Tutorials on thermal conduction experiments

Thermal instrumentation and measurements for micro and nano systems using electrical and optical methods

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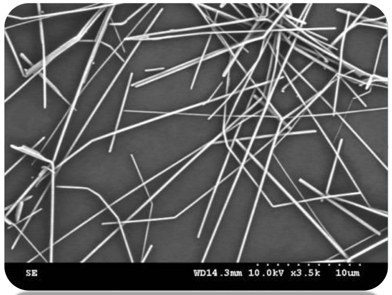
Outline of the lecture

1. Motivations and prerequisites: physics of electron and phonon heat transfer
2. A page of history: the starting point of thermal measurements in the XVIIth century
3. Thermometry down to the nanoscale: electrical experiments and others
4. DC and AC electrical techniques (3 omega PP, IP, nanowire)
5. Application of the techniques to the nanoscale
6. Nanofabrication of sensors for thermal measurements
7. Near field experiment of thermal conduction
8. Optical methods:
   1. Raman thermometry
   2. Time dependent thermoreflectance experiments

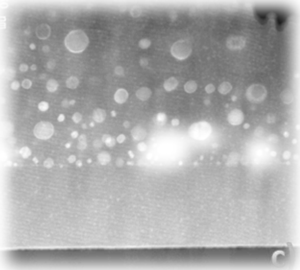
Motivations and prerequisites

Motivations: why sensitive experiments are needed

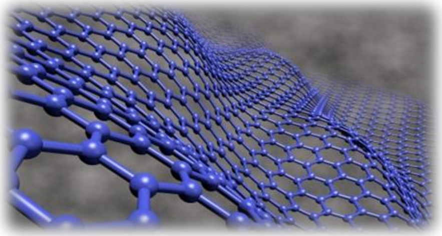
- Fundamentals of heat transport at the nanoscale
- Electronic transport
- Phononic transport
- (Photonic transport)=> see Radiative transfer
- Effect of low dimensions on the thermal transport



Nanoscale → Small Energy/Power



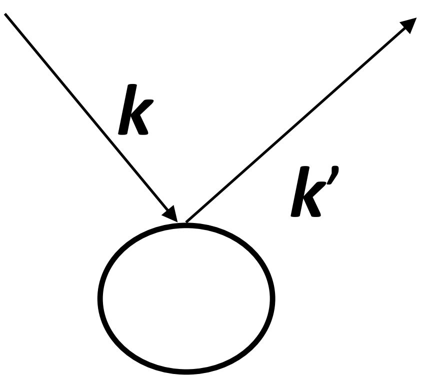
- Growing need of developing sensitive experimental tools
- Sensitive Tools
- Versatile tools
- Adapted to low dimensions



Prerequisite on thermal transport: two main heat carriers

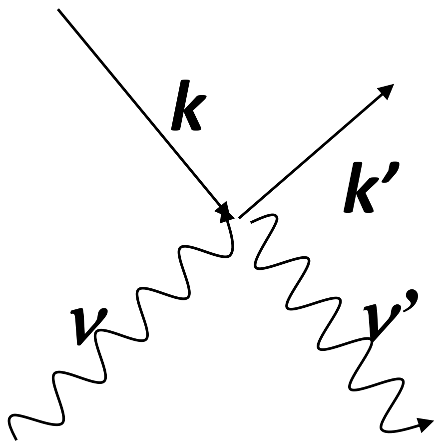
- Electronic heat transport :
  - Specificities
  - Important length (mean free path, Fermi wave length)
  - Characteristic energy $k_B T$ versus E_F
- Phononic heat transport :
  - Specificities
  - Important length (mean free path, dominant phonon wave length)
  - Characteristic energy $k_B T$ versus $\hbar\omega$

<u>Mean free path</u>



Phonon elastic scattering (impurity)

$|k| = |k'|$



Inelastic scattering

$|k| \neq |k'|$ and $|\upsilon| \neq |\upsilon'|$

<u>Wavelength</u>

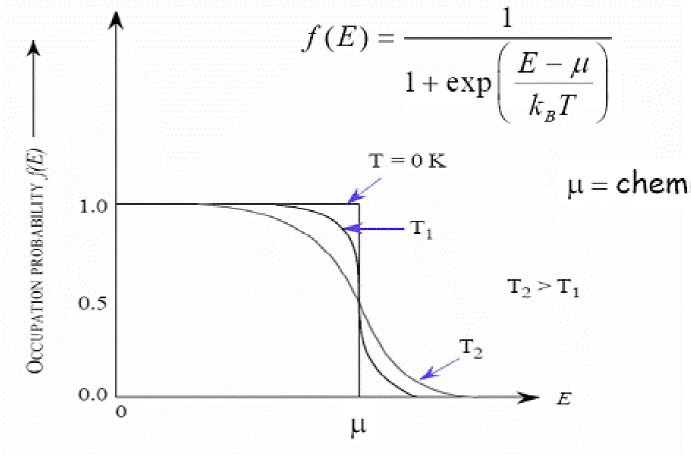
$$E_F = \hbar k_F$$

$$\lambda_{Dom} = \frac{h v_S}{4.25 k_B T}$$

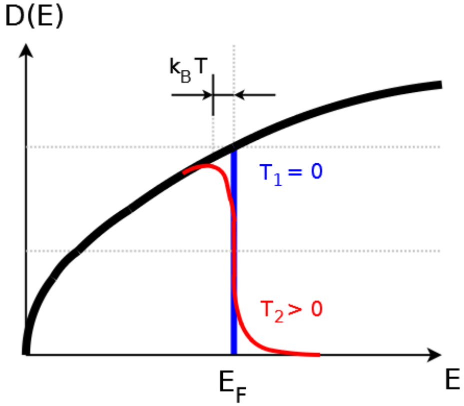
Electron thermal transport

Λ_{e-}~nm	•**Mean free path**
λ_F~0.1nm	•**Relevant wave length**
T	•**Temperature dependence (K, C)**
diffusive	•**Transport**
fermions	•**Statistic Fermi-Dirac distribution**

$k_BT << E_F$



μ = chemical potential



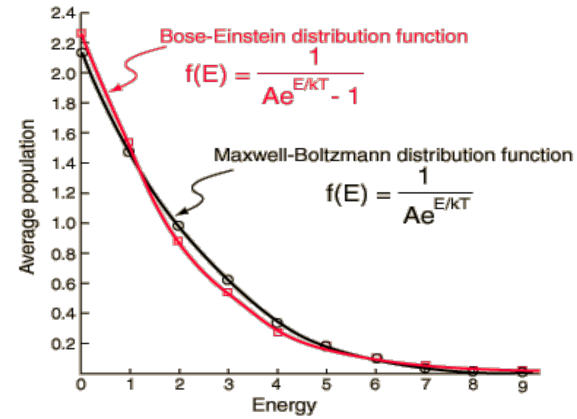
Drude model $\sigma_{e-} = \frac{\tau}{3} e^2 v_F^2 D(E_F)$

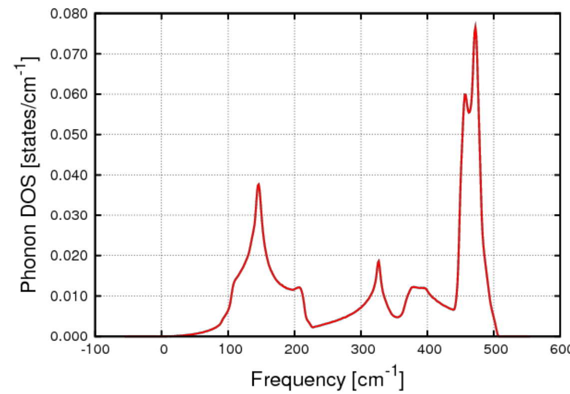
Boltzmann equation $k_{e-} = \frac{\pi^2}{6} k_B^2 v_F^2 \tau D(E_F) T$

Wiedemann Franz law $\frac{k_{e-}}{\sigma_{e-} T} = \frac{1}{3} \frac{\pi^2 k_B^2}{e^2}$

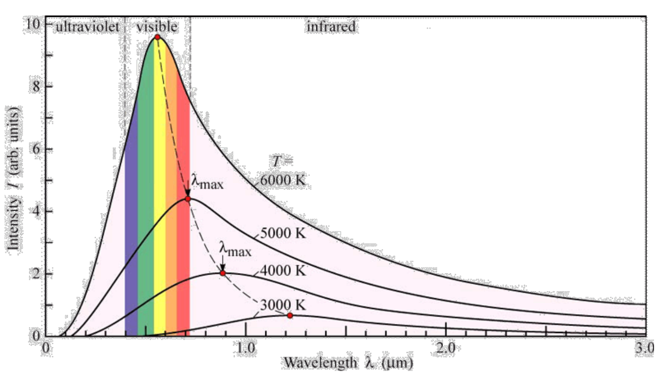
Phonon thermal transport

	At 1K	At 1K
•Mean free path	Λ_{ph}**~cm**	Λ_{ph}**~500nm**
•Relevant wave length	λ_{ph}**~100nm**	λ_{ph}**~1nm**
•Temperature dependence (K, C)	T^3	**NA**
•Transport	**Diffusive/ballistic**	
•Statistic Fermi-Dirac distribution	**bosons**	





In silicon



$$\lambda_{Dom} = \frac{hv_S}{4.25 k_B T}$$

A page of history

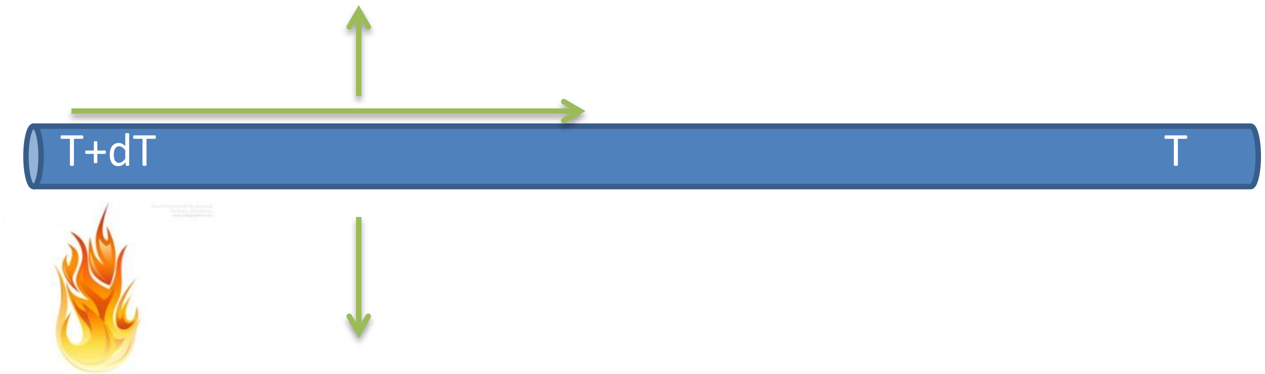
The origin of the C/K concept

- Early days : Guillaume Amontons (1663–1705): heat flow along the temperature gradient (no thermometer)

- Heat, matter and temperature: Latent heat,

Joseph Black (1728-1799)

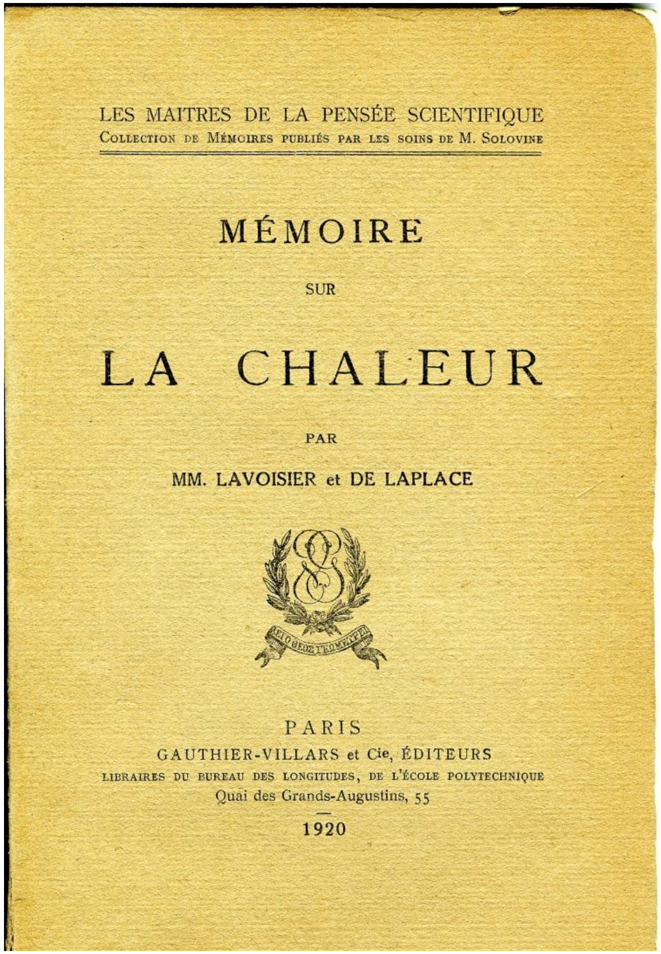
- First measurements: Jean-Henri Lambert (1728–77) published in 1779 : convection, geometry



Joseph Black

Jean-Henri Lambert

The first study on specific heat…1780



"Les Physiciens sont partagés sur la nature de la chaleur"

Nouveaux concepts : chaleur libre, capacité de chaleur, chaleur spécifique

"Plusieurs d'entre eux la regarde comme un fluide répandu dans la nature"…

"D'autres physiciens pensent que la chaleur n'est que le résultat des mouvements insensibles des molécules de la matière."

Lavoisier et Laplace (Mémoire sur la chaleur 1780).

Specific heat measurements

Pierre Simon de Laplace

Antoine Laurent Lavoisier

- Treatise on calorimetry published in 1782
- First experiment of heat capacity
- Ice calorimeter (temperature reference)

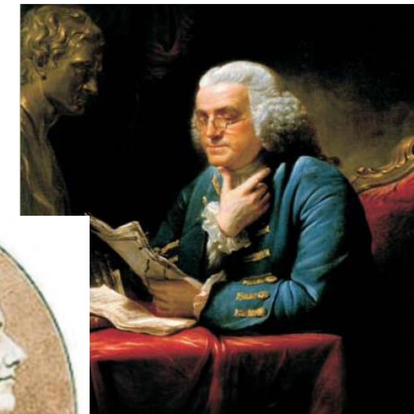
L (latent heat ice/water at T = 0°C)

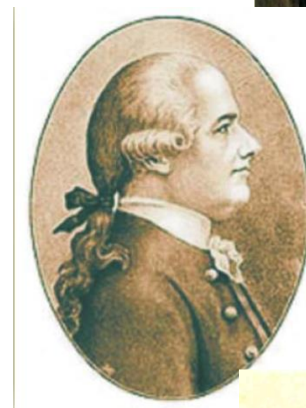
= 334 J/g ≈ 80 cal/g

L (latent heat water/water vapour at T = 100 °C) = 2260 J/g ≈ 539 cal/g

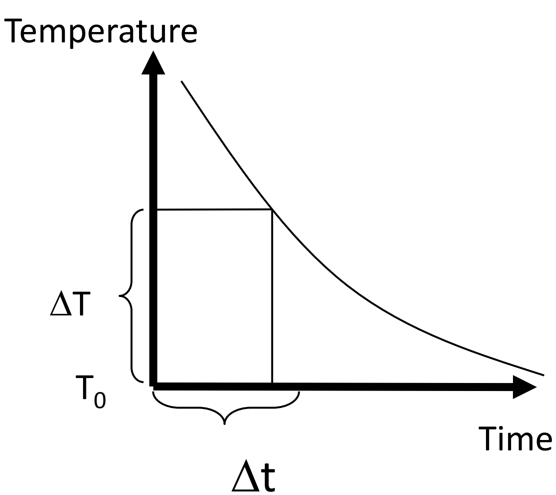
History of thermal conductivity measurements

- Benjamin Franklin (1706-1790) compared thermal conductivity of different metals
- Jan Ingen-Housz (1730-1799)
- Count Rumford (1753-1814) insulating materials
- Joseph Fourier (1768-1830)





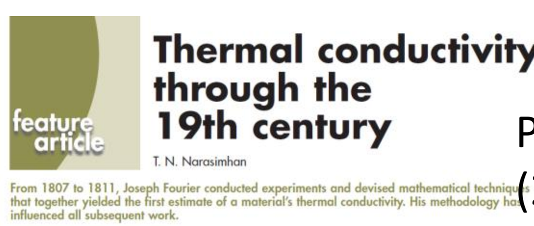
2 experiments: steady state (K,h), transient (C,h>>K)



$$\tau = \frac{C}{K}$$

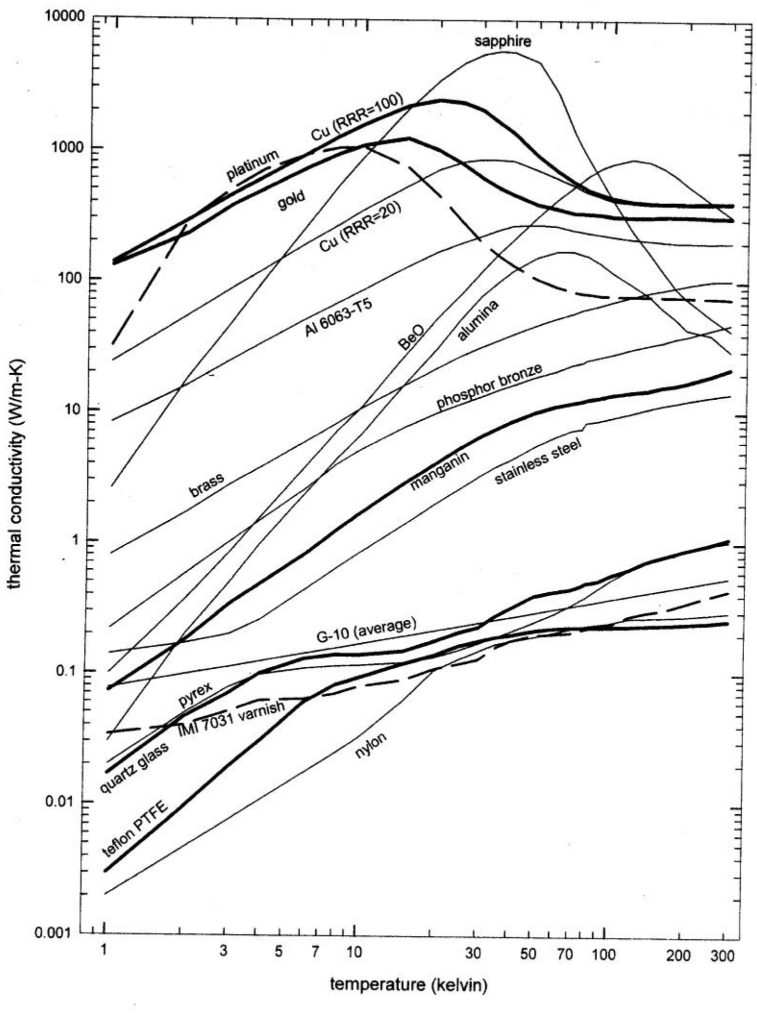
Joseph Fourier

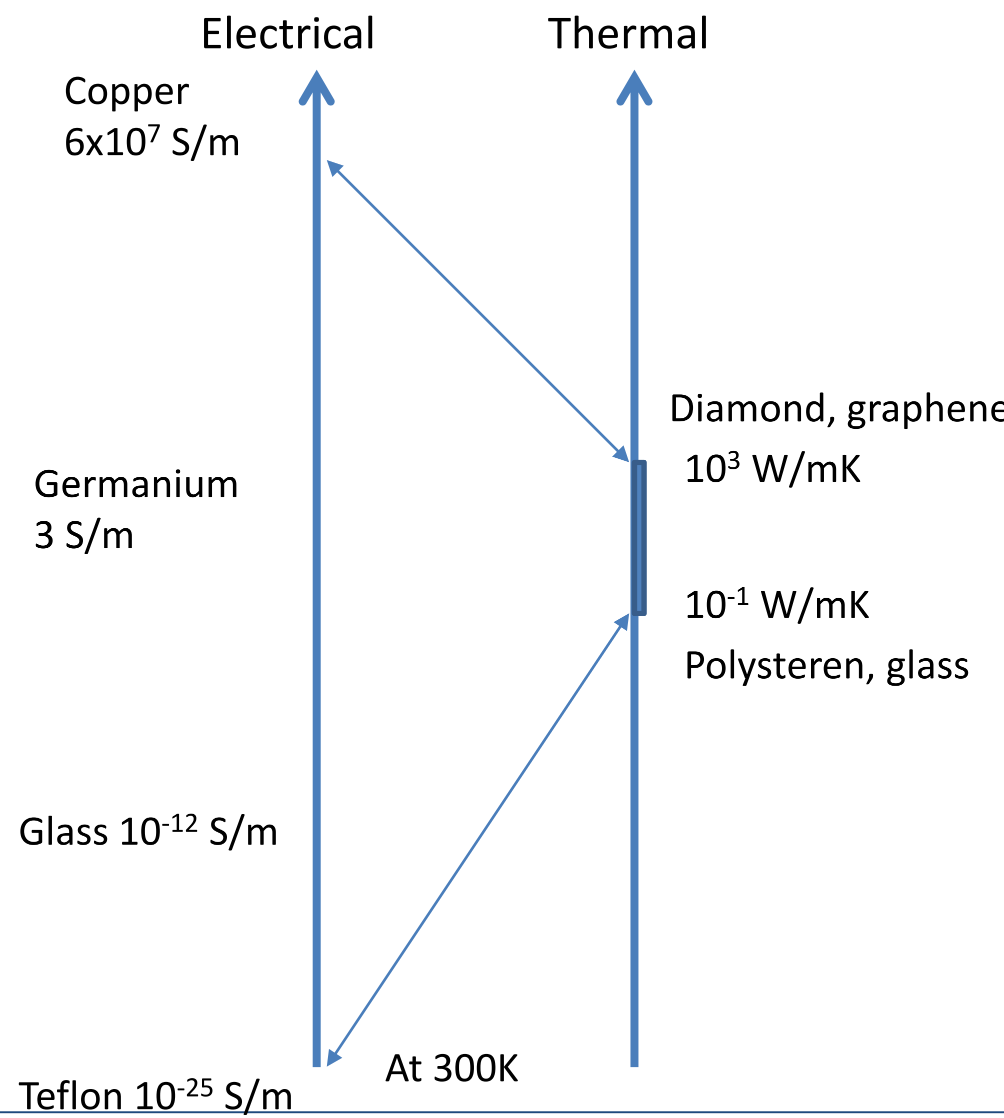
Time dependent experiments:


feature article
Thermal conductivity through the 19th century
T. N. Narasimhan
From 1807 to 1811, Joseph Fourier conducted experiments and devised mathematical techniques that together yielded the first estimate of a material's thermal conductivity. His methodology has influenced all subsequent work.

Physics Today **63**, 36 (2010)

Temperature behavior of the thermal conductivity



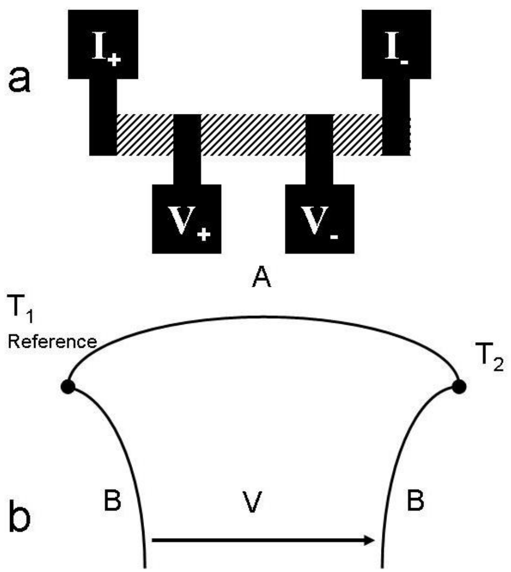


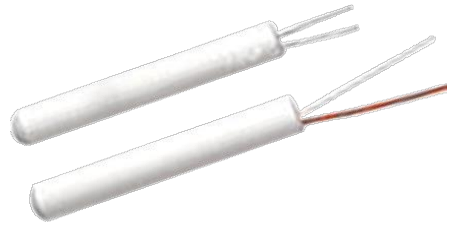
Thermometry down to the nanoscale: electrical experiments

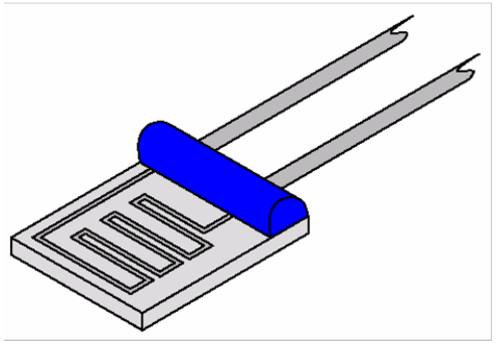
Choosing the correct thermometer

- Sensitivity of experiment: temperature coefficient
- Resistive thermometry (thin films)
- Thermocouple
- 4 wire measurement: separate the current and voltage leads
- Not primary thermometer, need calibration

$$\alpha = \frac{1}{\Psi}\frac{\partial \Psi}{\partial T}$$





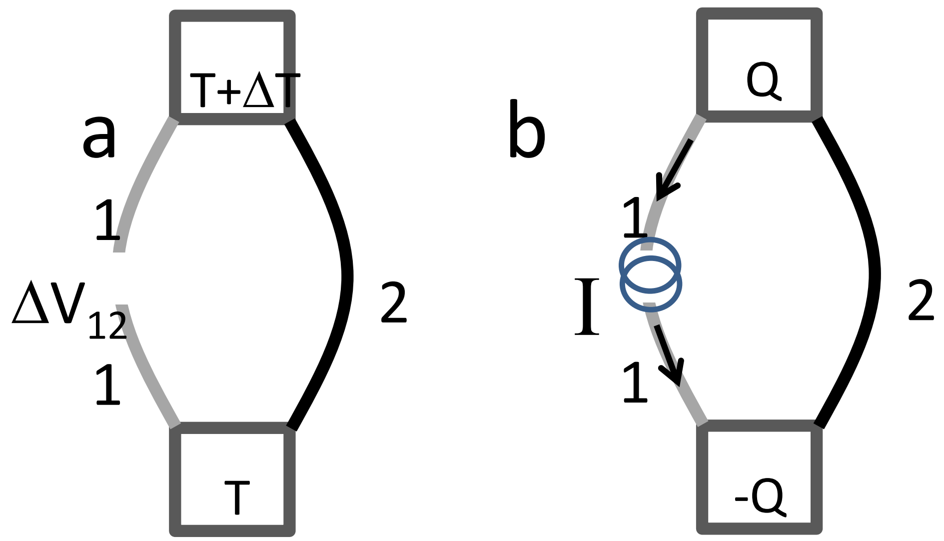


Thermocouple

- Involve two materials (1 and 2)
- $\Delta V = 200 \mu V/K$
- CrNi/AlNi
- Can be lithographied and down scaled

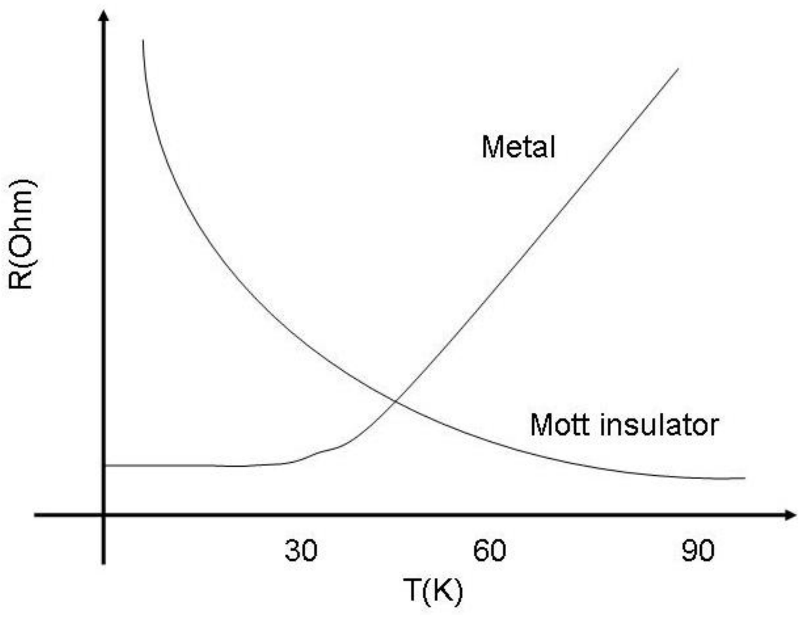
- Au/Cr

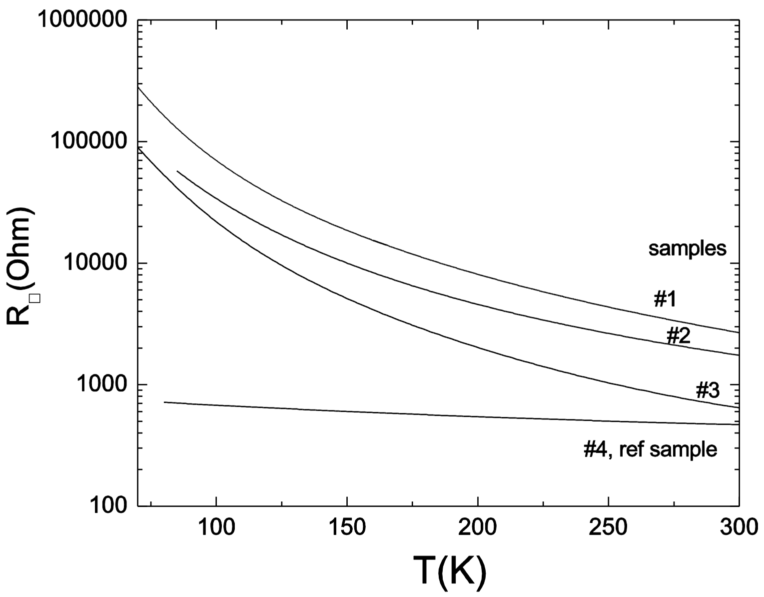
Metal	Seebeck Coefficient (µV/K)
Al	-1.66
Au	1.94
Cr	21.8
Ni	-19.5



Resistive thermometry

- Metal (Pt, Au, Ni) Temp=40K-1500K
- Semiconductor (Ge, Si) 0K-50K
- Mott-Anderson insulator (Metal to insulator transition: NbN, NbSi, AuGe) 0K-400K





For NbN thermometry Bourgeois et al., Review of Scientific Instrument **77**, 126108 (2006)

Performances and limitations

- Platinum: versatile, low impedance, thin films, can be downscaled, for temperature above 50K

$$\alpha = \frac{1}{\Psi}\frac{\partial \Psi}{\partial T}$$

- Germanium thermometer: very sensitive, macroscopic
- NbN or NbSi: very sensitive, versatile, high impedance, thin films only, can be downscaled, wide temperature range (0-400/500K)

Thermometer	High temp	Low temp	α at room T	α at 1K	Scalability at the nanometer	Impedance
Platinum	√	x	2×10^{-3}	x	√	100
Germanium	x	√	x	-10	x	1000
NbN, NbSi	√	√	-1×10^{-2}	-1	√	1000

Calibration needed !!

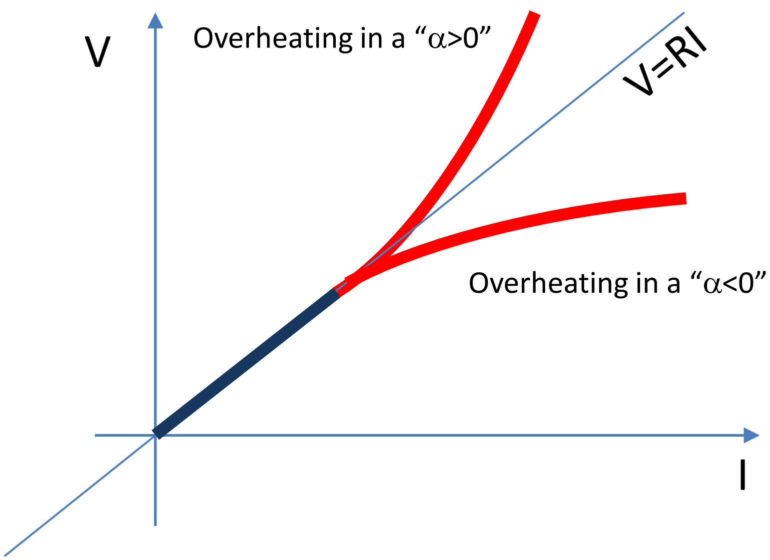
- DC measurement: current source and voltmeter
- DC Offset
- Low temperature

$$\frac{V(I_+) - V(I_-)}{2}$$

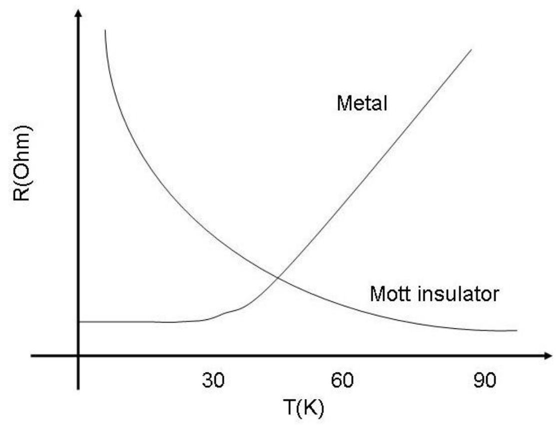
- AC measurements (lock-in Amplifier
- Experimental set-up more evaluated
- 3 omega method, ac calorimetry (2ω)

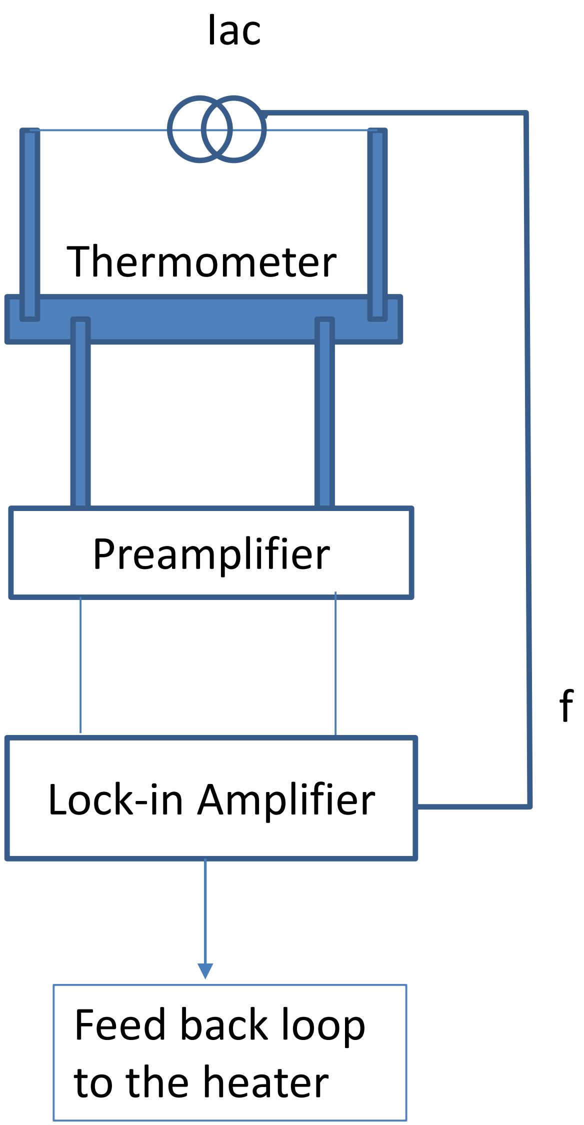
Error coming from overheating of the thermometer

$$P=RI^2 \text{ or } P=V^2/R$$



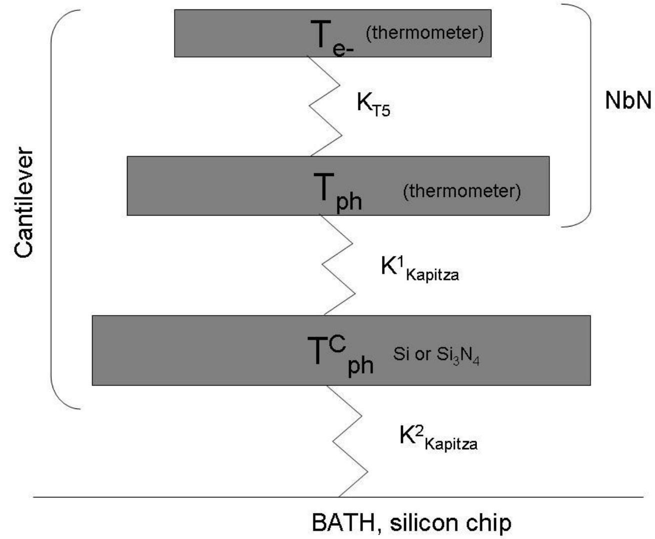
- Resistive thermometer
  - If $\alpha>0$ (Platinum) Regulation with voltage $P=V^2/R$
  - Regulation in current (avalanche)
  - If $\alpha<0$ (germanium) Regulation with current $P=RI^2$
  - Regulation in voltage (avalanche)



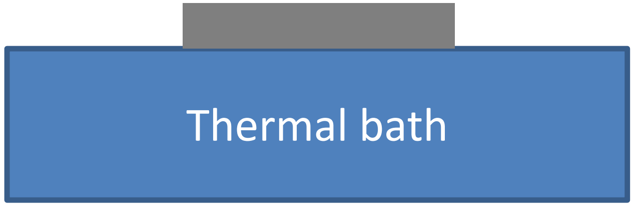


Measurement of downscaled system

- Sensitive measurement of the temperature
- Temperature measurement as close as possible to the system
- Decoupling: short time scale or low temperature
- Kapitza resistance
- Acoustic Mismatch Model (AMM) and Diffusive Mismatch Model (DMM)



$$K^{1}_{Kapit} = \frac{T^3}{12}\ \mathrm{W/K^4cm^2}$$



See Swartz and Pohl *Rev. Mod Phys* **61**, 605 (1989)

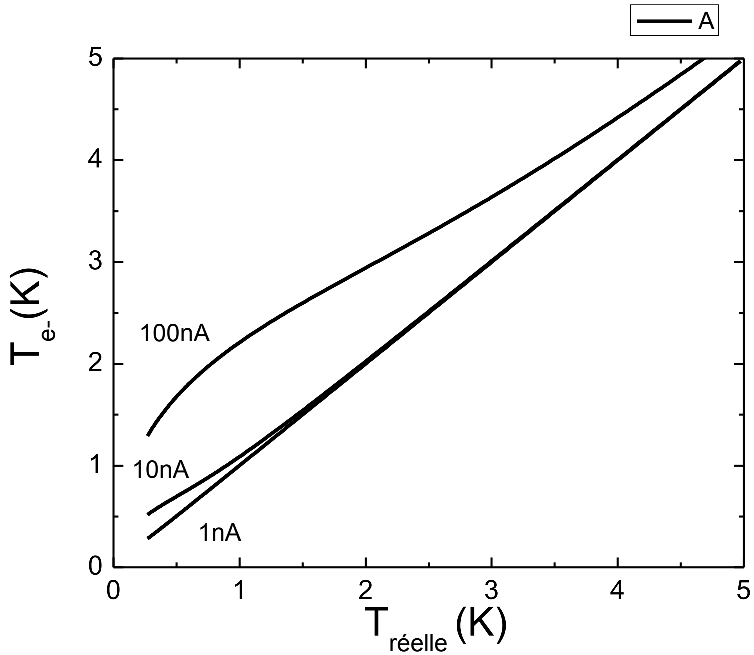
- Measurement of the electron temperature
- Under low electric field

$$T_{e^-} - T_{ph} = \frac{P_{e^-}}{K_{e^-/ph}} = \tau_{e^-/ph} \frac{V^2}{RC_{e^-}}$$

- High electric field

$$T_{e^-}^n - T_{ph}^n = \frac{P_{e^-}}{V g_{e^-/ph}}$$

- Work with high impedance thermometer



e^--phonon coupling constant (g_{e-}/ph)
1000W/K^5cm^3

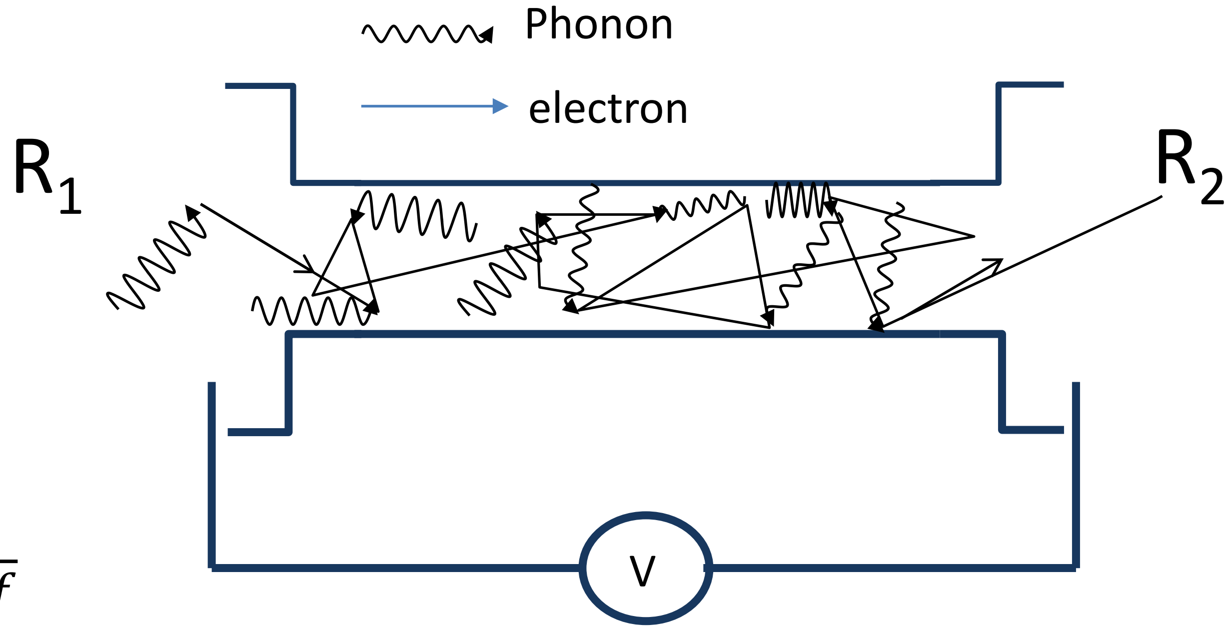
Example : silicon nanowire

<u>Exercise:</u> thermometer of 50nm, 100nm large, 10mm long
R=200kOhm, I_{ac}=1nA, T=0.5K, ΔT=??

Answer 4mK

- **Noise thermometry** ("The noise is the signal" Rolf Landauer *Nature* **392**, 658-659 (16 April 1998))

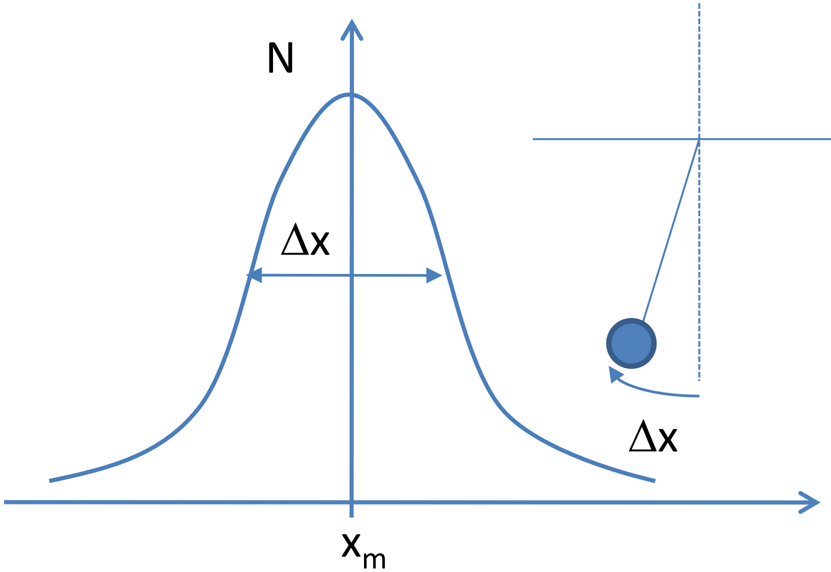
- Universal thermometry !!



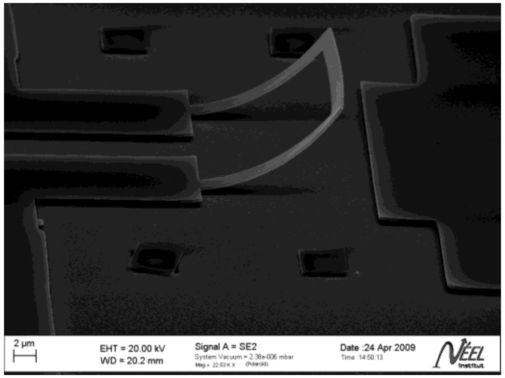
$$V\left(\frac{Volt}{\sqrt{Hz}}\right) = \sqrt{4k_B T R \Delta f}$$

$$\alpha \sim \frac{1}{T}$$ → Low temperature

- Measurement of thermal noise
- Vibration of a cantilever : NEMS
- No thermometer needed, universal thermometry FWHM~k_BT
- Macroscopic measurement of the global temperature of a beam

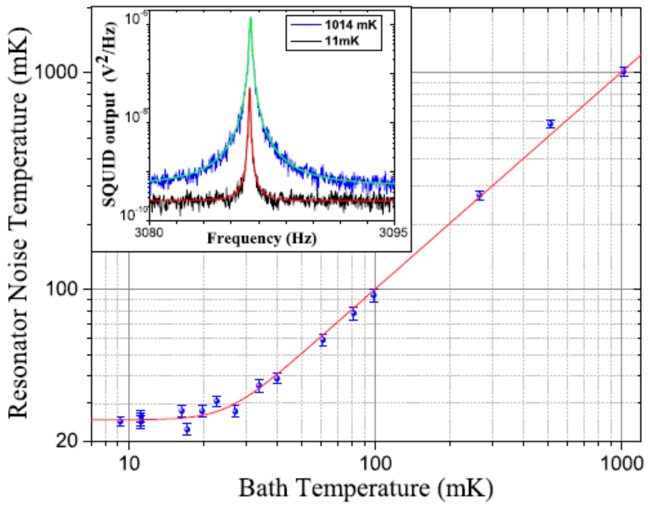


- Phonon thermometer !!



$$\frac{1}{2}k\langle \Delta x^2 \rangle = \frac{1}{2}k_BT$$

$$\langle \Delta x^2 \rangle = \frac{k_BT}{m\omega_0^2}$$



APPLIED PHYSICS LETTERS **98**, 133105 (2011)

SINIS junction for electron thermometry

REVIEWS OF MODERN PHYSICS, VOLUME 78, JANUARY 2006

Opportunities for mesoscopics in thermometry and refrigeration: Physics and applications

Francesco Giazotto*

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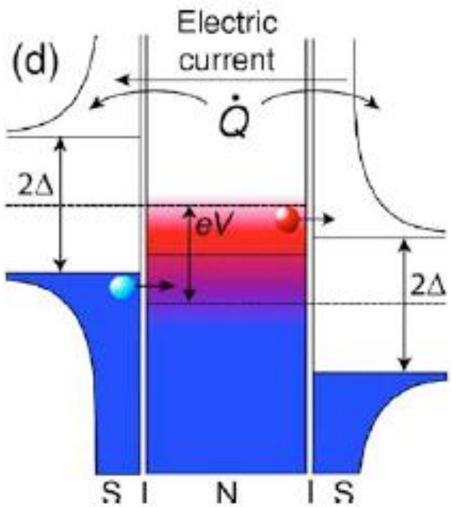
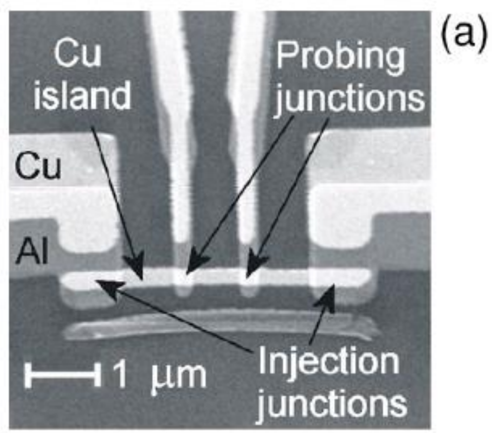
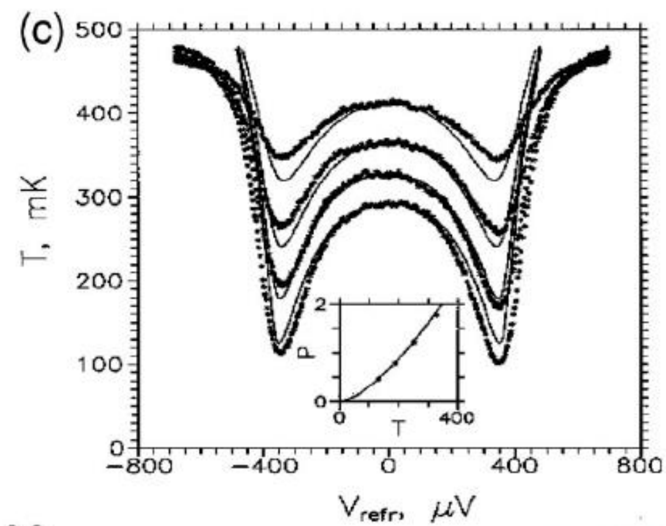
and NEST CNR-INFM and Scuola Normale Superiore, I-56126 Pisa, Italy

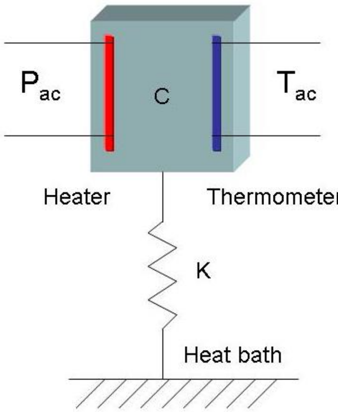


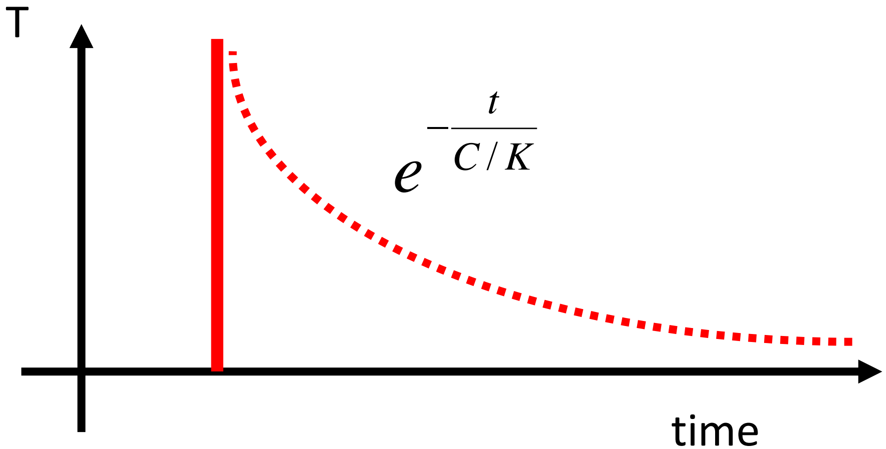


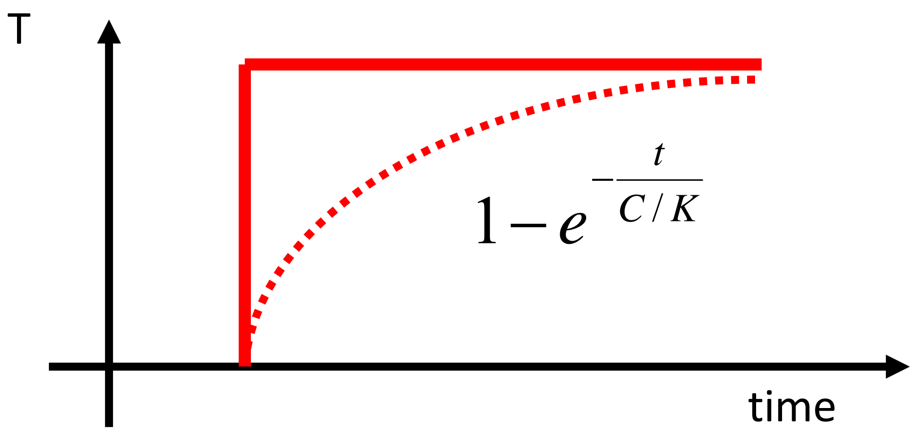


C/K

- Heat Pulse: exponential decrease of the temperature
- Heat step function: measurement of the thermalization of the system
- $\tau = C/K$
- Two properties can be measured C and K

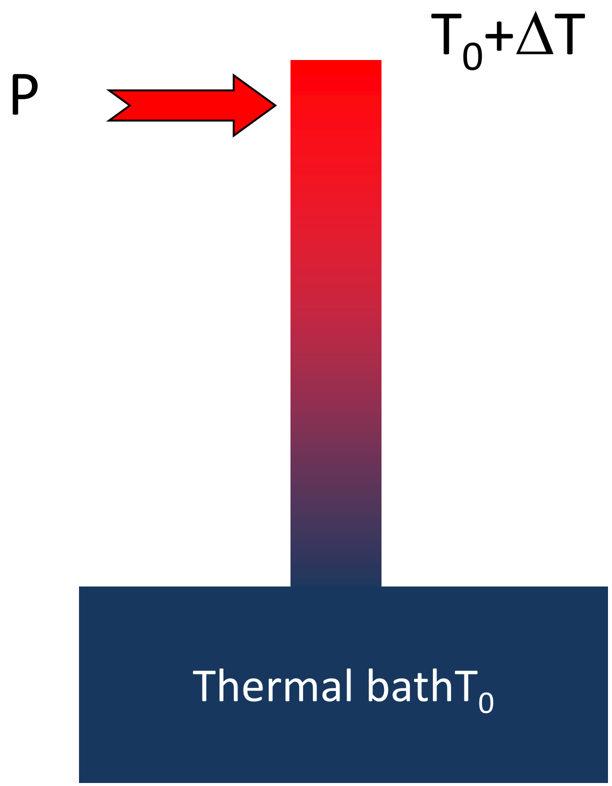






Thermal conductance

- Known power
- Measure of ΔT
- K is a mean value integrated over ΔT
- Dynamic measurement by modulating P
- Nanowire ??
- Connection to heat bath and to the thermometer
- Thermal contact



$$K = \frac{P}{\Delta T}$$

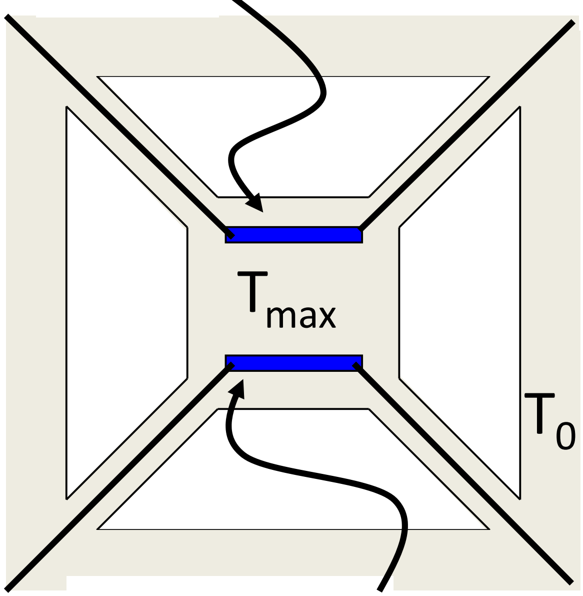
Nanowires

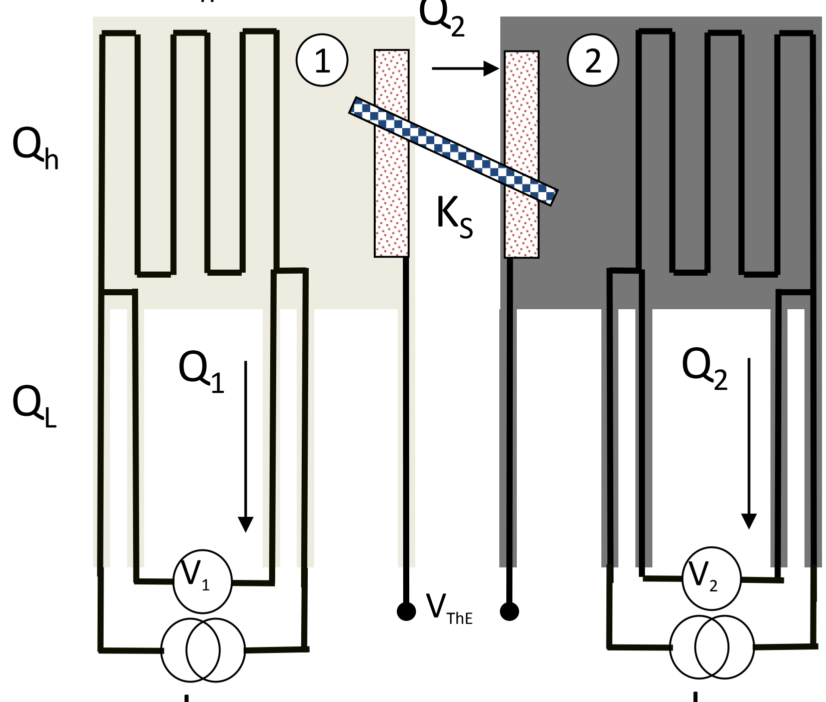
Thin films

Membranes

Principle of suspended sensors

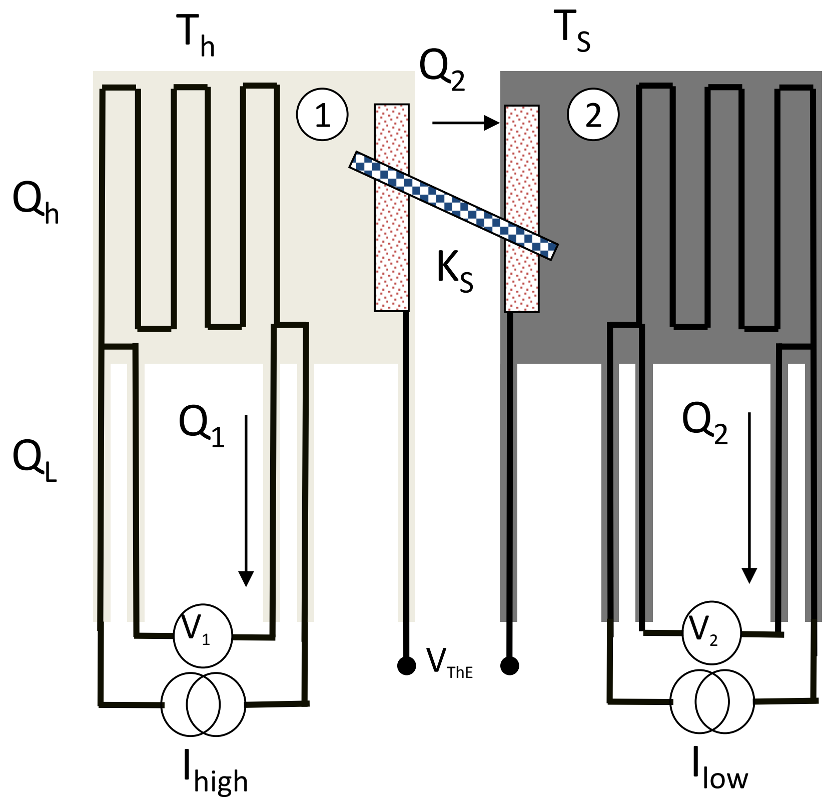
- Method based on a temperature gradient
- Between the membrane and a thermal bath
- Between two membranes

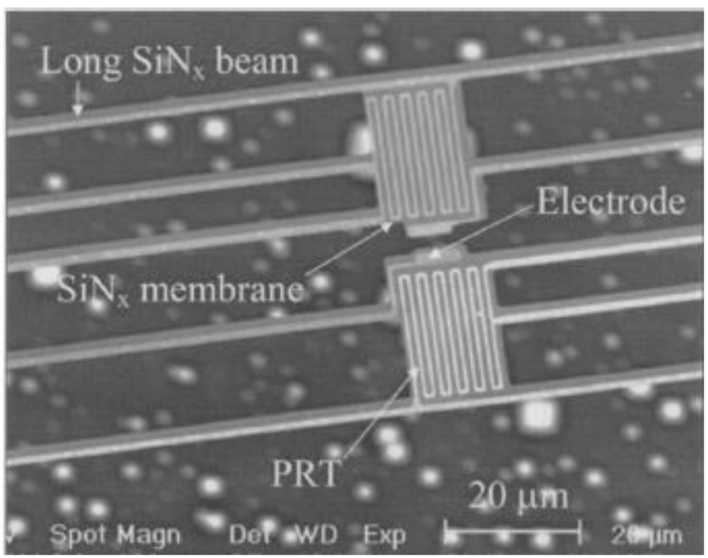




L.Shi/A. Majumdar method for nanowire

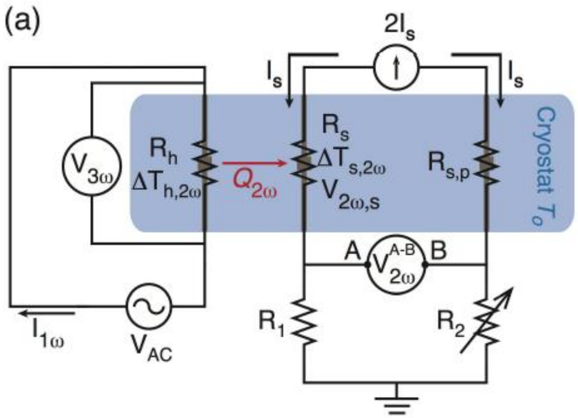
- Measure of K and C and of the thermoelectric power
- Platinum thermometry
- Adapted to self grown nano-objects
- Major difficulty: the thermal contact between the nanowire and the heat bath
- Thermal model needed

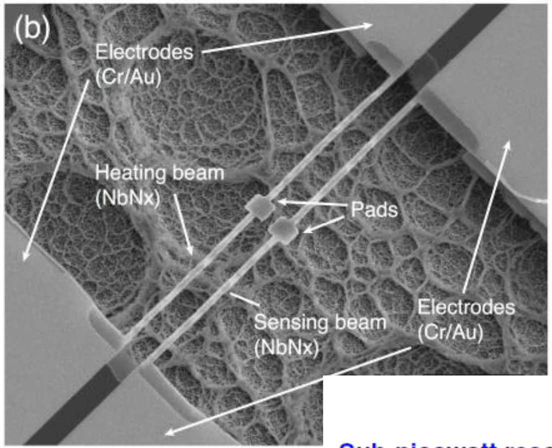




L. Shi, ……, P. Kim, A. Majumdar, Journal of Heat Transfer **125**, 881 (2003)

Extension with NbN thermometry: subpicoWatt

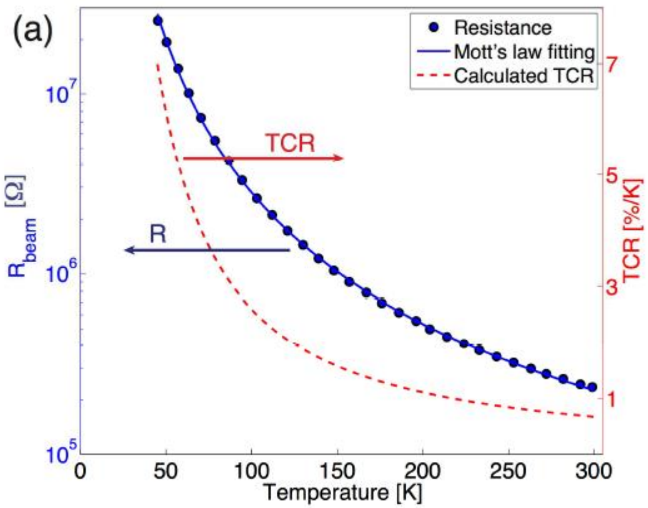


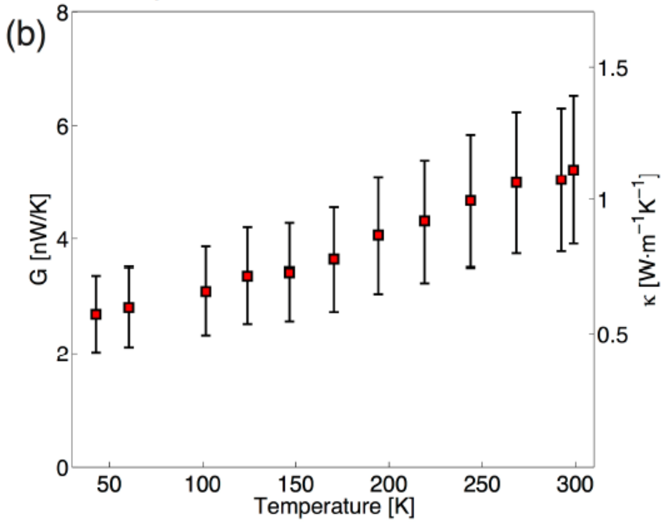


REVIEW OF SCIENTIFIC INSTRUMENTS **85**, 094903 (2014)

Sub-picowatt resolution calorimetry with niobium nitride thin-film thermometer

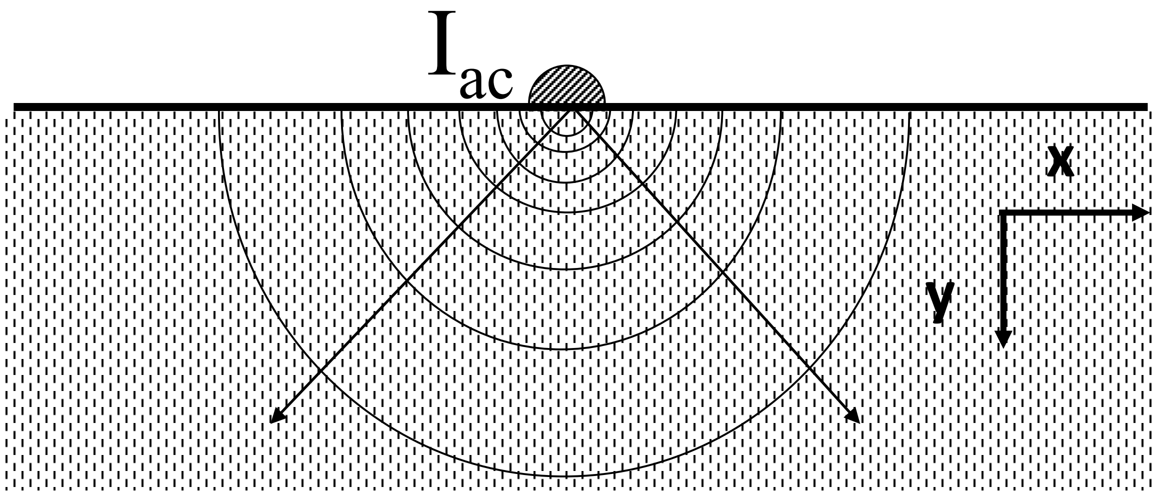
Edward Dechaumphai and Renkun Chen[a)]
Department of Mechanical and Aerospace Engineering, University of California, San Diego, La Jolla, California 92093, USA

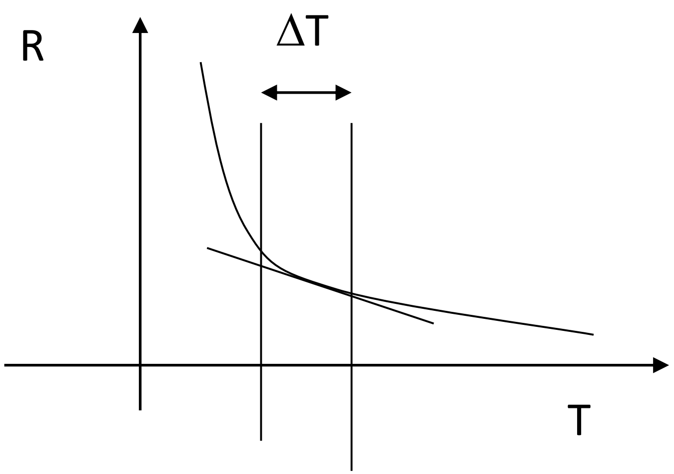




Thin films

- Heater=thermometer
- Thermal conductance measurement at low frequency (Cahill RSI 1990)
- Heat capacity measurement at high frequency) Birge & Nagel (RSI 58, 1464 (1987))





- $I_{ac} \sim 1\omega$ (Ohmic component)
- $T_{ac} \sim I^2 \sim 2\omega$
- $R \sim T \sim 2\omega$
- $V_{3\omega} \sim I_{ac}R \sim 3\omega$

D. G. Cahill, Rev. Sci. Instrum. **61**, 802 (1990)

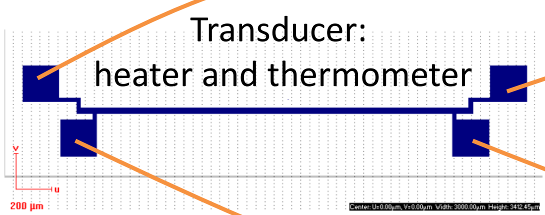
J.-Y. Duquesne, Phys. Rev. B. **79**, 153304 (2009)
Yanqing Liu, OB, J. Phys. Conf. Series (2014)

- Focus on electrical based measurement
- Problems related to measurement using continuous signal
- Preamplification of the signal before measurement
- AC thermal measurements: proposed since 1910 by O. Corbino
- Measure by lock-in amplifier
- Differential geometry


Orso Mario Corbino (1876-1937)

O.M. Corbino, Phys. Z. **11**, 413 (1910), *ibid.* **12**, 292 (1911)

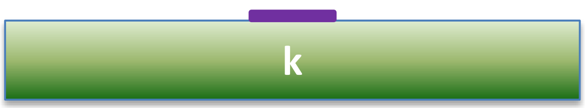
3 omega : Principles of measurement



$I_{AC} \sim \omega$

$P_{joule} \sim 2\omega$, $\Delta T_{ac} \sim 2\omega$, $\Delta R_{tr}(T) \sim 2\omega$

$V_{3\omega} \sim R_{tr}(T) \cdot I_{AC} \sim 3\omega$



Thermal penetration depth:

$$\mu = \sqrt{\frac{D}{\omega_{th}}}$$

D: thermal diffusivity

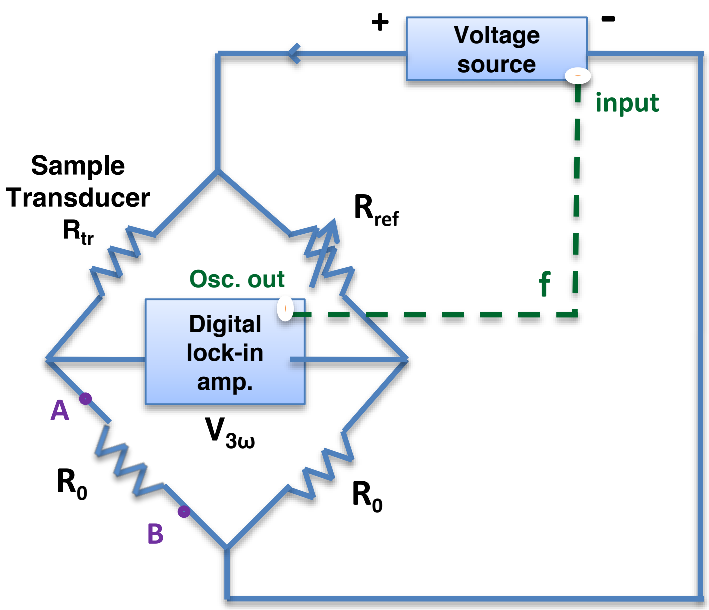
$\omega_{th} = 4 \cdot \pi \cdot f_e$

Low frequency regime: $\mu >> 2b$

High frequency regime: $\mu << 2b$

$V_{3\omega}(\omega)$ in low frequency regime --> k

Wheatstone bridge (biased by a voltage source)



$$k = \frac{\alpha \cdot R_{tr}^2 \cdot I^3}{4 \cdot \pi \cdot l} \left(\frac{dV_{3\omega_{inph.}}}{d \ln \omega} \right)^{-1} \cdot \frac{R_0}{R_{ref} + R_0}$$

$$I = \frac{V_{AB}}{R_0}$$ *(R_0 = 120 Ω)*

(Y. Liu, Jacques RICHARD, OB, TPS, Institut Néel)

3 omega : Principles of measurement

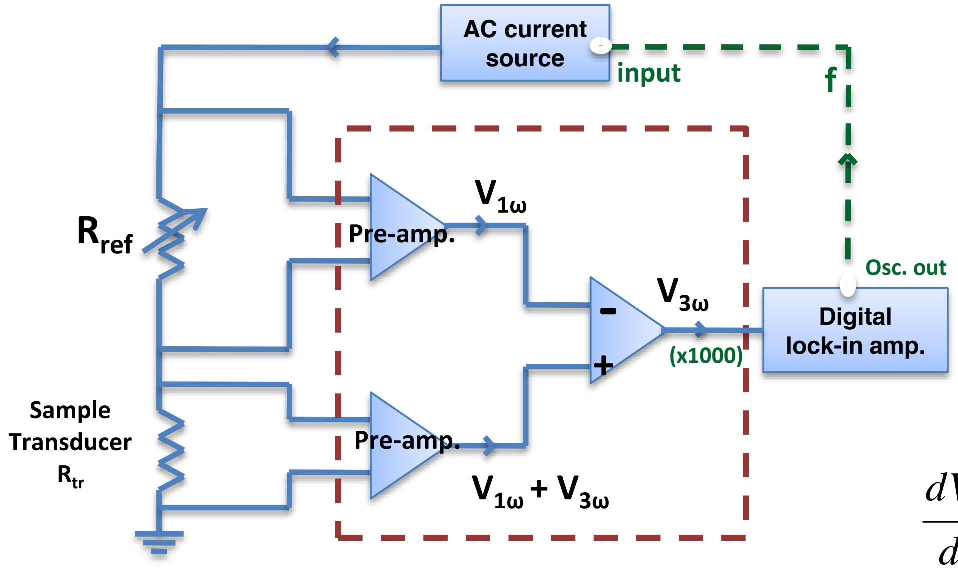
Differential bridge (biased by a current source)

$V_{measured} = V_{1\omega} + V_{3\omega}$

$\frac{V_{3\omega}}{V_{\omega}} \sim \frac{1}{1000}$

R_{ref} :
- independent of f_e
- Subtraction of $V_{1\omega}$ improving the ratio of $\frac{V_{3\omega}}{V_{\omega}}$
- $\frac{\Delta R}{R} \sim 10^{-3}$

f_e: 100 ~ 1000 Hz



Home made device
(Néel electronic service, Jean-luc Mocellin)

$$k = \frac{\alpha \cdot R_{tr}^2 \cdot I^3}{4 \cdot \pi \cdot l} \left(\frac{dV_{3\omega_{inph.}}}{d \ln \omega} \right)^{-1}$$

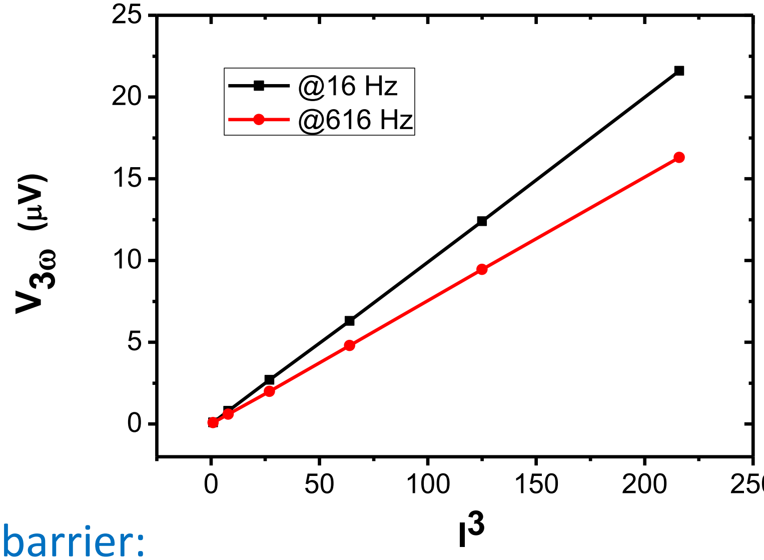
$\frac{dV_{3\omega_{inph.}}}{d \ln \omega}$: slope from the linaire fit of $V_{3\omega_{inph.}}(\ln \omega)$

(Y. Liu *et al.*, J. Phys. Conf. Series, 2014)

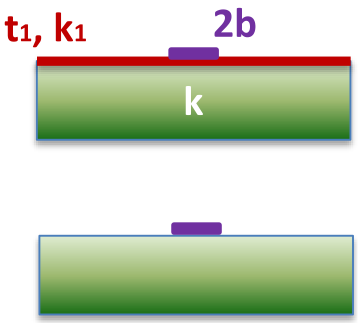
3 omega : Principles of measurement

✓ Verification of current range:

$$V_{3\omega} = a \cdot I^3$$



✓ Contribution of the thin films thermal barrier:



$t_1 << 2b$

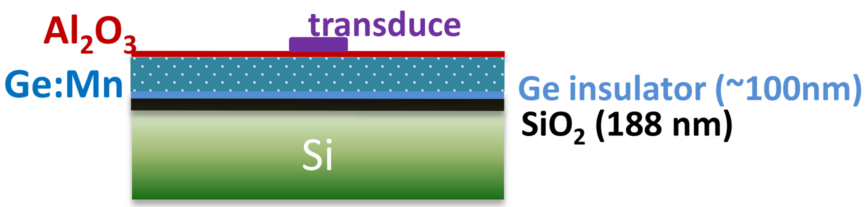
- one-dimensional thermal transport across the film
- Heat flux conserved through the thin film

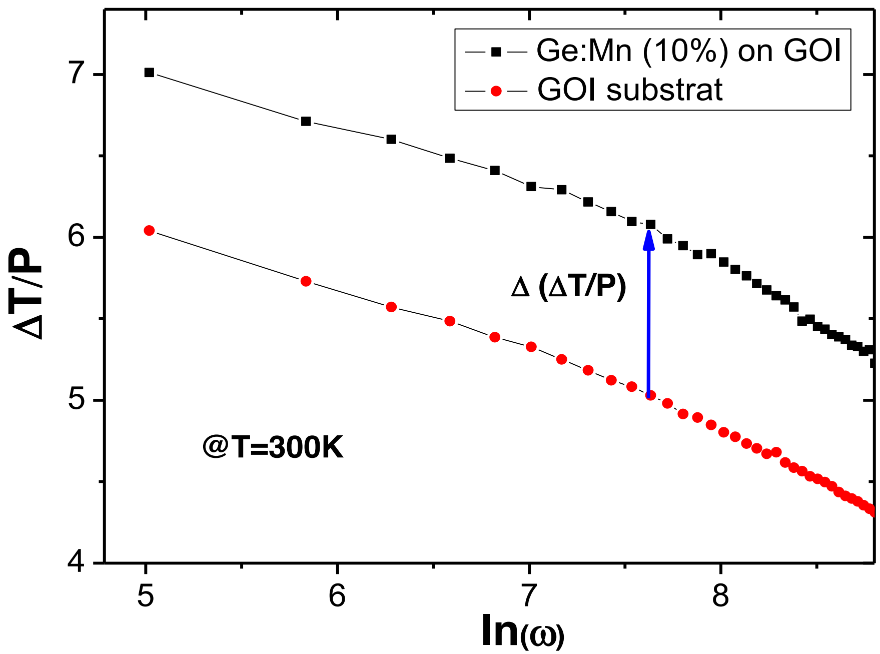
A thermal resistance barrier independent of f_e

A shift in real part of ΔT

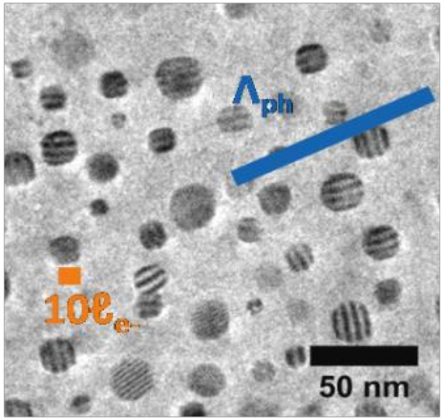
$$\Delta T = \frac{P_l}{\pi \cdot k_0}\left(-\frac{1}{2}\ln\frac{\omega}{\Omega} + \eta - i\frac{\pi}{4}\right) + \frac{P_l \cdot R'}{2b}$$

How to extract thermal conductivity





T (K)	300 K
k_{Si} $(Wm^{-1}K^{-1})$	130
$k_{Ge:Mn}$ $(Wm^{-1}K^{-1})$	3.3



@300K

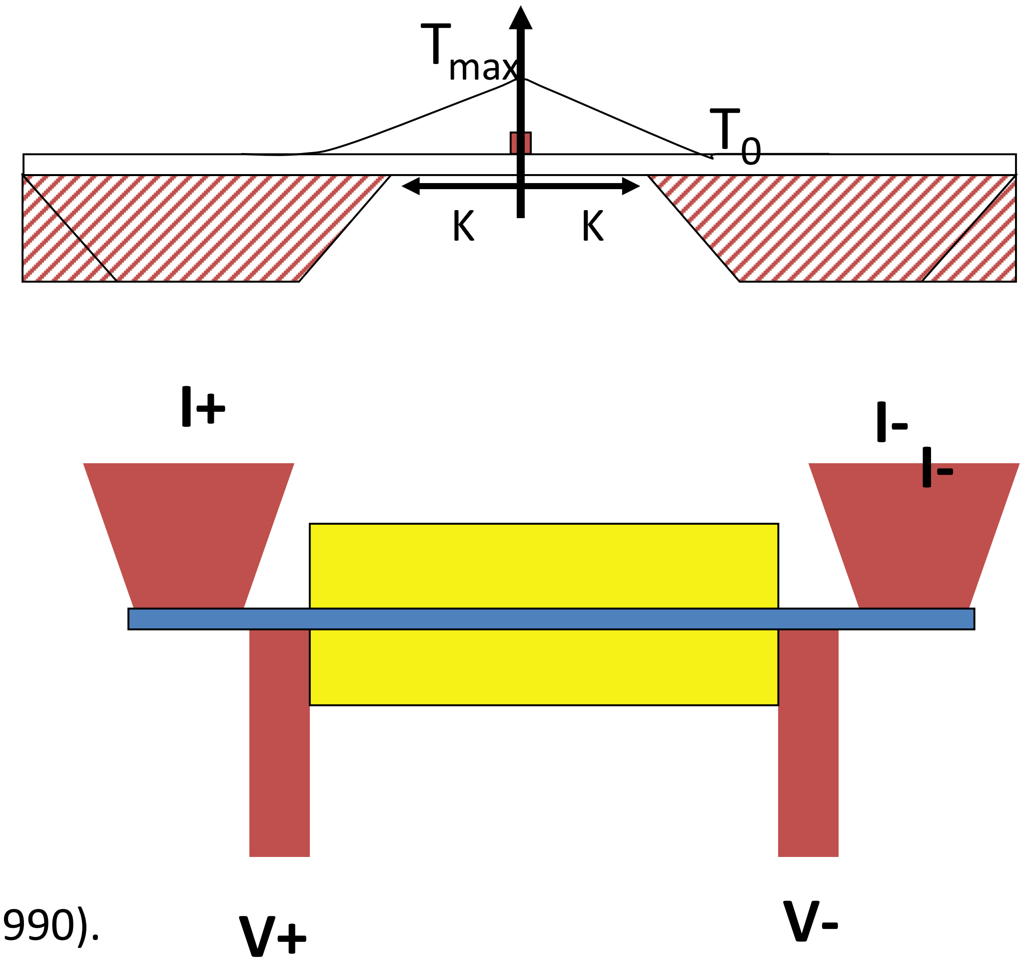
$k_{Ge} = 60\ Wm^{-1}K^{-1}$

$k_{Ge:Mn} = 3.3\ Wm^{-1}K^{-1}$

Reduction by a factor of 20

Membranes

- In plan thermal conductance measurement
- DC current
- Silicon nitride membrane

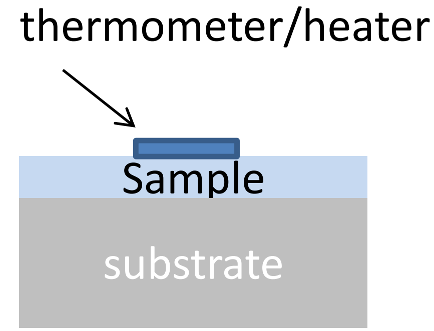


F. Völklein, Thin solid Films **188**, 27 (1990).

Method of measurements

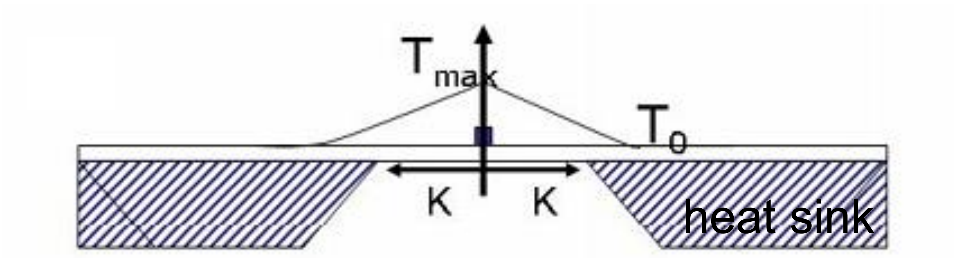
3ω method

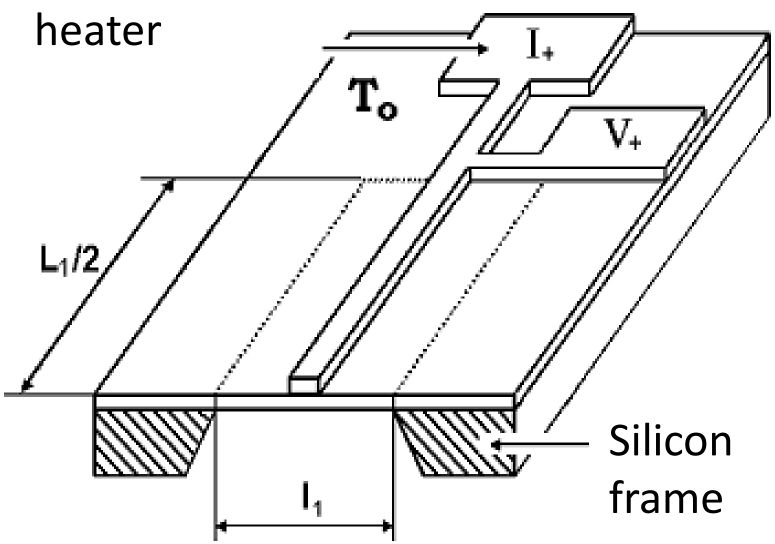
Metal line heated with $I_{AC} = f(\omega) = A\sin(\omega t)$
-> Dissipated power: $RI_{AC}^2 = f(2\omega)$
-> Resistance variation = f(2ω)
-> Voltage variation = f(2ω).f(ω)
-> Measured voltage: ohmic part V(ω)+ thermal part V(3ω)



Völklein method

-Direct current

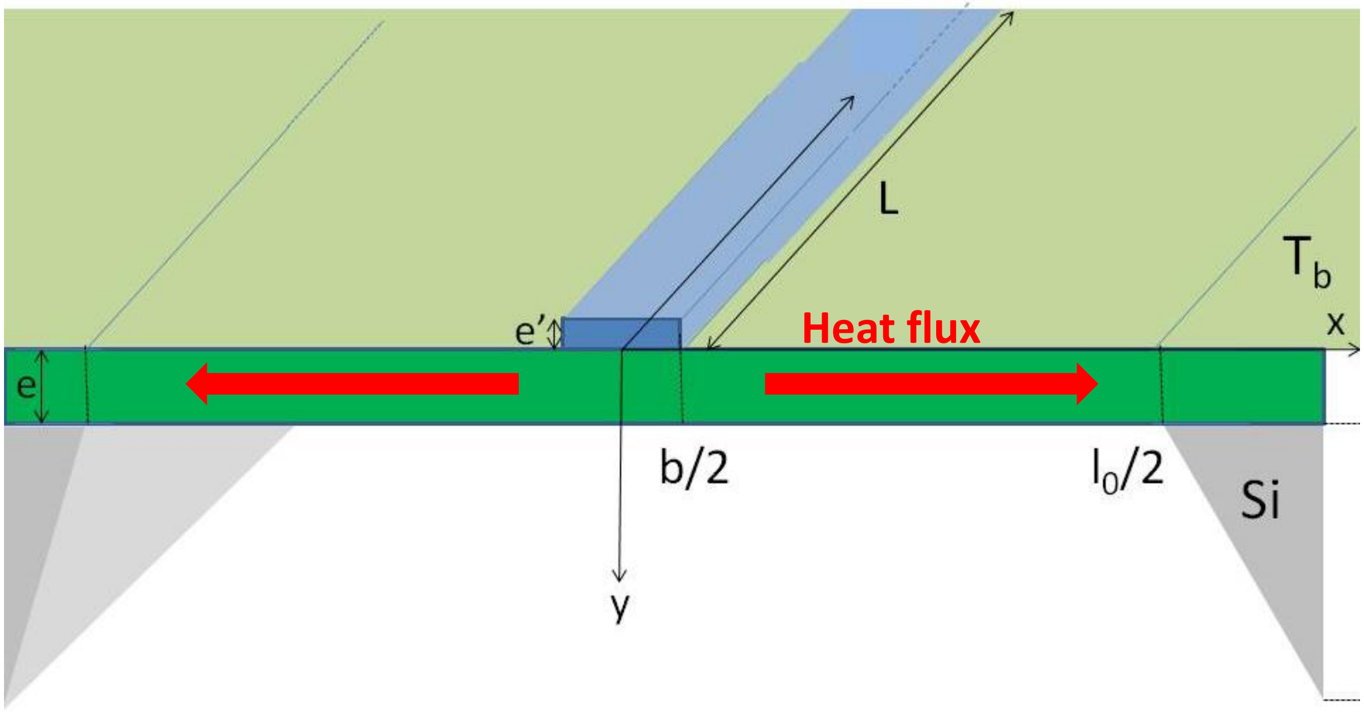




Heat balance: measurement of the thermal conductance in the plane

3ω / Völklein method: 3ω on membrane

$V(3\omega) = f(K,C)$



e = 100/400 nm
e' = ~ 50 nm
b = ~20 µm
L = ~1 mm
l_0 = 150/500 µm

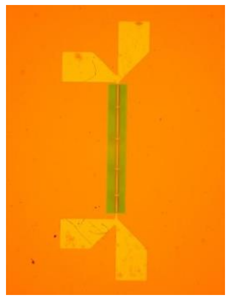
A. Jain and K. E. Goodson, Journal of Heat Transfer **130**, 102402 (2008).
A. Sikora, H. Ftouni, J. Richard.., and O. B, Rev. Sci. Instrum. **84**, 029901 (2013)
F. Völklein, H. Reith, and A. Meier, Phys. Status Solidi A **210**, 106–118 (2013)

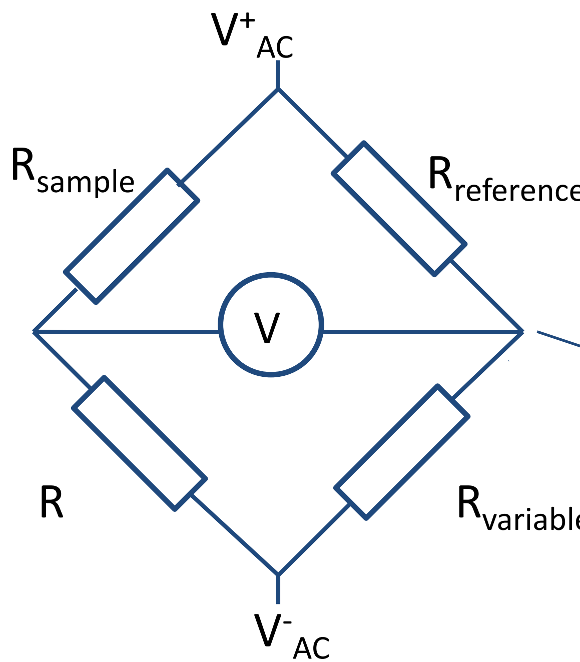
Signal treatment: Wheatstone bridge

Problem: $V(\omega) >> V(3\omega)$, $V(3\omega)/V(\omega) \approx 3 \times 10^{-4}$

Minimisation of $V(\omega)$ voltage thanks to a **Wheatstone bridge**

Membrane + NbN Thermometer/ heater





Reference: Thermometer/ heater NbN

$V_{3\omega}$ measurement

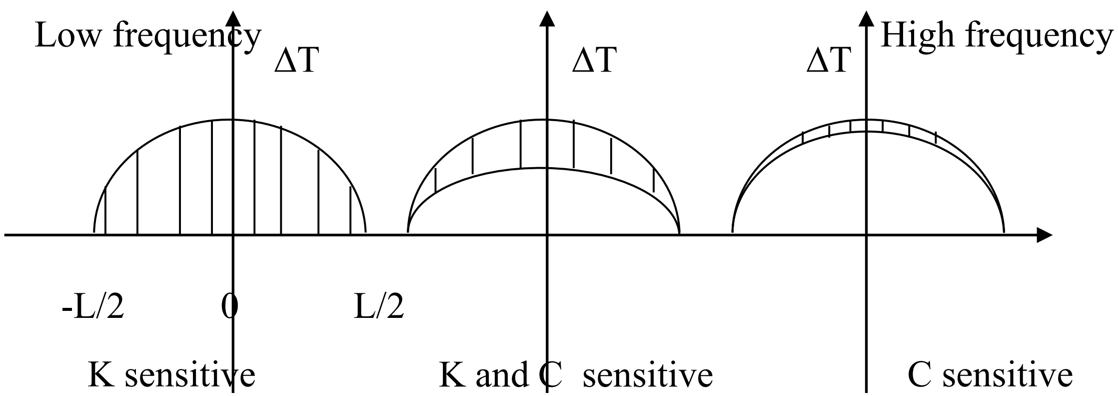
(bridge balancing)

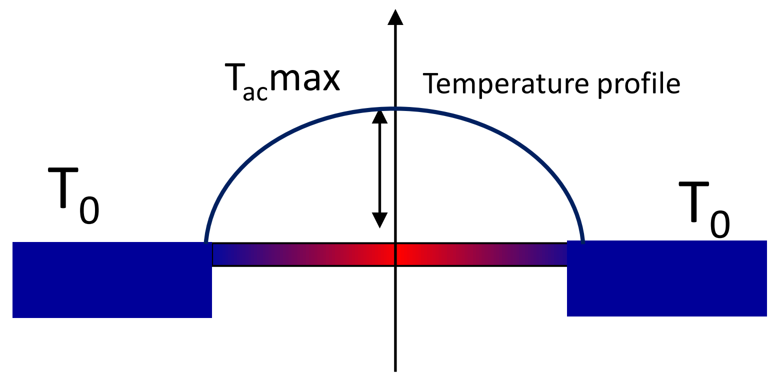
After balancing: $V(3\omega)/V(\omega) \approx 0.4$ (2V)

A. Sikora, H. Ftouni, J. Richard, C. Hébert, D. Eon, F. Omnès, and O. B, Rev. Sci. Instrum. **83**, 054902 (2012). erratum Rev. Sci. Instrum. **84**, 029901 (2013)

3 omega technique: equations

$$\rho C_P \frac{\partial}{\partial t}T(x,t) - k\frac{\partial^2}{\partial x^2}T(x,t) = \frac{I_0^2 \sin^2(wt)}{LS}[R + (\frac{dR}{dT})_{T_0}(\Delta T(x,t))]$$





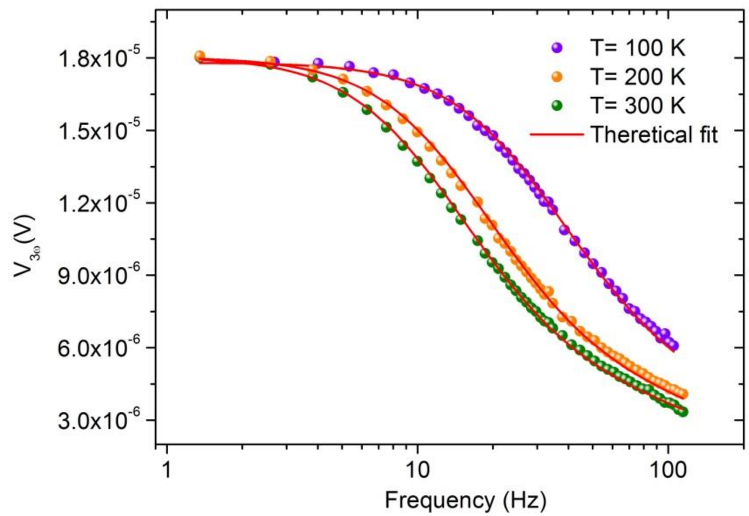
$$V_{3\omega} = \frac{4I_0^3R^2\alpha}{\pi^4 K(\sqrt{1+(2\omega\gamma)^2}}$$

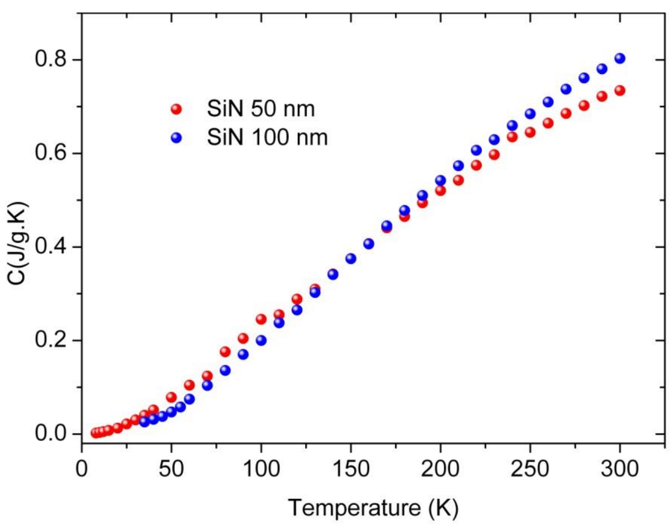
⇨

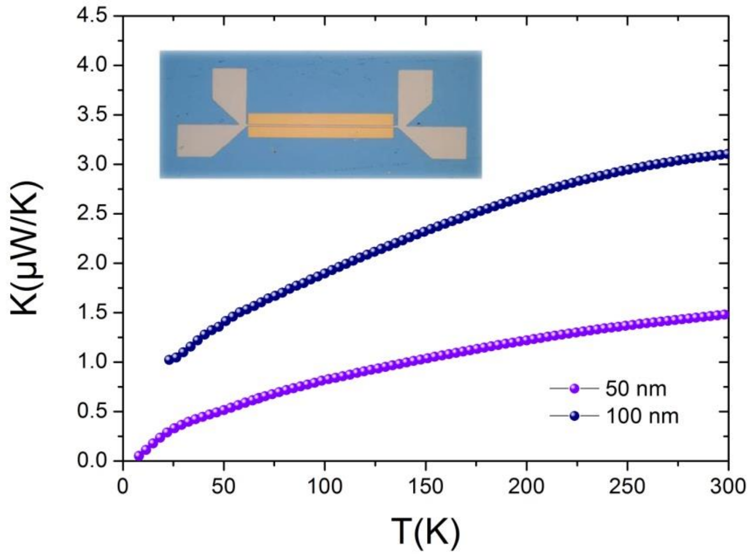
$$K = \frac{4I_0^3R^2\alpha}{\pi^4 V_{3\omega}}$$

O. Bourgeois, Th. Fournier and J. Chaussy, J. Appl. Phys, **101**, 016104 (2007)

Thermal conductivity and specific heat measurements on very thin membrane : 50nm thick



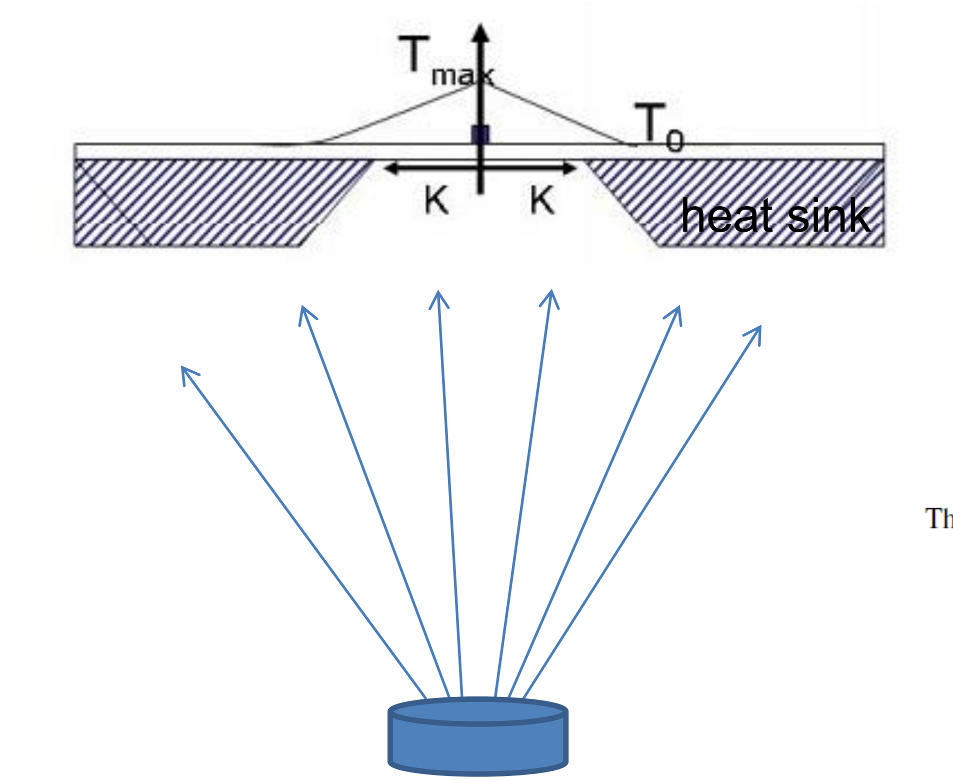




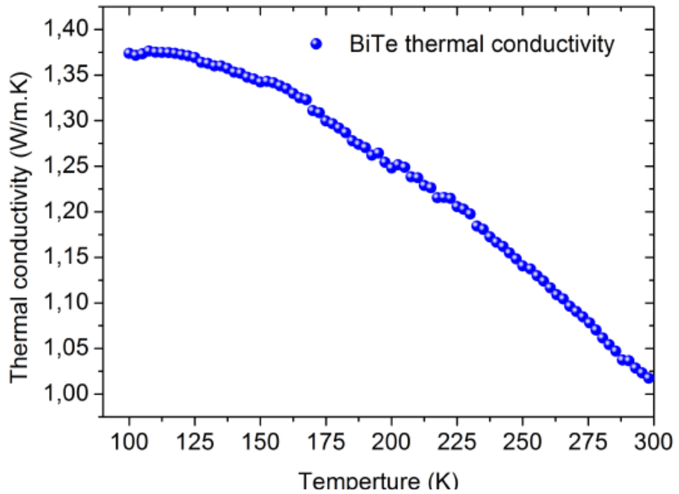
Hossein Ftouni, Dimitri Tainoff, Jacques Richard, Kunal Lulla, Jean Guidi, Eddy Collin, and O.B., Rev. Sci. Instrum. **84**, 094902 (2013).

Thesis of Hossein Ftouni (I. Néel, 2013)

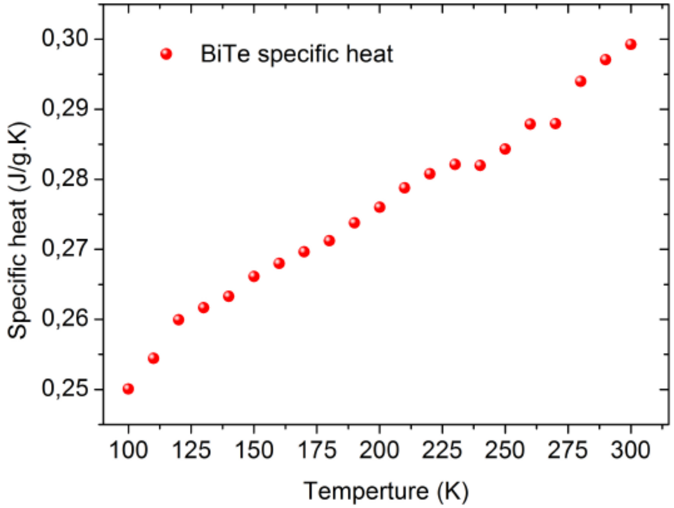
Generic method: measurement of thermal properties of evaporated materails



Thesis Hossein Ftouni (Néel I. sup. O.B.)



Thermal conductivity of 200 nm thick Bi_2Te_3 film as a function of temperature

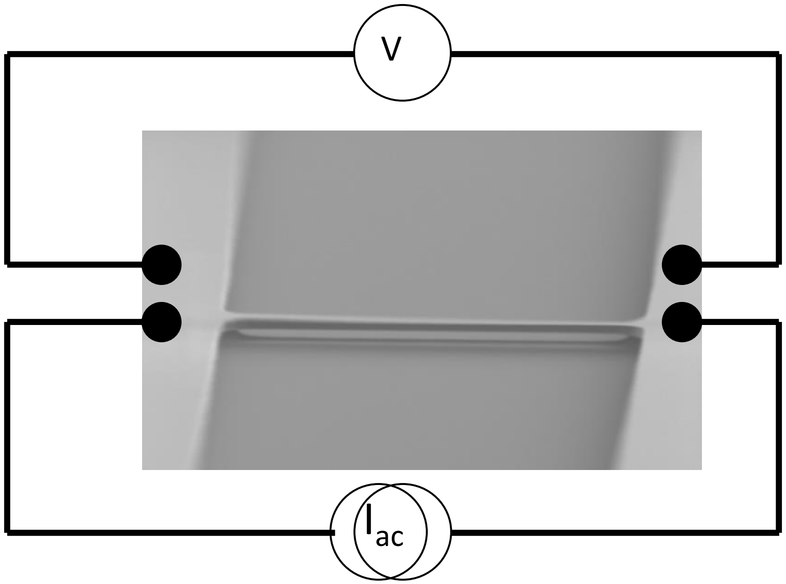


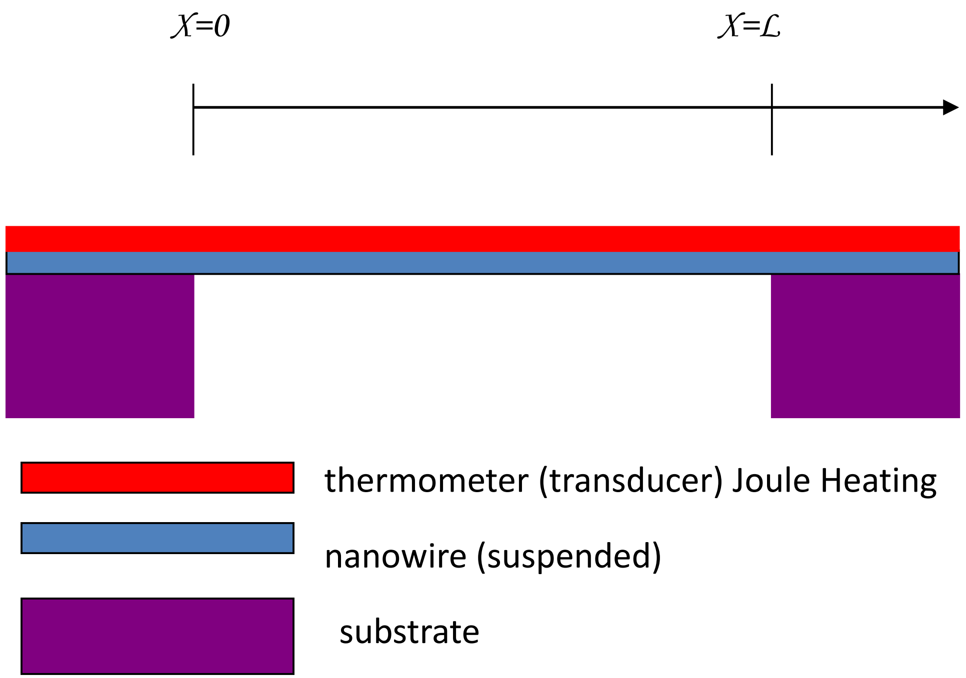
5.3: Specific heat of 200 nm thick Bi_2Te_3 film as a function of temperature.

3 omega at the nanoscale

3ω method Measure in plane (longitudinal)

- Proposed by Lu et coll.
- Adapted for suspended nanowires
- Low frequency limit



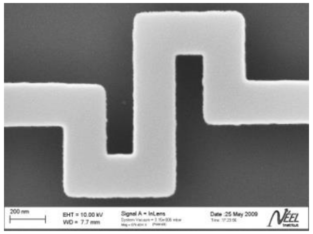


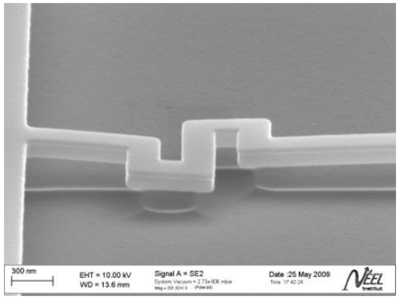
Lu L, Yi W and Zhang DL 2001 *Rev. Sci. Instrum.* **72** 2996

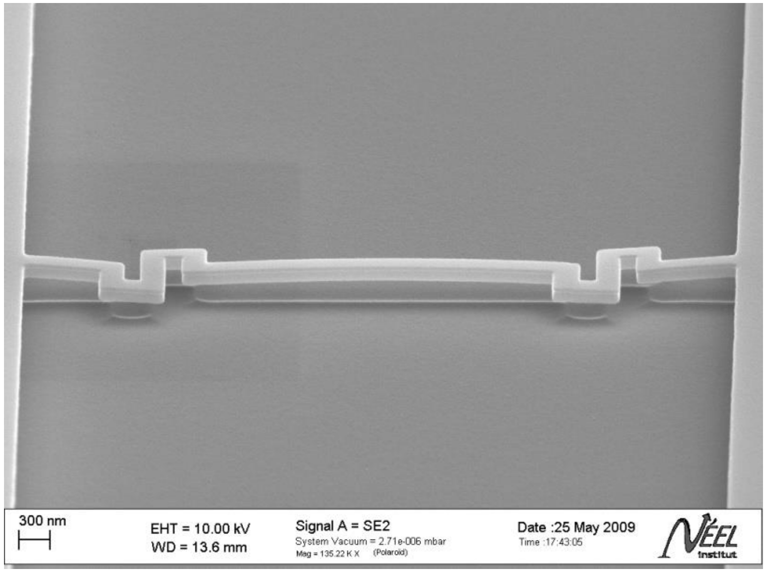
O. B., Th. Fournier and J. Chaussy, J. Appl. Phys, **101**, 016104 (2007)

Blocking phonons at the nanoscale

- Introducing of a serpentine nanostructure in the suspended nanowire (5µm long)
- Length scale 200nm
- Blocking only the ballistic phonons
- Reduce the thermal conductance

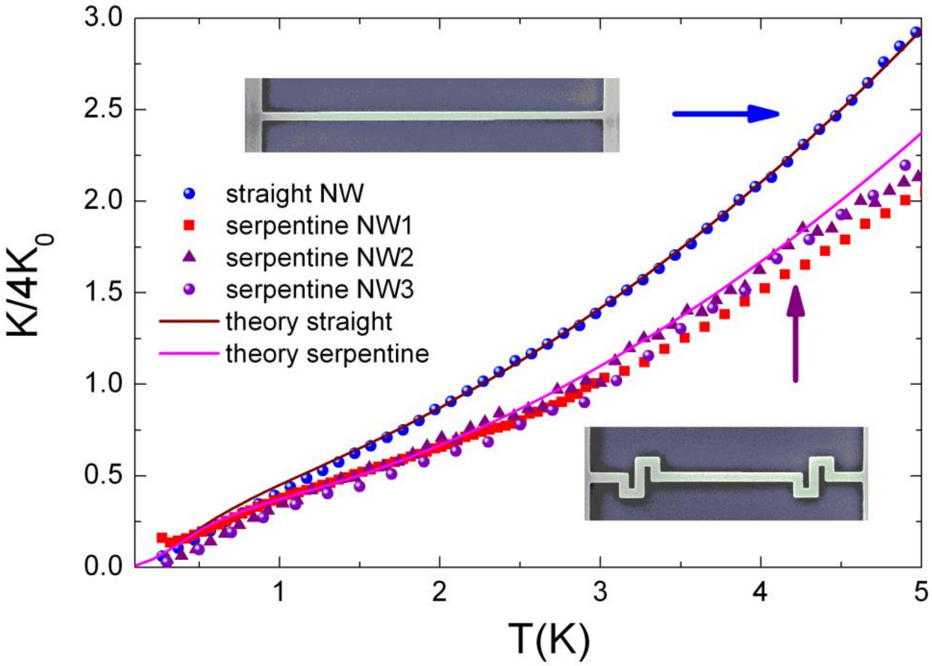






Strong reduction of thermal conductance

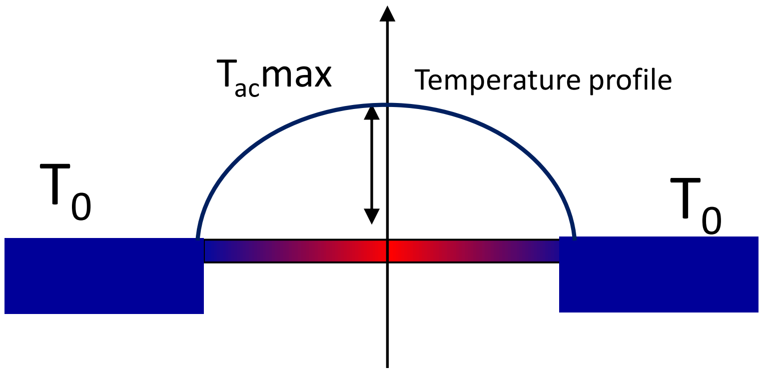
- Reduction of up to 40% of the thermal conductance
- Model this system by transmission function analysis
- Very good agreement between the model and the data
- Concerning ballistic phonons the reduction is of the order of 80%



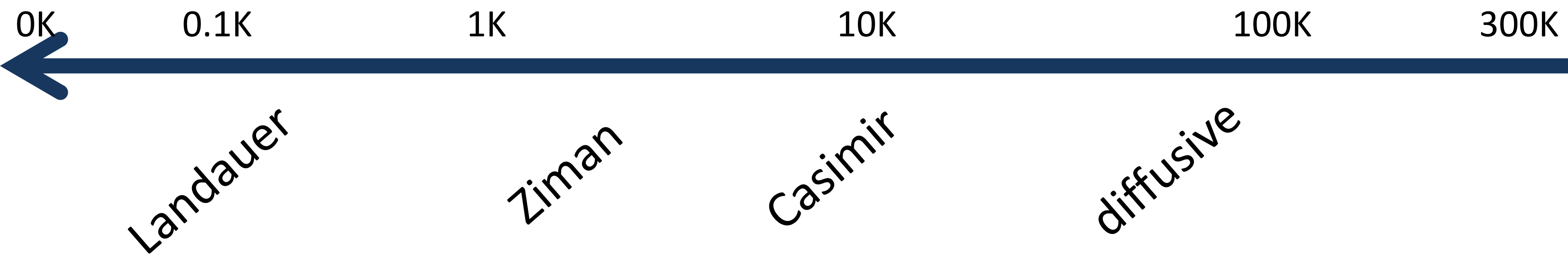
J-S. Heron, T. Fournier, N. Mingo, and O. B., Nano Letters, **9**, 1861 (2009)
J-S. Heron, C. Bera, T. Fournier, N. Mingo, and O. B., Phys Rev B **82, 155458** (2010)
C. Blanc, A. Rajabpour, S. Volz, O. B, Appl. Phys. Lett. **103**, 013104 (2013).

3 omega: Limitations at very low temperature

$$\rho C_P \frac{\partial}{\partial t} T(x,t) - k \frac{\partial^2}{\partial x^2} T(x,t) = \frac{I_0^2 \sin^2(wt)}{LS} [R + (\frac{dR}{dT})_{T_0} (\Delta T(x,t))]$$



Phonon transport : specificity of low temperature



Silicon nanowire of diameter: 100nm

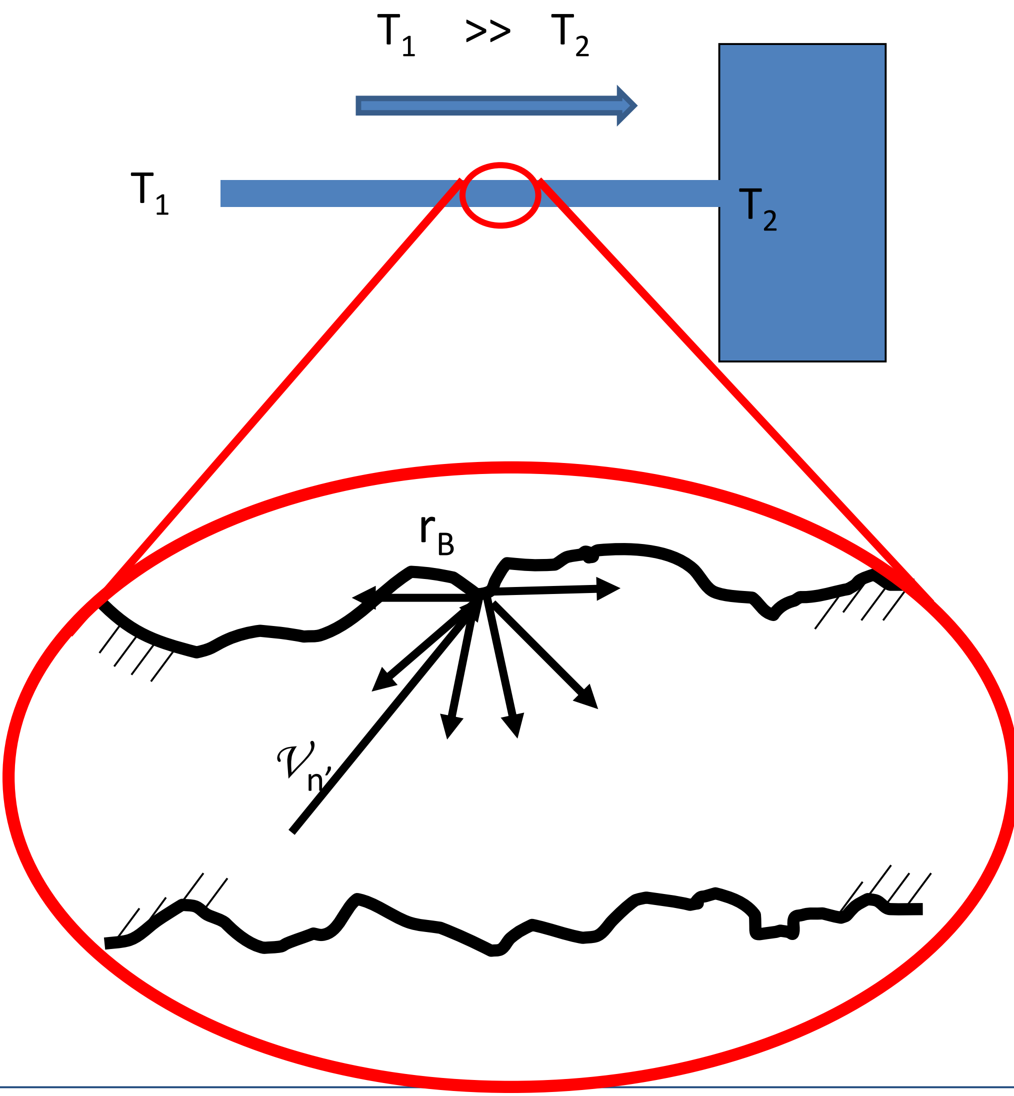
λ_{dom} (T) < Λ_{ph} (T) < L	Diffusive regime	Maxwell Boltzmann : $k = \frac{\upsilon_s}{3}\int c(\omega)\Lambda(\omega)d\omega$
λ_{dom} (T) < L ~ Λ_{Cas} < $\Lambda_{ph\text{-}bulk}$ (T)	Casimir regime	Casimir model $K_{Cas} = \beta\Lambda_{Cas}T^3$
λ_{dom} (T) ~ L < Λ_{Ziman} (T)	Ziman regime	Ziman model $\Lambda_{Ziman} = \frac{1+p}{1-p}\Lambda_{Cas}$
L < λ_{dom} (T) < Λ_{ph} (T)	Ballistic regime	Landauer : $T = \frac{1}{1+\Lambda_{eff}/L}$

Finite size effect: Casimir theory for phonon transport

- Mean free path Λ_{ph}
- Λ_{Cas}=D (Diameter of the nanowire)
- Boundary scattering: black body radiation for phonons
- Still diffusive
- Comment on the specific heat (kinetic equation)

$$K(T) = 3.2 \times 10^3 \left(\frac{2\pi^2 k_B^4}{5\hbar^3 v_s^3}\right)^{(2/3)} \frac{S\Lambda_{Cas}}{L} T^3$$

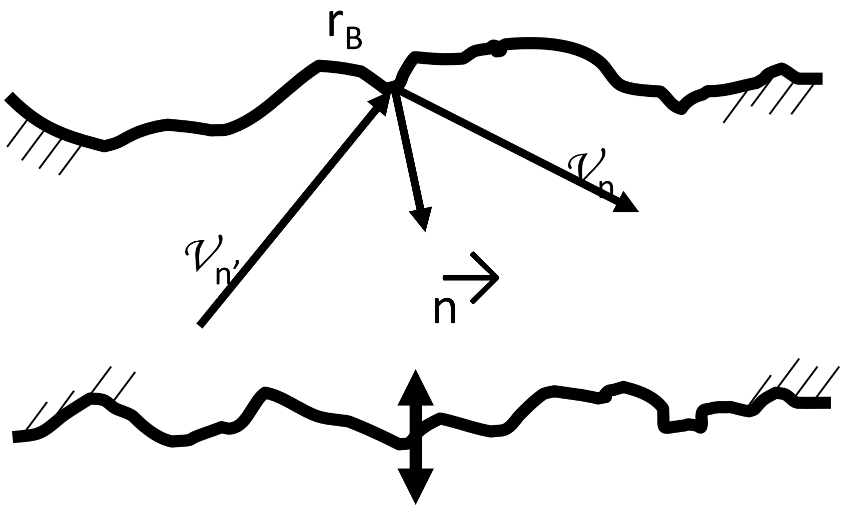
Breakdown of the concept of thermal conductivity



Casimir theory and beyond

$$\lambda_{Dom} = \frac{h v_S}{4.25 k_B T}$$

- At low temperature, the dominant phonon wave length is increasing:
- Probability of specular reflection $p(\lambda_{dom})$ depending on λ_{dom} (phenomenological parameter)
- $p(\lambda_{dom})=0$ (perfectly rough surface) $\lambda_{dom} \ll \eta_0$ Casimir model
- $p(\lambda_{dom})=1$ (perfectly smooth surface) $\lambda_{dom} \gg \eta_0$



η_0 is the root mean square of the asperity

J.M. Ziman *Electrons and phonons* (Clarendon Press, Oxford, 2001)

Casimir Theory and beyond : the Ziman model

- Ziman-Casimir model

$$\Lambda_{ph} = \frac{1+p}{1-p}\Lambda_{Cas}$$

where *p* probability of specular reflection

If *p*=0 transport is diffusive (Casimir), if *p*=1 ballistic transport

$$p(\lambda) = \int P(\eta) e^{\frac{-16\pi^3\eta^2}{\lambda_{dom}^2}} d\eta$$

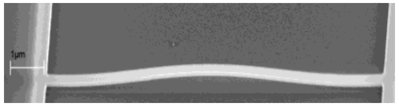
Probability distribution of asperity

$$P(\eta) = \frac{1}{\eta_0} e^{-\eta/\eta_0}$$

$$K(T) = 1.35 \times 10^{-5} \left(\frac{2 - e^{-4\pi\lambda_{dom}(T)/\eta_0}}{e^{-4\pi\lambda_{dom}(T)/\eta_0}} \right) \Lambda_{Cas} T^3$$

Ziman model of phonon transport: ballistic contribution

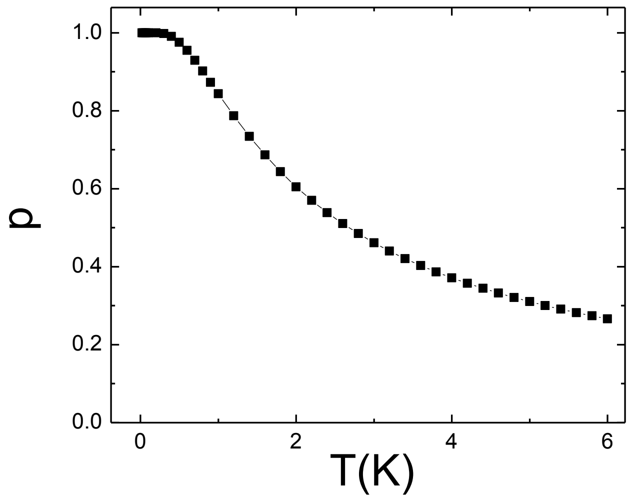
$$K = 3.2\times 10^{-3}\left(\frac{2\pi^2 k_B^4}{5\hbar^3 v_s^3}\right)^{2/3}\frac{S\Lambda_{eff}}{L}T^3$$

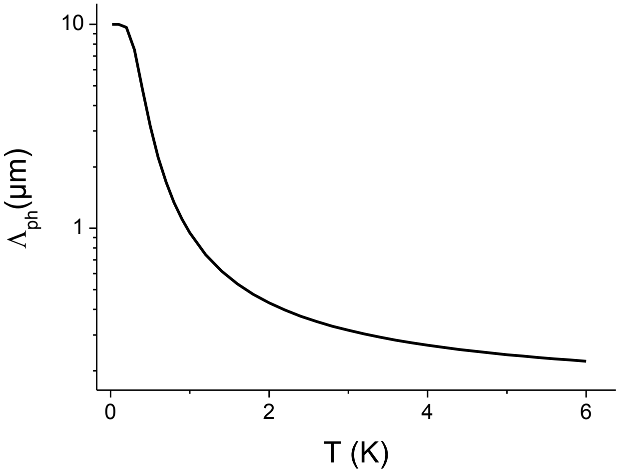


$$\lambda_{Dom} = \frac{hv_S}{4.25k_B T}$$

$$\Lambda_{eff} = \frac{1+p}{1-p}\Lambda_{Cas}$$

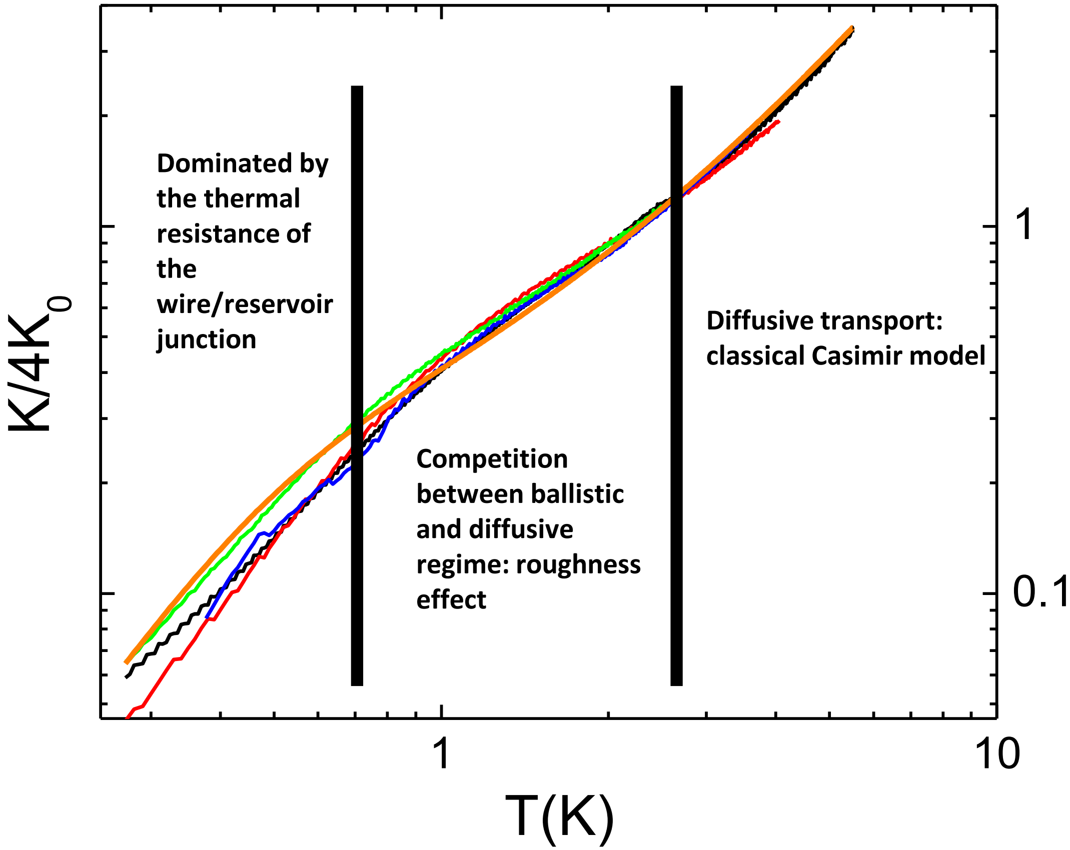
$$\Lambda_{ph}^{-1} = \Lambda_{eff}^{-1} + L^{-1}$$

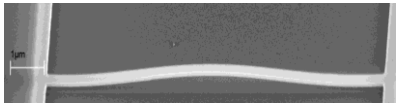




J.-S. Heron, T. Fournier, N. Mingo and O. Bourgeois, Nano Letters **9**, 1861 (2009).

Evidence of contribution from ballistic phonons





<u>Fitting parameter:</u>

- •Roughness h=4nm
- •Speed of sound 9000m/s
- •Contribution of the contact (Chang, C.; Geller, M. *Phys. Rev. B* **2005**, *71*, 125304.)

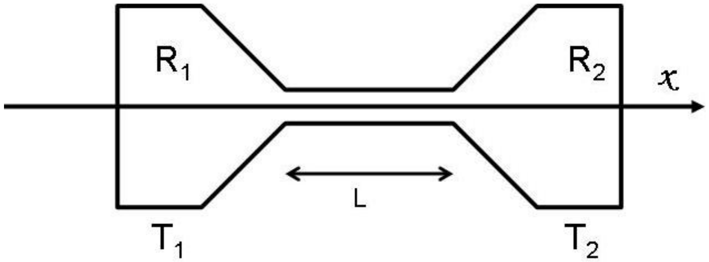
J.-S. Heron, T. Fournier, N. Mingo and O. B, Nano Letters **9**, 1861 (2009).

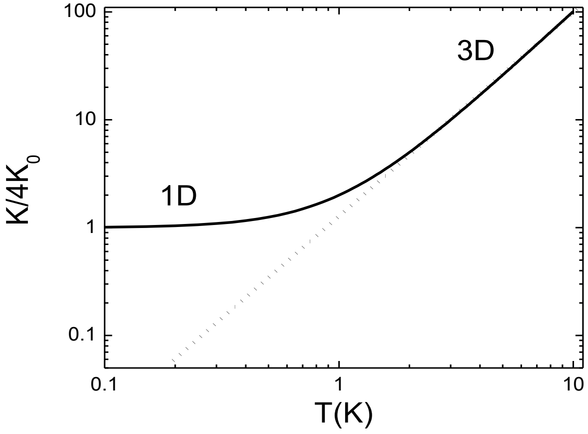
Beyond the 3 omega at low temperature

- Λ>>d (temperature defined over a volume Λ^3)
- **λ_{dom}>>d**
- 4 acoustic phonon modes

$$K_0 = \frac{4\pi^2 k_B^2 T}{3h}$$

- Conduction channel (Similar to the Landauer model of electrical conductance)
- Not dependant on the materials
- Valid whatever the heat carrier statistic
- Pendry, Maynard: flow of entropy or information



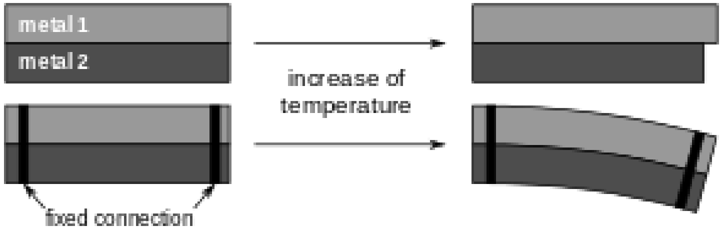


J.B. Pendry, J. Phys. **16**, 2161 (1983)

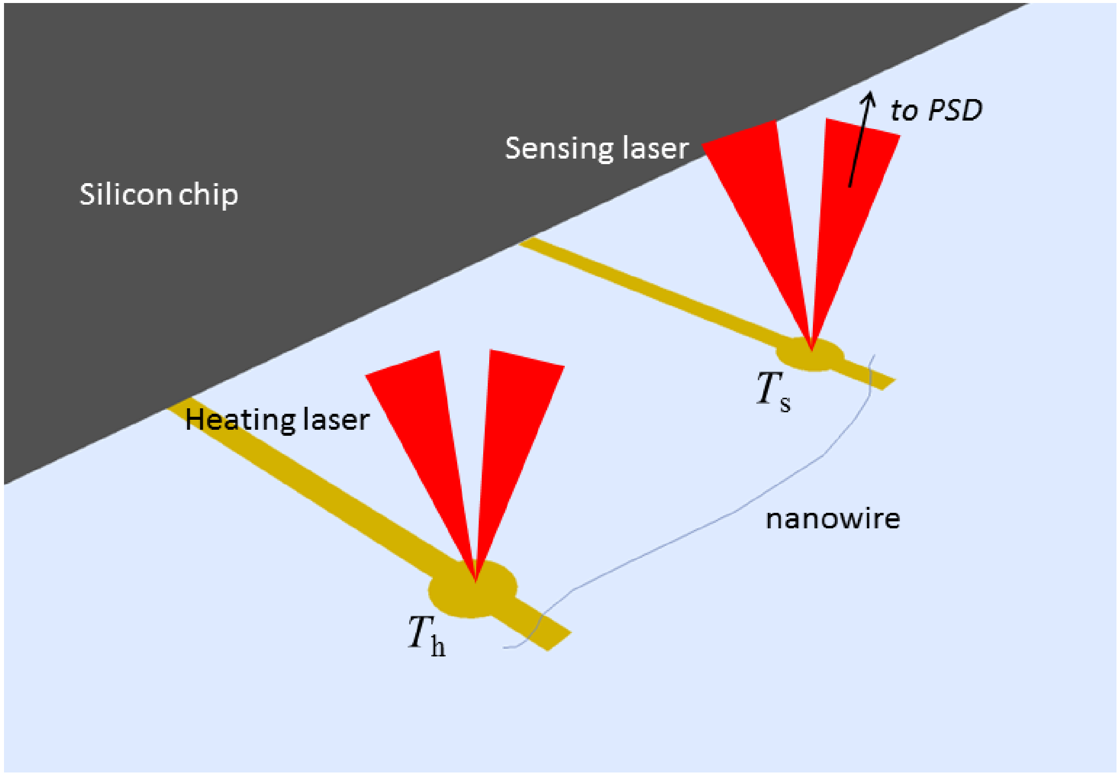
R. Maynard and E. Akkermans, Phys. Rev. B **32**, 5440 (1985)

L.G.C. Rego and G. Kirczenow, Phys. Rev. Lett. **81** 232 (1998)

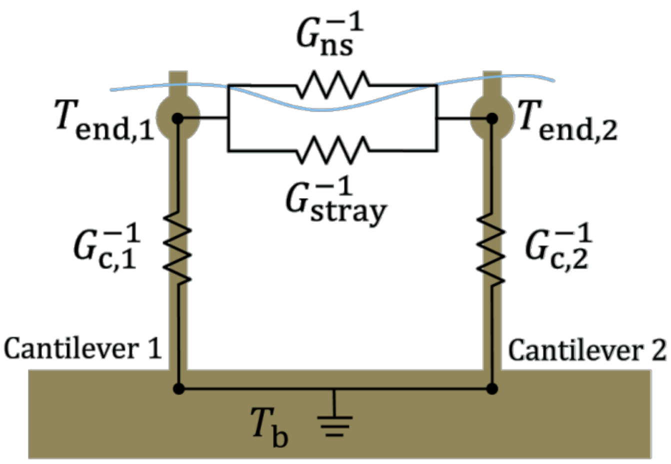
Mechanical technique based on bimetal cantilever: an old thermometry



Dual Cantilever Technique



C. Canetta and A. Narayanaswamy, Rev. Sci. Instrum., 84, 105002 (2013)



- G_{ns} – nanostructure conductance
- G_{stray} - stray conductance
- $G_{c,1}$, $G_{c,2}$ – cantilever conductances. Usually $G_{c,1} \approx G_{c,2}$

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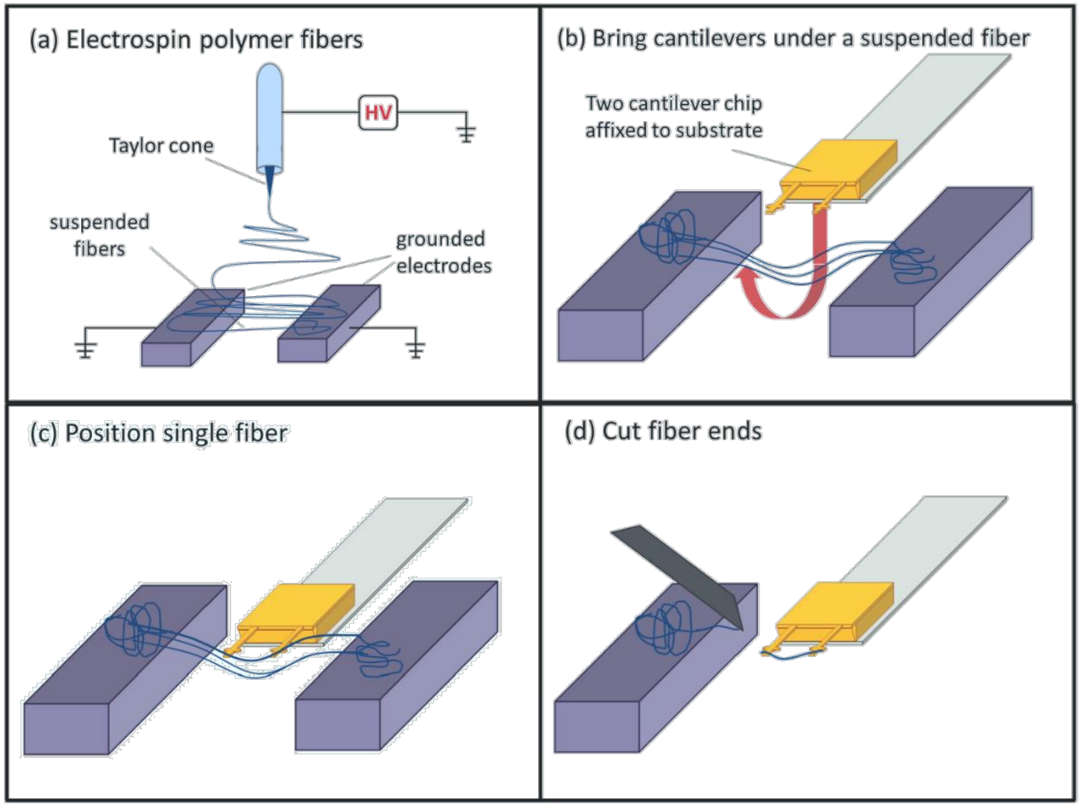
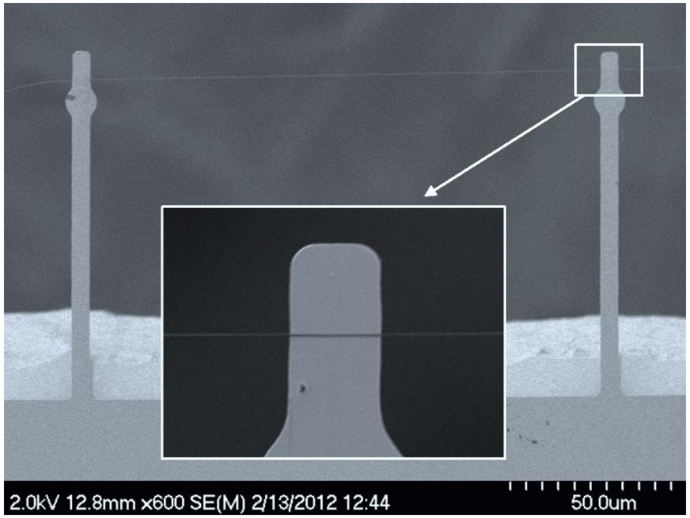
Method of attaching nanowires to cantilevers





Polystyrene fiber, diameter < 200 nm

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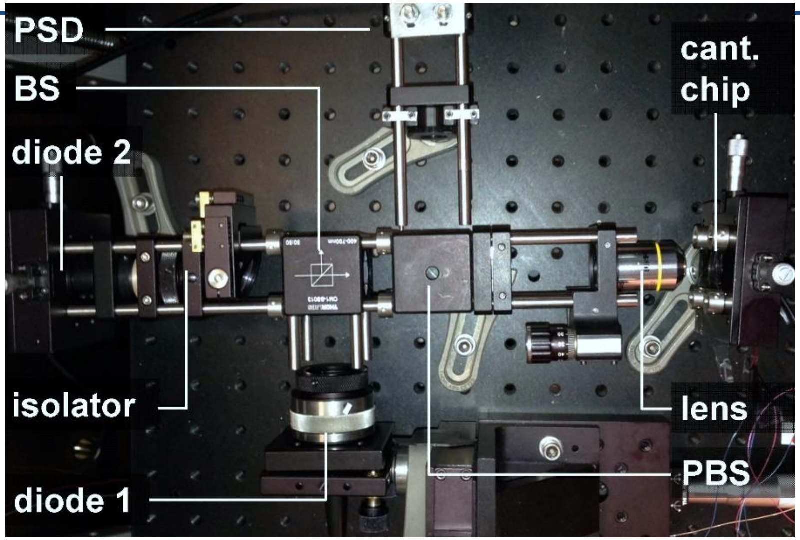
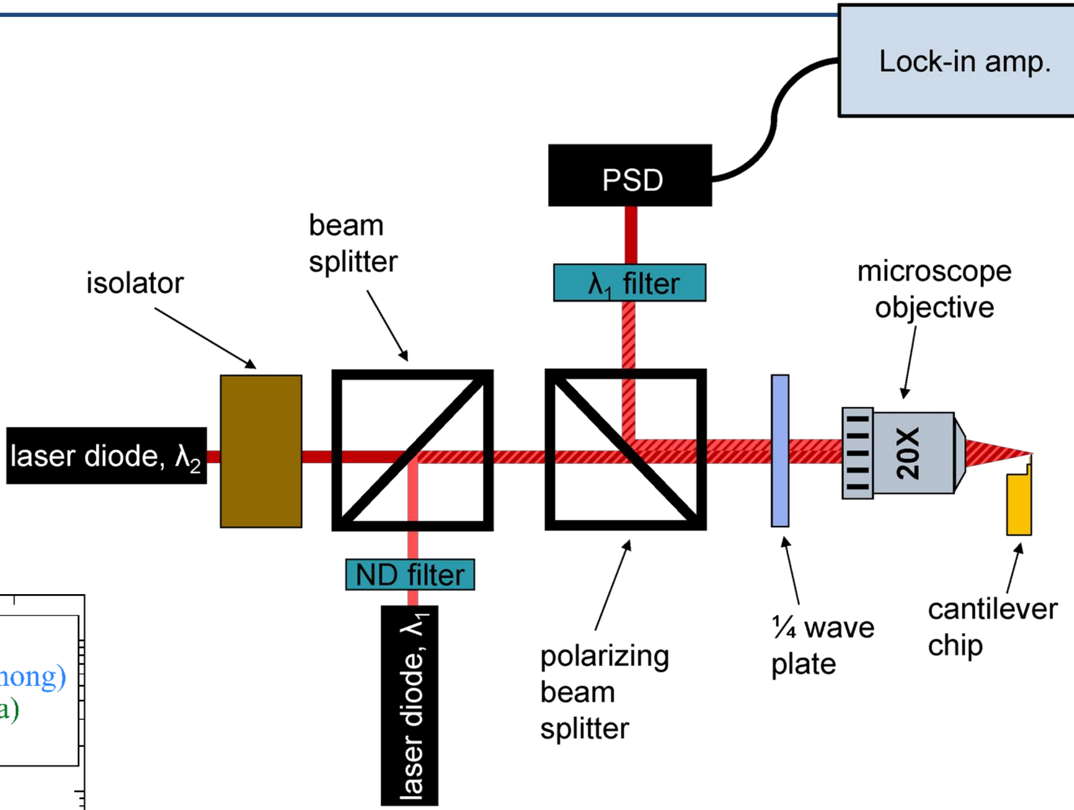
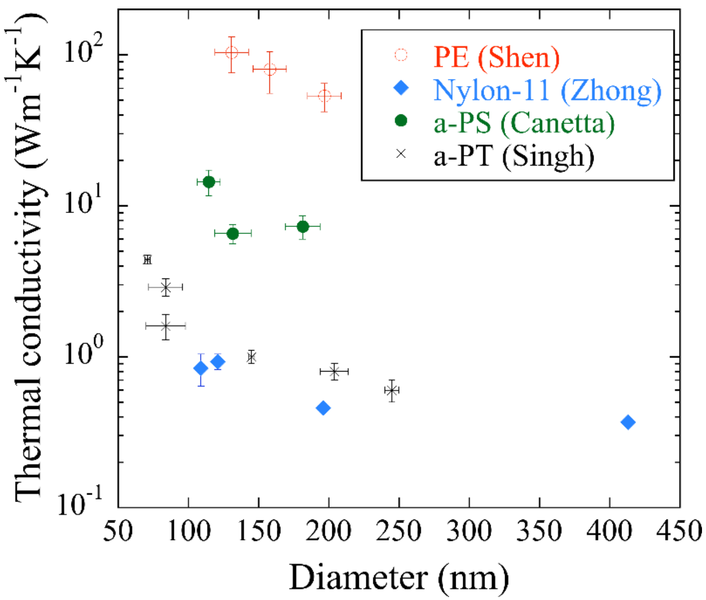
Measurement set-up







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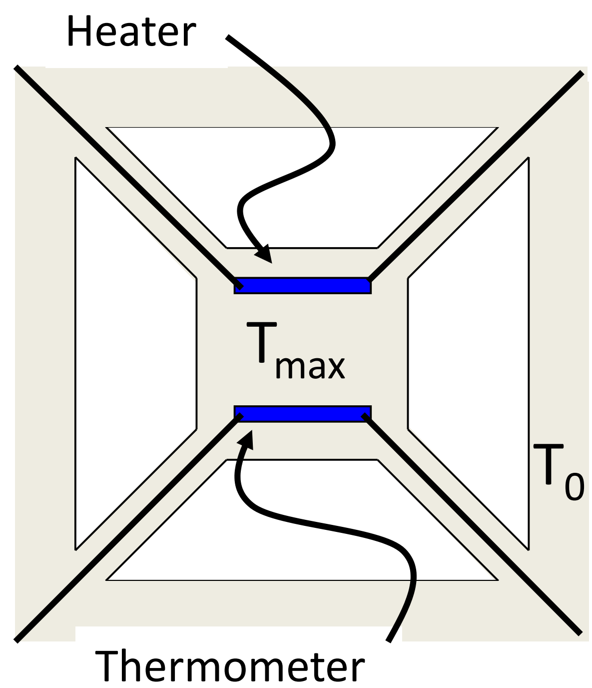
Micro and Nanofabrication

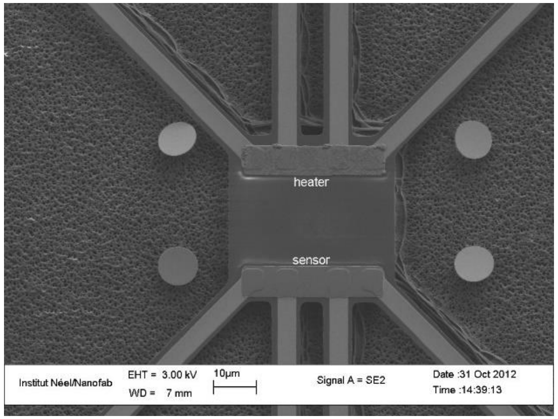
Clean room facilities

- A need for thermally isolated samples
- Suspended objects (difficult)

- Optical photolithography (UV light, monochromatic)
- Laser lithography (UV laser)
- E-beam lithography (electron beam)

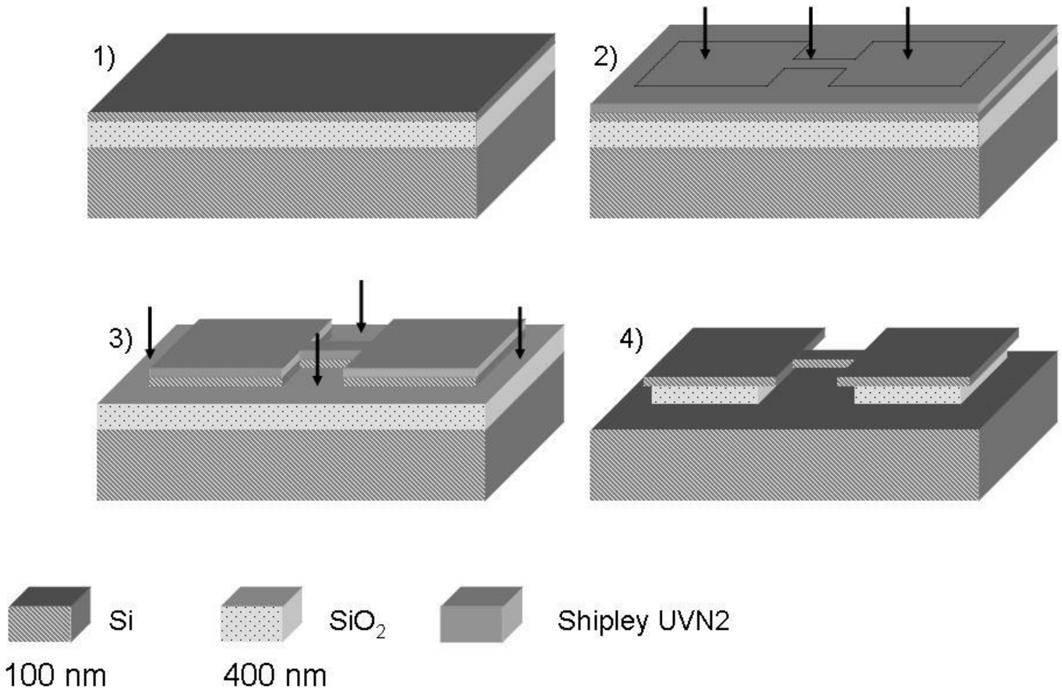
- Chemical etching (vapour or liquid), HF, KOH, XeF2, CH3CF4

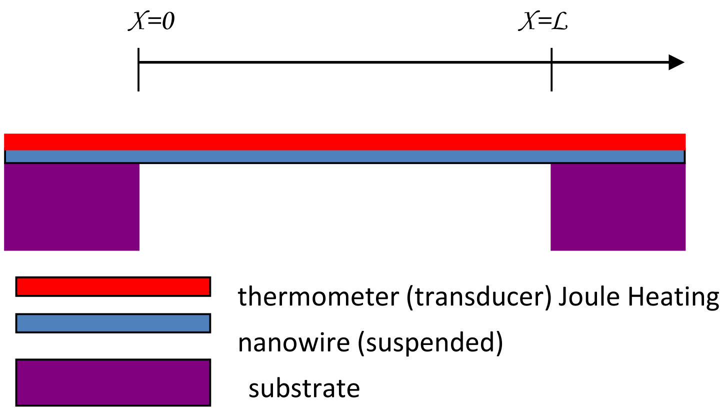




An exemple: SOI substrate

- SOI: silicon on insulator
- The fabrication of the silicon nanowire are realized by e-beam lithography on SOI substrate.
- Spinning e-beam resist
- e-beam isolation (structuration)
- Reactive Ion Etching (RIE)
- HF etching, nanowire suspended
- Deposition of thermometer

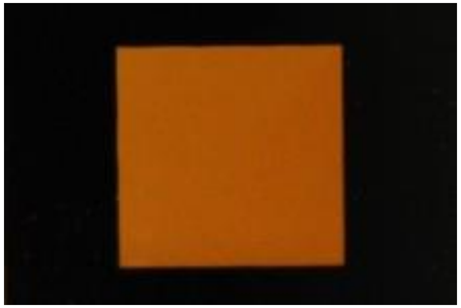




Si or Silicon nitride membrane

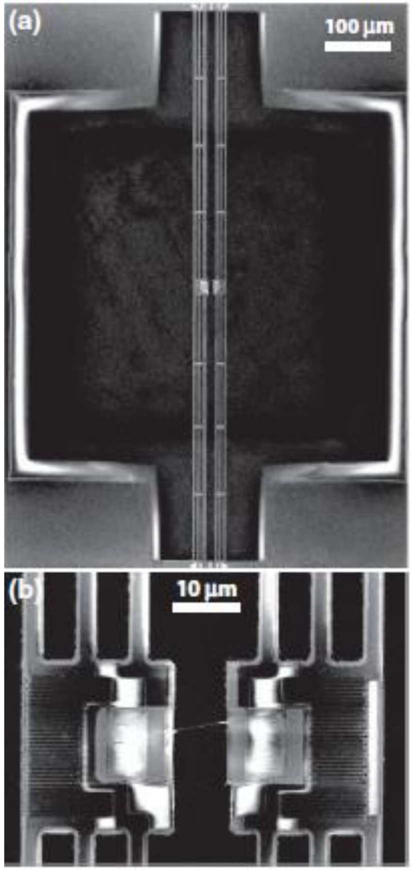
- KOH (liquid) need critical point drying, super-critic CO_2
- XeF_2 (vapour) avoid sticking effect

Si low stress membrane (VTT, Finland)

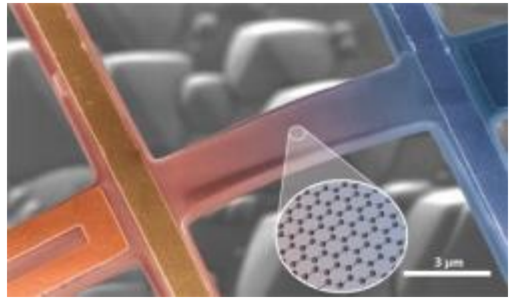


Si membrane stressed by SiN (VTT, Finland)
Chavez, APL Mat 2014

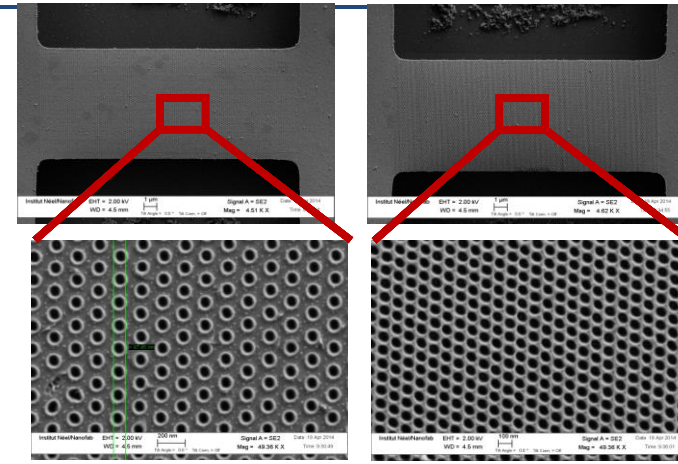
More examples...



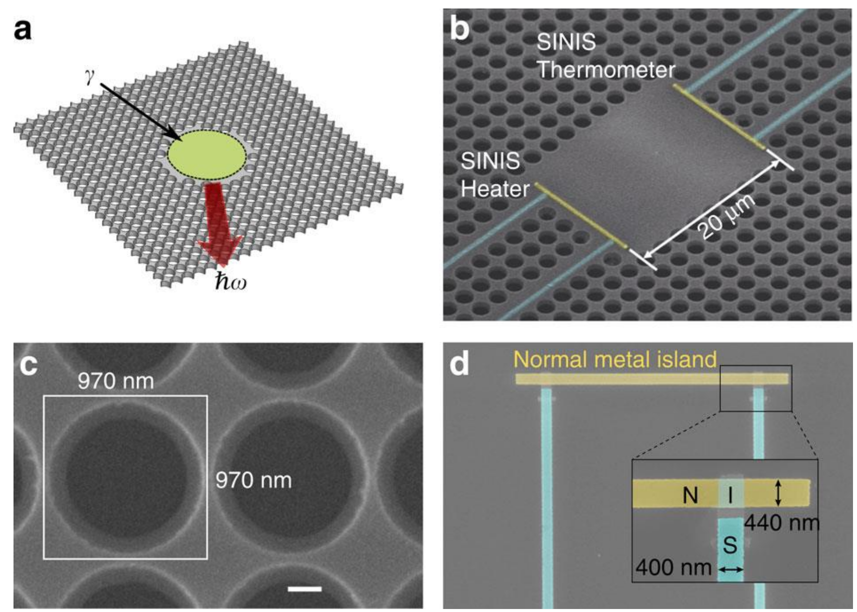
SiN structure from Group of Li Shi, Univ of Texas at Austin



L. Shi Science 2010

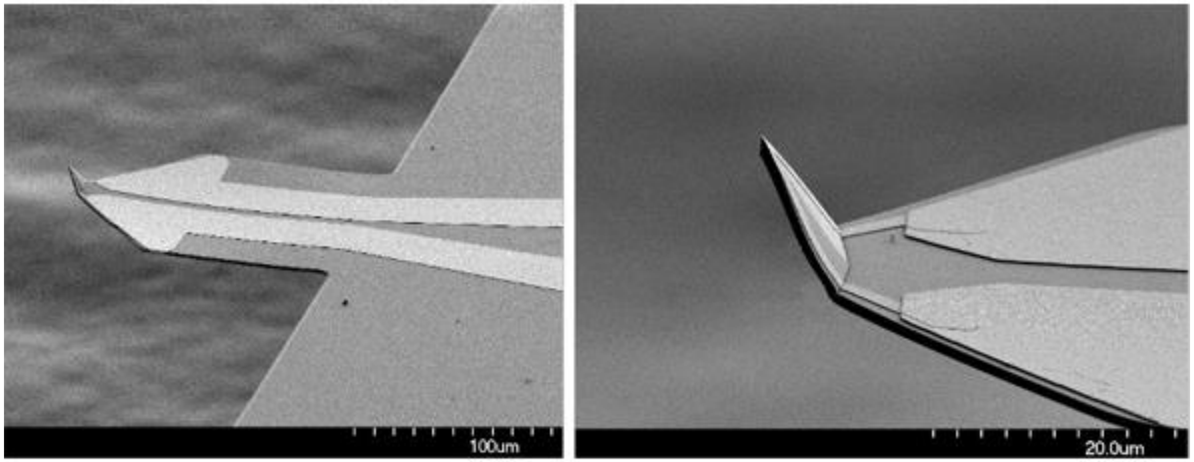


Germanium structure O.B. group Néel CNRS

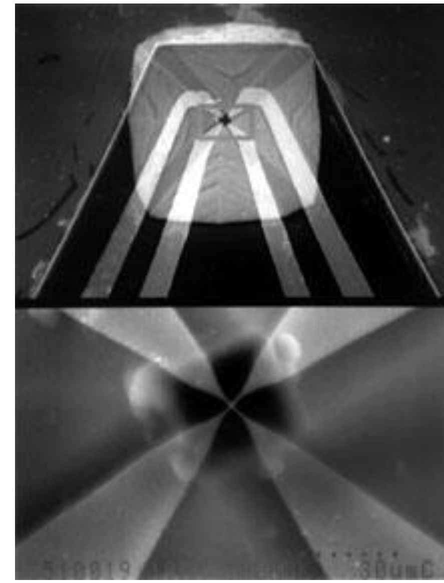


SiN from Group of Maasilta Jyvaskyla Univ., Nature Com 2014

Nanoscale tip for SThM

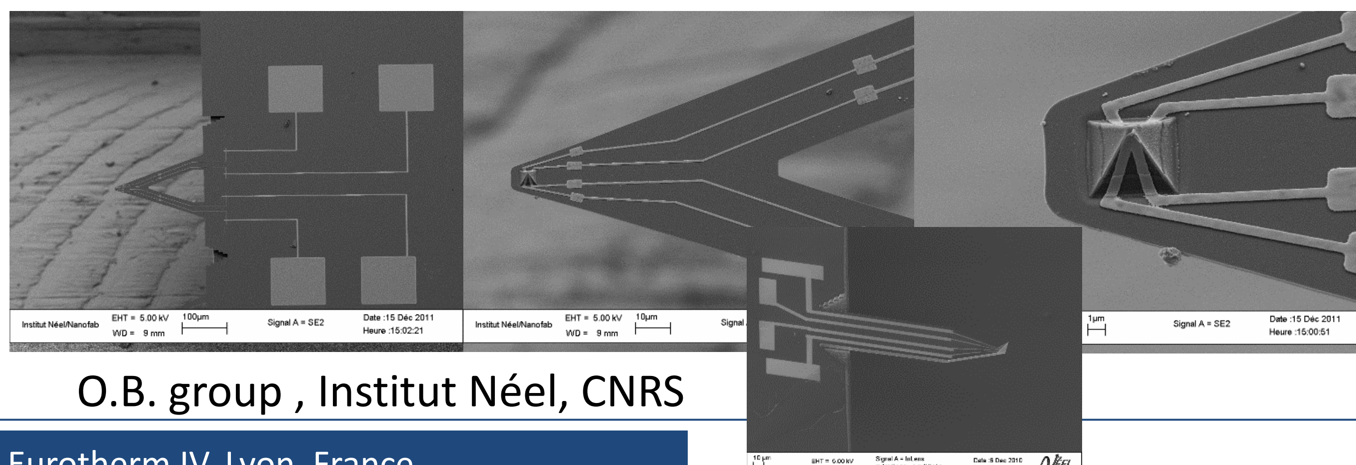


Kelvin probe



http://web.eng.gla.ac.uk/groups/nano/afm/TCouplePage.html

AFM tip: First test with NbN thermometer

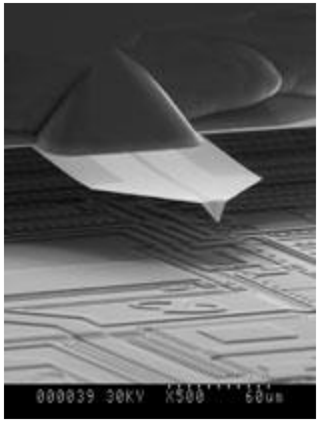


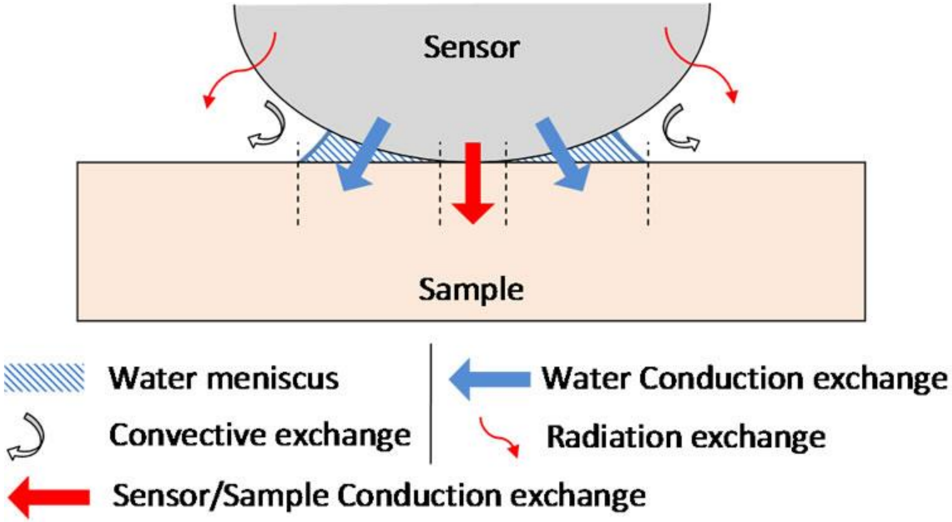
O.B. group , Institut Néel, CNRS

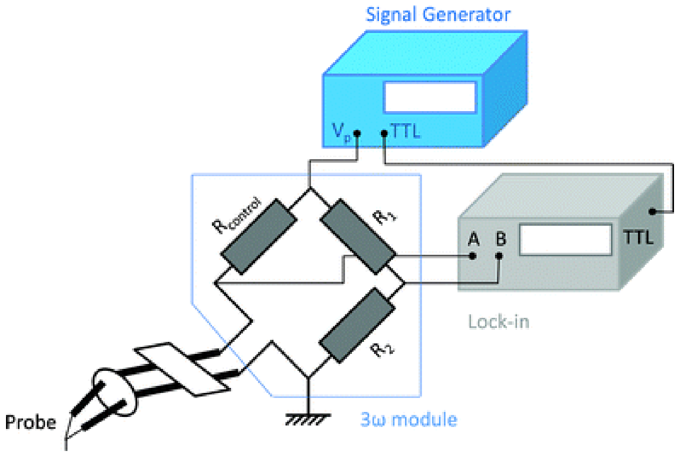
Near field Thermal measurement

Coupling an AFM with a thermometer

- Allows local thermal characterization
- Mapping of surface (temperature or by 3 omega the thermal conductance of the surface)
- Need an elaborated thermal model
  - Radiation
  - Convection if not under vacuum
  - Contribution of the contact
  - Conduction through the tip itself to the heat bath
  - Estimation of the thermal contact



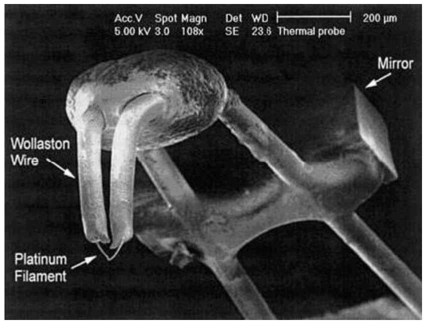


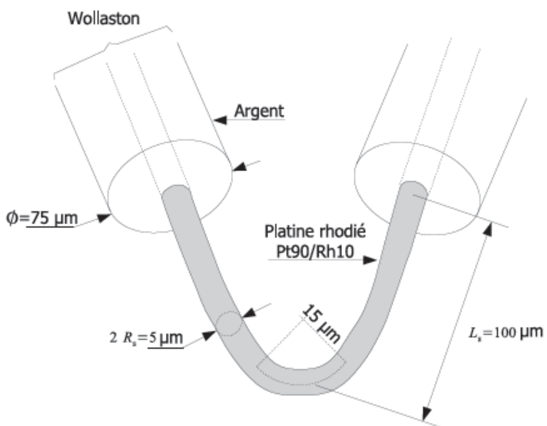


Nanoscale, 2012,**4**, 4799-4829

The tip and the experimental set-up

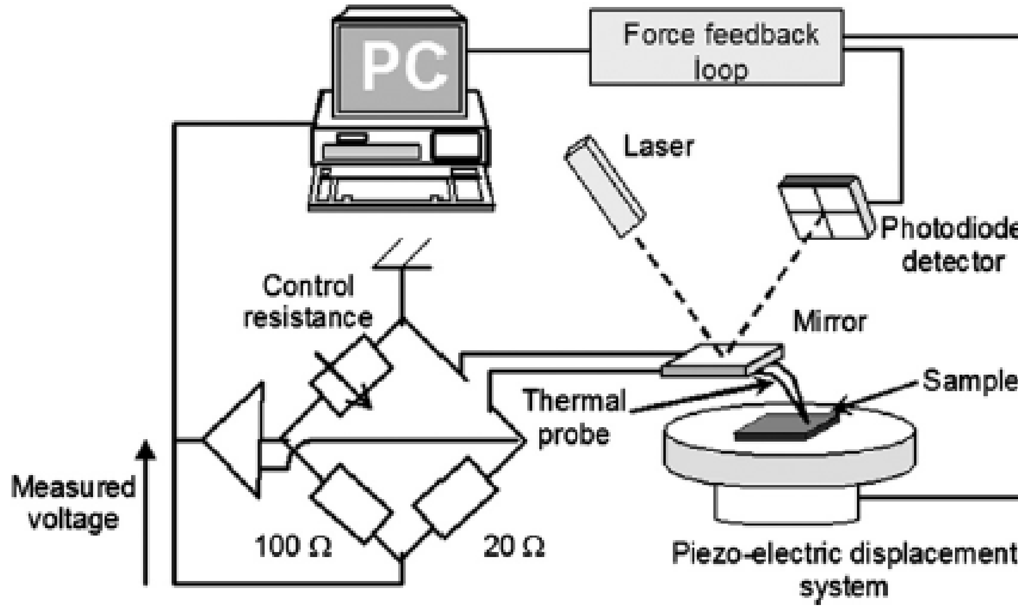
Wollaston tip

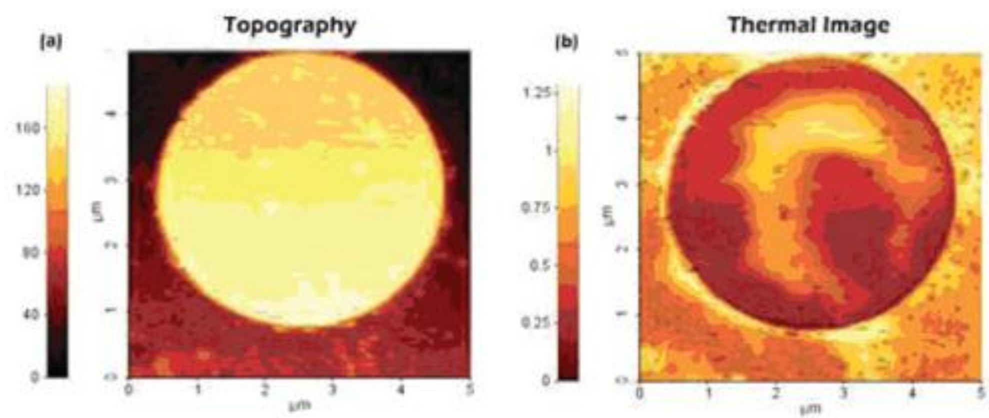




Lefèvre thesis 2004, Ali Assi thesis 2014

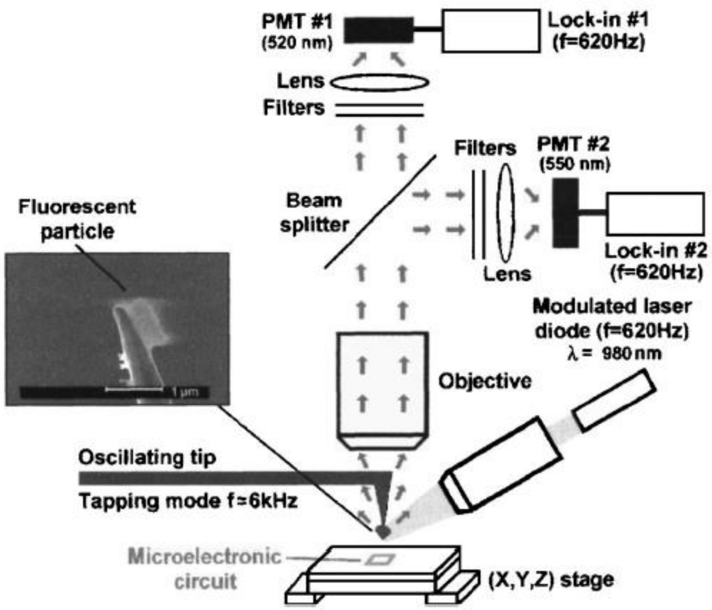
Group S. Gomes,
Microelec. Journ. 44, 1129 (2013)

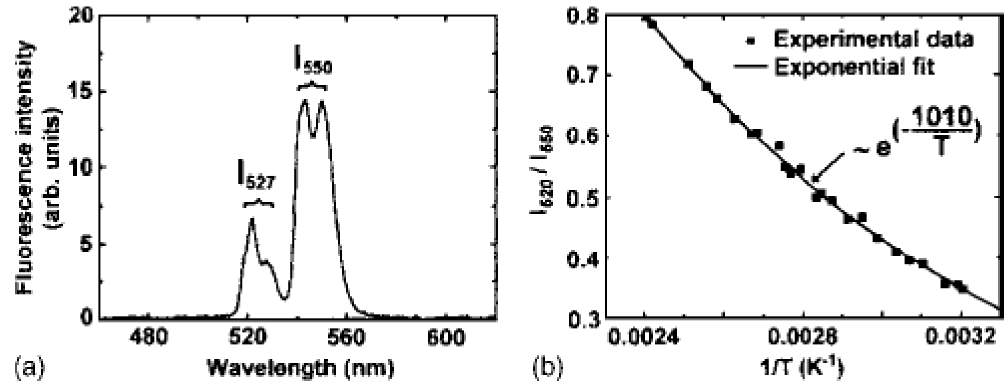


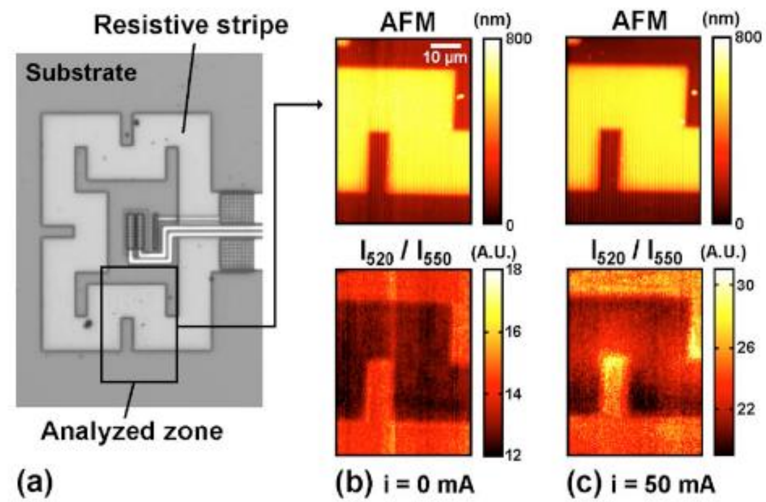


SThM based on fluorescence

- Fluorescence of nanoparticles of Erbium/Ytterbium





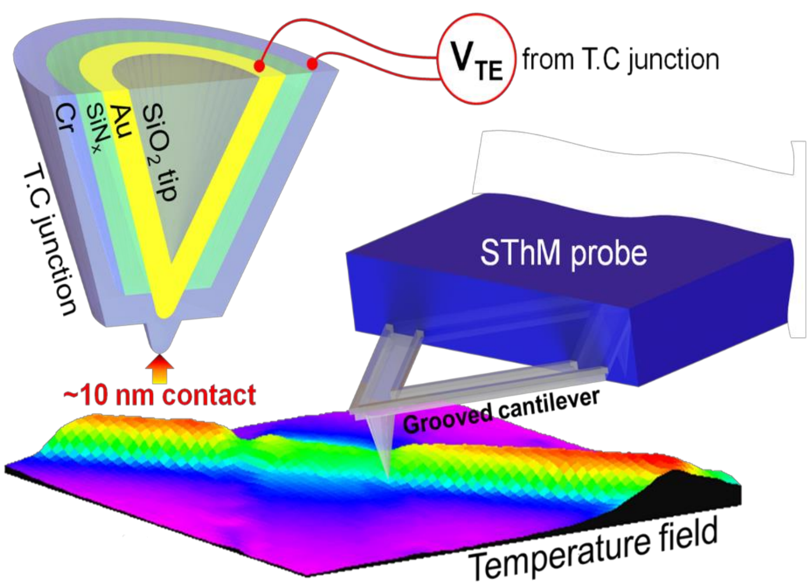


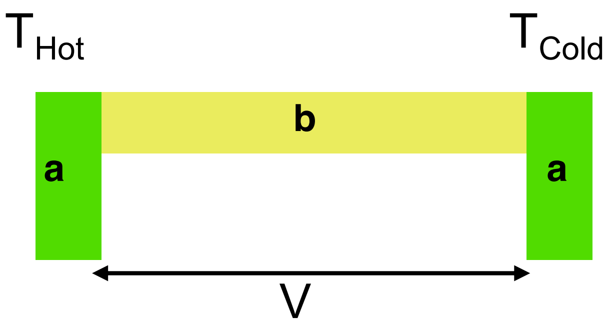
APPLIED PHYSICS LETTERS 87, 184105 (2005)

L. Aigouy, G. Tessier (ESPCI)

SThM with thermocouple

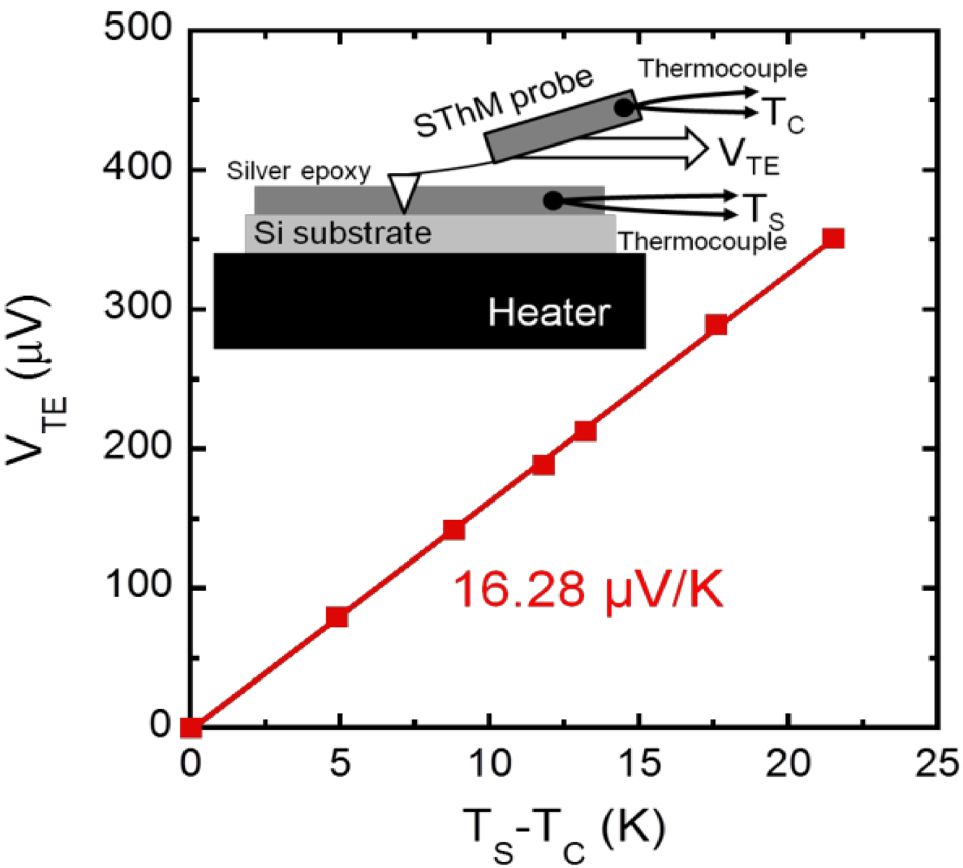
- Couple Au/Cr: 20µV/K
- Another junction serves as references
- Pramod Reddy Univ. Michigan

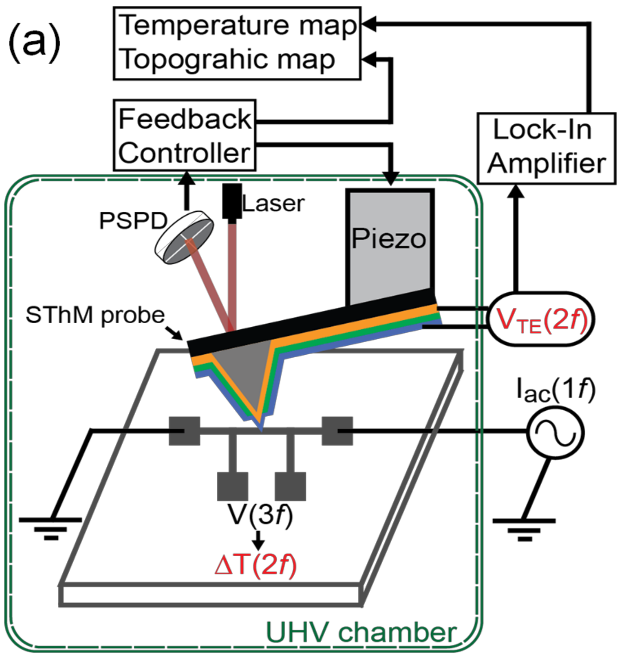




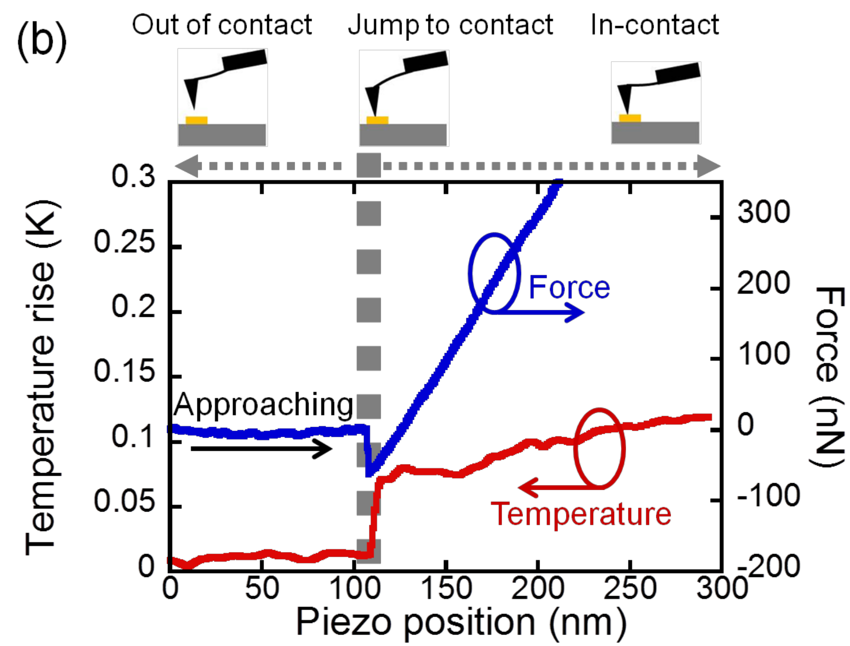
Thermopower, $S = V/\Delta T$

Metal	Seebeck Coefficient (µV/K)
Al	-1.66
Au	1.94
Cr	21.8
Ni	-19.5


500
400
300
200
100
0
0
5
10
15
20
25
V_{TE} (µV)
T_S-T_C (K)
SThM probe
Thermocouple
T_C
V_{TE}
Silver epoxy
T_S
Si substrate
Thermocouple
Heater
16.28 µV/K

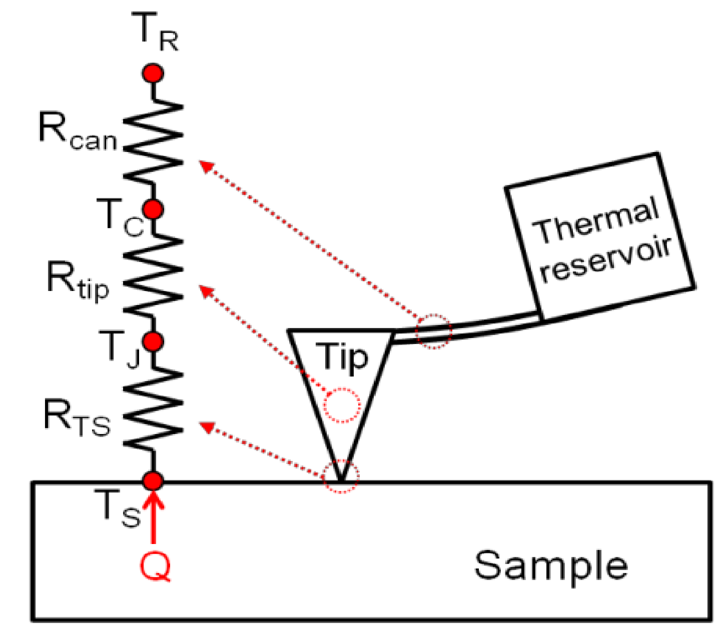


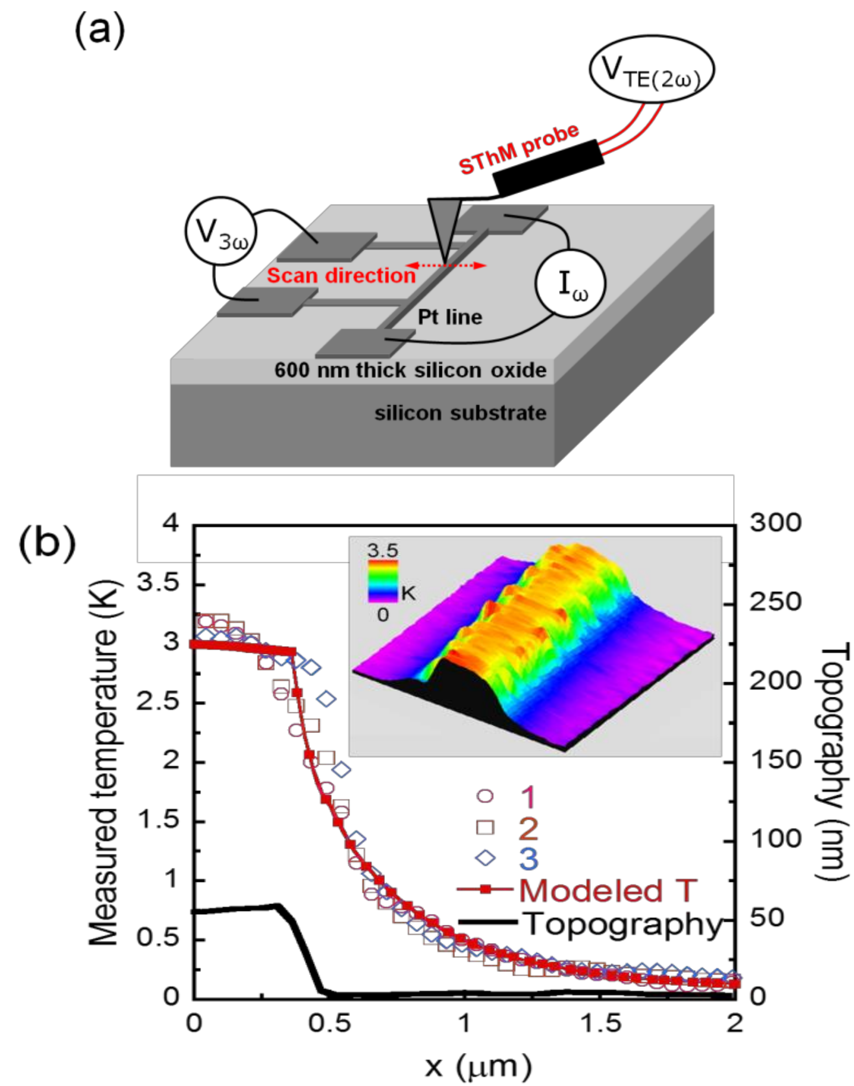
(a)
Temperature map
Topograhic map
Feedback Controller
Lock-In Amplifier
PSPD
Laser
Piezo
SThM probe
V$_{TE}$(2f)
I$_{ac}$(1f)
V(3f)
ΔT(2f)
UHV chamber



(b)
Out of contact
Jump to contact
In-contact
Temperature rise (K)
Force (nN)
Piezo position (nm)
Approaching
Force
Temperature

Sensitivity and spatial resolution





Optical methods: Raman thermometry and thermoreflectance

Advantage of contact less method for fragile membranes

Introduction

- Interaction between light and matter
- Elastic scattering Rayleigh
- What is Raman scattering ? Inelastic scattering
- Brillouin scattering ?

L. N. Brillouin

1889 – 1969

Main achievements

1. Brillouin Scattering (1922)
2. Brillouin Zones (Solids)
3. WKB method (diff. eqs.)
4. Perturbation Theory

C. V. Raman

1888 – 1970

Main achievements

1. Raman effect
   28 of February 1928
   Nobel Prize (1930)

L. I. Mandelstam

1879 – 1944

Main achievements

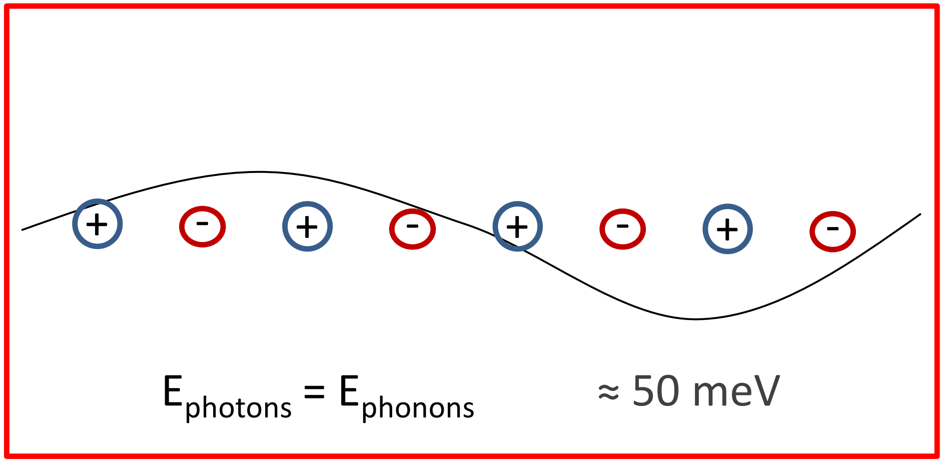
1. Inelastic Combinatorial Scattering
   21 February 1928

IR absorption and Raman effect in simple words….

IR ABSORPTION

Incident photon

E_i

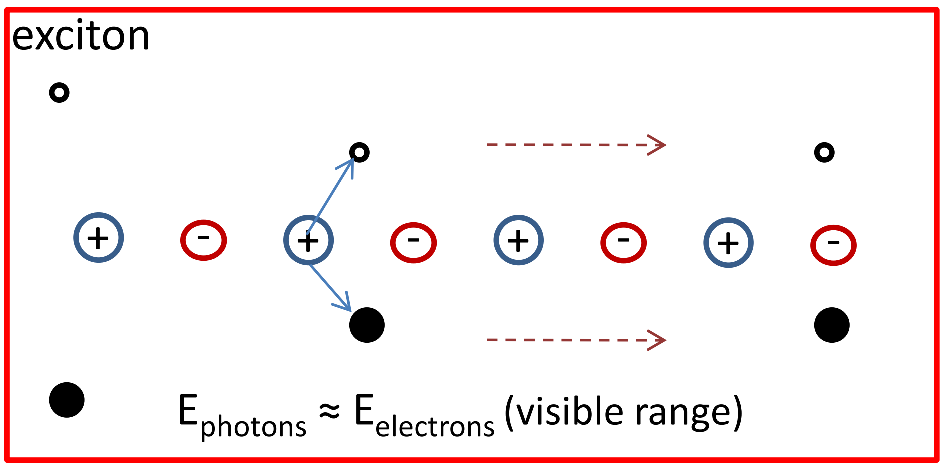


scattered photon

RAMAN EFFECT

Incident photon

E_i

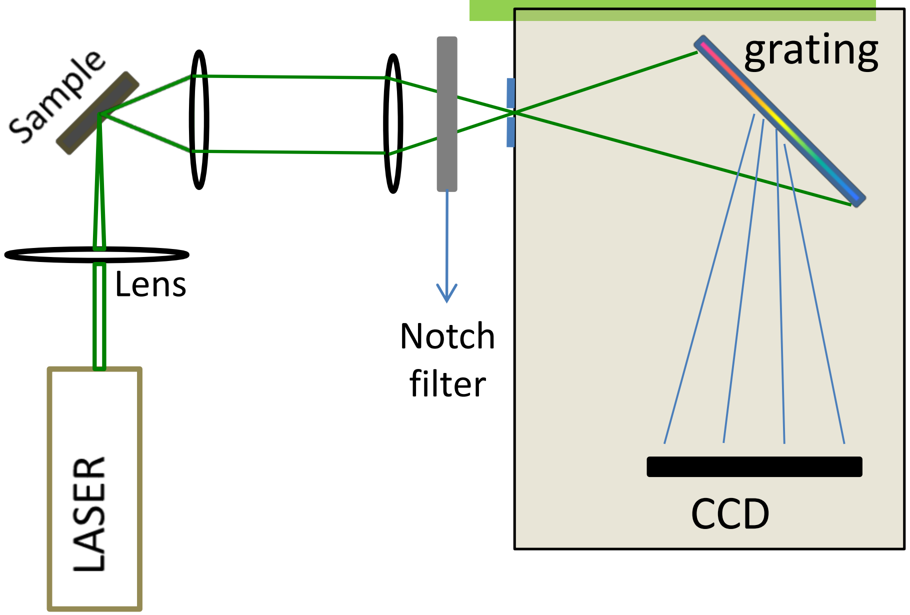


scattered photon

E_f

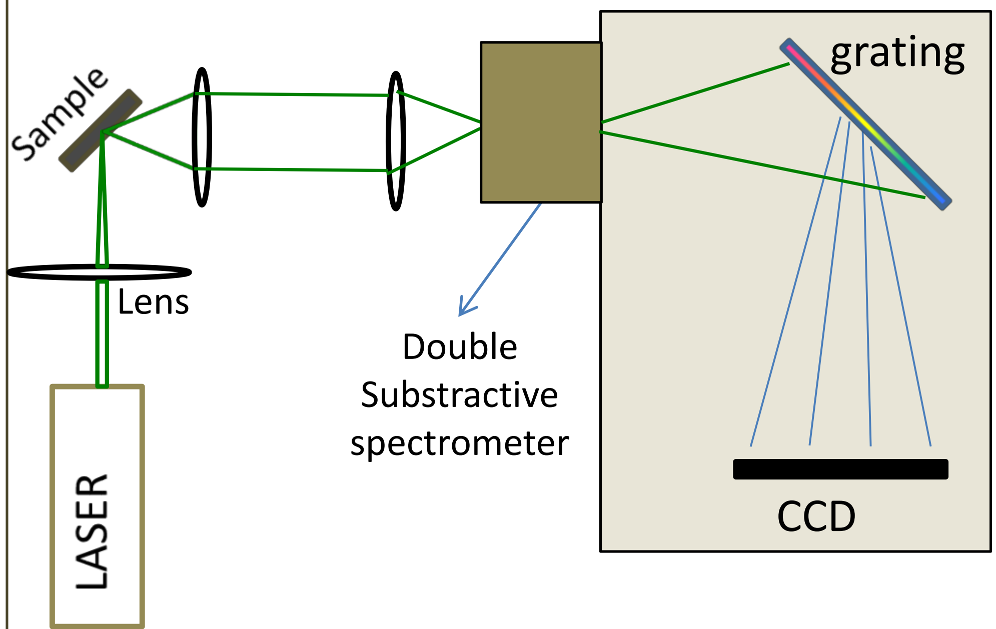
$E_i - E_f = E_{phonon}$

LASER + Single + edge or notch filter



i) High luminosity.
ii) Capable to achieve 100 cm^{-1} from the incident laser.
iii) Suitable for **optical** phonons.
iv) Usually only Stokes capable.
v) Extremely *user friendly*.

LASER + Triple spectrom.



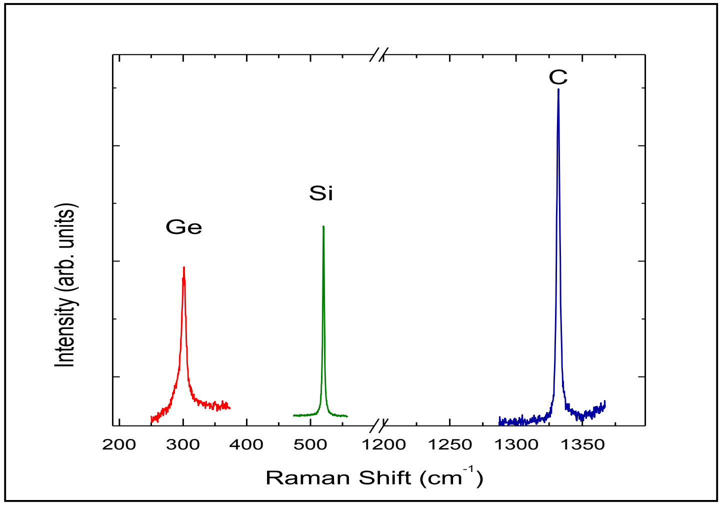
i) Lower luminosity.
ii) Capable to achive 10 cm^{-1} from the incident laser.
iii) Suitable for **acoustic or optical** phonons.
iv) Stokes and anti-Stokes

The Raman process takes the following steps:

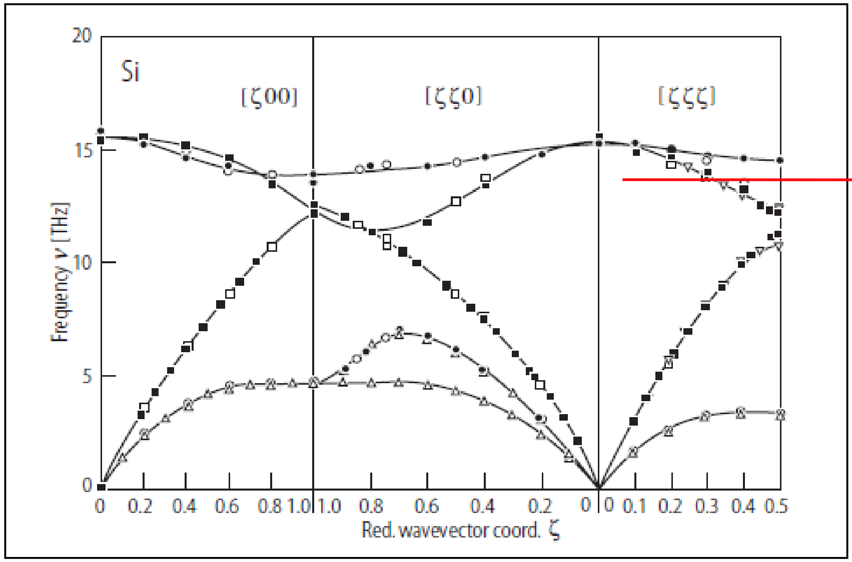
1) The incident photon excites the semiconductor creating an electron-hole pair (exciton).

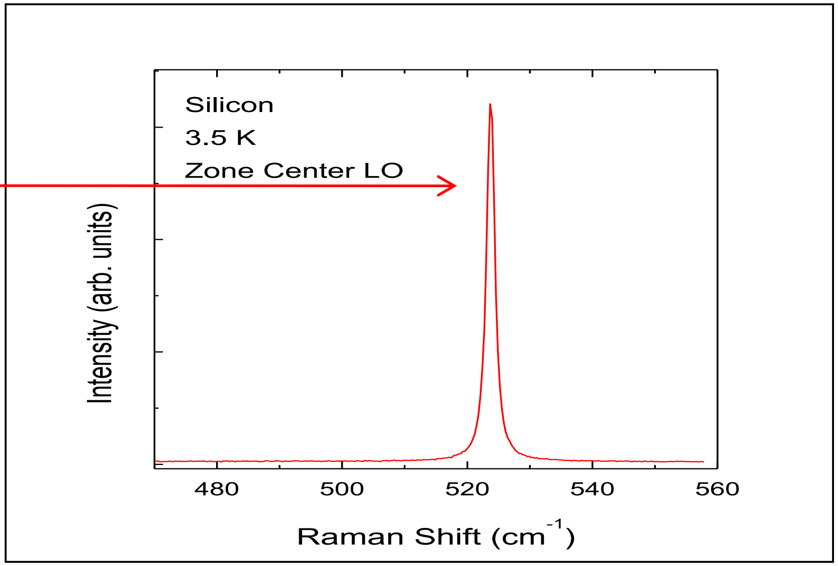
2) The exciton is scattered to a different state through the interaction with a phonon.

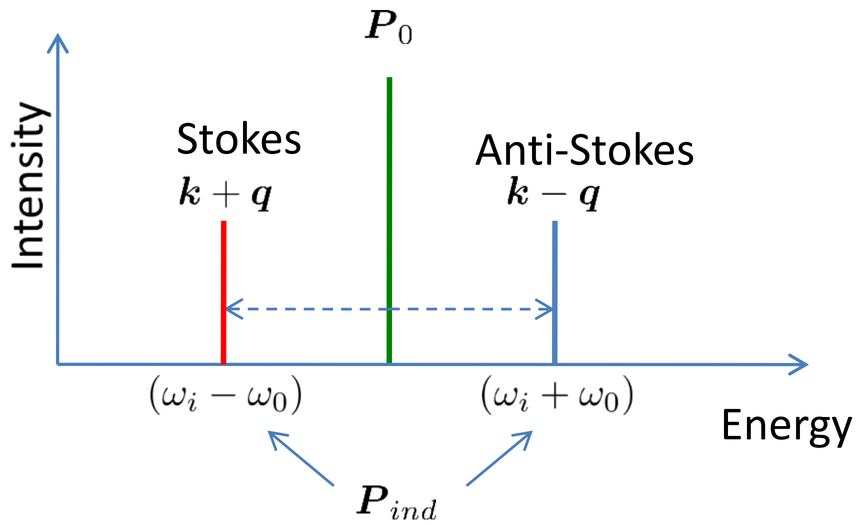
3) The exciton recombines radiatively with the emission of a photon.



Raman scattering: probing optical phonons

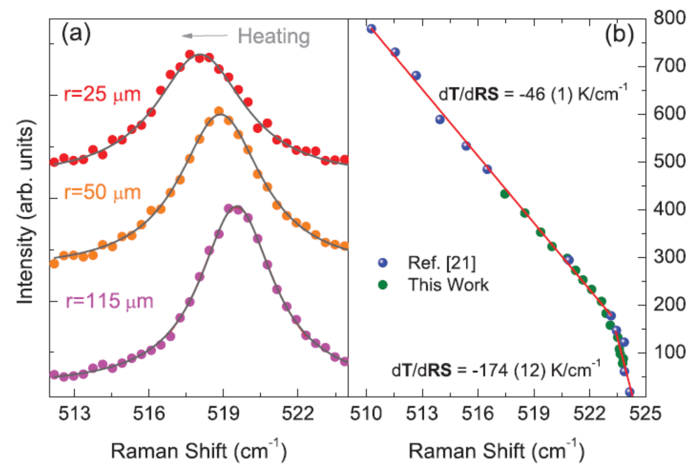


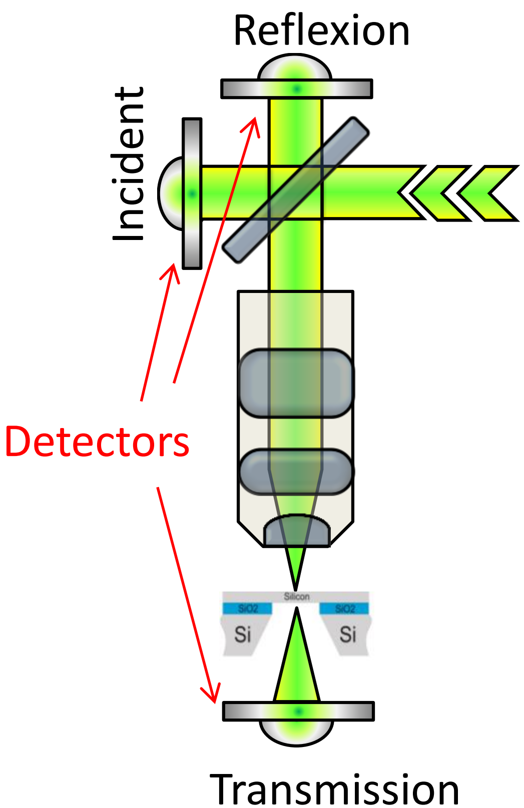


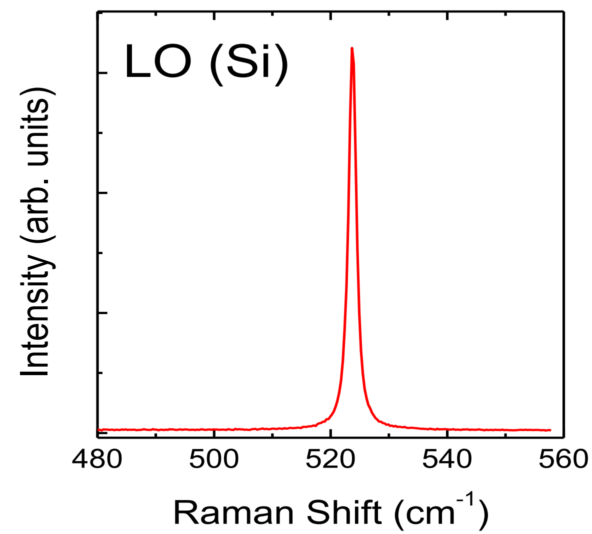


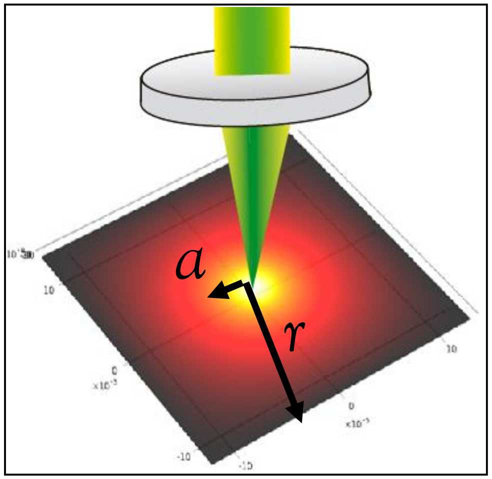
RAMAN THERMOMETRY: EXPERIMENTAL SET-UP

1) Accurate definition of the absorption coefficient A by measuring incident (Pi), reflected (Pr) and transmitted (Pt) power
2) Measuring the Raman shift of the Si LO phonons as function of the absorbed power
3) Calibration of the T dependence of the Si LO Raman mode
4) Solving the 2D heat equation using FE simulations with T and P_A as known parameters





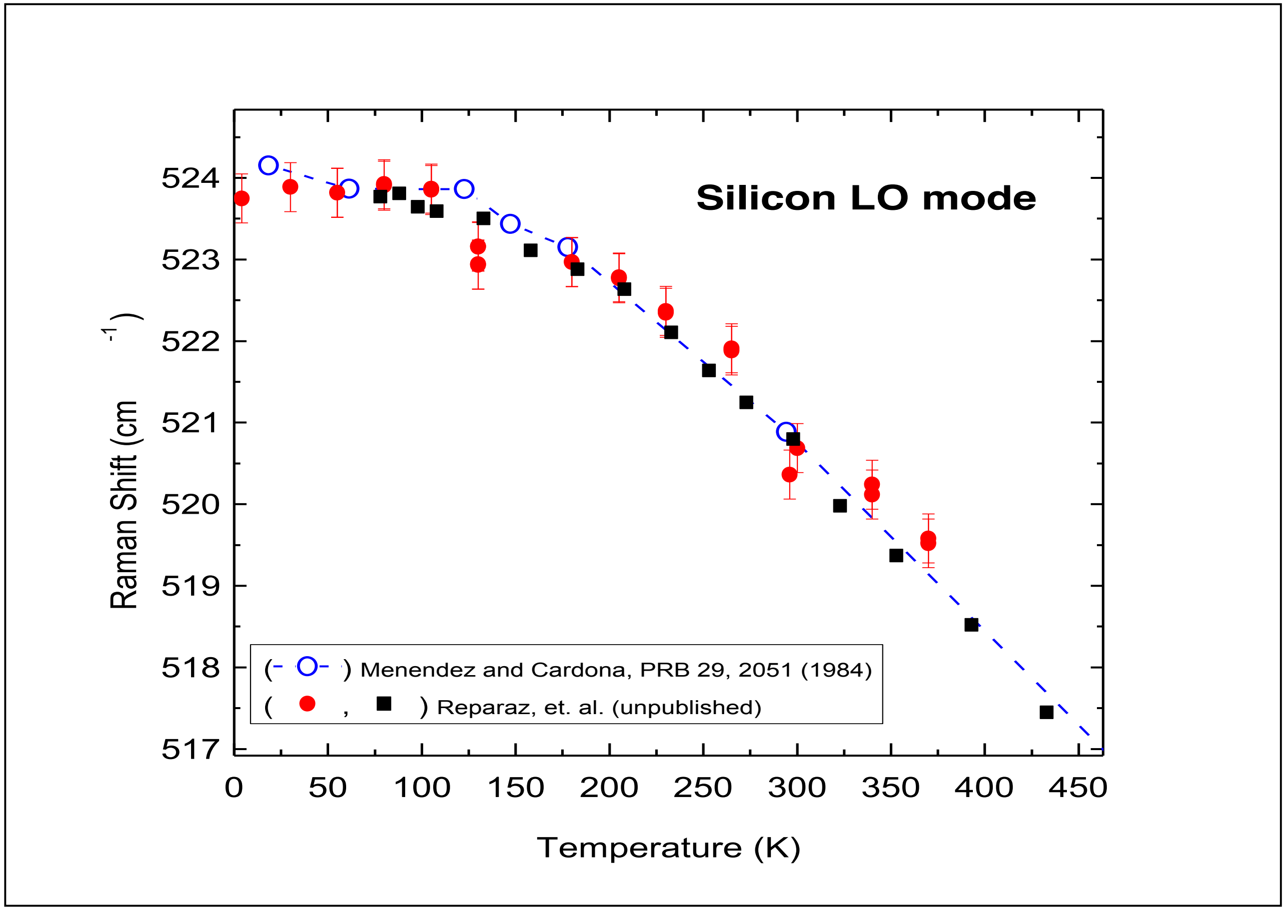




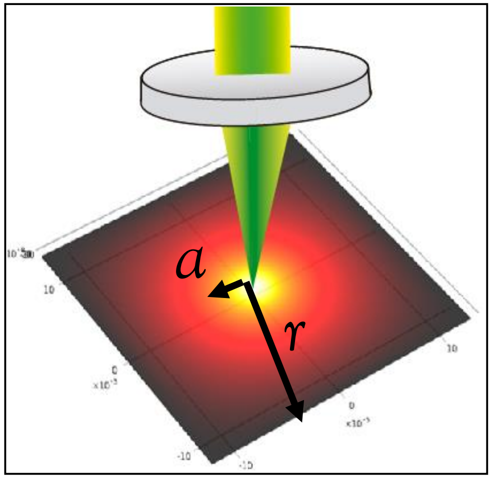
$$\nabla^2 \mathbf{T} = \frac{P_{abs}}{\kappa \pi a^2 d} \exp(\mathbf{r}^2/a^2)$$

J. S. Reparaz et al., *Rev. Sci Instrum.* 85, 034901 (2014)

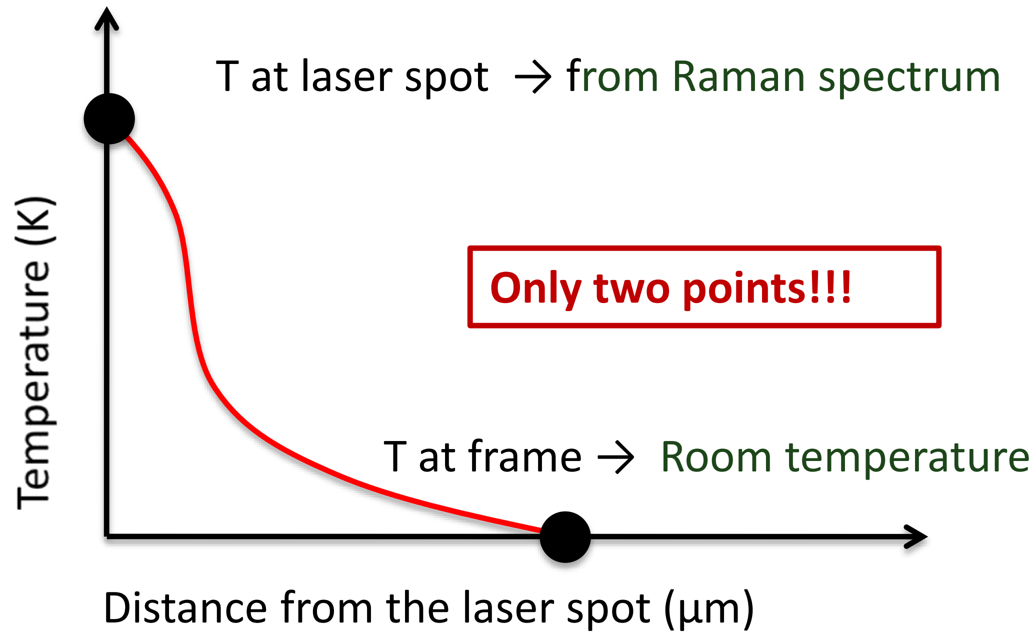
Sensitivity versus temperature



DRAWBACK OF THE TECHNIQUE

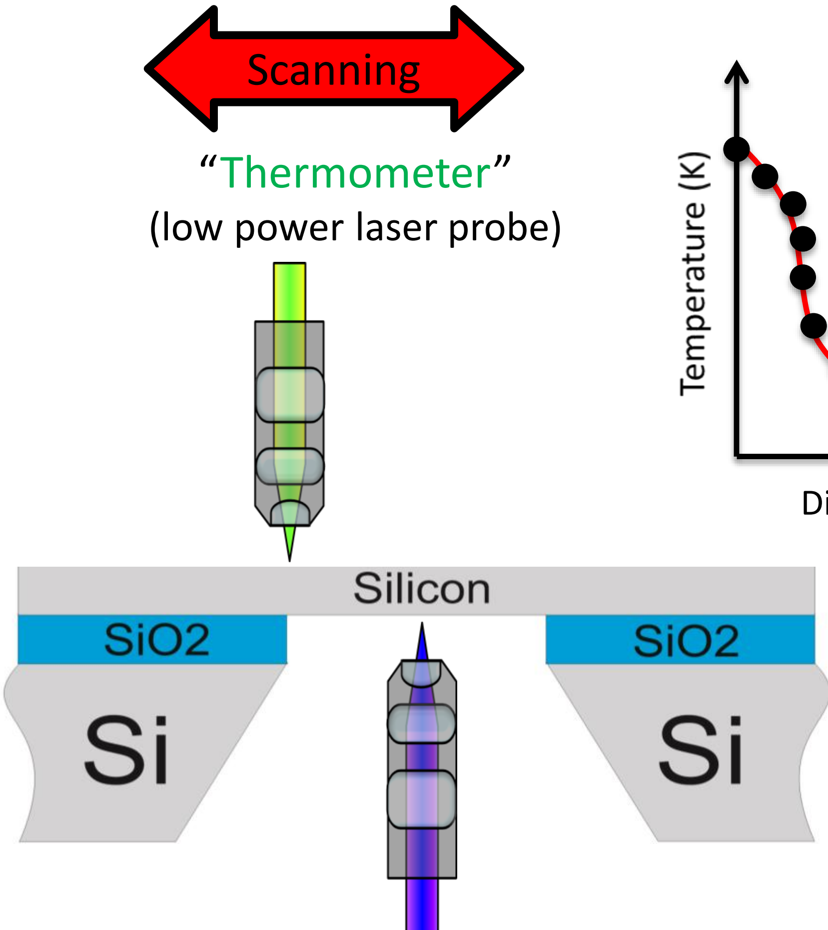


$$\nabla^2 \mathbf{T} = \frac{P_{abs}}{\kappa \pi a^2 d} \exp(\mathbf{r}^2/a^2)$$

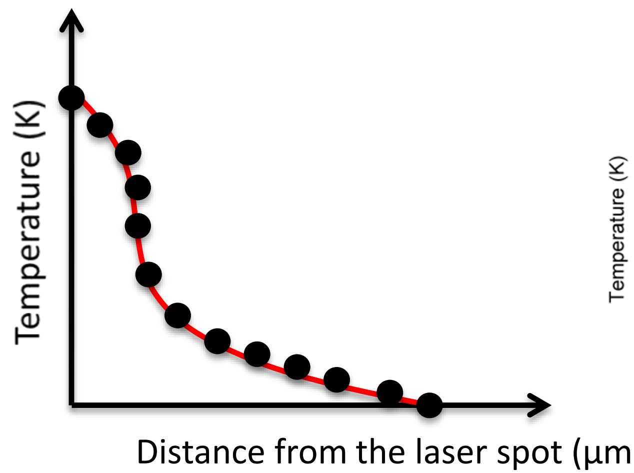


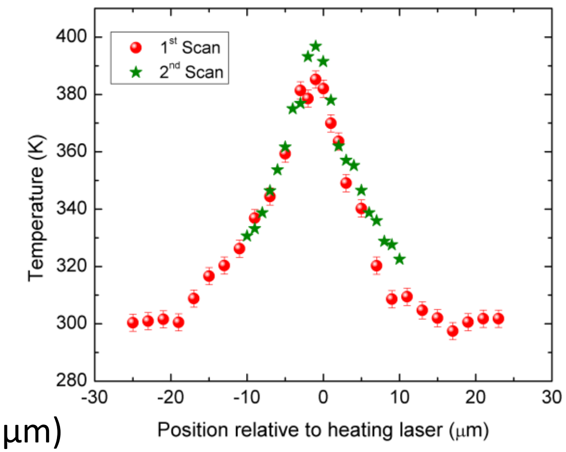
- Temperature profile is highly non-linear
- No information of heat transport regime

"2-LASER RAMAN THERMOMETRY"



"Heating Laser" (High power fixed laser)

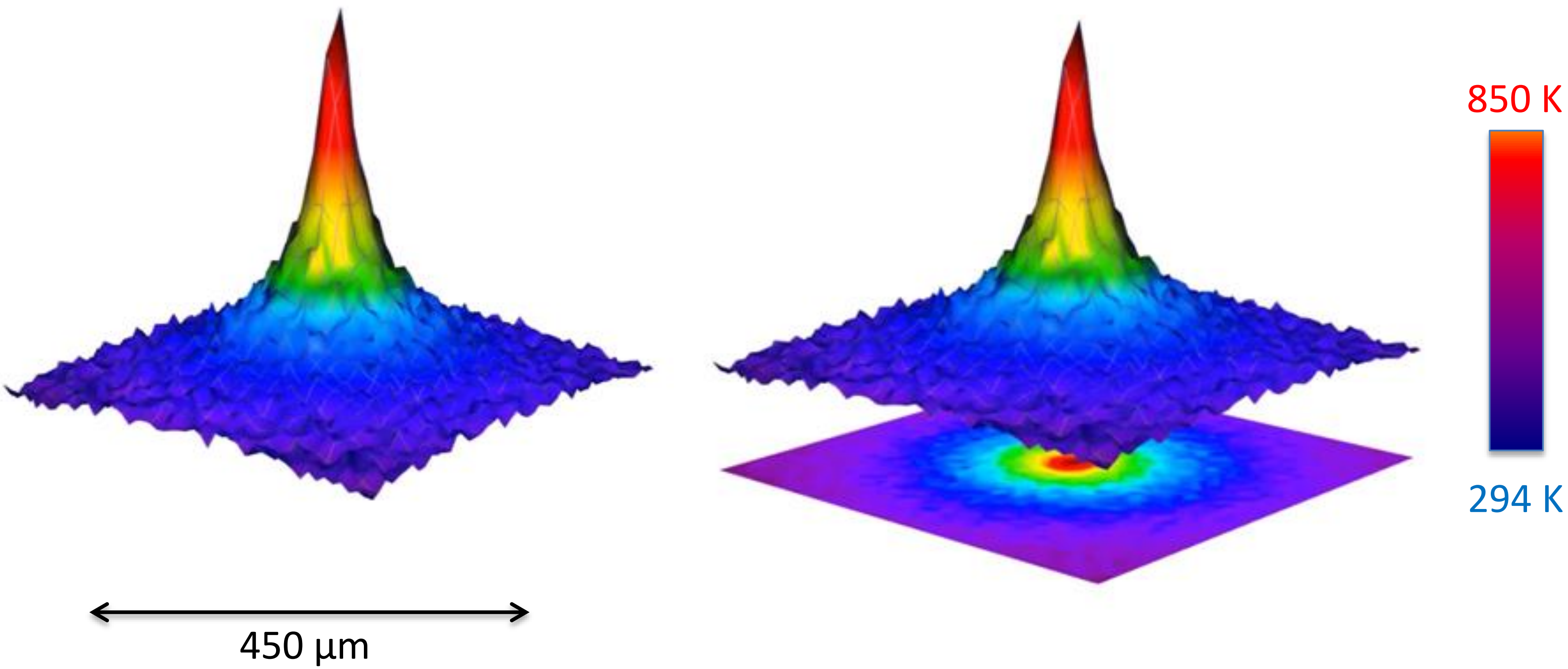




Solution:

Scan of the heat distribution profile by independent heating and probing lasers!

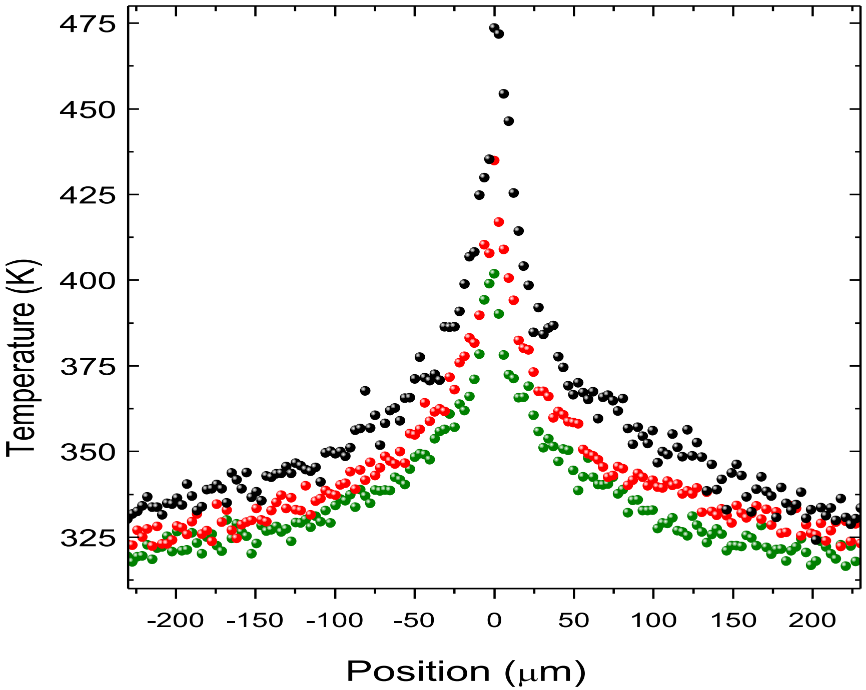
2-Dimensional Thermal Temperature Maps

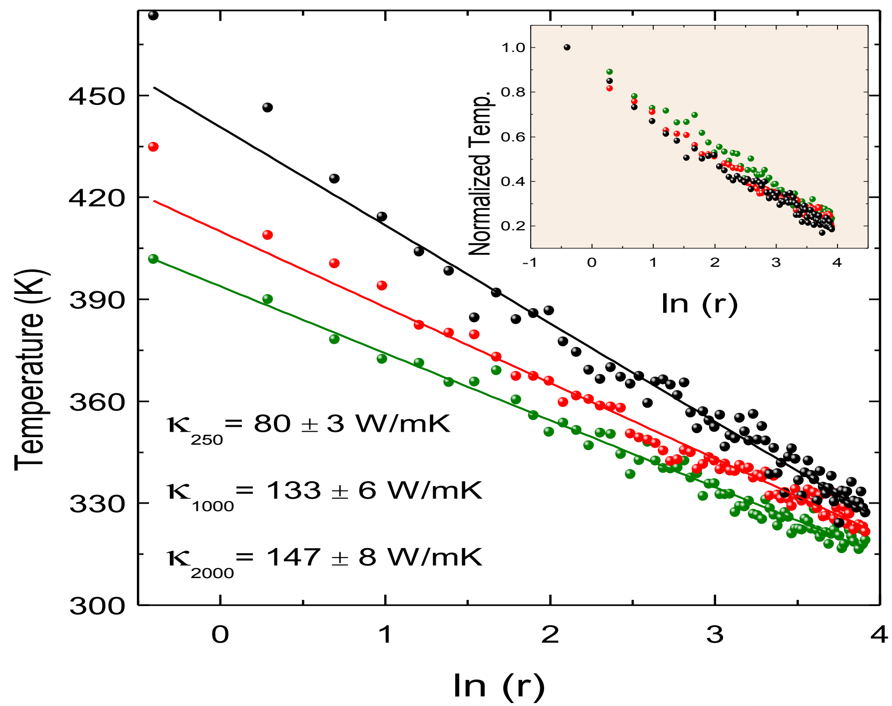


- The temperature distribution on the membrane is accurately probed
- The thermal field is isotropic since Si is isotropic.

J. S. Reparaz *et al.*, Rev. Sci. Instr. 85, 034901 (2014)

Power Dependence – 250 nm Si Membrane





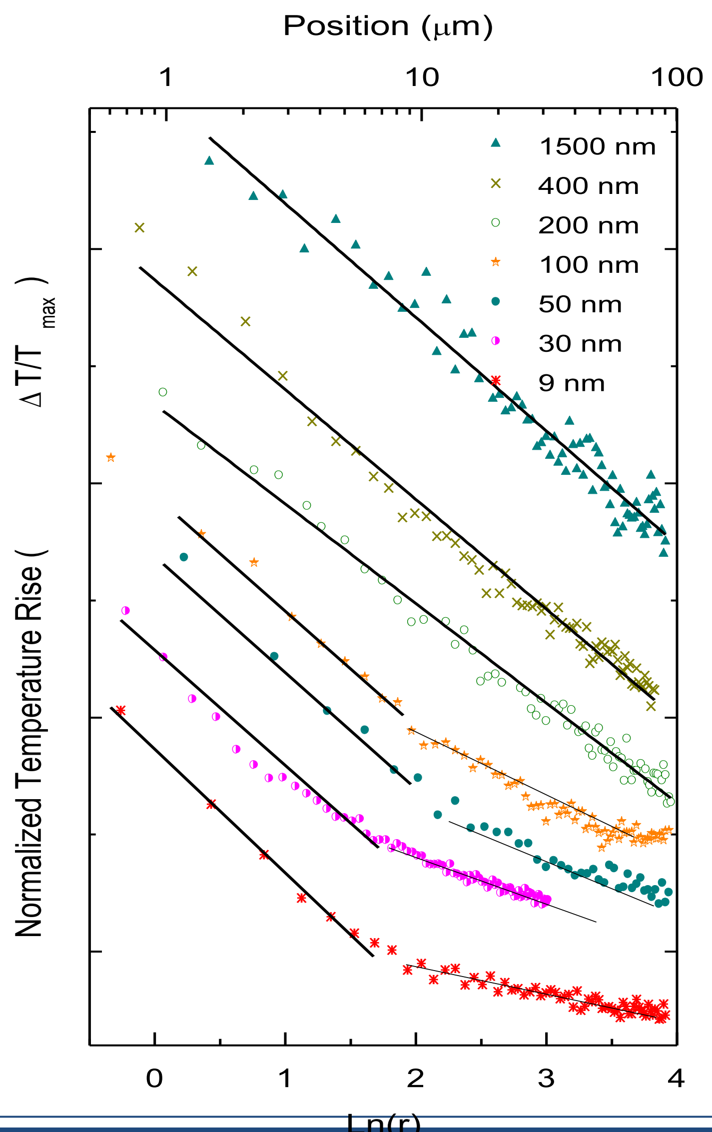
$$Q/A = -\kappa \nabla T$$

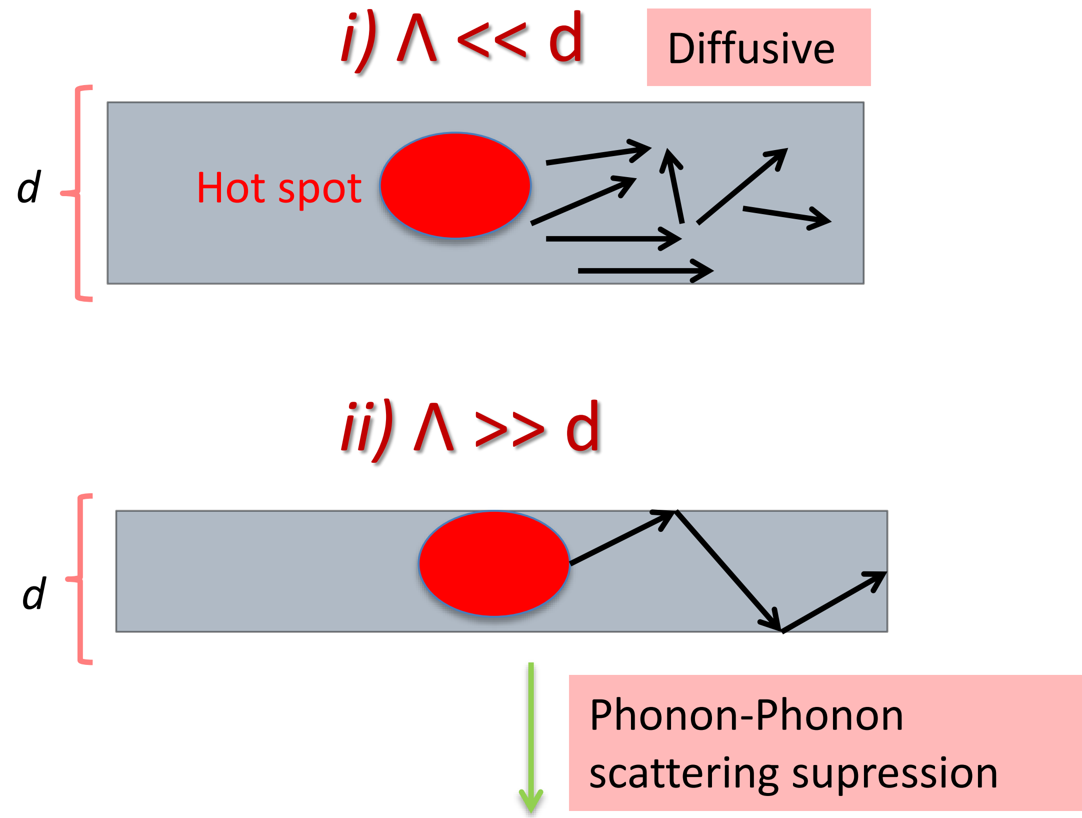
$$(A = 2\pi r d)$$

$$T(r) = T_0 - \frac{Q}{2\pi d \kappa_0} \ln(r/r_0) \quad \rightarrow \quad \kappa = \kappa_0$$

$$T(r) = T_0 \left(\frac{r}{r_0}\right)^{-Q/2\pi d a} \quad \rightarrow \quad \kappa = \frac{a}{T}$$

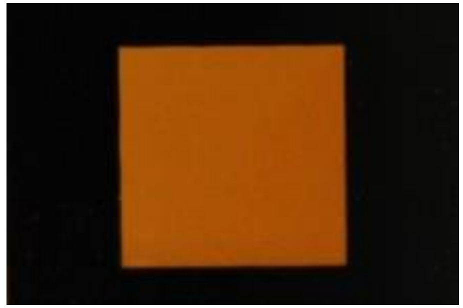
EXAMPLE 2: Size Dependence of Thermal Transport in Silicon Membranes

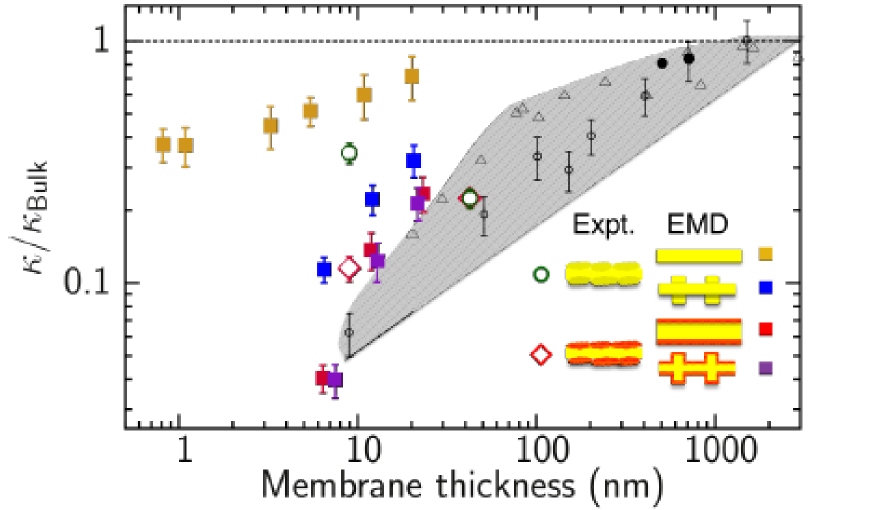


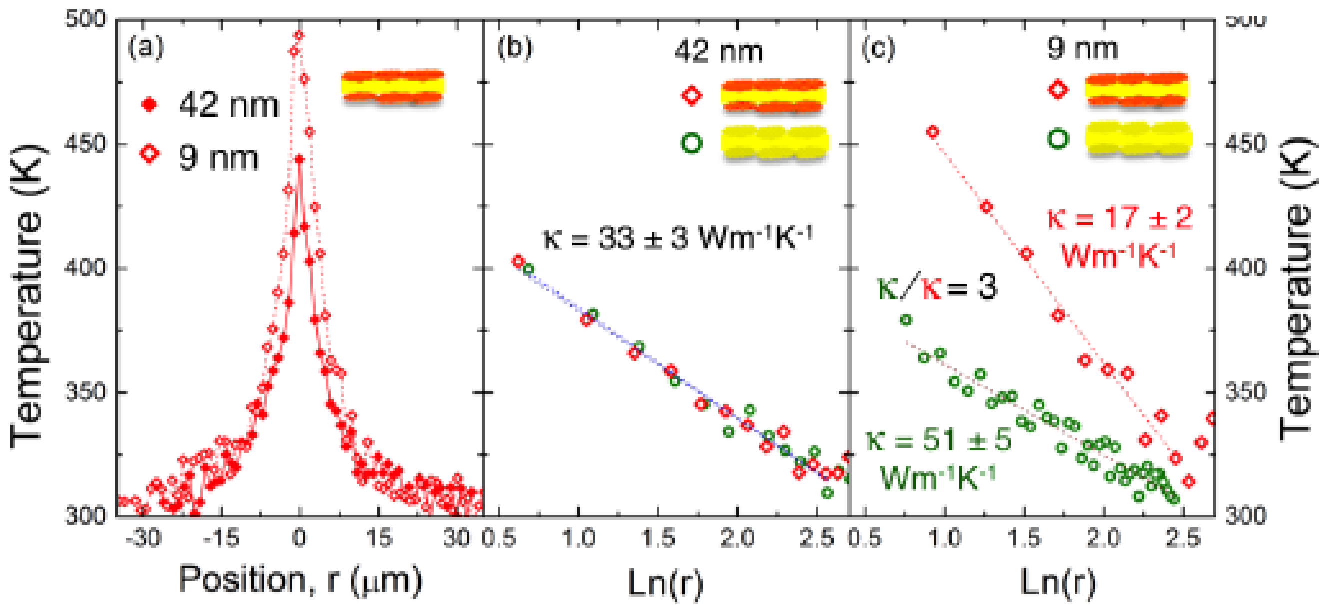


- Suppression of phonon-phonon scattering due to size effects.
- Role of surface roughness?

Thermal properties of 10 nm thick silicon membrane

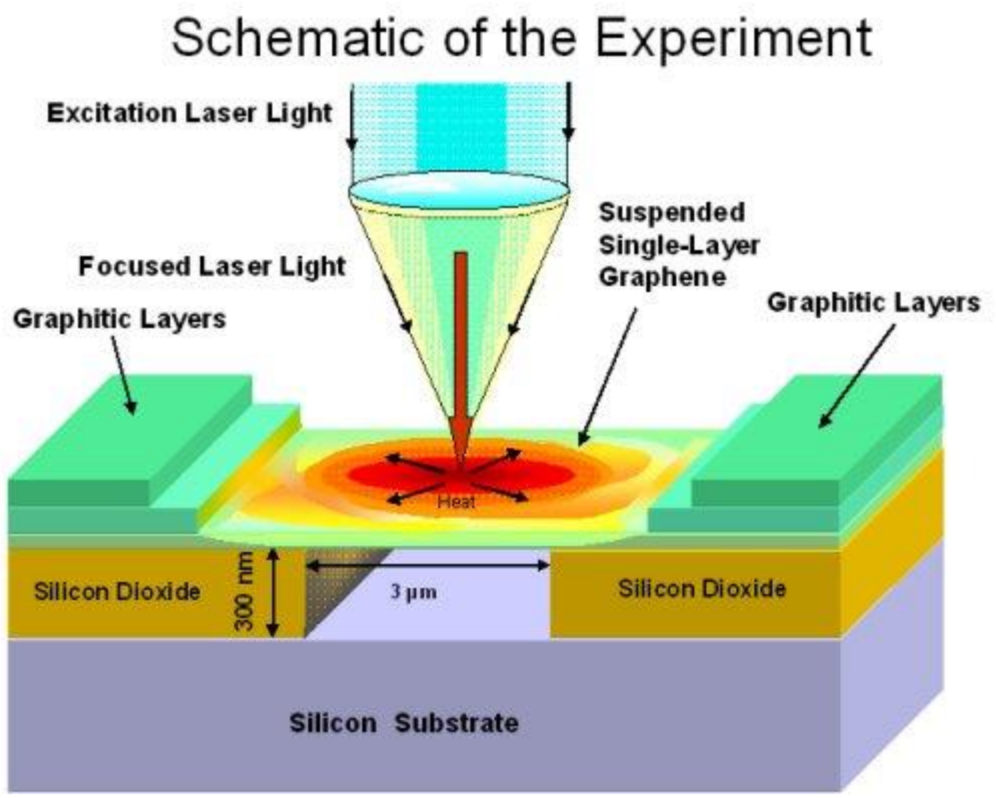






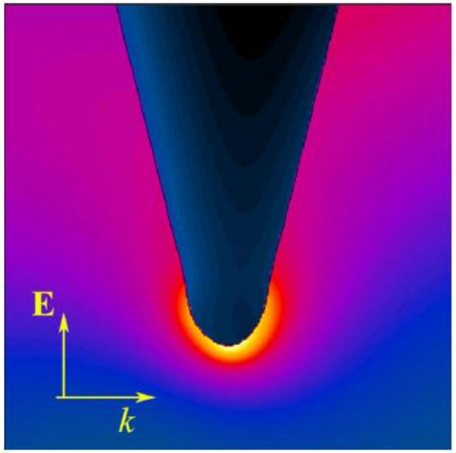
Group of Clivia Sotomayor-Torres in ICN (Barcelona, Spain)

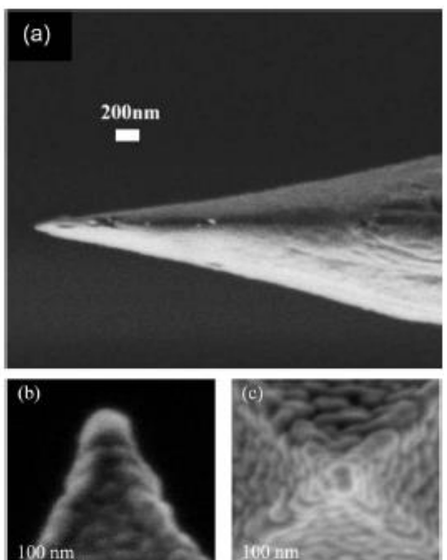
Measurement of thermal properties of suspended graphene



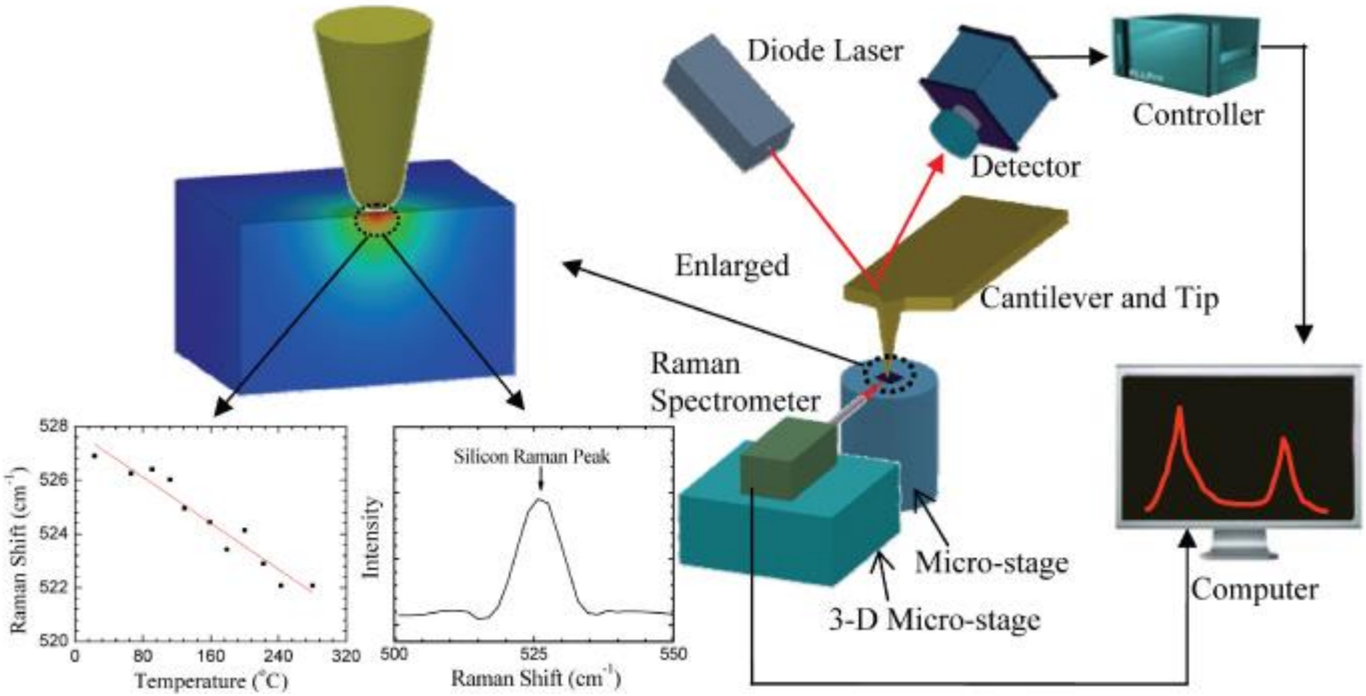
A.A. Balandin et al. APL, Nature Nano., PRB etc…

Graphene sheet





- A way to improve the resolution
- Tip enhance Raman Scattering (TERS)



Nano Reviews, Vol 3 (2012)

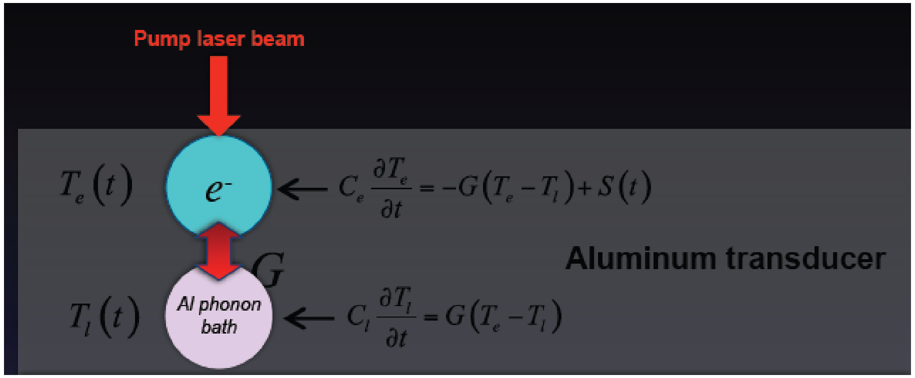
J. Wessel
J. Opt. Soc. Am. B **2,** 1538 (1985)

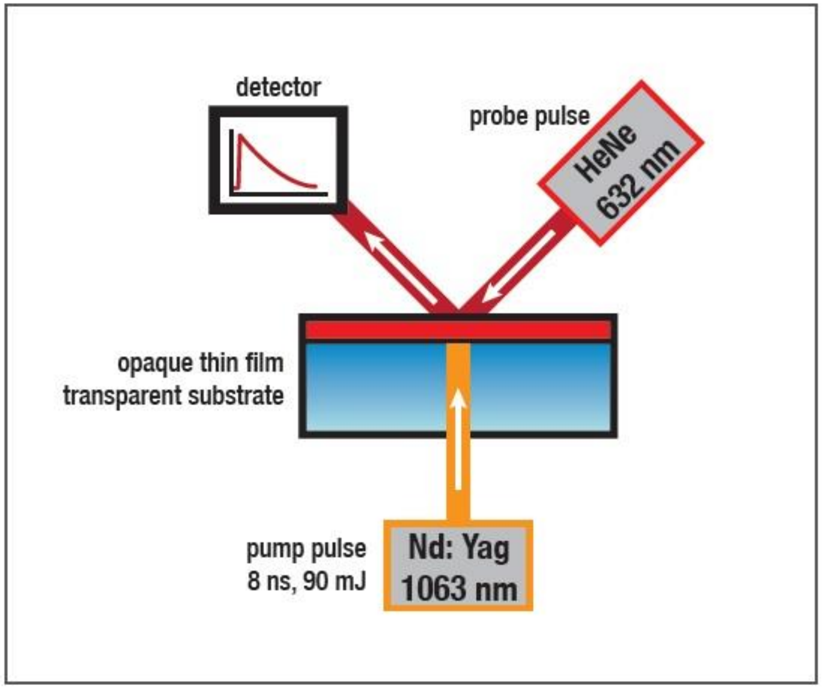
Time domain thermoreflectance

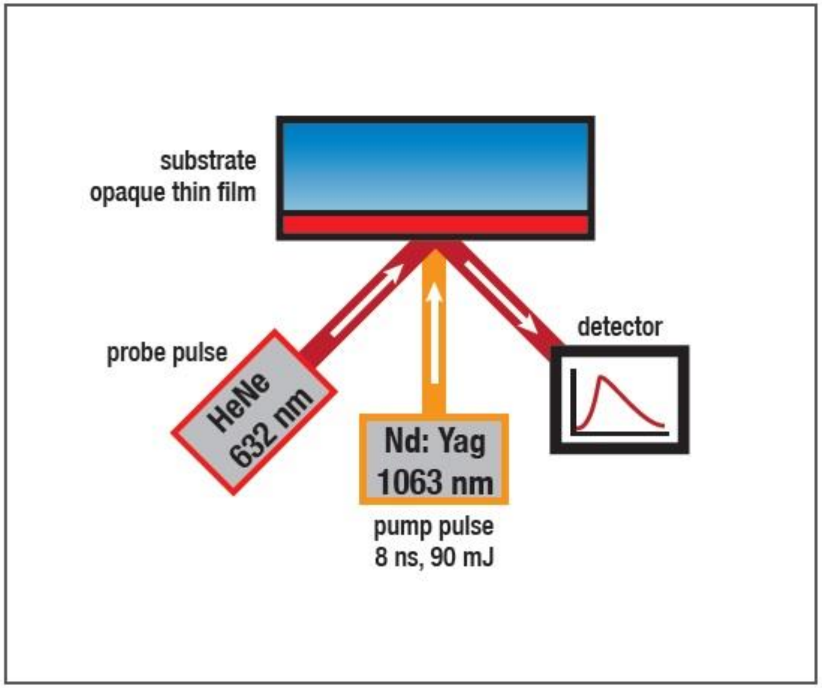
Experts: Bernard Perrin (INSP, acoustic properties), Stefan Dilhaire and co. w (thermal properties, Univ. Bordeaux, LOMA), David Cahill (thermal properties, Univ. Illinois at Urbana Champ.),

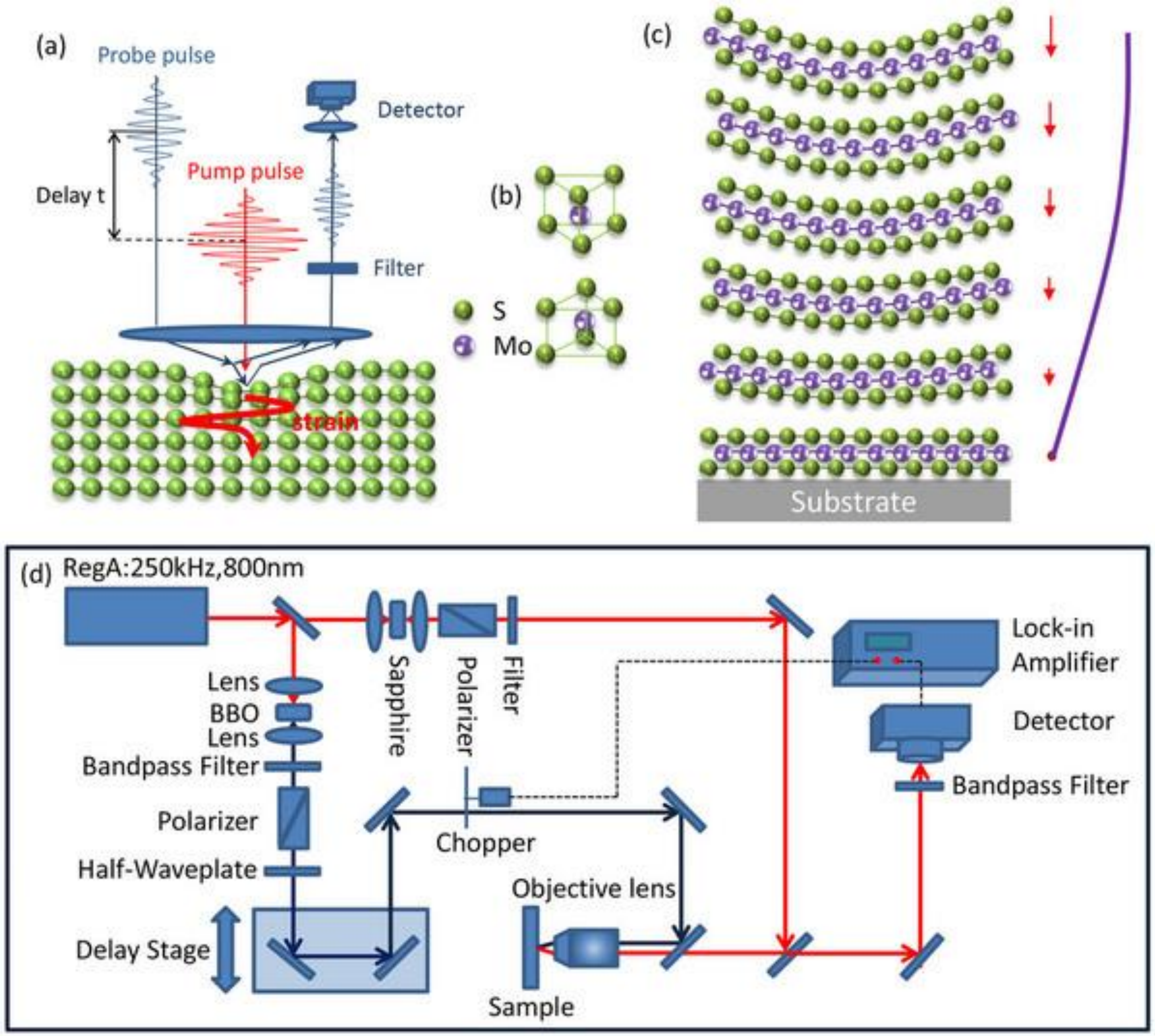
Time domain thermoreflectance

- Thermometry: temperature variation of the coefficient of reflection of the materials
- Aluminium: $\Delta R/R=10^{-5}$

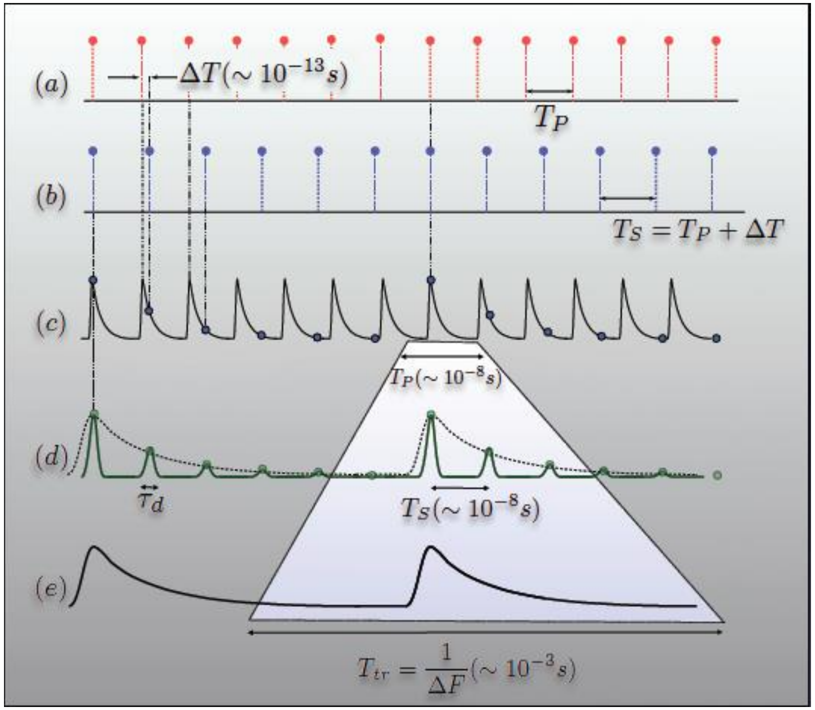


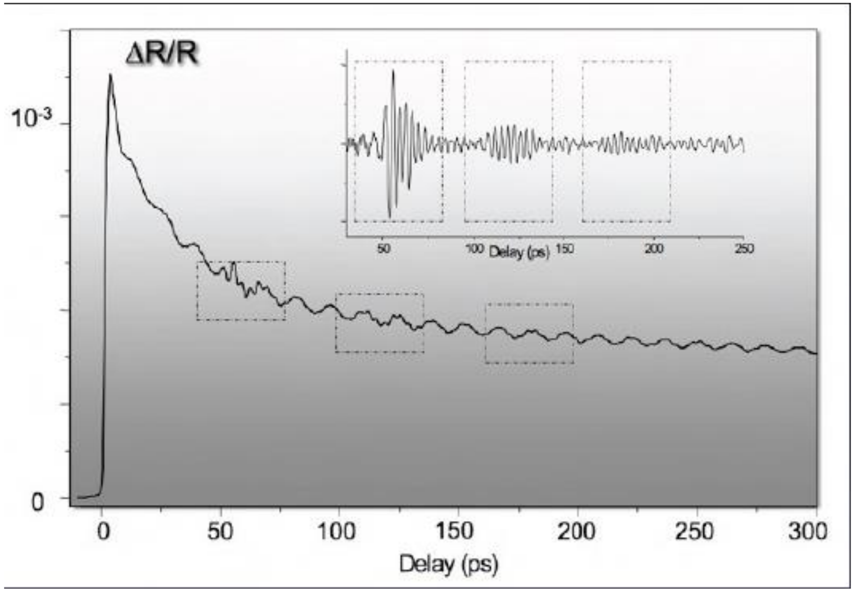





(a)
Probe pulse
Detector
Delay t
Pump pulse
Filter
strain
(b)
S
Mo
(c)
Substrate
(d) RegA:250kHz,800nm
Sapphire
Polarizer
Filter
Lock-in
Amplifier
Detector
Lens
BBO
Lens
Bandpass Filter
Polarizer
Half-Waveplate
Chopper
Bandpass Filter
Objective lens
Delay Stage
Sample

How to measure the thermal decay





Performances Picosecond Thermoreflectance

- Temperature sensitivity $100\mu K$
- Thermal Lateral resolution $1\mu m$
- Time resolution $100fs$

Taken from Stefan Dilhaire (LOMA)

TDTR versus 3 omega

• Accuracy is typically limited to several percent
due to uncertainties in the many experimental TDTR ypparameters
– Metal film thickness
– Heat capacity of the sample if film is thick
• But many experimental advantages
– No need for electrical insulation
– Can separate the metal/film interface thermal
conductance from the thermal conductivity
conductance from the thermal conductivity
– High spatial resolution
– Only need optical access: high pressures,
high magnetic fields, high temperatures

See papers by Cahill Univ. Illinois Urb. Champ.

Conclusions
Measurement at the nanoscale : comparison and numbers

Comparisons of the various techniques

	Work T	ΔT	Has to be suspended	Thin films	membrane	nanowire	1/x dX/dT at RT	ΔK	ΔE/ΔP	ΔK/K
Differential	0K-400K	<1mK	Yes	No	Yes	Yes	10^{-2}	nW/K	pW	0.1/1%
3 omega 1D/2D	0K-400K	<1mK	Yes	Yes	Yes	Yes	10^{-2}	pW/K	fW	0.1%
3 omega Semi-infinite	0K-400K	<10mK	No	Yes	No	Yes/No	10^{-2}	nW/K	nW	1 %
SThM resis.	RT	Few K	/	Yes	?	Y/N	10^{-3}	µW/K	?	1/10%
Fluoresc. SThM	RT	1K	/	Yes	Yes	Yes	10^{-5}	µW/K	?	10 %
Bimetal	RT	<1mK	Yes	No	No	Yes	10^{-3}	10 nW/K	pW	10 %
Raman	100k<T	20K	Yes	?	Yes	No	10^{-5}	??	??	??
TDTR	4K-300K		Y/N	Yes	Yes	No	10^{-5}	??	??	??

APPLIED PHYSICS LETTERS 102, 203702 (2013)

Thermal conductivity of single biological cells and relation with cell viability

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[1]*Department of Mechanical Engineering, POSTECH, Pohang, 790-784, South Korea*
[2]*School of Interdisciplinary Bioscience and Bioengineering, POSTECH, Pohang, 790-784, South Korea*

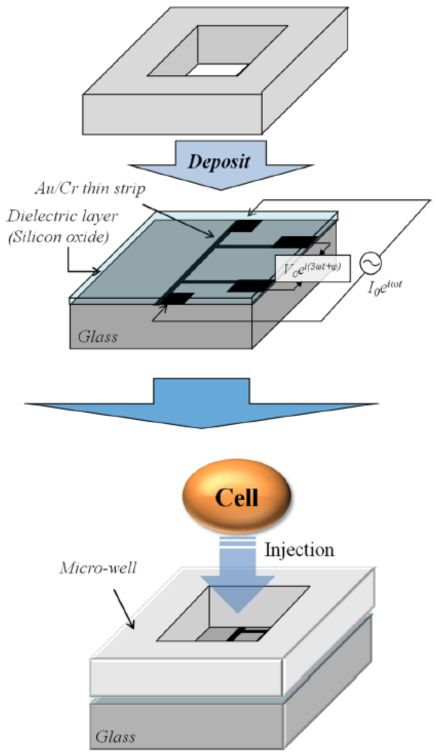


FIG. 1. Schematic design of the instrument.

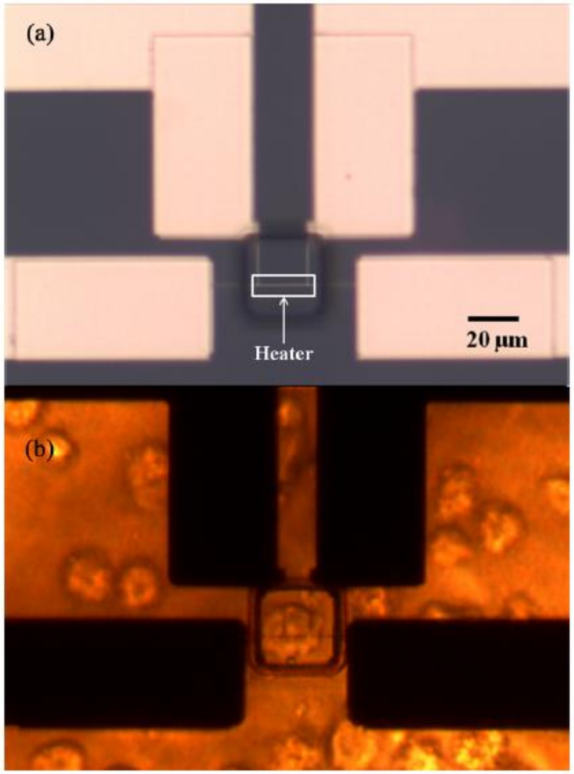


FIG. 2. Photographs of deposited NIH-3T3 J2 cell on sensor surface.

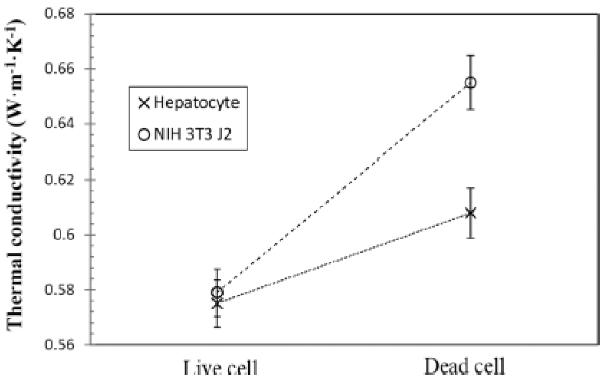


FIG. 4. Thermal conductivities of live and dead cells.

The thermal transport is strongly affected by the denaturation of the DNA strands

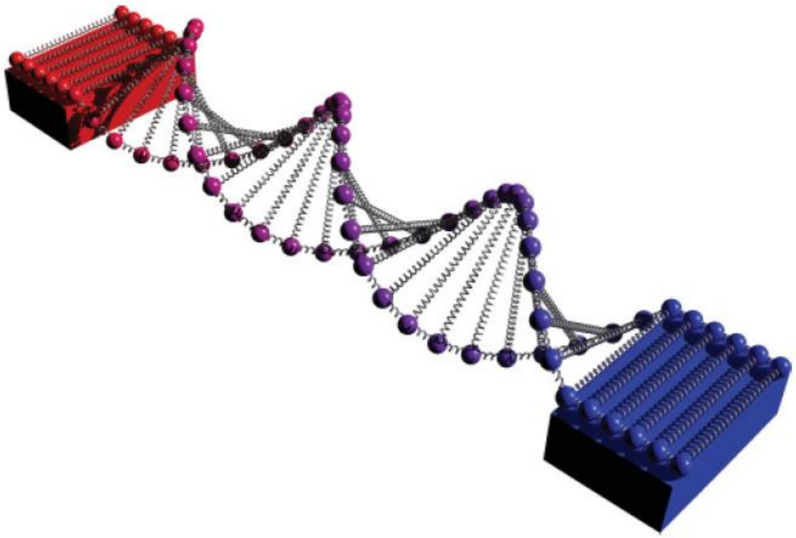
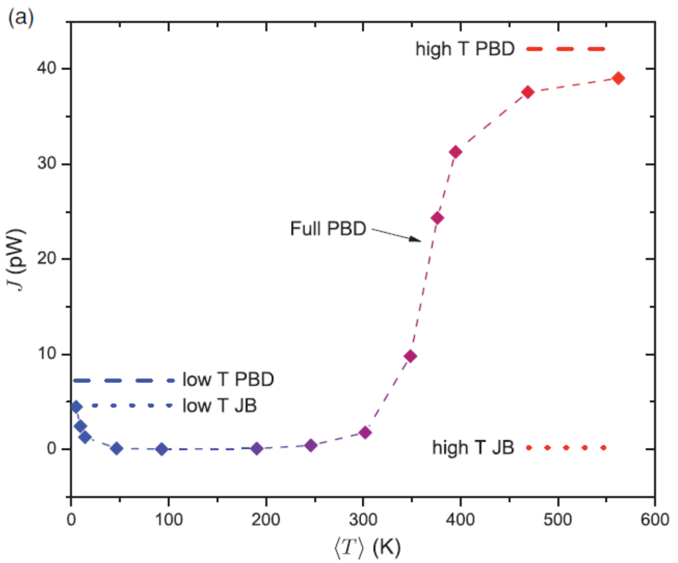


FIG. 1. (Color online) Schematic of DNA between two heat reservoirs that probe its structure via an energy current.



Polymeras chain reaction (PCR)

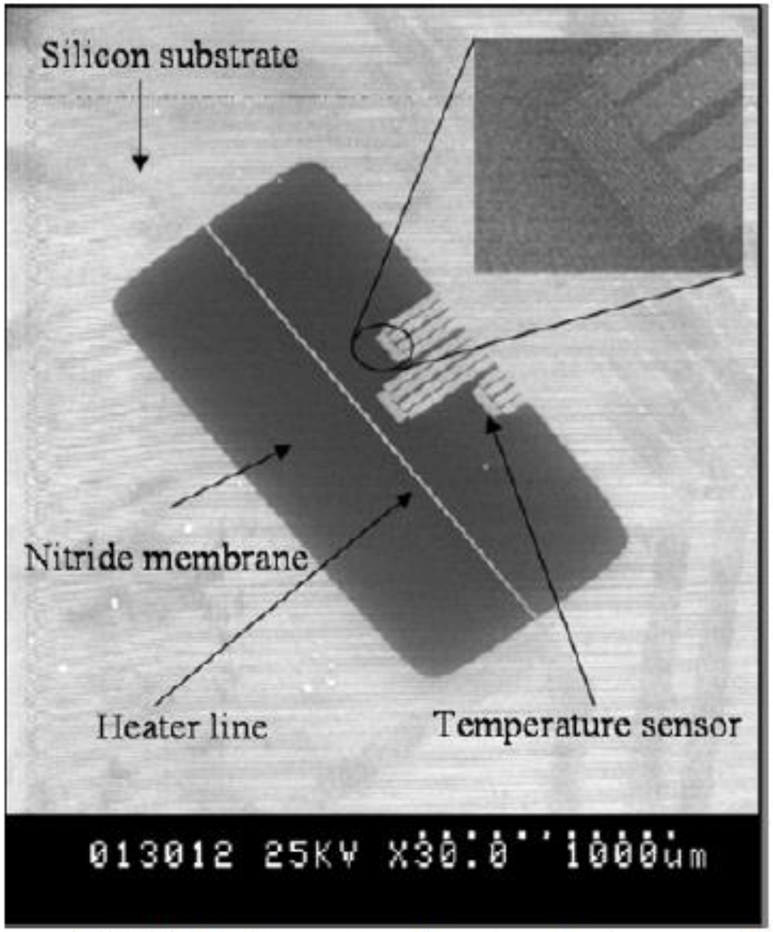


Figure 5. SEM of a microheater device used for studying the effect of thermal gradients on nerve cells [Jain, *et al.* (2009)].

- Technique used to duplicated DNA

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Nanometer-scale heat-conductivity measurements on biological samples

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Received 5 October 2005; accepted 6 January 2006

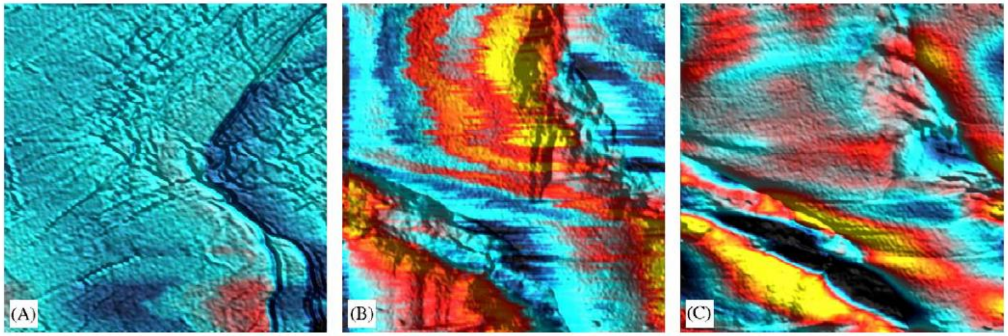

Fig. 4. Image A depicts a 2.2-μm scan on a human hair that was bleached and pretreated with strong detergents. The color texture corresponds to variations in the heat flux of about 100 nW from dark blue to violet, with decreasing heat conductance towards violet. Images B and C depict 8.8 μm scans of an untreated hair. The texture in B shows heat flux differences whereas in C it shows differences in friction. All pictures were made with the cantilever at 300 °C.

- Garden J., Bourgeois O.: Nanocalorimetry. In: Bhushan B. (Ed.) Encyclopedia of Nanotechnology: SpringerReference (www.springerreference.com). Springer-Verlag Berlin Heidelberg, 2012. DOI: 10.1007/SpringerReference_340054 2012-08-21 14:19:29 UTC.