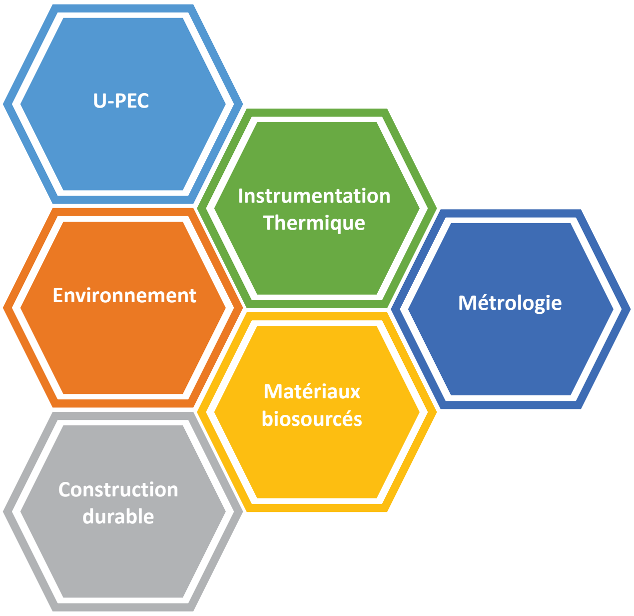

Ecole d'automne du GdR

Transferts hygrothermiques dans les matériaux biosourcés
10 au 14 novembre à la Rochelle

Abderrahim BOUDENNE

Centre d'Études et de Recherche en Thermique Environnement et Systèmes

Université Paris Est Créteil

Caractérisation et modélisation

Métrologie et caractérisation thermophysique & thermohydrique

Modélisation numérique thermophysique & thermohydrique

Echelle du matériau

Echelle de la paroi

Caractérisation et modélisation

Métrologie et caractérisation thermophysique & thermohydrique

Echelle du matériau

Transferts de chaleur et de masse dans les parois biosourcés

Les données d'entrée du modèle ?

Modèle de Kunzel

Transfert d'humidité

$$\xi_\varphi \frac{d\varphi}{dt} = \frac{\partial}{\partial x}\left(\frac{\delta_a}{\mu}\frac{\partial (p_{sat}\varphi)}{\partial x} + D_l \frac{\partial \varphi}{\partial x}\right)$$

Dérivée de la courbe de sorption (ξ_φ)

Facteur de résistance sec (μ)

Région hygroscopique (0% < φ < 95%)

$$D_{l,\varphi} = P_{sat}\delta_a\left(\frac{1}{\mu^*(\varphi)} - \frac{1}{\mu}\right)$$

Facteur de résistance humide

Région capillaire (φ > 95%)

Coefficient d'absorption capillaire

$$D_{l,ws} = \left(3.8\left(\frac{A_c}{W_f}\right)^2 .1000^{\frac{w}{w_f}-1}\right)\xi_\varphi$$

Teneur de saturation libre

Transfert de chaleur

$$(\rho\, c_p + c\, w)\frac{dT}{dt} = \frac{\partial}{\partial x}\left(\lambda \frac{\partial T}{\partial x}\right) - l_v \frac{\partial}{\partial x}\left(\frac{\delta_a}{\mu}\frac{\partial p_{sat}\varphi}{\partial x}\right)$$

Masse volumique et capacité thermique ($\rho\, c_p$)

courbe de sorption (w)

Conductivité thermique (λ)

Transferts de chaleur et de masse dans les parois biosourcés

Les données d'entrée du modèle ?

Modèle de Kunzel

Thermal conductivity	λ(T et HR)
Specific heat	$\rho\ c_p$(T et HR)
Thermal Diffusivity	α(T et HR)=($\lambda/\rho\ c_p$)

Transfert de chaleur

$$(\rho c_p + c_l w)\frac{dT}{dt} = \frac{\partial}{\partial x}\left(\lambda \frac{\partial T}{\partial x}\right) - l_v \frac{\partial}{\partial x}\left(\frac{\delta_a}{\mu}\frac{\partial p_{sat}\varphi}{\partial x}\right)$$

Masse volumique et capacité thermique

Conductivité thermique

Measurement methods

Classification on steady and unsteady states is also manly used

We classify the measurement methods in 2 categories

measurement methods of a single parameter	measurement methods of a several parameters
conductivity diffusivity effusivity specific heat	conductivity & diffusivity effusivity & diffusivity diffusivity & specific heat

Measurement methods

Measurement methods of a single parameter

- **Thermal conductivity**
  - Hot plate method
  - Hot wire method
  - 3w method
- **Thermal diffusivity**
  - Flash method
- **Thermal effusivity**
  - Photoacoustic methods
  - Mirage effect
- **Heat capacity**
  - Differential Scanning Calorimetry

Some examples

most used methods

of course there are other methods and devices

Measurement methods

Measurement methods of a single parameter

- **Thermal conductivity**
  - Hot plate method
  - Hot wire method
  - 3w method
- Thermal diffusivity
  - Flash method
- Thermal effusivity
  - Photoacoustic methods
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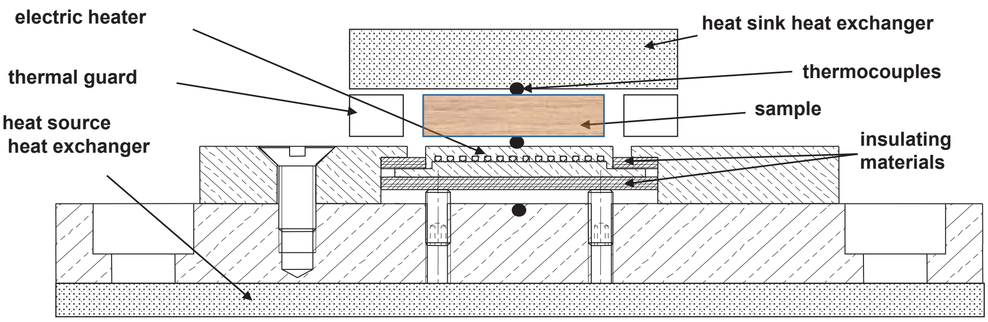
- Heat capacity
  - Differential Scanning Calorimetry

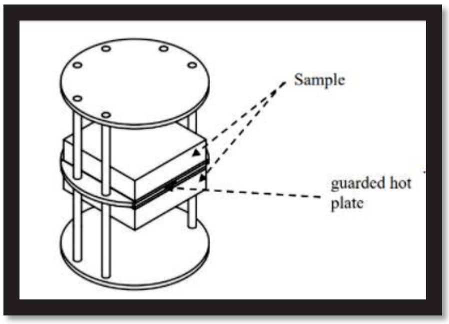
Measurement methods

Measurement methods of a single parameter

Thermal conductivity

Hot-plate method (ISO 8302)





Suitable for *insulating materials*

- Reach the steady state (time of 24 hours in some cases)
- Get one-way flow in the sample
- Measurements of flow and temperature accurately

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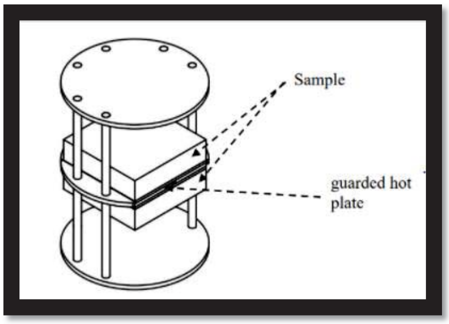
Measurement methods

Measurement methods of a single parameter

Thermal conductivity

Hot-plate method (ISO 8302)



Conductivity is a thermal property which is defined as being the coefficient of proportionality between the heat flux and the temperature gradient:

$$\vec{\phi} = \lambda \cdot \overrightarrow{\mathrm{grad}}(T)$$

When the heat transfers are in one direction the choice is to transcribe in 1D these transfers this heat. The heat equation is therefore written in the following form:

$$\phi = -\lambda \cdot \frac{dT}{dx}$$

If the heat source is a power P uniformly distributed over a surface S we have: $\Phi = \frac{P}{S}$

$$\lambda = \left| \frac{P}{S \cdot \frac{dT}{dx}} \right|$$

Φ is the heat flow in Wm^{-2}
λ the thermal conductivity in $Wm^{-1}.K^{-1}$
T the temperature in K
x the direction of propagation of the heat flow.

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Measurement methods

Measurement methods of a single parameter

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Measurement methods

Measurement methods of a single parameter

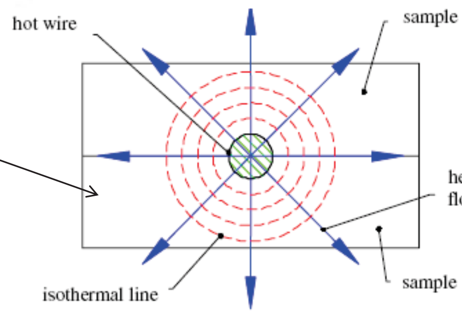
Thermal conductivity

Hot-wire method (ISO 8894)

Developed for measuring the conductivity of liquids

Extended to the characterization of solid

sample



Measurement methods

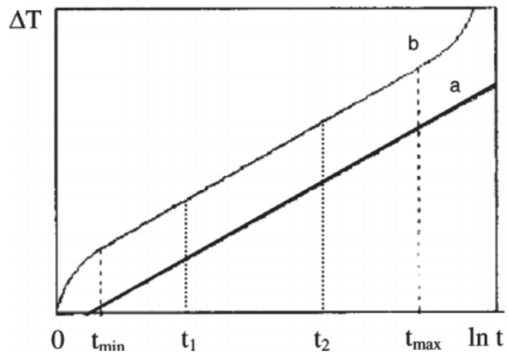
Measurement methods of a single parameter

Thermal conductivity

Hot-wire method (ISO 8894)

The mathematical model is based on the assumption that the hot wire be an infinitely thin and long line heat source. It produces a thermal pulse for a finite time with constant heating power and generates cylindrical coaxial isotherms in an infinite homogeneous medium initially at equilibrium

$$T(r,t) = \frac{Q}{4\pi\lambda}\left[\ln\left(\frac{4\alpha \cdot t}{r^2}\right) + \frac{r^2}{4\alpha t} - \frac{1}{4}\left(\frac{r^2}{4\alpha t}\right) - \cdots - \gamma\right]$$



λ is the thermal conductivity (W/m K);
Q (W/m) is the power supply per unit length of the heating line source;
α is the thermal diffusivity (m^2/s) of the sample;
r is the radial position where the temperature is measured;
$\gamma = 0.5772156$ is Euler's constant.

$$\lambda = \frac{Q}{4\pi[T(t_2) - T(t_1)]} \ln\left(\frac{t_2}{t_1}\right)$$

Measurement methods

Measurement methods of a single parameter

Thermal conductivity

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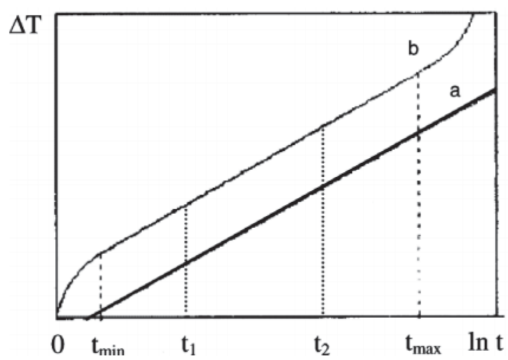

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Measurement methods

Measurement methods of a single parameter

- **Thermal conductivity**
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  - **3w method**
- Thermal diffusivity
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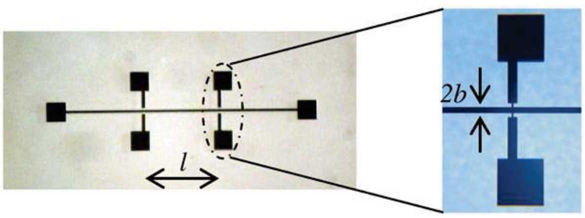
  - Photoacoustic methods
  - Mirage effect
- Heat capacity
  - Differential Scanning Calorimetry

Measurement methods

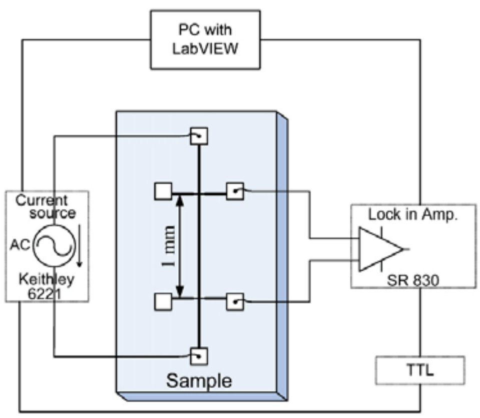
Measurement methods of a single parameter

Thermal conductivity

3-ω method



Photograph of the metal heater on an MgO (100) substrate



- **Applied of an alternating current pulse (ω) in the metal band.**
- **Measurement of a triple pulse (3ω) which will allow to extract the oscillation amplitude and the phase shift compared to the current.**

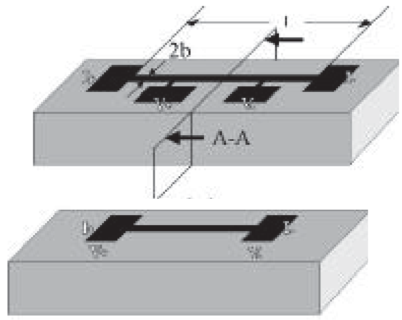
Measurement methods

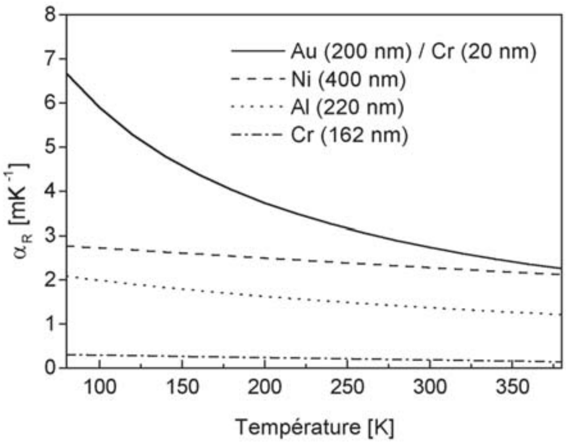
Measurement methods of a single parameter

Thermal conductivity

3-ω method

- The amplitude and phase of the temperature oscillation dependent on the pulsation (ω) of thermal conductivity of the materials under the metal strip.
- The measurement is carried out in the frequency domain.





Measurement methods

Measurement methods of a single parameter

- Thermal conductivity
  - Hot plate method
  - Hot wire method
  - 3w method
- Thermal diffusivity
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- Thermal effusivity
  - Photoacoustic methods
  - Mirage effect
- Heat capacity
  - Differential Scanning Calorimetry

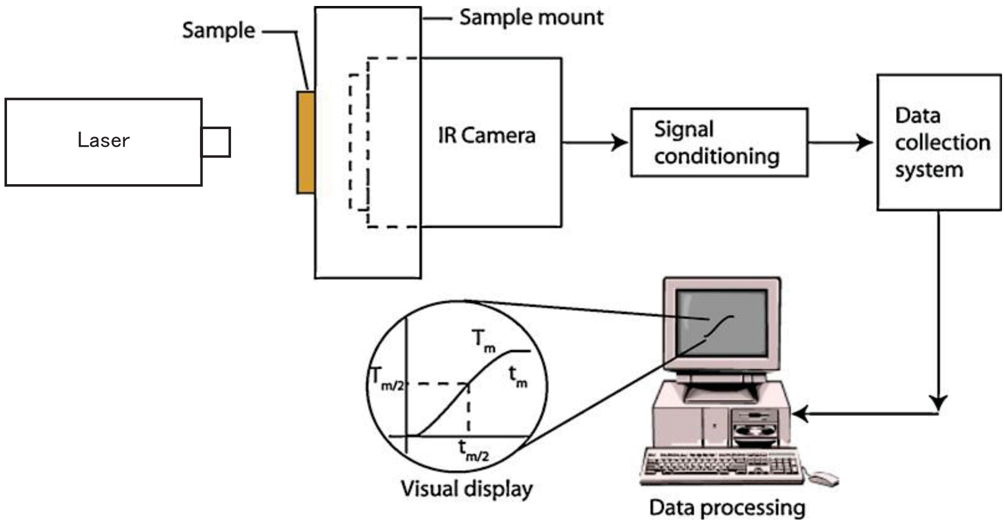
Measurement methods

Measurement methods of a single parameter

Thermal diffusivity

Non-contact measurement

Laser flash method

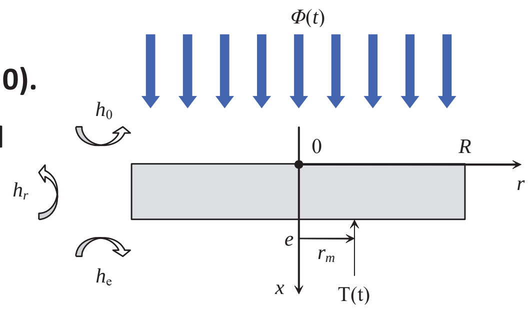


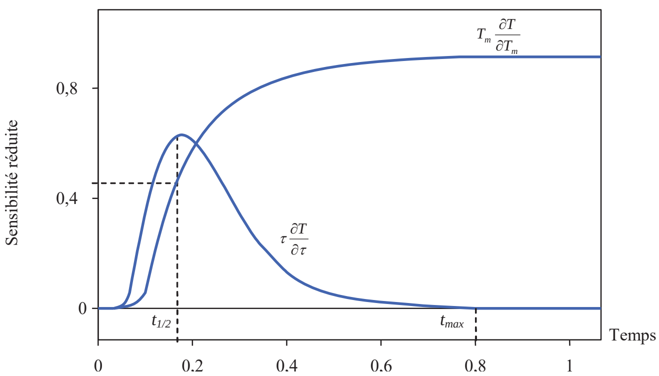
Measurement methods

Measurement methods of a single parameter

Thermal diffusivity

- The measurements are achieved in transient state
- A heat pulse of uniform flux density is imposed on a sample (x = 0).
- The temperature variation of the sample (side x = e) is measured





Thermal diffusivity is deduced by comparing the experimental thermogram with the thermokinetic model

Measurement methods

Measurement methods of a single parameter

- Thermal conductivity
  - Hot plate method
  - Hot wire method
  - 3w method

- Thermal diffusivity
  - Flash method

- Thermal effusivity
  - **Photoacoustic methods** (can also used for determination of α)
  - **Mirage effect**

- Heat capacity
  - Differential Scanning Calorimetry

Measurement methods

Measurement methods of a single parameter

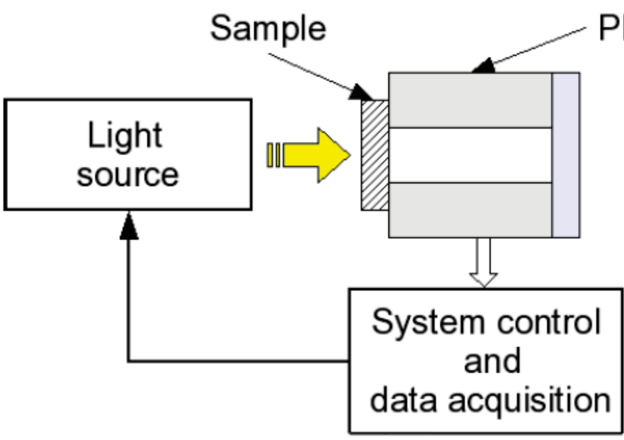
Thermal effusivity

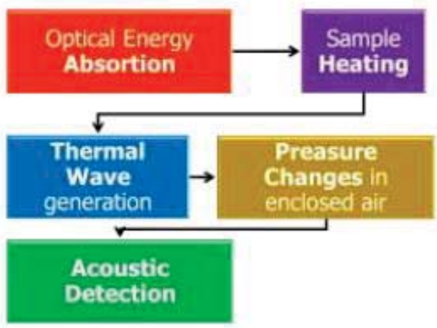
Photoacoustic methods

Proposed by Rosencwaig et Gesho en 1976

Non-contact measurement

Photothermal techniques are based on the conversion of absorbed optical energy into thermal energy.





Used for Liquids ➔ can also used for solids

Measurement methods

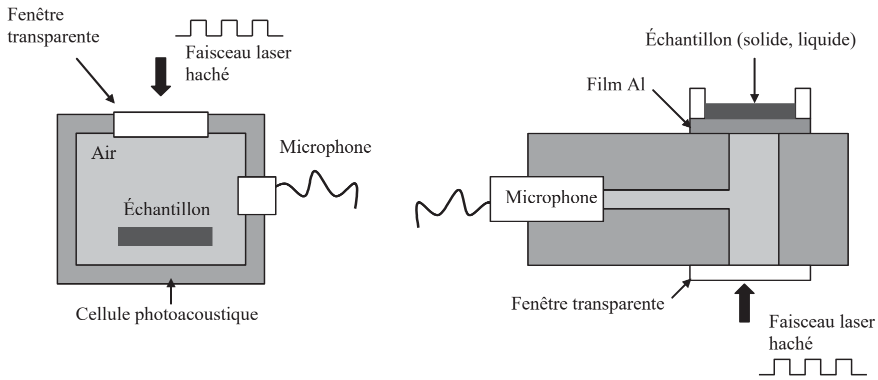
Measurement methods of a single parameter

Thermal effusivity

Photoacoustic methods

Non-contact measurement

- ❑ The sample is placed inside a small cell containing a gas, usually air
- ❑ The sample is excited through a window with a modulated beam of a laser or a halogen lamp
- ❑ A microphone records the resulting pressure variations for the volume of gas in the cell



Measurement methods

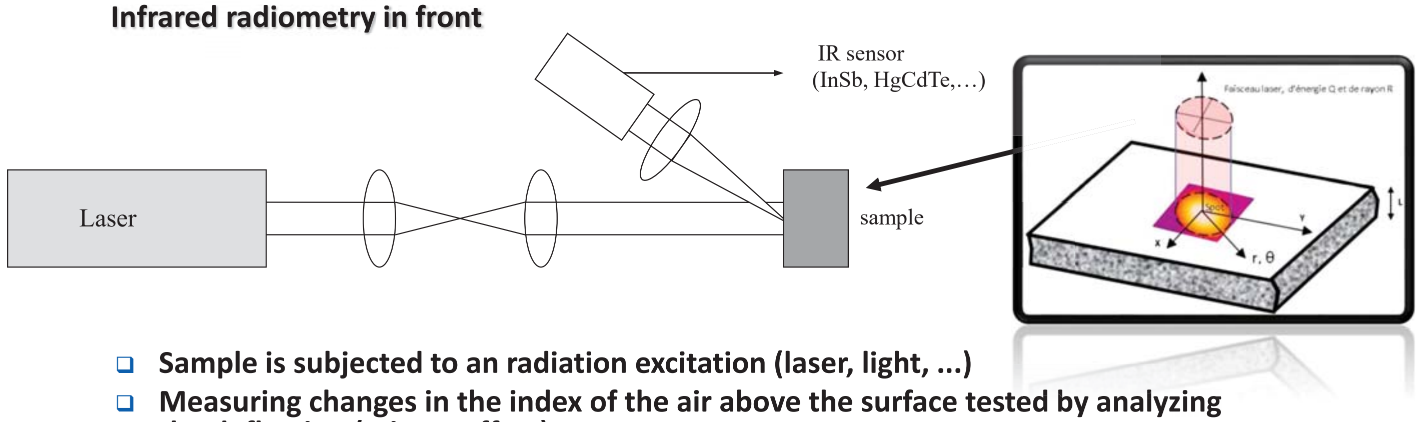
Measurement methods of a single parameter

Thermal effusivity

Mirage effect

Non-contact measurement

Infrared radiometry in front



- ❑ Sample is subjected to an radiation excitation (laser, light, ...)
- ❑ Measuring changes in the index of the air above the surface tested by analyzing the deflection (mirage effect)

Used for thin materials

Measurement methods

Measurement methods of a single parameter

Thermal effusivity

Mirage effect

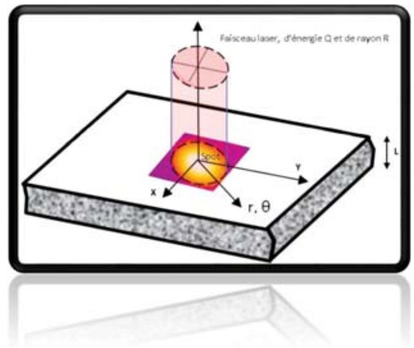
Non-contact measurement

$$T(r,0,t) \approx \frac{2Q}{b\sqrt{\pi^3 t}} \cdot \frac{1}{R^2}$$

Measurements in the center of the sample (r = 0)

$$Q' = \frac{2Q}{\pi R^2}$$

$$T(t) = \frac{Q'}{b\sqrt{\pi t}}$$



t is the time, b is thermal effusivity,
Q is rotal laser beam energy, R laser spot radius

Used for thin materials

Measurement methods

Measurement methods of a single parameter

- Thermal conductivity
  - Hot plate method
  - Hot wire method
  - 3w method
- Thermal diffusivity
  - Flash method
- Thermal effusivity
  - Photoacoustic methods
  - Mirage effect
- **Heat capacity**
  - **Differential Scanning Calorimetry**

Measurement methods

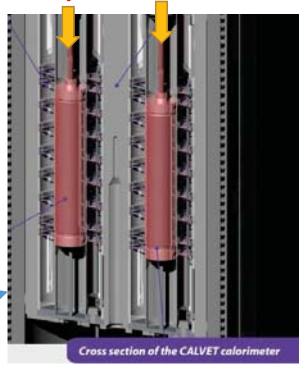
Measurement methods of a single parameter

heat capacity

Calorimeters Device

Calorimeters is used for the characterization of heat capacity c_p of materials across wide temperature ranges

Sample Reference



The sample, within a measurement cell, is placed directly into the center of the measurement zone.

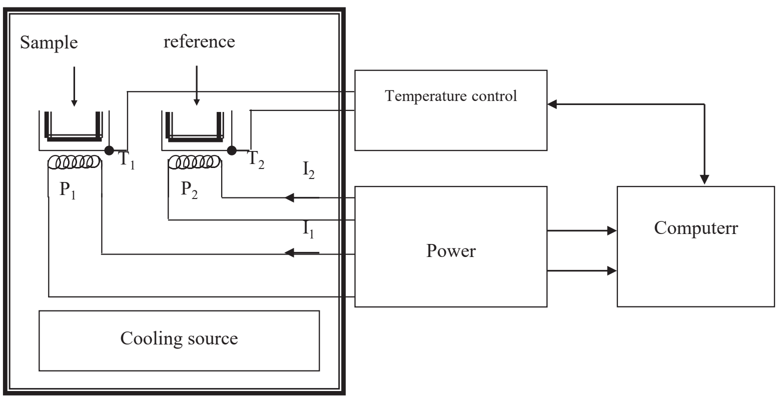
Measurement methods

Measurement methods of a single parameter

heat capacity

Differential Scanning Calorimetry

Measuring cell is thermally coupled with a reference cell, the sensor is arranged to minimize the influence of perturbations of the block calorimeter



Measurement methods

Measurement methods of a single parameter — heat capacity

Differential Scanning Calorimetry

Measuring cell is thermally coupled with a reference cell, the sensor is arranged to minimize the influence of perturbations of the block calorimeter

$$c_{pe}(T) = \frac{\Phi_e - \Phi_b}{\Phi_r - \Phi_b} \cdot \frac{m_r}{m_e} \cdot c_{pr}(T)$$

m_e = masse de l'échantillon (kg) ;

m_r = masse du matériau de référence (kg) ;

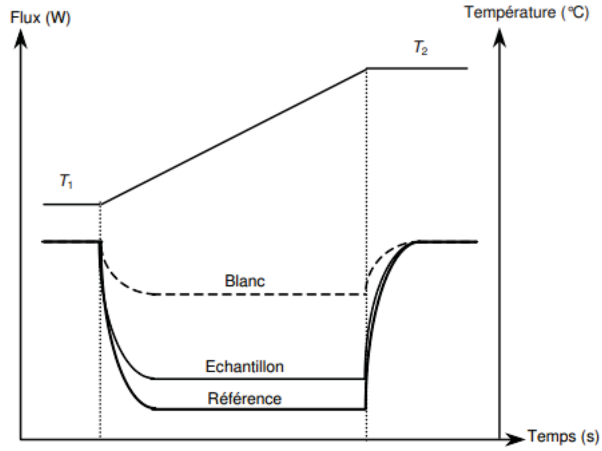
Φ_b = flux mesuré lors de l'essai sans échantillon (W) ;

Φ_r = flux mesuré lors de l'essai avec le matériau de référence (W) ;

Φ_e = flux mesuré lors de l'essai avec l'échantillon (W) ;

$c_{pr}(T)$ = capacité thermique du matériau de référence ($J \cdot K^{-1} \cdot kg^{-1}$) ;

$c_{pe}(T)$ = capacité thermique de l'échantillon ($J \cdot K^{-1} \cdot kg^{-1}$).



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Measurement methods

We classify the measurement methods in 2 categories

measurement methods of a single parameter	measurement methods of a several parameters
conductivity diffusivity effusivity capacity	conductivity & diffusivity effusivity & diffusivity diffusivity & specific heat

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Measurement methods

Measurement methods for several parameters

of course, there are other devices and methods

- ***Conductivity and diffusivity***
  - **Hot Disk method**
  - Transient hot-wire
  - Transient hot-bridge
  - Periodic method [DiCo]
- ***Diffusivity and specific heat***
  - Netzsch « Microflash »
- ***Diffusivity and effusivity***
  - Photoacoustic and Photopyroelectric methods

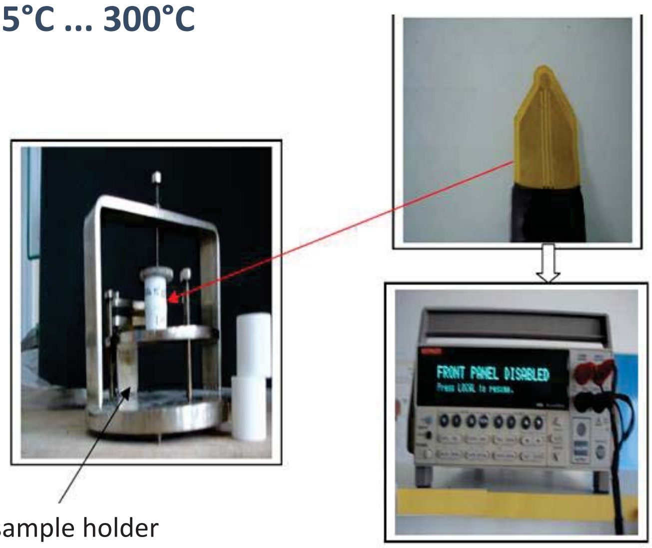
Measurement methods

Measurement methods for several parameters

Hot Disk method

Thermal conductivity & diffusivity

-35°C … 300°C



sample holder

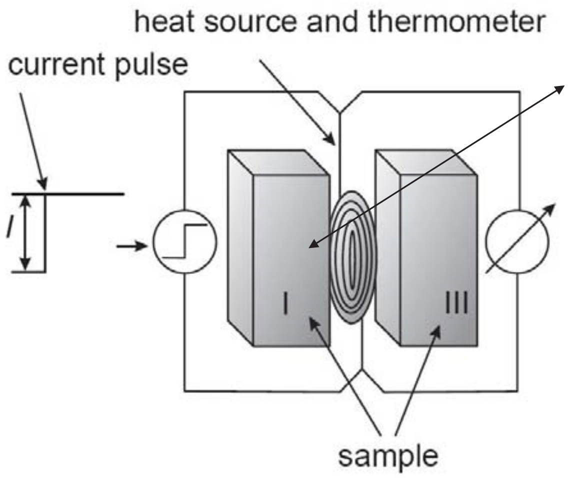
Measurement methods

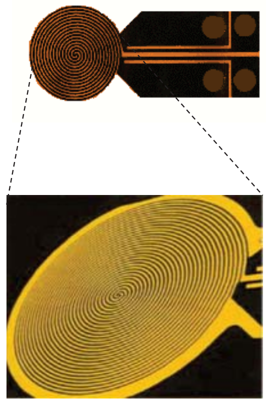
Measurement methods for several parameters

Hot Disk method

Thermal conductivity & diffusivity

-35°C ... 300°C





Measurement methods

Measurement methods for several parameters

Hot Disk method

Thermal conductivity & diffusivity

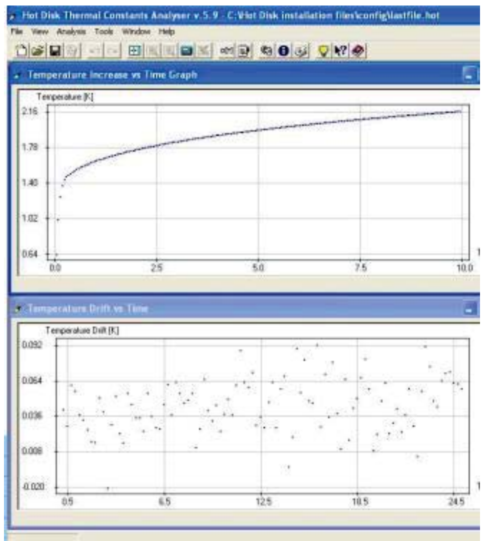
$$R = R_0(1+\alpha\,\overline{\Delta T(\tau)})$$

TCR

$$\Delta T(\tau) = P_0(\pi^{3/2} r \lambda)^{-1} * D_s(\tau)$$

$$D_s(\tau) = [m(m+1)]^{-2} * \int_0^{\tau} d\sigma * \sigma^{-2} * \sum_{l=1}^{m} l * \sum_{k=1}^{m} k * \exp\left(\frac{-(l^2+k^2)}{4m^2\sigma^2}\right) I_0\left(\frac{lk}{2m^2\sigma^2}\right)$$

$$\tau = \left(\frac{t * a}{r^2}\right)^{1/2}$$



In this model ➔ thermal conductivity is inversely proportional to thermal diffusivity

Measurement methods

Measurement methods for several parameters

- *Conductivity and diffusivity*
  - Hot Disk method
  - Transient hot-wire
  - Transient hot-bridge
  - Periodic method [DiCo]
- Diffusivity and effusivity
- Photoacoustic methods
- *Diffusivity and specific heat*
  - Netzsch « Microflash »

Measurement methods

Measurement methods for several parameters

Transient hot-wire method

Thermal conductivity & diffusivity

- The system is involving a vertical and cylindrical symmetry 1D where in the wire both provides heating and serves as a thermometry.
- The mathematical model is expressed for that of a boundless line source of heat suspended vertically in a boundless medium.
- The equation of the specified solution of Fourier's law is as follows:

$$T(t) - T_{\text{ref}} = \Delta T = \frac{q}{4\pi\lambda} \ln\left(\frac{4K}{a^2C}t\right)$$

where $T(t)$ is the temperature of the wire at time t; T_{ref} is the reference temperature; ΔT is the temperature of the cell; q is the applied power; λ is the thermal conductivity, a function of both temperature and density; K is thermal diffusivity; a is the radius of the wire; and $InC = \gamma$, where γ is the Euler constant.

Measurement methods

Measurement methods for several parameters

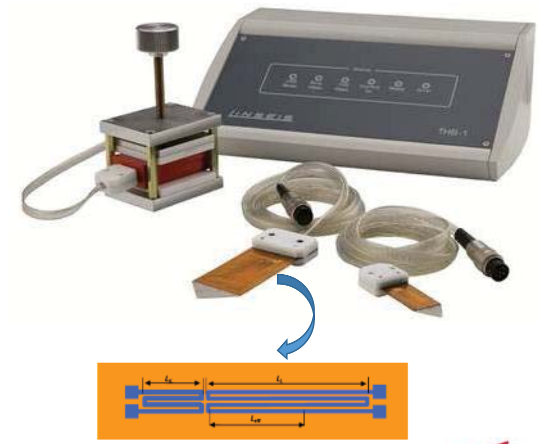
Transient Hot-Bridge (THB) method

Thermal conductivity & diffusivity

-35°C ... 200°C

THB is an enhancement of the Hot Wire or the Transient Hot Strip method

- An electrical current causes a temperature difference between the sample center and edge
- Thermal conductivity (λ) and thermal diffusivity (α) identification



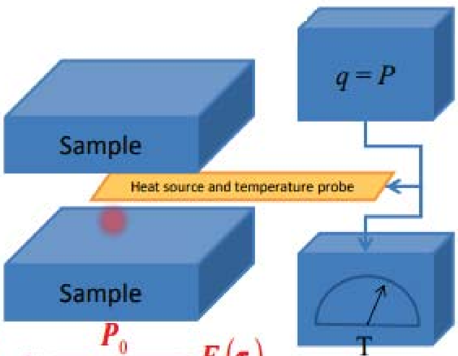
Measurement methods

Measurement methods for several parameters

Transient Hot-Bridge (THB) method

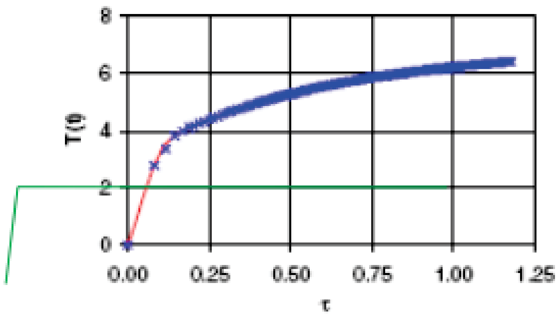
Thermal conductivity & diffusivity

-35°C ... 200°C



$$\lambda_m \sim \frac{P_0}{L \cdot \Delta T(\tau)} \cdot F(\tau)$$

$$\tau = \sqrt{\frac{a \cdot t}{r_0^2}}$$



The value of the thermal conductivity is nearly inversely proportional to temperature rise.
Diffusivity determines measurement duration, the needed time to reach the steady state.

Measurement methods

Measurement methods for several parameters

- ***Conductivity and diffusivity***
  - Hot Disk method
  - Transient hot-wire
  - Transient hot-bridge
  - Periodic method [DiCo]
- ***Diffusivity and specific heat***
  - Netzsch « Microflash »

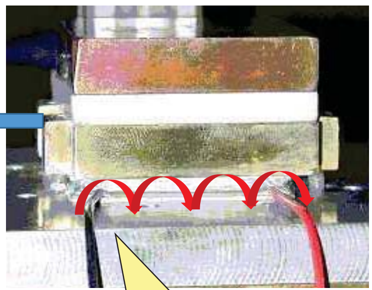
Measurement methods

Measurement methods for several parameters

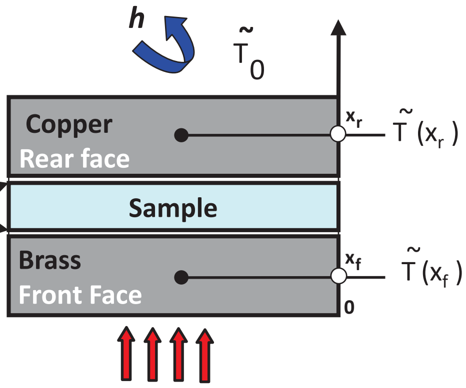
Periodic Method [DiCo]

Thermal conductivity & diffusivity

-35°C ... 200°C



Thermal grease



$$V(t) = V_{moy} + \sum_{n=1}^{5} V_n . \sin\left(2\pi 2^{n-1} f_0 t\right)$$

Measurement methods

Measurement methods for several parameters

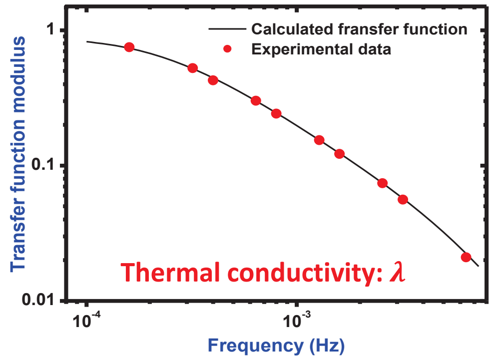
Periodic Method [DiCo]

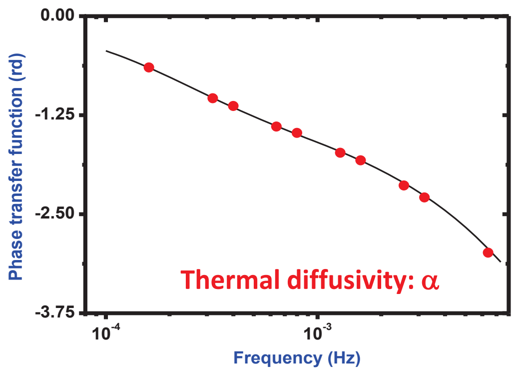
Thermal conductivity & diffusivity

-35°C ... 200°C

Inverse method (Levenberg-Marquardt)

$$S(\hat{\beta}_{k,a}) = \sum_{i=1}^{N} \left[\left(\tilde{H}_{real}(f_i) - H_{real}(f_i) \right)^2 + \left(\tilde{H}_{imag}(f_i) - H_{imag}(f_i) \right)^2 \right]$$





Measurement methods

Measurement methods for several parameters

- *Conductivity and diffusivity*
  - Hot Disk method
  - Transient hot-wire
  - Transient hot-bridge
  - Periodic method [DiCo]
- ***Diffusivity and specific heat***
  - **Netzsch « Microflash »**

Measurement methods

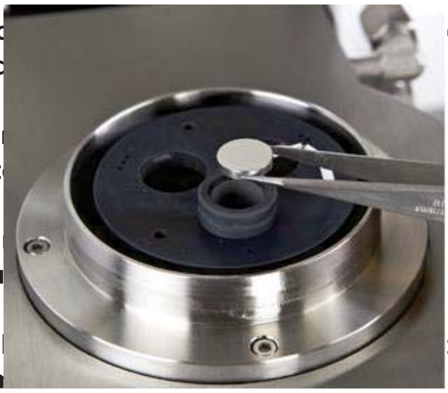
Measurement methods for several parameters

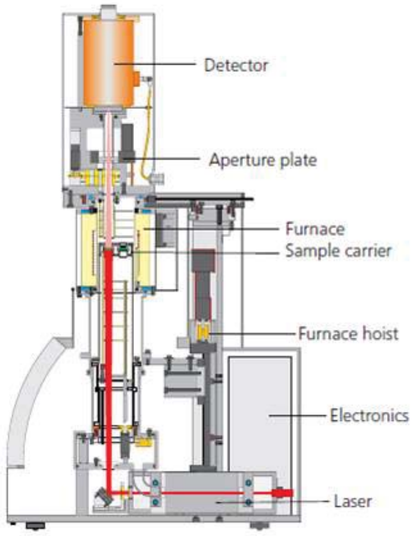
Netzsch « Microflash »

Diffusivity & Specific heat

-125°C ... 500°C, RT ... 1100°C.

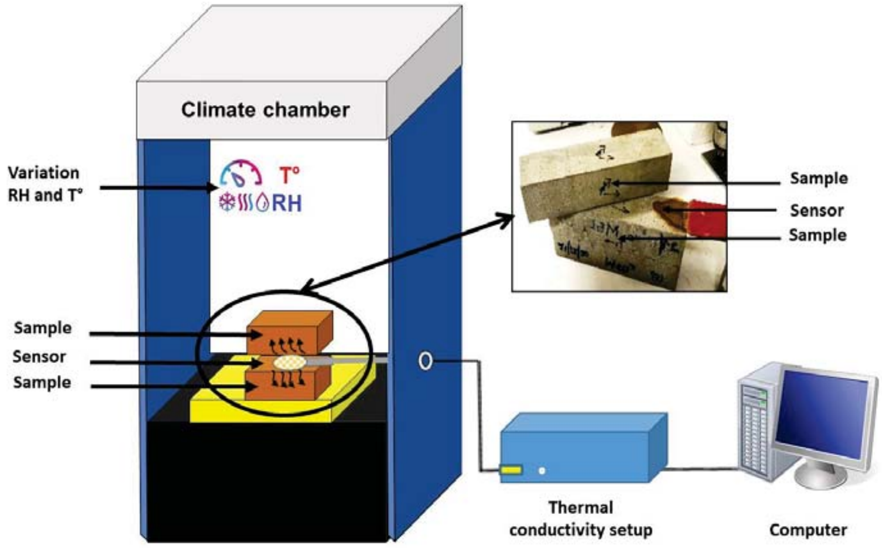
- The fr… …le is heated…
- The te… …red detect…
- (*Cp*) a… measu…
- With t… …be determ…





Measurement methods (T and RH)

Thermal conductivity	λ(T et HR)
Specific heat	$\rho\, c_p$(T et HR)
Thermal Diffusivity	α(T et HR)=($\lambda/\rho\, c_p$)



There are other methods and devices

Measurement methods

Conclusion

- Many measurement methods have been developed → experimental tools and models of varying complexity
- The validity of each method depends on its reliability and of the margin of error calculated from a model described

- what is the method that I will used?
- for which kind of sample?

- These methods work well, but generally depend on some parameters → geometry, thermal properties, boundary conditions,…

Thank you

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