute temperature = 1% error in conductivity

Measurement on an aluminium grade

Conclusion

- It is possible to measure thermal conductivity at low temperatures with an uncertainty of better than 3%. To do this, the temperature measurement chain must be mastered
- Metallic materials vary in conductivity by more than 30% depending on annealing or thermal history
- The data in the bibliography are not sufficient given the variability of the parameter measured

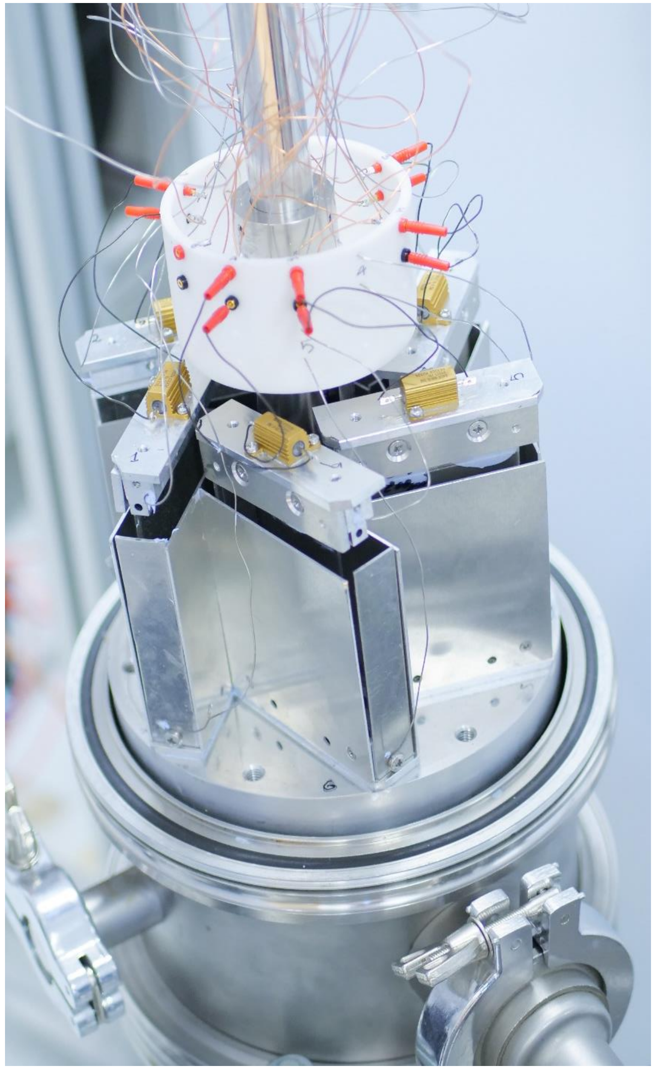
Measurement on composites

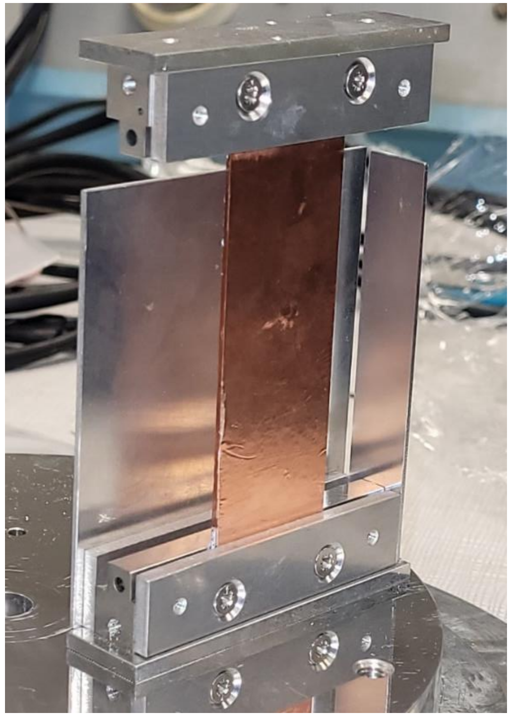


A	Glass slide
B	Glass slide with film to measure
C	Heater
D	Bulk at constant temperature

The aim of this experiment is to determine the thermal conductivity of the deposited film to increase the thermal conductivity of the support.

Measurement on composites





Measurement on composites

The conduction in the glass slide is :

$$P = \frac{\lambda \cdot S}{l} \cdot \Delta T$$

Where P is the injected power, λ the conductivity of the glass, S the section of the glass blade and ΔT the temperature difference between the upper and lower part

$$C_{verre} = \lambda \cdot S = \frac{P \cdot l}{\Delta T}$$

Measurement on composites

The power passing through a sample placed on a glass plate is expressed:

$$P = \frac{\lambda_{echantillon} \cdot S_{échantillon} + C_{verre}}{l_{echantillon}} \cdot \Delta T$$

We can write :

$$C_{échnatillon=}\lambda_{echantillon} \cdot S_{échantillon} = \frac{P \cdot l_{echantillon}}{\Delta T} - C_{verre}$$

The conductivity is :

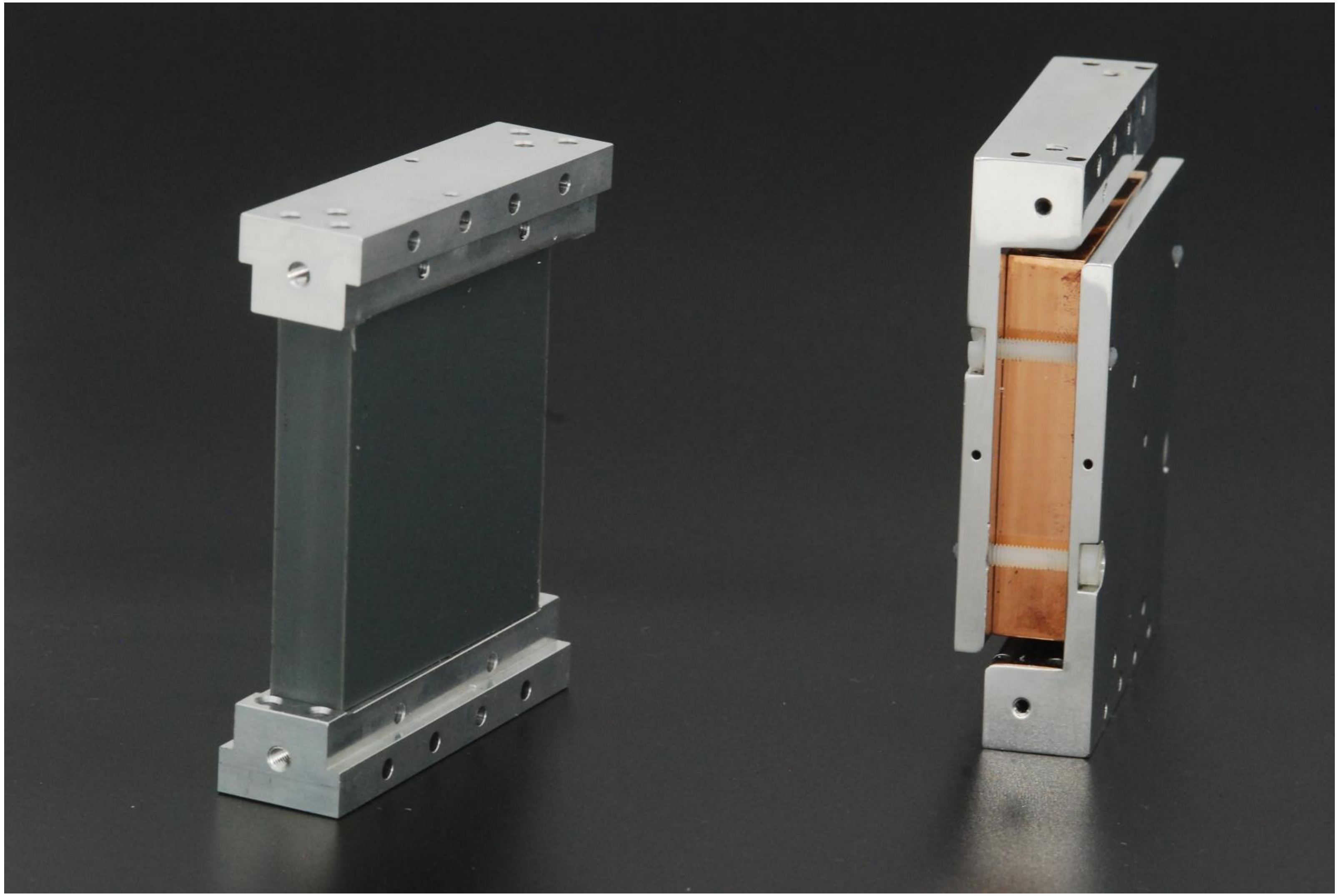
$$\lambda_{echantillon} = \frac{C_{échnatillon}}{S_{échantillon}}$$

Measurement on composites

	Graphite	Cu	C3	Glass
C-brut (P*l/ΔT) (W.m/K)	1.06E-02	6.72E-04	4.91E-04	4.63E-04
Longueur (mm)	65.21	65.8	65.55	65.6
Epaisseur (m)	9.00E-04	1.80E-05	2.00E-04	
Largeur (m)	0.0506			
C corrigé (W.m/K)	9.83E-03	0.0002087	2.7854E-05	
λ(W/m/K)	215.8	463.8	5.571	

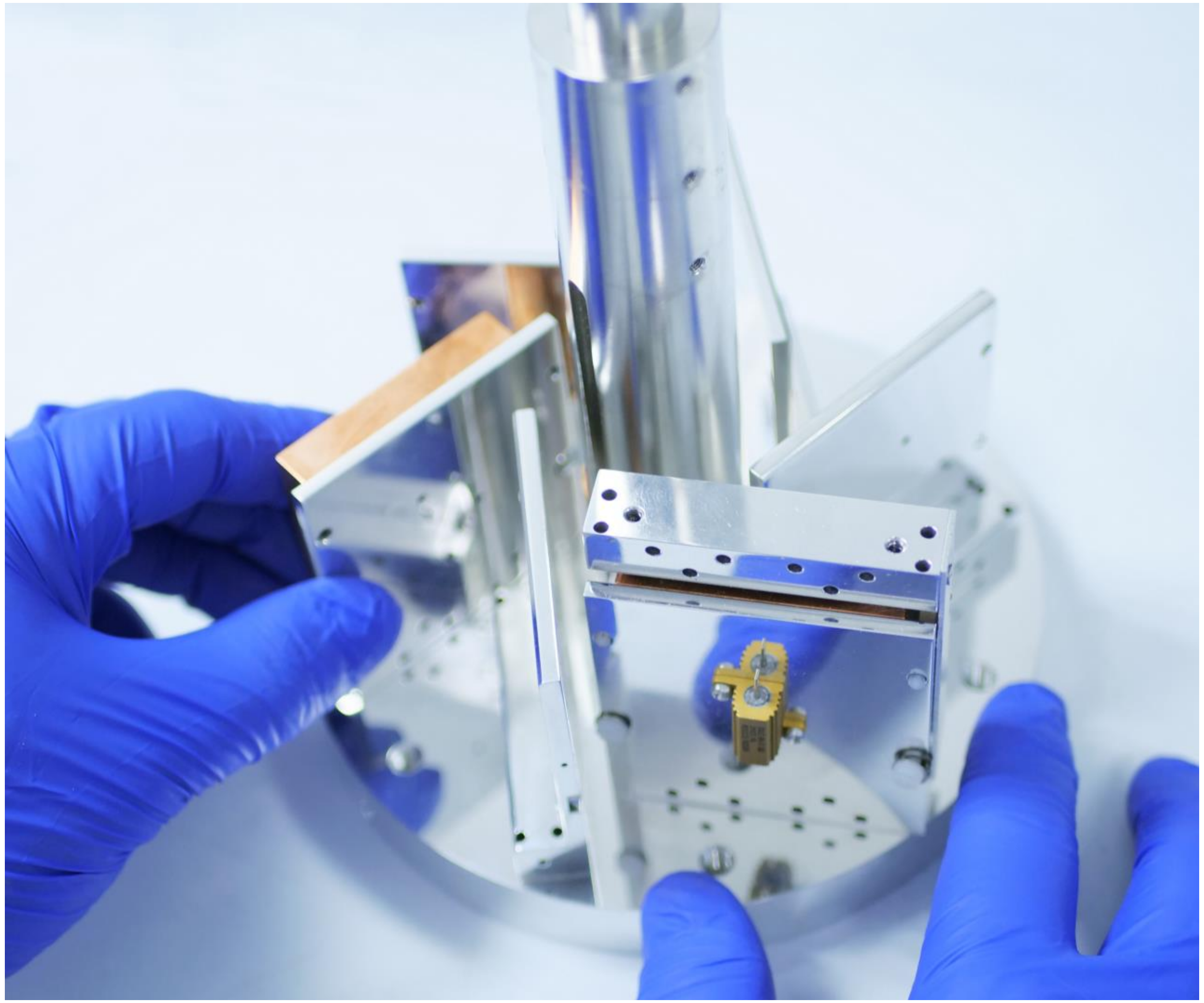
- C3 is a polymer loaded with carbon nano-tube
- The graphite is a commercial form of compacted graphite

Measurement of thermal conductivity and heat capacity on composites

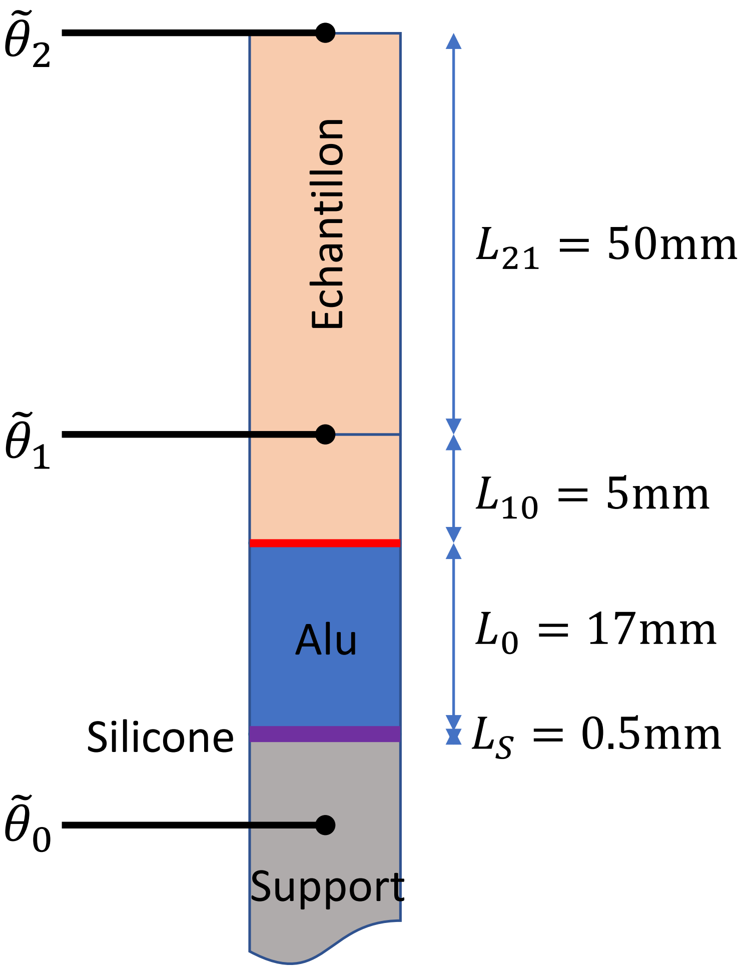


Epoxy carbon composites are anisotropic and it is necessary to measure in both directions.
Two configurations are required.

Measurement of thermal conductivity and heat capacity on composites



Measurement of thermal conductivity and heat capacity on composites



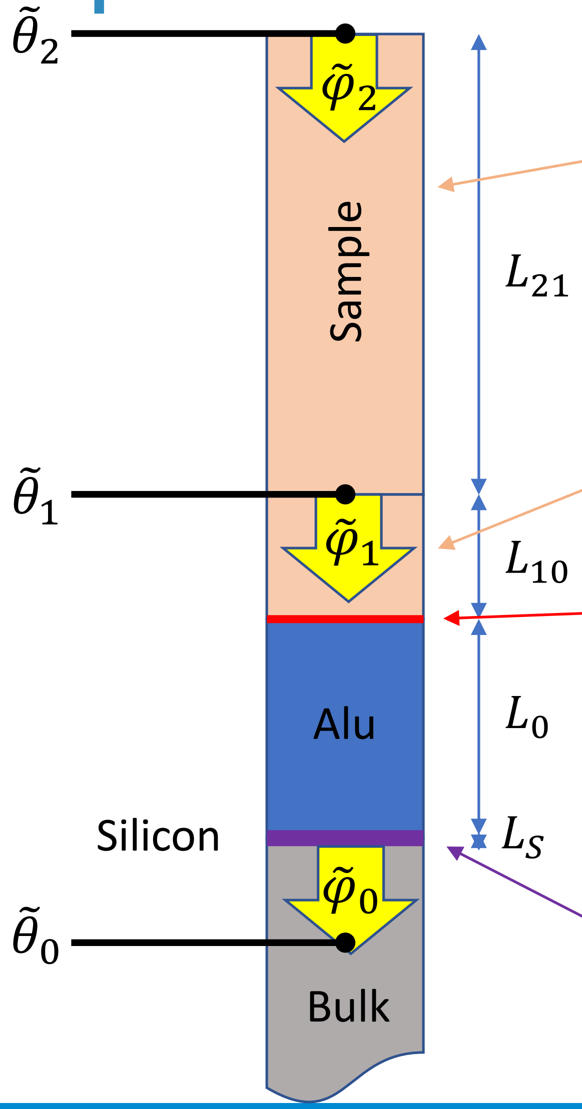
Hypotheses:

- 1D transfers
- No lateral losses
- Isothermal support ($\tilde{\theta}_0 = 0$)
- Silicone = material without inertia = thermal resistance
- Thermal contact resistance “sample-aluminum”: Rc = 10-4 m².K.W-1
- Negligible probe contact resistance

Valeurs utilisées (cas à 77K)

Material	$\lambda\ (\mathrm{W.m^{-1}.K^{-1}})$	$\rho\ (\mathrm{kg.m^{-3}})$	$c_p\ (\mathrm{J.kg^{-1}.K^{-1}})$	$a\ (\mathrm{m^2.s^{-1}})$
Sample (1)	4	1730	940	2.46×10^{-6}
Aluminum (0)	83.5	2700	348	88.87×10^{-6}
Filled silicone	3	-	-	-

Measurement of thermal conductivity and heat capacity on composites



$$Q_{21} = \begin{pmatrix} \cosh(\alpha_1 L_{21}) & \dfrac{\sinh(\alpha_1 L_{21})}{\lambda_1 \alpha_1} \\ \lambda_1 \alpha_1 \sinh(\alpha_1 L_{21}) & \cosh(\alpha_1 L_{21}) \end{pmatrix}$$

Avec $\alpha_1 = \sqrt{\dfrac{j\omega}{a_1}}$

$$Q_{10} = \begin{pmatrix} \cosh(\alpha_1 L_{10}) & \dfrac{\sinh(\alpha_1 L_{10})}{\lambda_1 \alpha_1} \\ \lambda_1 \alpha_1 \sinh(\alpha_1 L_{10}) & \cosh(\alpha_1 L_{10}) \end{pmatrix}$$

$$Q_{Rc} = \begin{pmatrix} 1 & R_c \\ 0 & 1 \end{pmatrix}$$

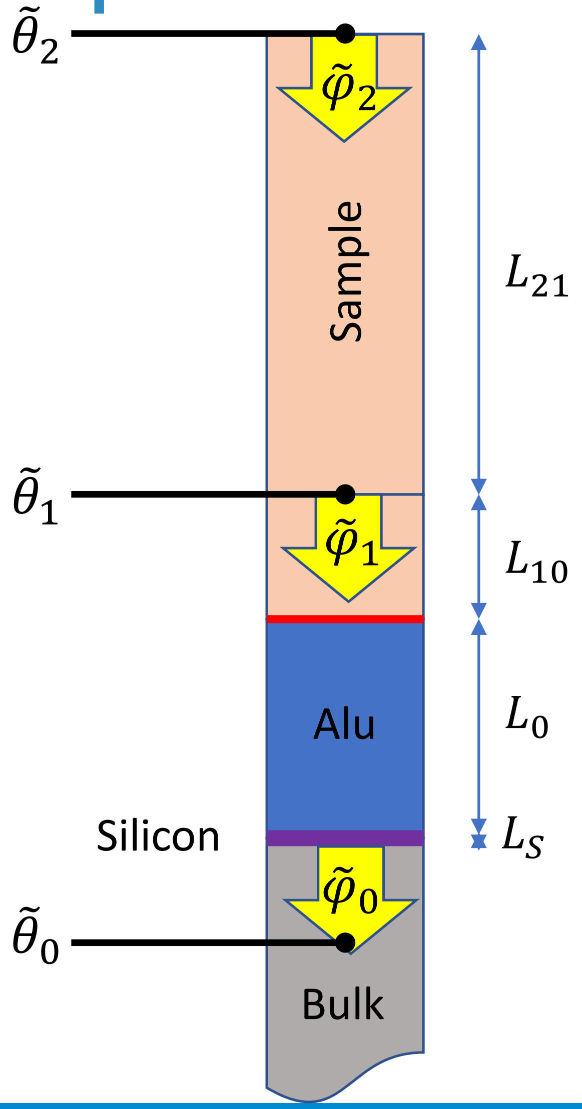
$$Q_0 = \begin{pmatrix} \cosh(\alpha_0 L_0) & \dfrac{\sinh(\alpha_0 L_0)}{\lambda_0 \alpha_0} \\ \lambda_0 \alpha_0 \sinh(\alpha_0 L_0) & \cosh(\alpha_0 L_0) \end{pmatrix}$$

Avec $\alpha_0 = \sqrt{\dfrac{j\omega}{a_0}}$

$$Q_{Rs} = \begin{pmatrix} 1 & R_S \\ 0 & 1 \end{pmatrix}$$

Avec $R_S = \dfrac{L_S}{\lambda_S}$

Measurement of thermal conductivity and heat capacity on composites



Quadripole chain T_2 to T_0:

$$\begin{pmatrix}\tilde{\theta}_2\\ \tilde{\varphi}_2\end{pmatrix} = [M_{20}] \times \begin{pmatrix}\tilde{\theta}_0\\ \tilde{\varphi}_0\end{pmatrix} = [M_{20}] \times \begin{pmatrix}0\\ \tilde{\varphi}_0\end{pmatrix}$$

With: $[M_{20}] = \begin{pmatrix}A_{20} & B_{20}\\ C_{20} & D_{20}\end{pmatrix} = [Q_{21}] \times [Q_{10}] \times [Q_{Rc}] \times [Q_0] \times [Q_{Rs}]$

We obtain: $\tilde{\theta}_2 = B_{20} \times \tilde{\varphi}_0$

Transfer function calculation

From T_1 to T_0:

$$\begin{pmatrix}\tilde{\theta}_1\\ \tilde{\varphi}_1\end{pmatrix} = [M_{10}] \times \begin{pmatrix}\tilde{\theta}_0\\ \tilde{\varphi}_0\end{pmatrix} = [M_{10}] \times \begin{pmatrix}0\\ \tilde{\varphi}_0\end{pmatrix}$$

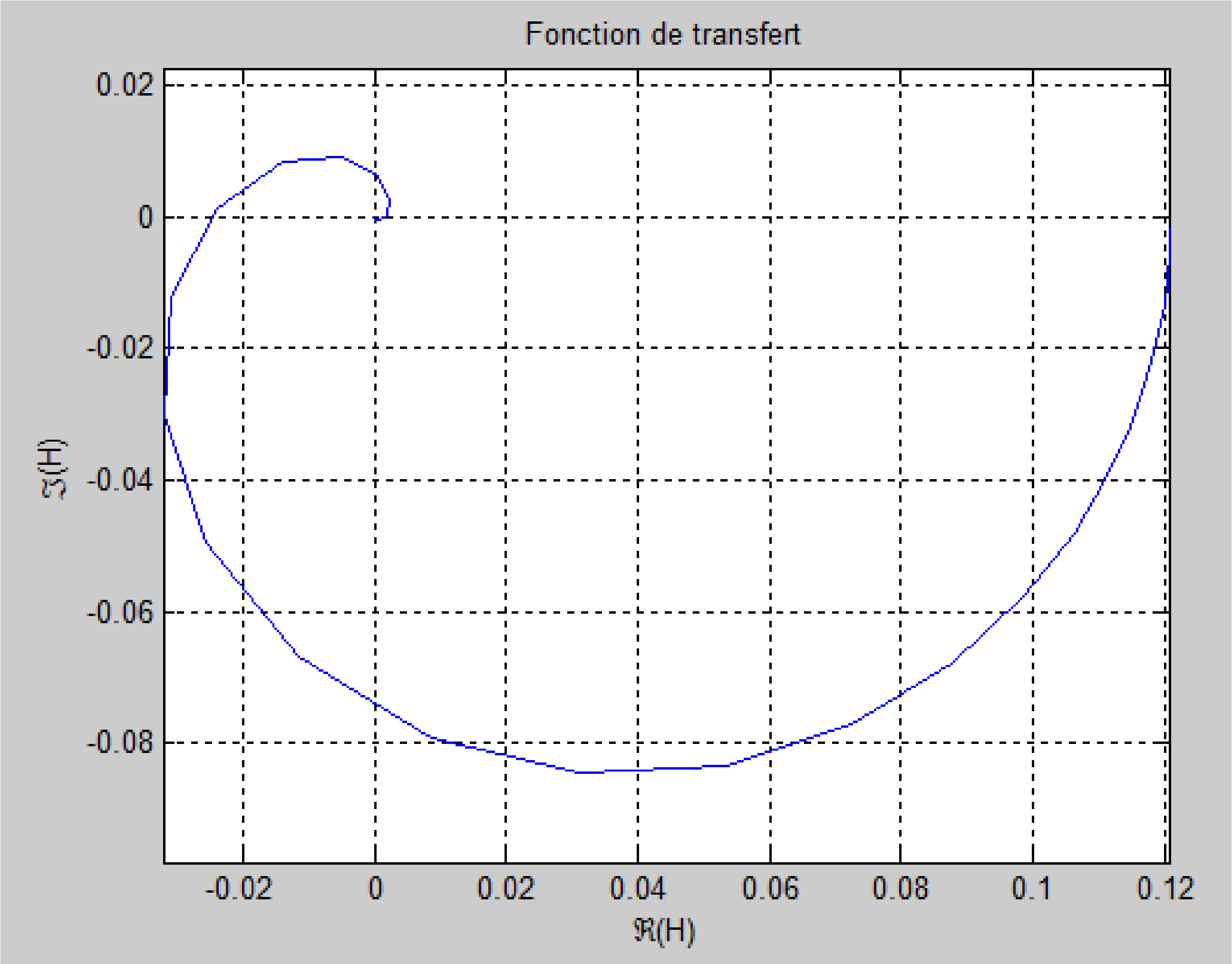
With: $[M_{10}] = \begin{pmatrix}A_{10} & B_{10}\\ C_{10} & D_{10}\end{pmatrix} = [Q_{10}] \times [Q_{Rc}] \times [Q_0] \times [Q_{Rs}]$

We obtain: $\tilde{\theta}_1 = B_{10} \times \tilde{\varphi}_0$

Transfer function

$$H(f) = \frac{\tilde{\theta}_1}{\tilde{\theta}_2} = \frac{B_{10}}{B_{20}}$$

Measurement of thermal conductivity and heat capacity on composites



For data at 77K

From:

$$10^{-5}\text{Hz} \leq f \leq 10^{-1}\text{Hz}$$

"Static" verification: estimation of $\lambda 1$ from the value of the low-frequency transfer function

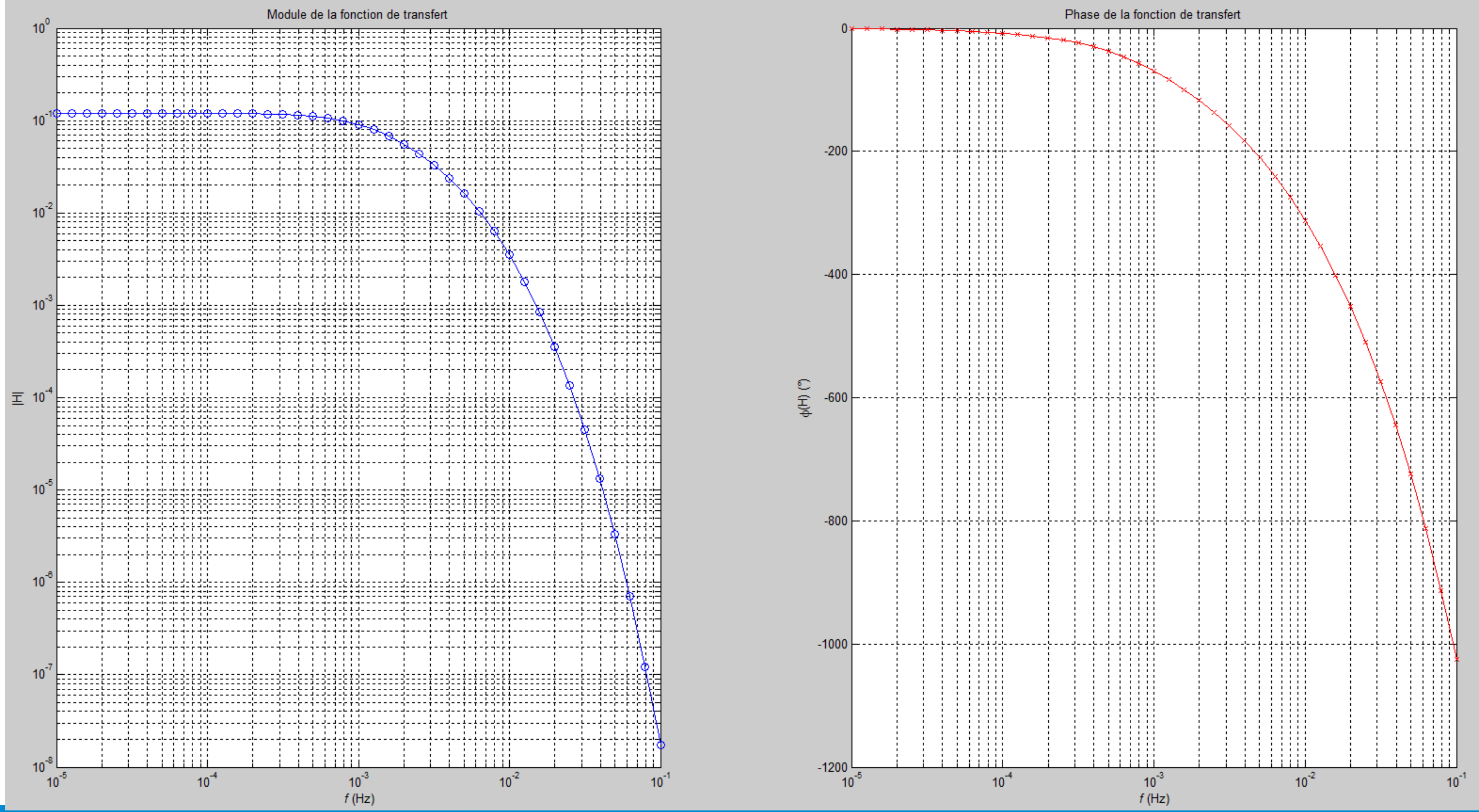
$$\lambda_{1,estim} = \frac{L_{21}}{(R_{10} + R_c + R_0 + R_s) \times \left(\frac{1}{\Re(H(0))} - 1\right)}$$

We obtain:

$$\lambda_{1,estim} = 3.9994\ \text{W.m}^{-1}.\text{K}^{-1}$$

Instead of: $\lambda_1 = 4\ \text{W.m}^{-1}.\text{K}^{-1}$

Measurement of thermal conductivity and heat capacity on composites



For data at 77K

Measurement of thermal conductivity and heat capacity on composites

Sensitivity study

For data at 77K

Vector of parameters to consider (9 parameters in total):

$$\boldsymbol{p} = \{\lambda_1; a_1; L_{21}; L_{10}; R_c; \lambda_0; a_0; L_0; R_S\}$$

Calculation of reduced sensitivity coefficients:

$$C_i = p_i \times \frac{\partial H}{\partial p_i}$$

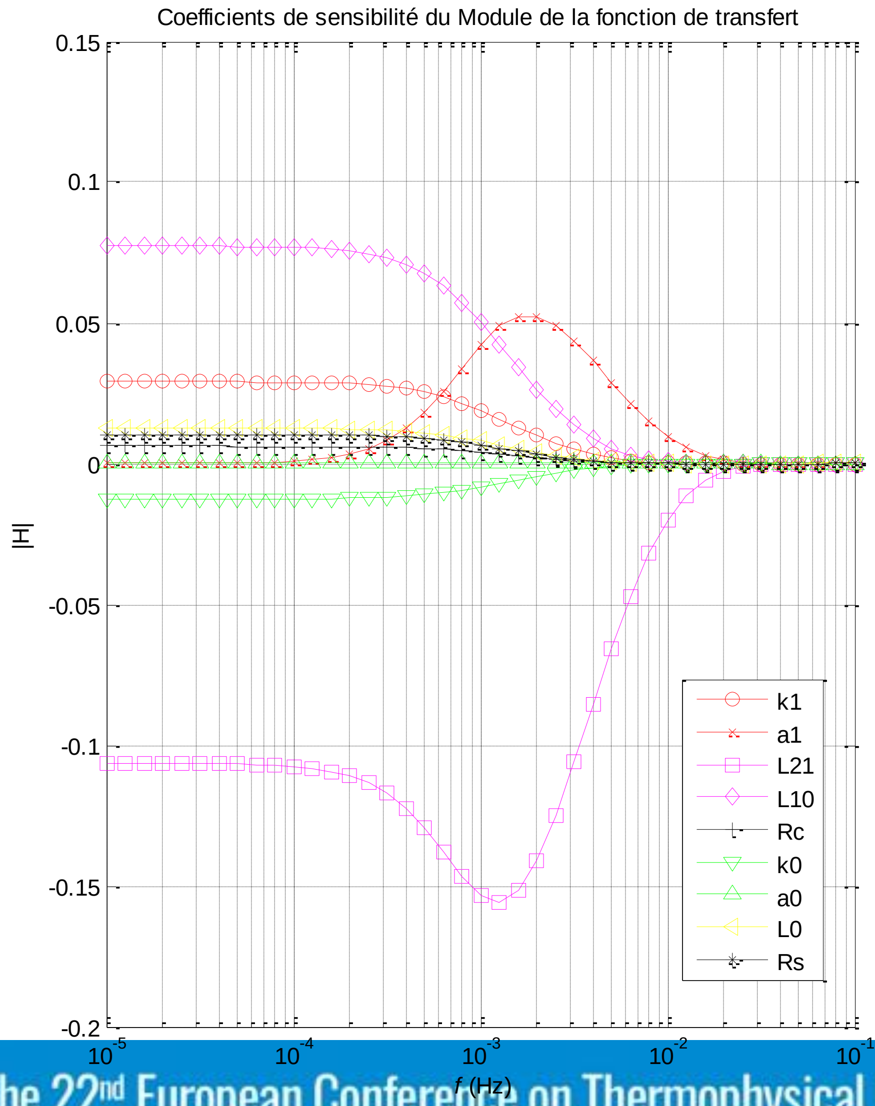
As H is not analytically differentiable "simply", we approximate the sensitivity coefficients from the calculation of the transfer function for two values p_i^+ and p_i^- of the parameter p_i:

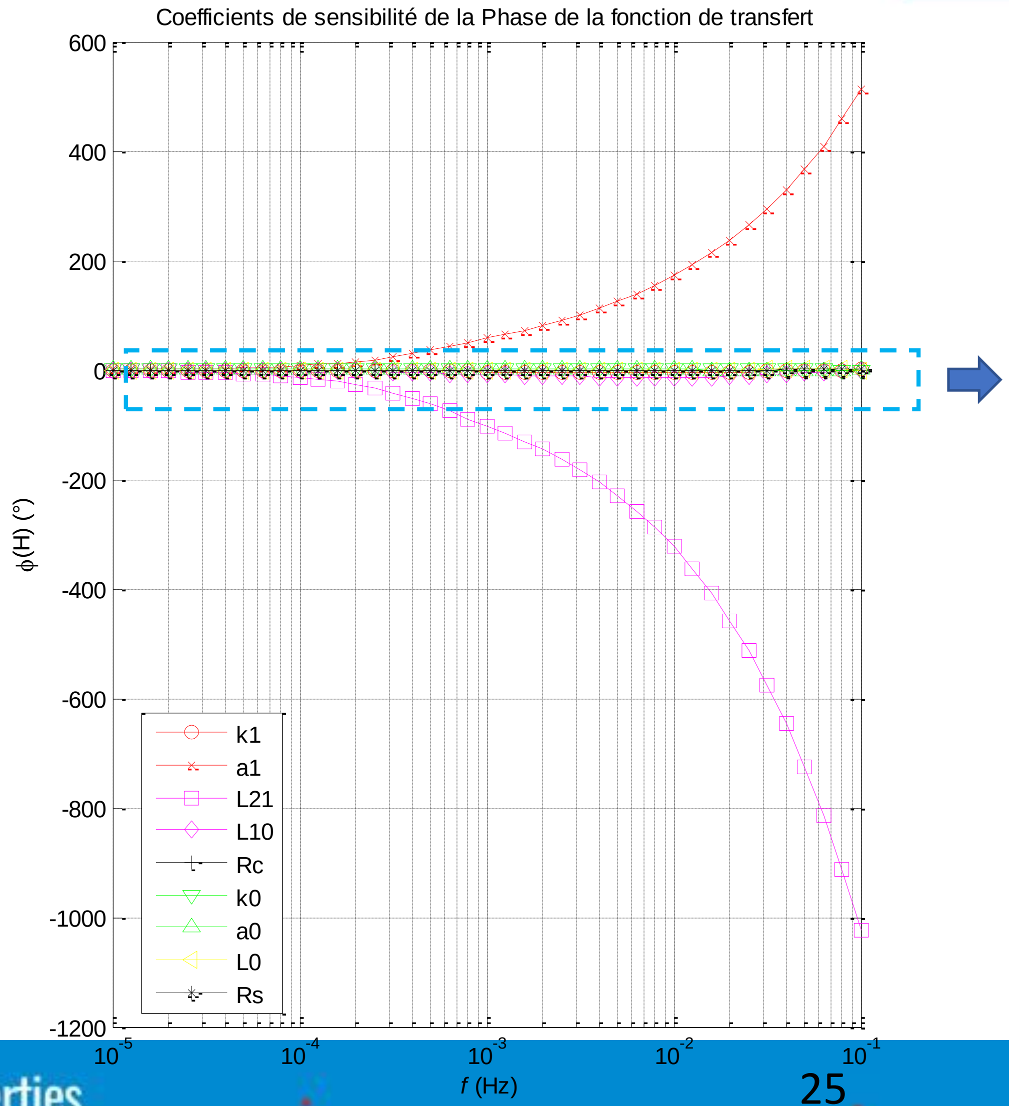
$$C_i \cong p_i \times \frac{H(p_i^+) - H(p_i^-)}{p_i^+ - p_i^-}$$

$p_i^+ = 1.01 \times p_i$ *and* $p_i^- = 0.99 \times p_i$

Measurement of thermal conductivity and heat capacity on composites

Sensitivity study





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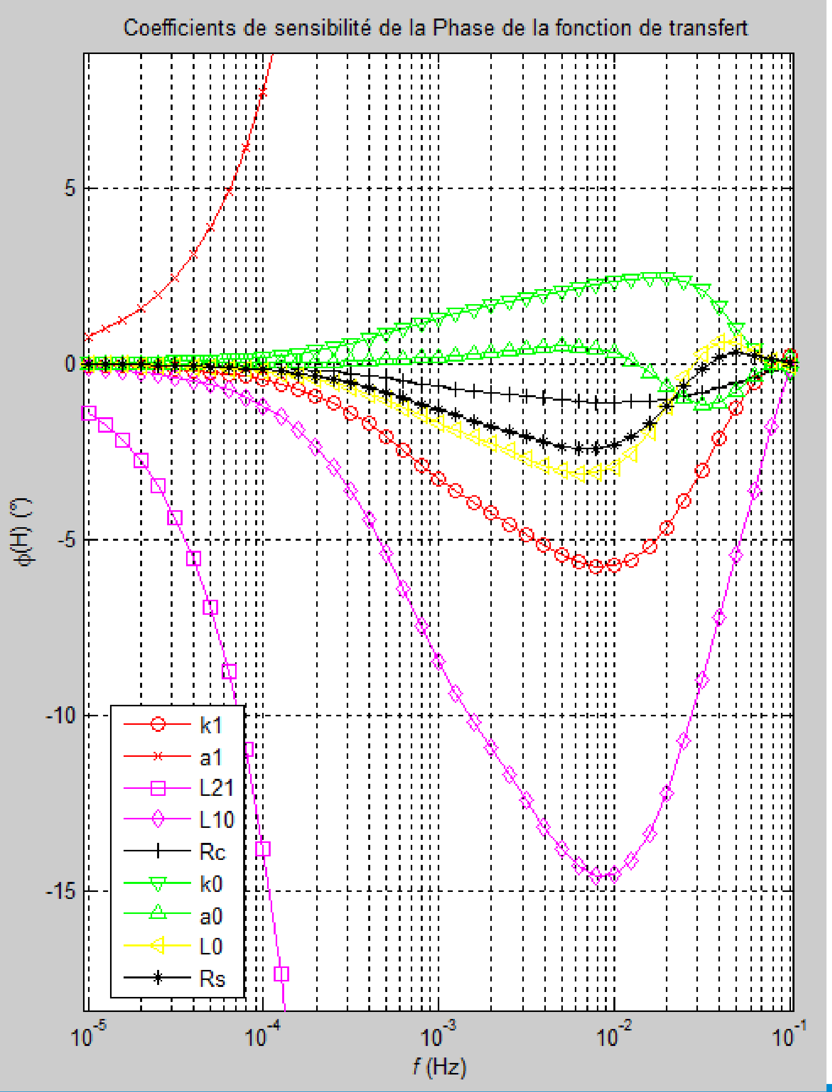
Measurement of thermal conductivity and heat capacity on composites



- Modulus of transfer function :
  - Mainly sensitive to L_{21} et L_{10}
  - Sensitive to diffusivity of sample for central frequency
  - Sensitive to thermal conductivity at low temperature

- for $f > 2 \times 10^{-2}\text{Hz}$, modulus is not sensitive

- Le module de la fonction de transfert n'est pas sensible à la diffusivité thermique de l'aluminium

- Les paramètres suivants sont corrélés:
  - Conductivité thermique de l'échantillon
  - Conductivité thermique de l'aluminium
  - Résistance de contact et Résistance du film silicone
  - Longueurs L_{10} et L_0

- The **phase** of transfer function :
  - sensitive to L_{21} and thermal diffusivity (corelated)
  - Less sensitive to the other parameters
  - Not sensitive for all parameter for $f < 10^{-4}\text{Hz}$

Measurement of thermal conductivity and heat capacity on composites

Conclusion:

- The low-temperature measurement chain needs to be mastered.
- Bibliographic data is not sufficient for modelling systems.
- For certain metals the thermal conductivity changes after having undergone annealing cycles.

Thank you for your attention

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Measurement on an aluminium grade

Uncertainties

Calculation of uncertainties:

- The uncertainty in the voltage has been neglected as it is very small.
- The uncertainty on the current I is taken as 1% according to the characteristics of the device.
- The heat losses taken into account correspond to the conductive and convective exchanges for a primary vacuum, i.e. a convection coefficient equal to 2 $W.m^{-2}.K^{-1}$ ($P=10^{-3}$ mBar)
- The error on temperature difference is take at 0.02°C
- Dimensional uncertainty assessed by taking into account the max and min areas of several section measurements.
- Negligible uncertainty on the distance between the two thermocouple