Scanning Thermal Microscopy (SThM): Nanoscale thermal measurements

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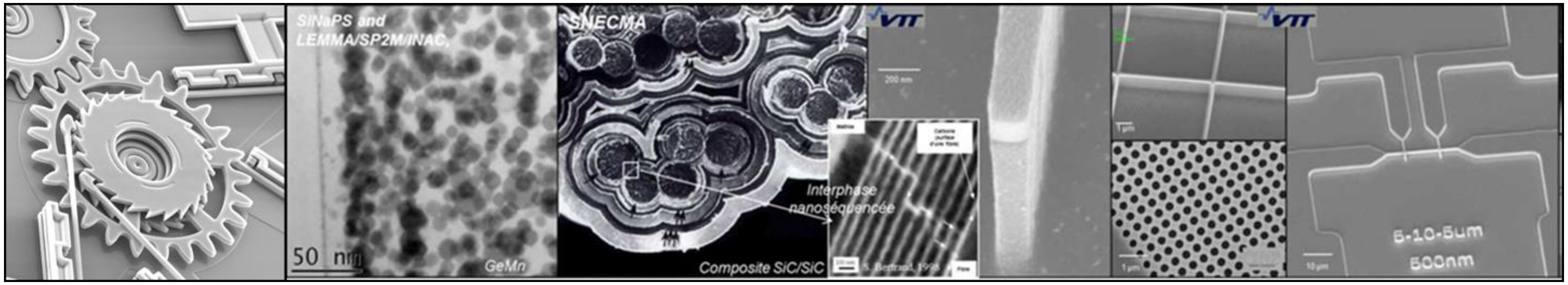
Context and motivation

- Micro and nanotechnologies
- New micro- and nano- structured materials & systems



- Many sectors of sciences and industry concerned

Life **Energy**

Electronics **IT Communication** **Transport**

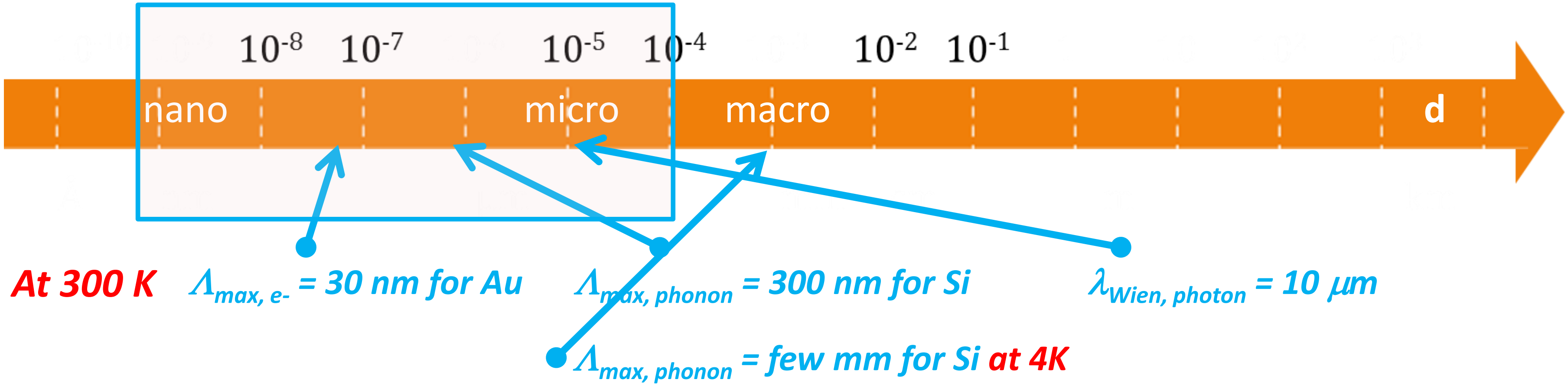
Context and motivation

On micro- and nano- scales
invalidity of macroscopic laws and models of heat transfer

phonons
electrons
molecules (gas)
photons

Λ Mean free path
λ wavelength
d Media dimension

f(material, *T*)



At 300 K $\Lambda_{max,\, e-}$ = 30 nm for Au $\Lambda_{max,\, phonon}$ = 300 nm for Si $\lambda_{Wien,\, photon}$ = 10 μm

$\Lambda_{max,\, phonon}$ = few mm for Si **at 4K**

When d ↘ : increasing impact of surface and interface / volume impact

Scanning Thermal Microscopy (SThM): Nanoscale thermal measurements

1. **Instrumentation and modes**
2. **Influence factors on measurements** (simple model) & **methodologies used to calibrate the technique**
3. **SThM analysis of a nanostructure**: main measurement steps
4. **Conclusion and main challenges**

Scanning Thermal Microscopy (SThM): Nanoscale thermal measurements

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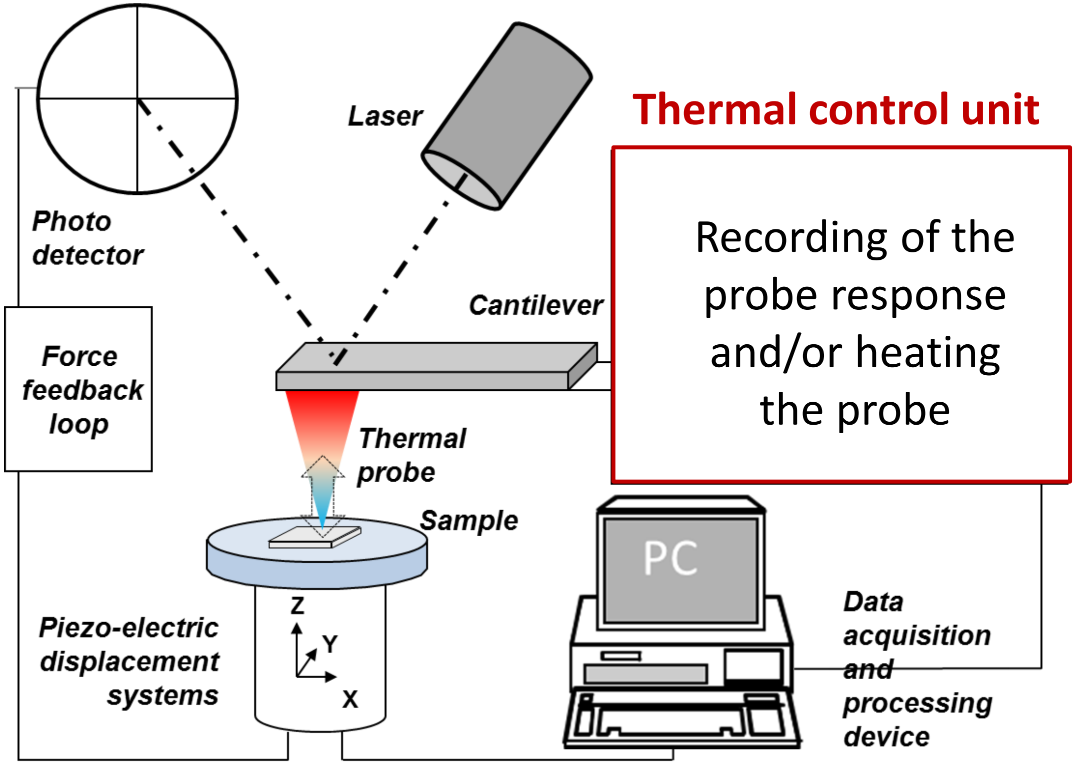
SThM principle

Atomic Force Microscope (AFM) + a thermal AFM probe

AFM probe equiped with a thermal sensor

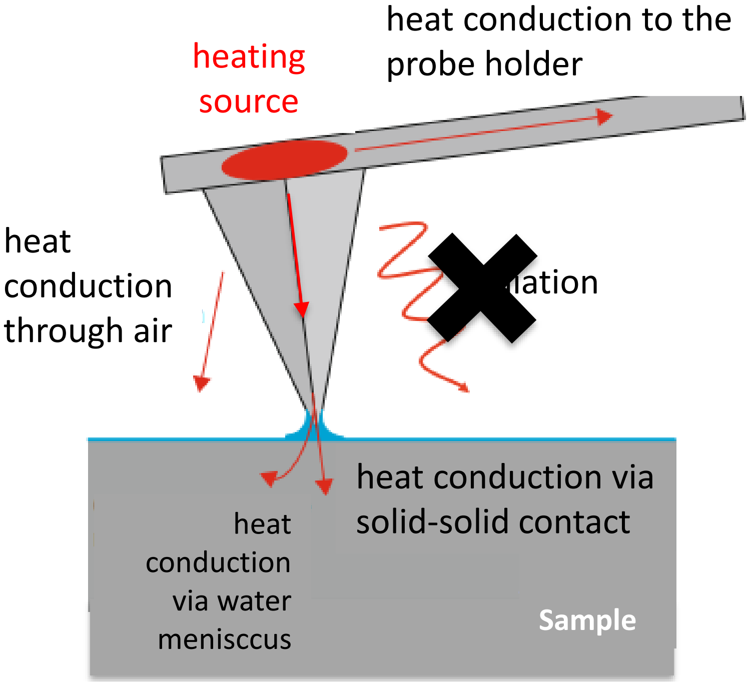


Thermal control unit

Recording of the probe response and/or heating the probe

P.S.S.A, 212, 477–494 (2015)

Probe-sample interaction



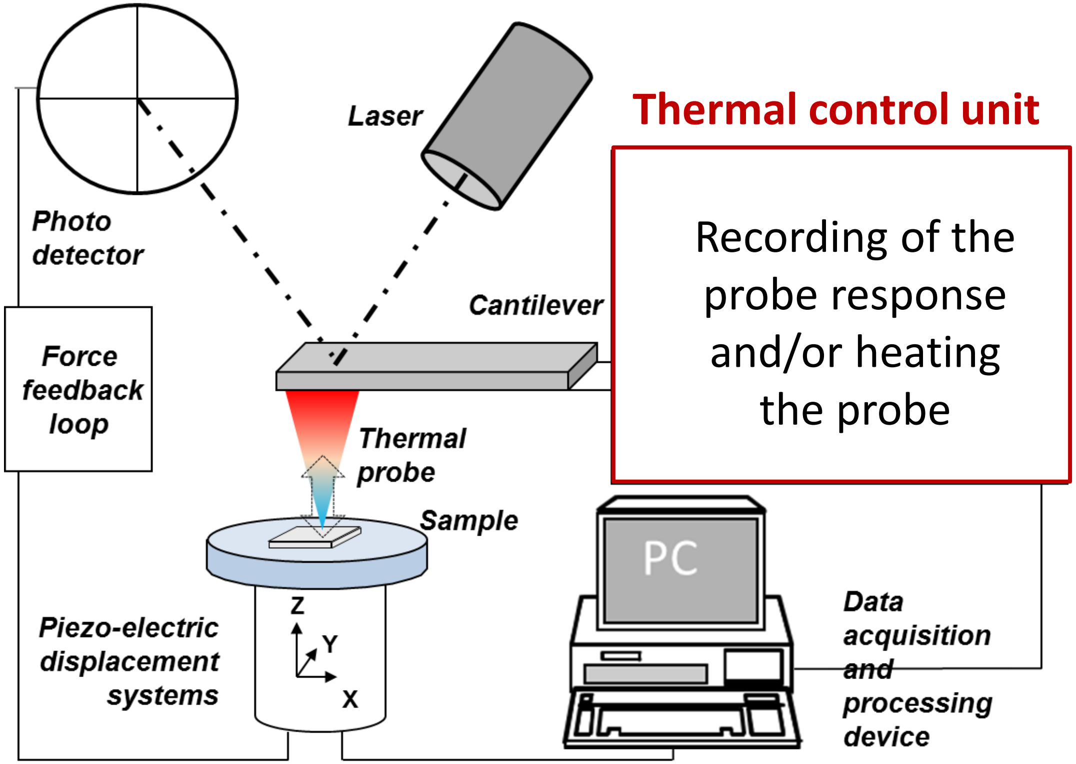
Active mode

Environment

Air, vacuum (and liquid)

SThM principle

Atomic Force Microscope (AFM) + a thermal AFM probe



Thermal control unit

Recording of the probe response and/or heating the probe

P.S.S.A, 212, 477–494 (2015)

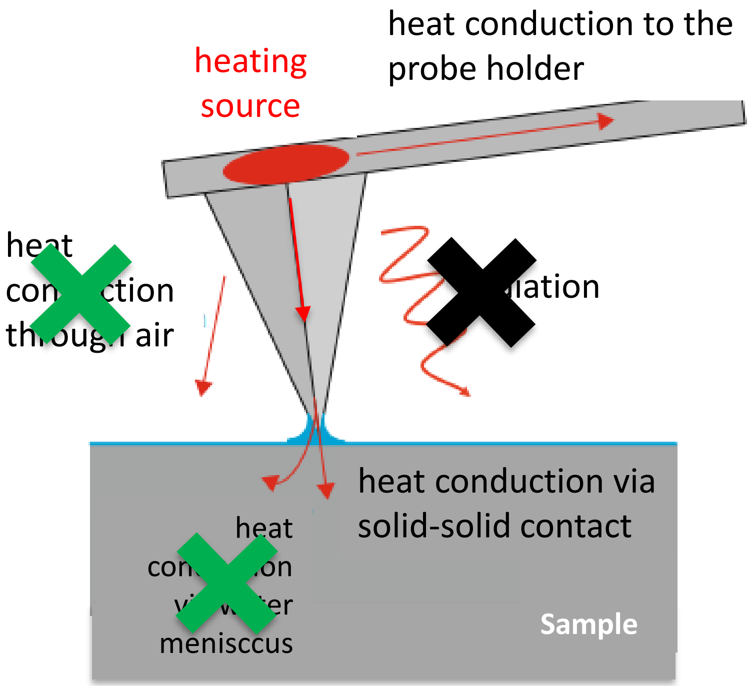
Environment

Air, **vacuum** (and liquid)

pressure lower than 10^{-2} Torr

AFM probe equiped with a thermal sensor

Probe-sample interaction

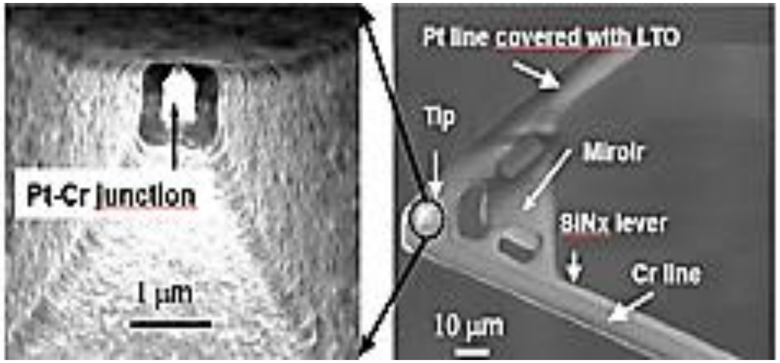


Active mode

SThM principle

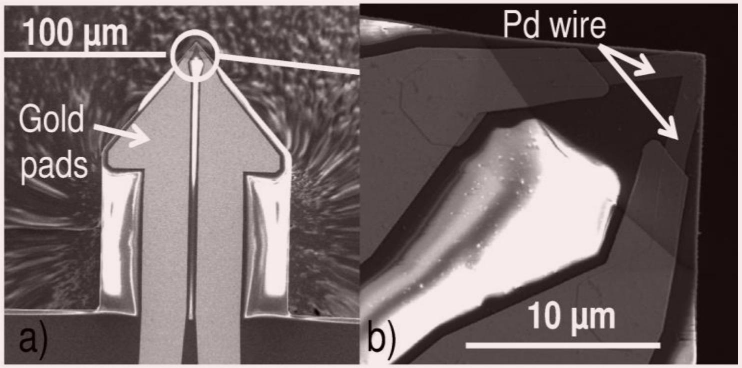
Some types of SThM probes

Thermocouple $S_{Th} \propto \Delta V \propto S_{m_1-m_2}\Delta T$



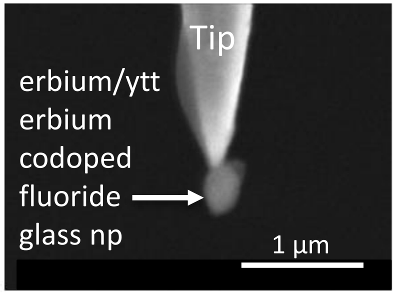
Annual review of materials science, 1999. 29(1): p. 505-585.

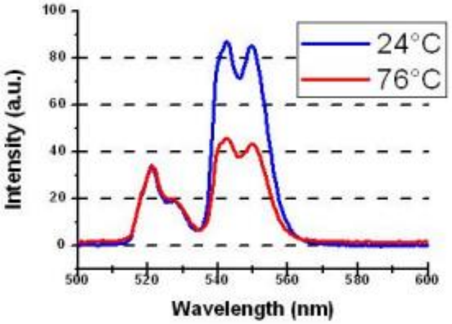
Resistor $R_p = R_{p0}(1 + \alpha_p(T_p - T_{p0}))$



KelvinNanoTechnology (KNT)

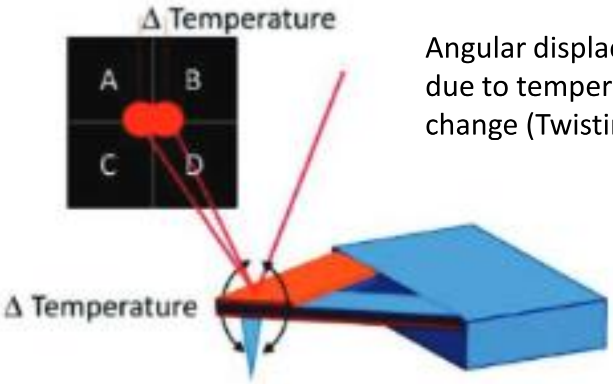
Fluorescent nanoparticle at the apex of the AFM tip





Journal of physics: conference series Volume: 92 Issue 1 (2007)

Bimaterial lever



Angular displacement due to temperature change (Twisting mode)

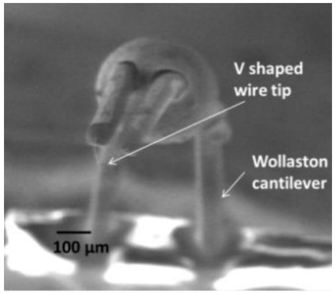
Nano Lett. 2012. 12: p. 1218

SThM principle

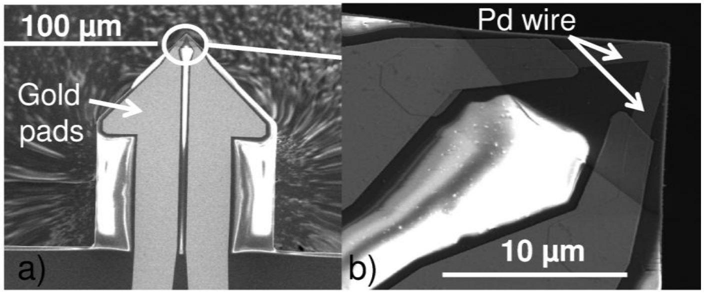
Resistive SThM probes

$$R_p = R_{p0}\,(1 + \alpha_p\,(\overline{T}_p - T_{p0}))$$

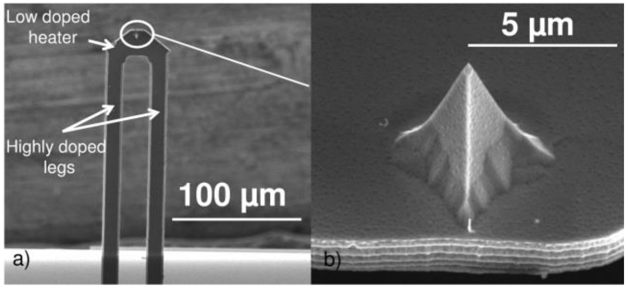
Metallic probe and sensor



Wollaston wire probe



KelvinNanoTechnology (KNT)



Thermal lever (Si doped probe)

	WW probe	KNT probe	DS probe
Probe electrical resistanc (Ω)	~ 2.3	~ 350	~ 1200
Temperature coefficient of electrical resistance: α_p (.10^{-3} K^{-1})	~ 1.66	~ 1.2	~ 2.3 [350-550K]
Electrothermal sensitivity (Ω. K^{-1})	~ 0.004	~ 0.4	~ 2.8
Time thermal resolution (ms)	~ 5.2	τ_{NiCr} ~ 7 τ_{Pd} ~ 0.2	τ_1 ~ 14 τ_2 ~ 0.26

Guen (E.) Thesis Université de Lyon (2020)

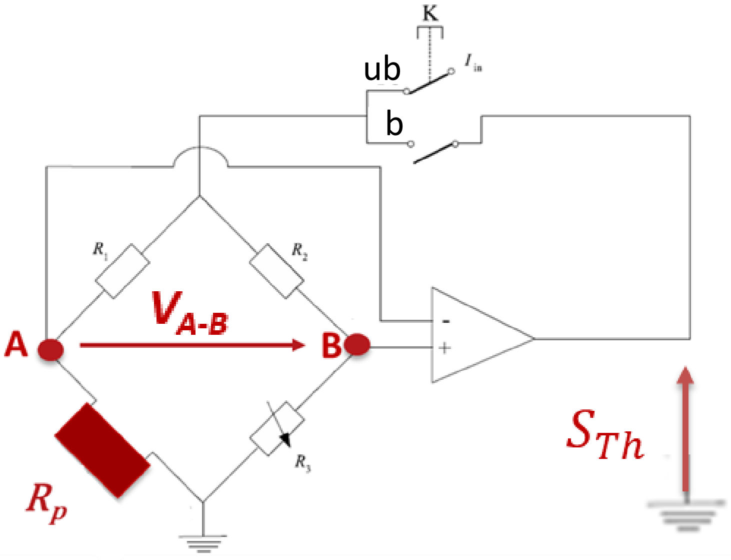
Indicative values
Calibration needed to determine them

SThM principle

Thermal control units

Wheatstone bridge or **differential bridge**

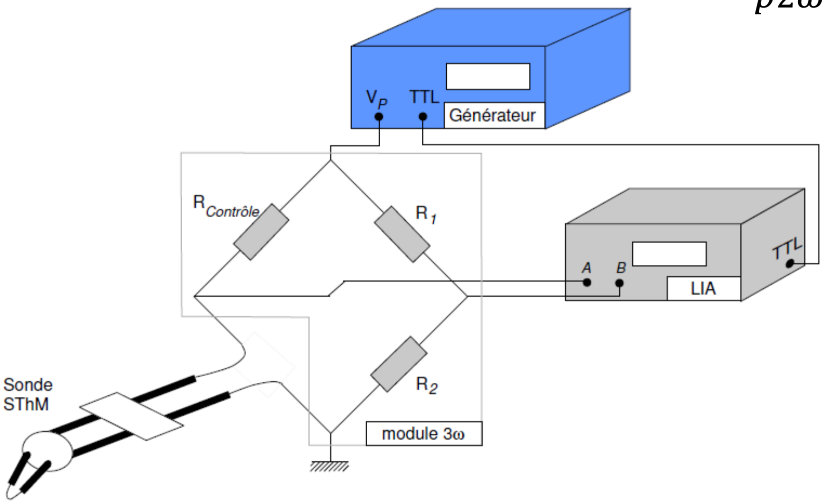
Wheatstone bridge



Lefèvre (S.) Thesis Université de Poitiers (2002)

- Passive and active modes by adjusting the electrical current through the tip sensor
- In the two modes:
    - ○ dc, ac or dc-ac regimes
    - ○ 3ω SThM mode in active mode:

$$\Delta\overline{T}_{p2\omega} = \frac{2V_{p,3\omega}}{\alpha R_{p0} I_0}$$



SThM principle

Atomic Force Microscope (AFM) + **a resistive SThM probe +TCU**

Topography imaging

Thermal imaging

While scanning the sample surface, **detection of variation of probe electrical resistance** ΔR_p ***i.e. probe temperature*** $\Delta \bar{T}_p$

Basic modes

Passive mode $\Delta \bar{T}_p$ → Local heating at the surface of active components

Active mode $\Delta \bar{T}_p$ → Variation of the probe-sample system thermal conductance

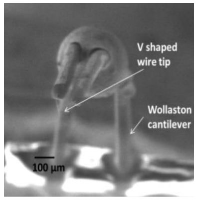
→ Local heating at the surface of active components

Refer to

- *Menges et al., Nature communication 2015*
- *TI 2023, R2770 v2*

SThM principle

Active mode

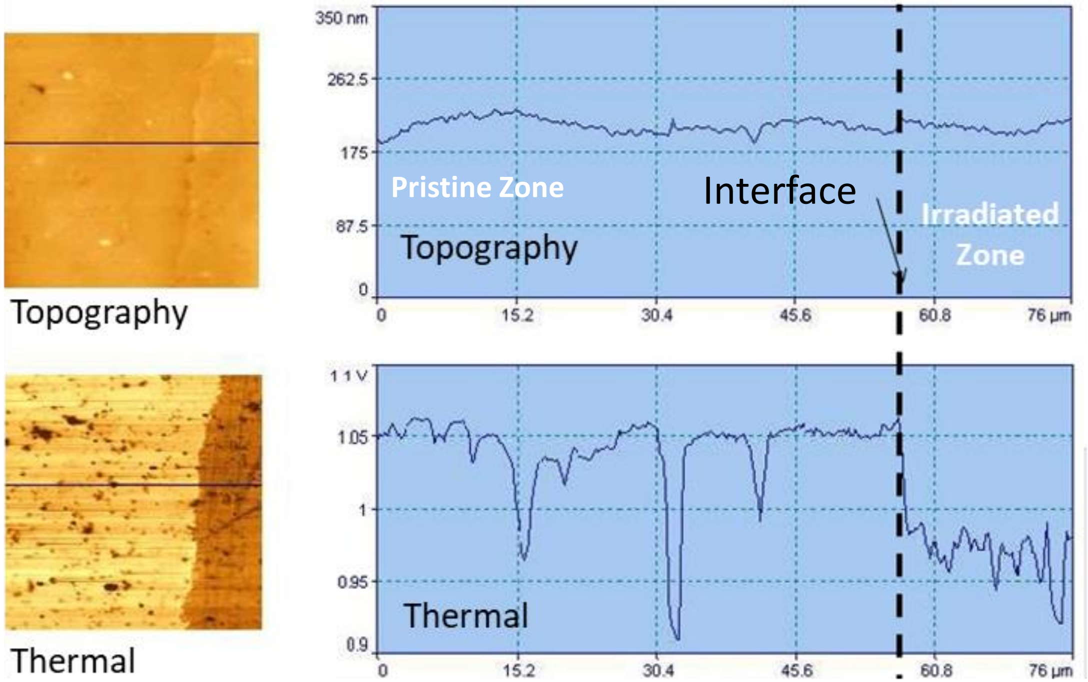


$\bar{T}_p \cong 100^\circ C$

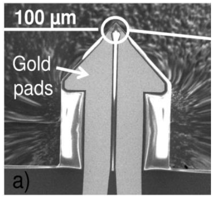
Ambient air conditions

TiC locally irradiated with heavy ions

(Kr with 85 MeV energy)



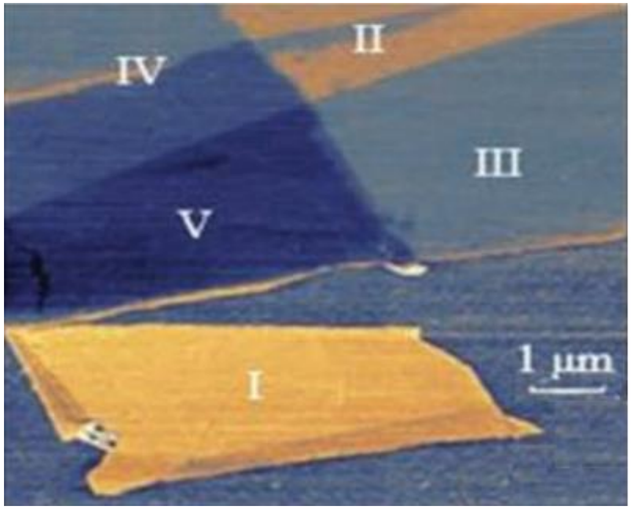
David (L.) Thesis Université de Lyon (2005)



$\bar{T}_p \cong 100^\circ C$

Vacuum conditions

MoS_2 layers



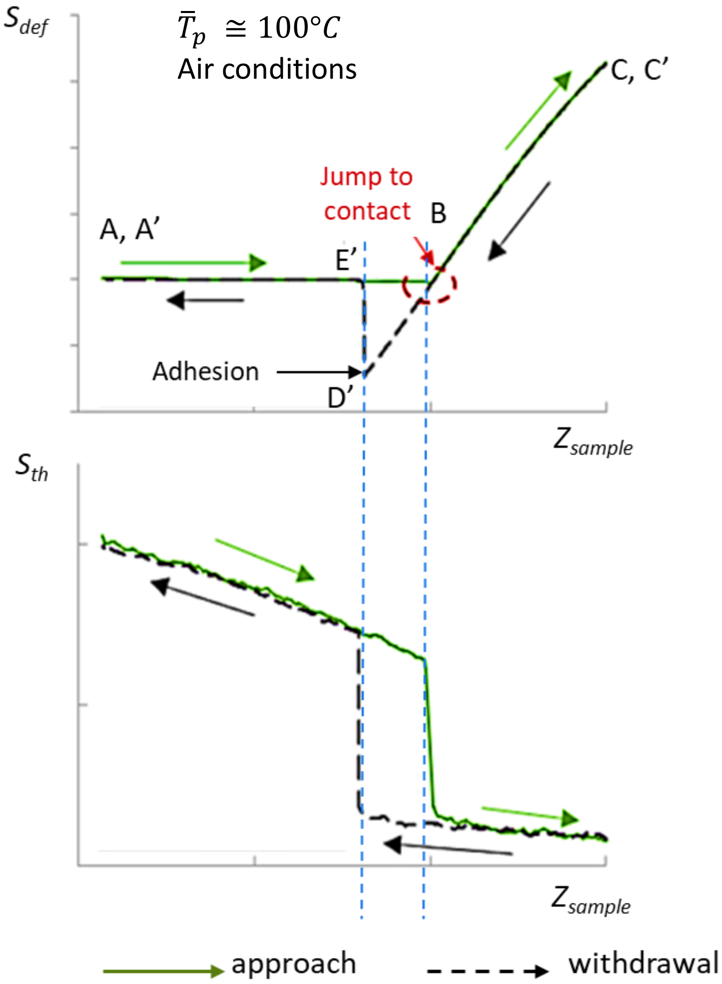
Thermal

Phys. Rev. Lett. 2013, 111, 205901

SThM principle

Simultaneous complementary analyses

Imaging	Topography, roughness	Thermal contrast
Point measurements	Probe-sample force	*Probe temperature* = *f*(probe-sample dist. & force)



Guen (E.) Thesis
Université de Lyon (2020)

+ Combined with other scanning probe microscopy modes: mechanical, electrical…

How to measure the thermal properties of a sample from S_{Th}?

How to calibrate the technique for such measurements ?

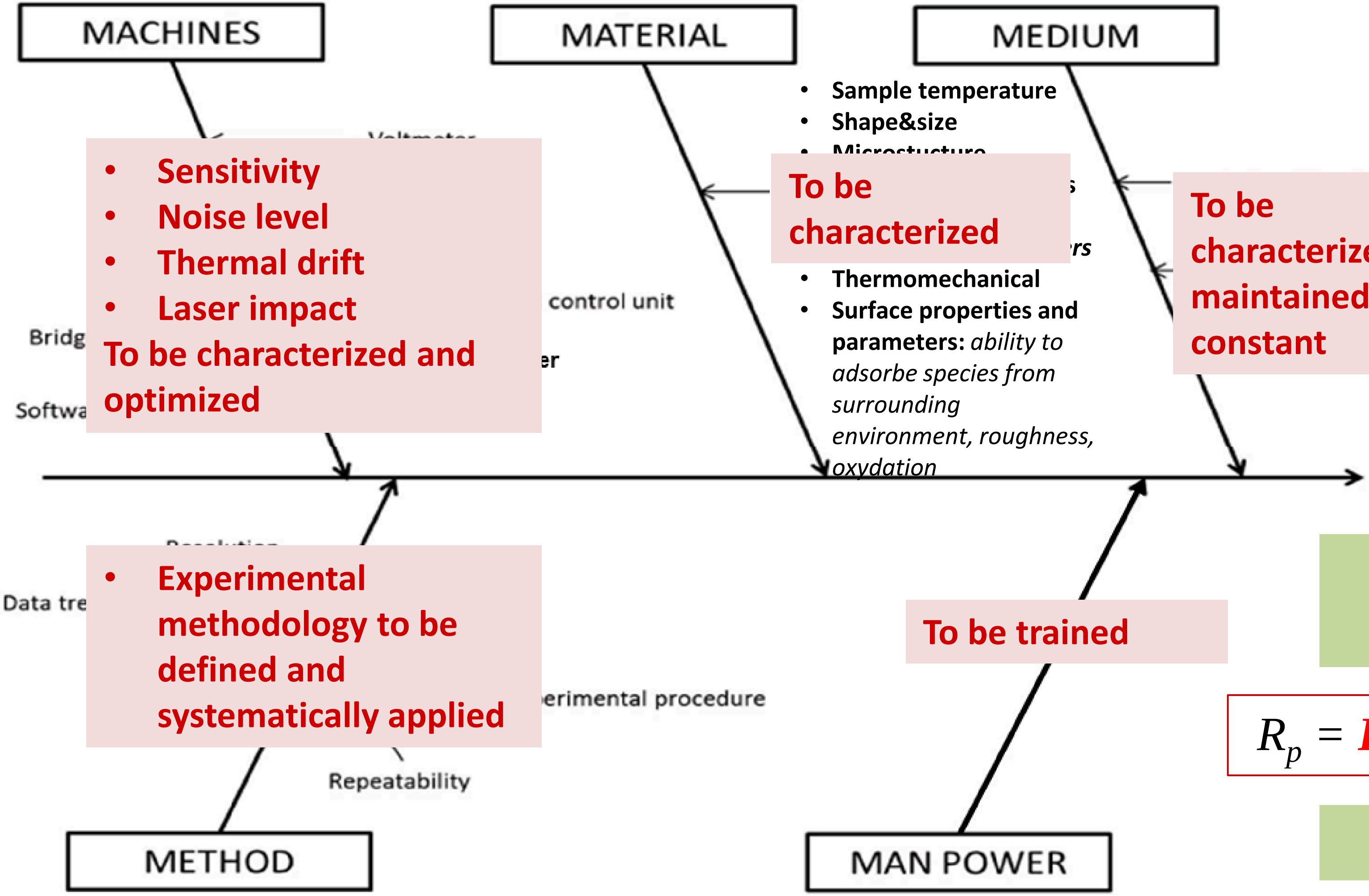
Scanning Thermal Microscopy (SThM): Nanoscale thermal measurements

1. Instrumentation and modes
2. **Influence factors on measurements** (simple model) & **methodologies used to calibrate the technique**
3. SThM analysis of a nanostructure: main measurement steps
4. Conclusion and main challenges

Influence factors

Simplified Ishikawa's diagram for $S_{th} \propto \Delta R_p \propto \Delta \bar{T}_p$

FOR A GIVEN PROBE



- Sensitivity
- Noise level
- Thermal drift
- Laser impact

To be characterized and optimized

To be characterized

- **Sample temperature**
- **Shape&size**
- **Microstucture**
- **Thermomechanical**
- **Surface properties and parameters:** *ability to adsorbe species from surrounding environment, roughness, oxydation*

To be characterized and maintained constant

- Experimental methodology to be defined and systematically applied

To be trained

$S_{th} \propto \Delta R_p \propto \Delta \bar{T}_p$

How to link ΔR_p to ΔT_P ?

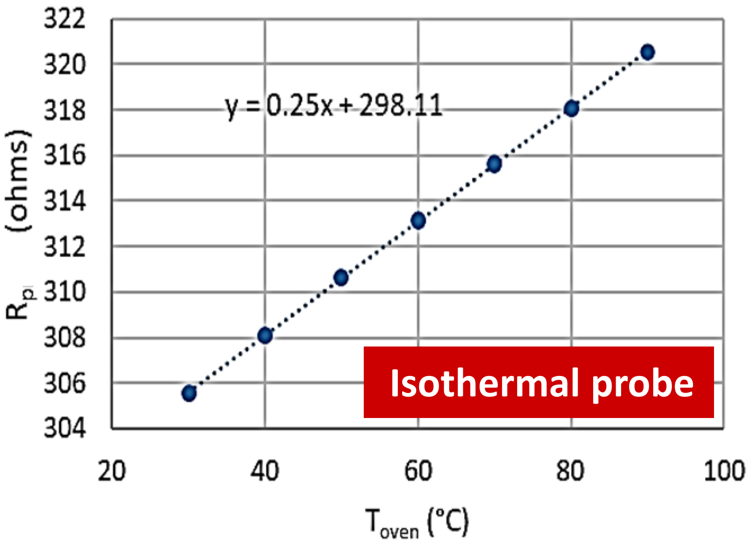
$$R_p = R_{p0}\,(1 + \alpha_p\,(\bar{T}_p - T_{p0}))$$

Calibration n°1

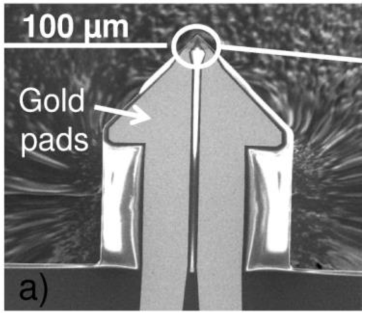
MST, 2017, 28 (11), pp.115010.

Calibration of probe sensor

T - sensor calibration in an oven



$$R_p = \boldsymbol{R_{p0}}\,(1 + \boldsymbol{\alpha_p}\,(T_{oven} - T_{p0}))$$



$$R_P = f\,(R_{sensor},\ R_{electrical\ lead})$$

$$R_{sensor} = \boldsymbol{R_{sensor,T0}}\,(1 + \alpha_{sensor}\,(\overline{T}_{sensor} - T_0))$$

$$R_{electrical\ leads} = R_{l0}\,(1 + \alpha_l\,(\overline{T}_l - T_0))$$

2 equations 6 unknown parameters

Different strategies

R_{l0}
$R_{sensor,T0}$
} measured when possible or calculated with known dimensions of the probe components →dimensions to be determined

α_{sensor}
α_l
} from scientific litterature + T - probe calibration

$$\Rightarrow \quad R_P = f\,(\boldsymbol{\overline{T}_{sensor} - T_0})$$

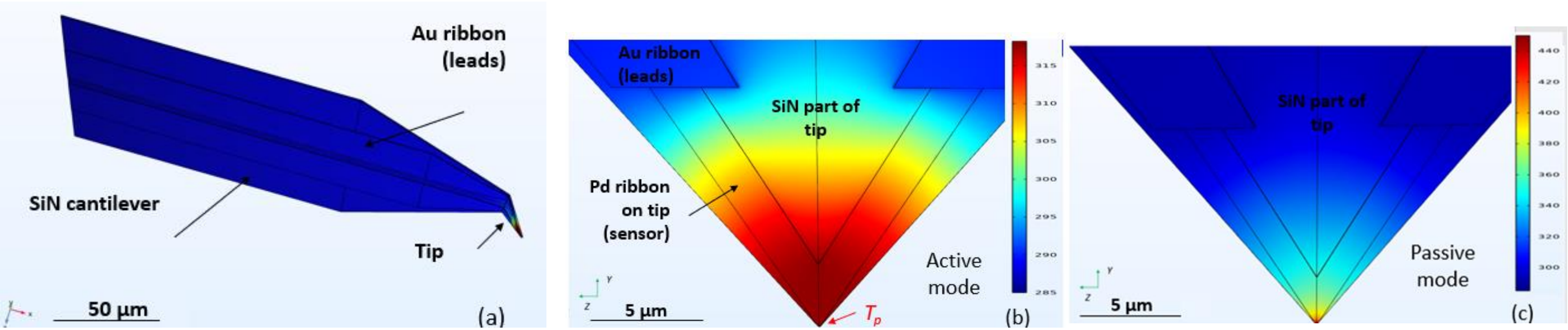
⚠ But experimental conditions differing than those during measurements

Guen (E.) Thesis Université de Lyon (2020)

Calibration of probe sensor

While measuring a sample the probe is not isothermal

3D FEM modelling of a Pd probe in vacuum conditions



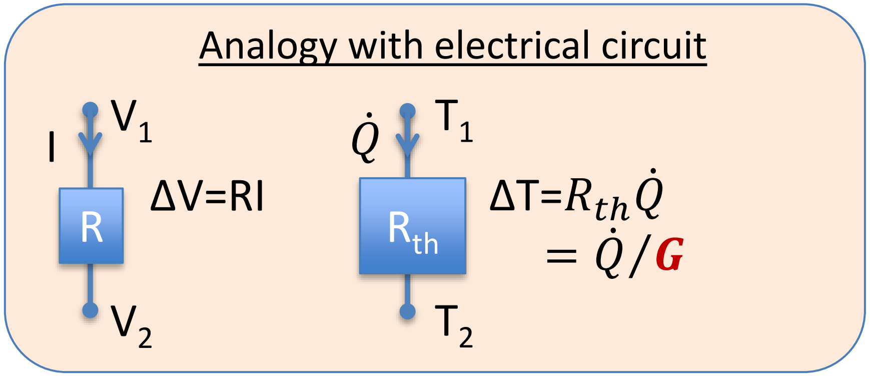
TI 2023, R2770 v2

Temperature profile along the resistive Pd sensor completely different in passive and active modes

A probe cannot be calibrated by using a heating sample if we want to measure the thermal properties of a sample

Influencing factors

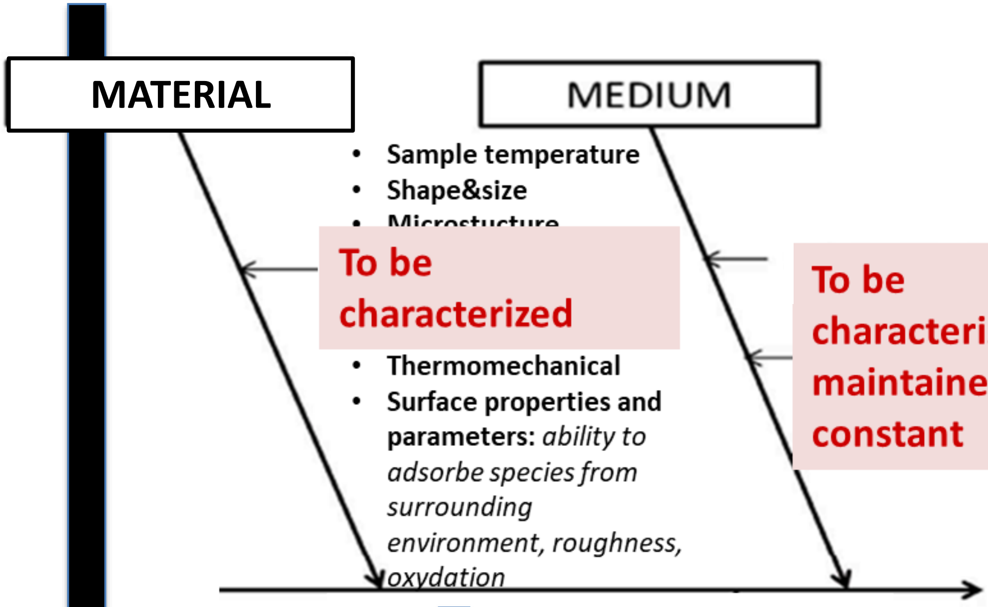
Simplified Ishikawa's diagram for $S_{th} \propto \Delta R_p \propto \Delta T_P$
FOR A GIVEN PROBE



Analogy with electrical circuit

$\Delta V = RI$

$\Delta T = R_{th}\dot{Q} = \dot{Q}/G$



MATERIAL

MEDIUM

- **Sample temperature**
- **Shape&size**
- **Microstucture**
- **Thermomechanical**
- **Surface properties and parameters:** *ability to adsorbe species from surrounding environment, roughness, oxydation*

To be characterized

To be characterized and maintained constant

Probe-sample interaction. heat conduction through: gas?, liquid meniscus? …

$S_{th} \propto \Delta R_p \propto \Delta \bar{T}_{Sensor}$

T_s

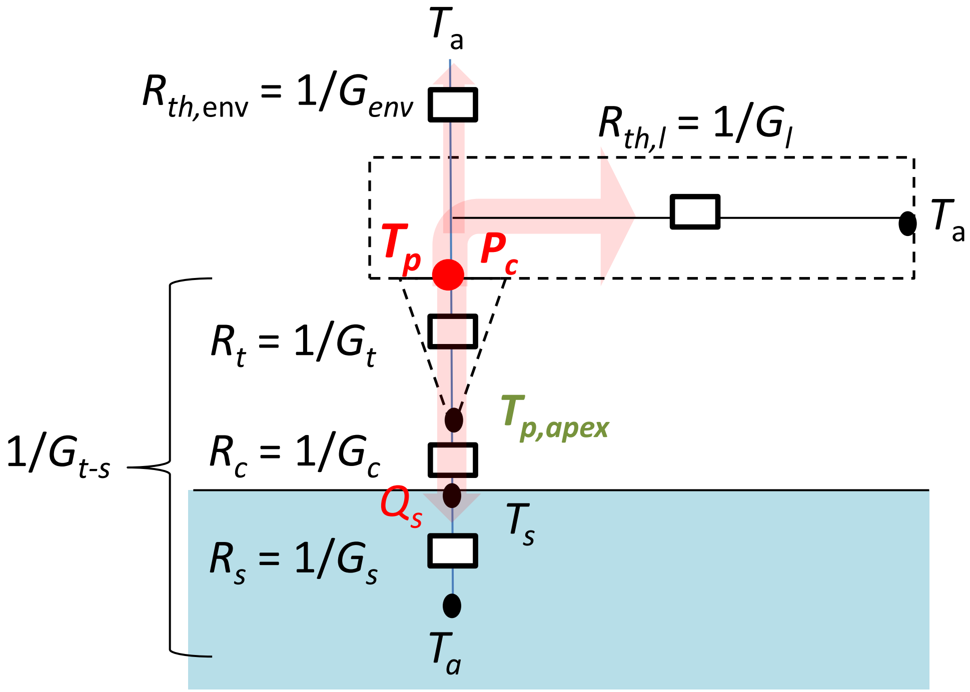
$G_s(k_s, \text{sample geometry})$

Probe-sample interaction. heat conduction through: solid-solid multicontacts ?, Oxide layer? …

Model of the probe interacting with sample needed,
It depends on: G_p , G_s and G_c

Simple model – active mode

- **Probe electrically heated with an electrical current I_p**



- **Probe in contact with sample (index ic)**

$$G_{probe,ic} = P_c/(\boldsymbol{T}_{p,apex} - T_a)$$

$$= R_p I_p^2/(\boldsymbol{T}_{p,apex} - T_a)$$

$$= \boldsymbol{G}_l + \boldsymbol{G}_{env,ic} + \underline{G_{t\text{-}s}}$$

$$\frac{1}{G_{t-s}} = \frac{1}{R_{th,t} + R_{th,c} + R_{th,s}}$$

$\boldsymbol{T_p}$ **can be measured but** $T_{p,apex}$?

G_l? $R_{th,t}$? G_c? $G_{env,ic}$?

- **More advanced model needed for determining the thermal gradient along the probe**
- **Calibrations**

Guen (E.) Thesis Université de Lyon (2020)

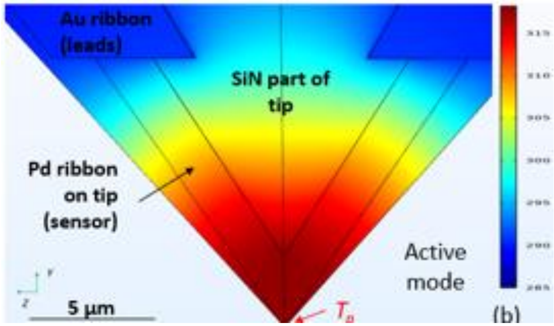
Model – active mode

Determining the thermal gradient along the probe

- **1D and 3D models proposed based on the resolution of the heat equation with a power dissipated in the sensor *P.***

$$P + k_p \Delta T = \rho C_p \frac{dT}{dt}$$

k sensor thermal conductivity (W/(m·K))
C_p specific heat capacity (J/(kg·K))
ρ density (kg/m3)



TI 2023, R2770 v2

TO BE CALIBRATED as they depend on geometrical and physical properties of the materials constituting the probe

Calibration:

Fitting measurements with simulations
for a **probe out of contact** and **in different experimental conditions :**

- probe temperature,
- electrical current amplitude
- frequency...

P.S.S.A, 212, 477–494 (2015)

$\cancel{G_l}$? $\cancel{R_{th,t}}$? G_c ? $G_{env,ic}$?

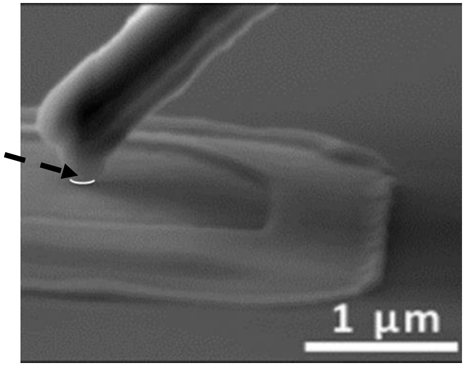
$T_{p,apex}$ **while the probe in contact ?**

$G_{env,ic}$ → 0 W/K in air and vacuum conditions

Probe in contact - $G_{t\text{-}s}$ – active mode – vacuum conditions

$$\frac{1}{G_{t-s}} = \frac{1}{R_{th,c}+R_{th,s}} \qquad G_{t\text{-}s} = \frac{G_c}{1+G_c/G_s}$$

G_c ?



At the level of a spatially limited nanometric in size contact

G_c depends on:

- Difference in phonon dispersion between the **two materials in contact**
- Electron-phonon coupling at interface
- Atomic and nanometric roughness of tip and sample
- Possible native oxide

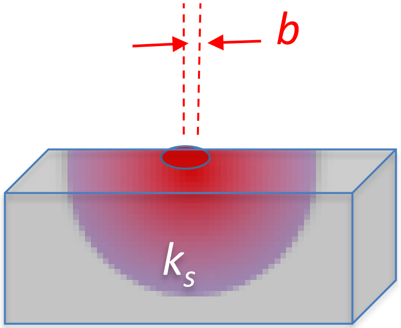
Calibration: Using reference sample(s) with well known thermal conductivity k_s and roughness

How to estimate G_s as a function k_s?

Constriction in case of bulk samples

$$1/G_s = R_{s,diffusive} + R_{s,balistic}$$

$$= \frac{1}{4\,Kbk_s} + \frac{4\Lambda}{3\pi b^2 k_s}$$



b ?

- ProZh. Eksp. Teor. Fiz. 48, 984 (1965).
- Phys. Soc. London 89, 927 (1966).
- Physical Review B 60, 3963 (1999).

G_c and *b* – active mode – vacuum conditions

G_c and *b* crutial parameters for a given model of the probe

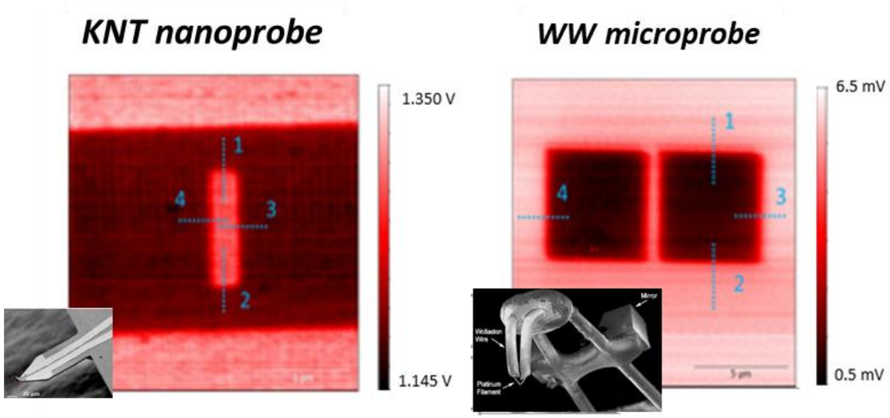
Determining *b*

- Nanocontact mechanical models depending on SThM probes
- Using specific samples

Determining G_c using reference sample(s)

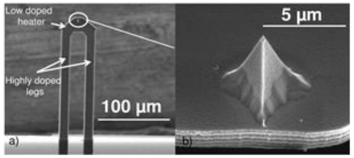
G_c depends on the studied sample

- *Approximation G_c constant*
- *Uncertainty on the measurement to be precised by the user depending on its determination method*



Zone	KNT (nm)	WW (nm)
1	104	480
2	72	380
3	72	800
4	110	760

Quantiheat Appl. Note 2017



10 nm in UHV

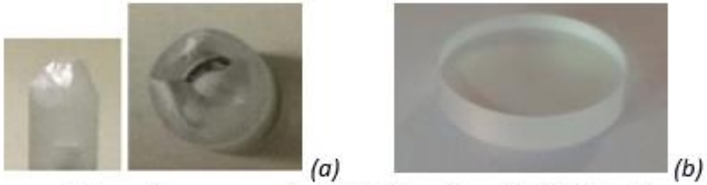
IBM

Calibration, active mode

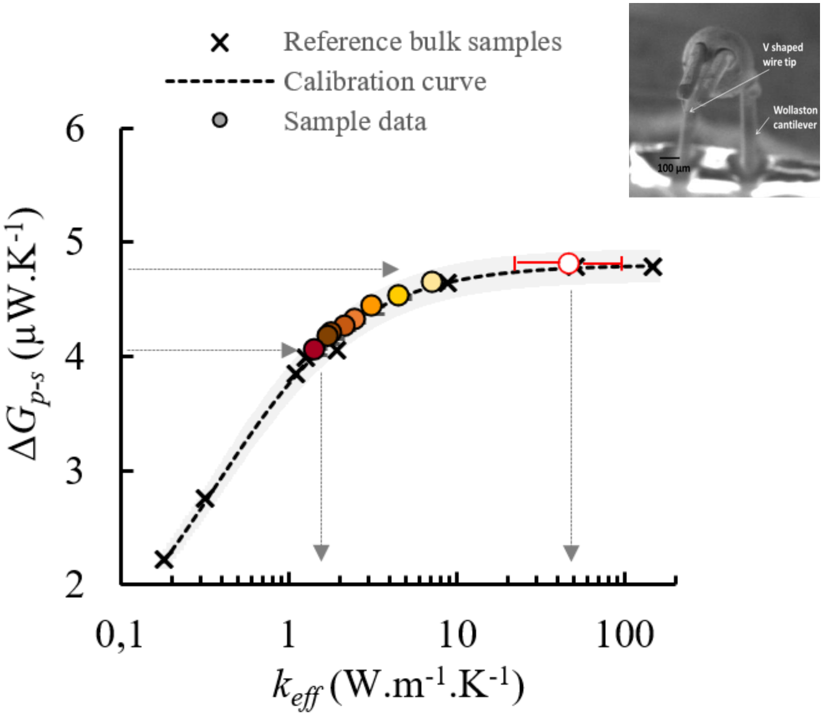
Journal of Applied Physics, 2020, vol. 128, no 23.

Reference samples with known thermal conductivity, roughness and mechanical property

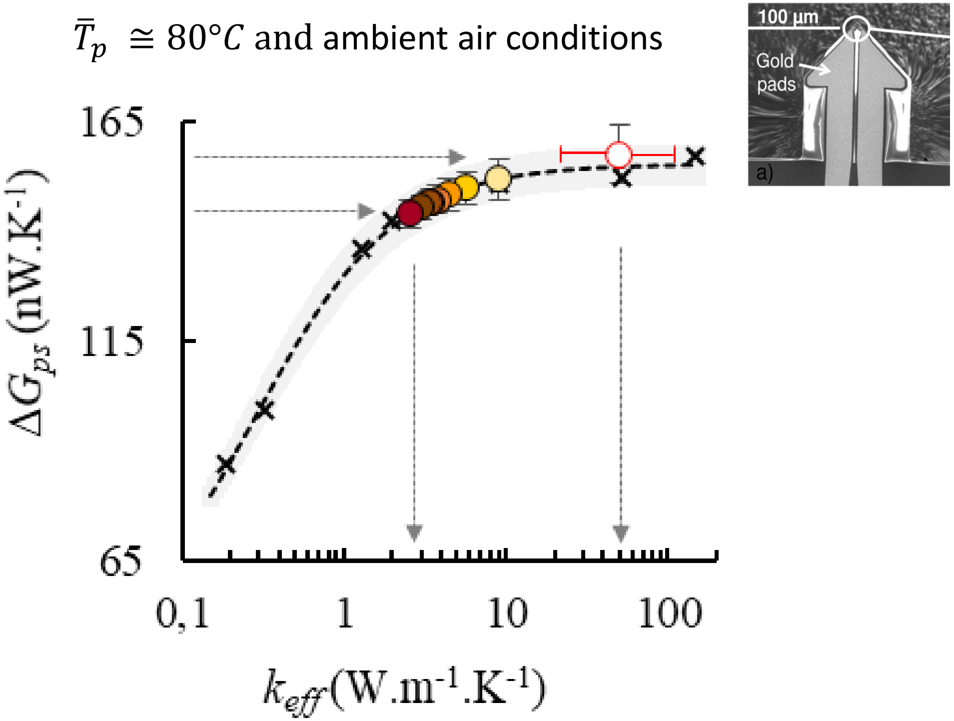
Using a calibration curve: $\frac{\boldsymbol{A}}{1+\boldsymbol{B}/k_s}+c$



Picture of some samples (a) S-TH-cal1 and (b) S-TH-cal3

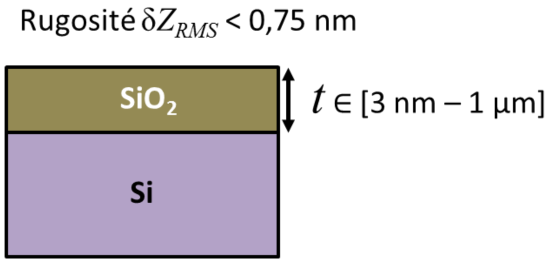


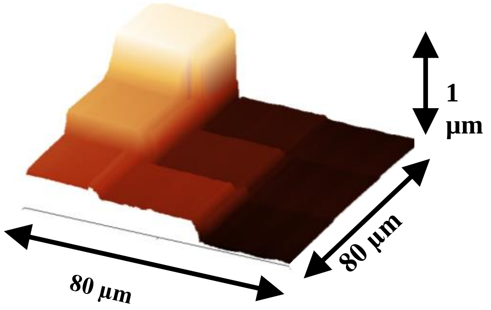
$\bar{T}_p \cong 80°C$ and ambient air conditions

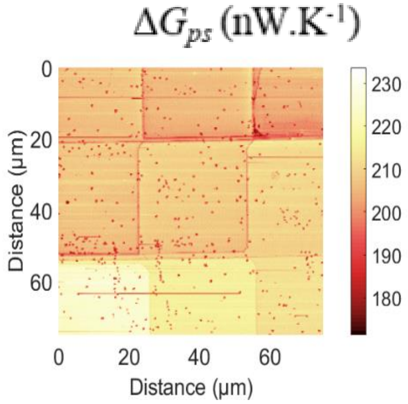


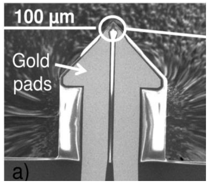
Oxide effect

Journal of Applied Physics, 2020, vol. 128, no 23.

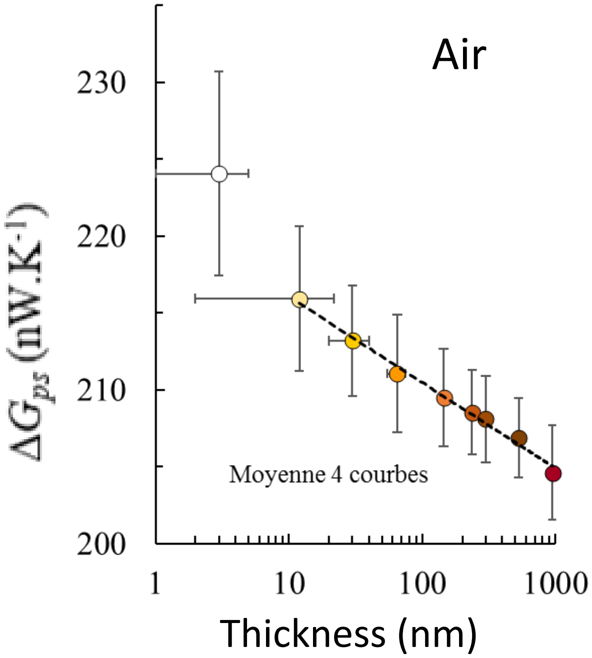


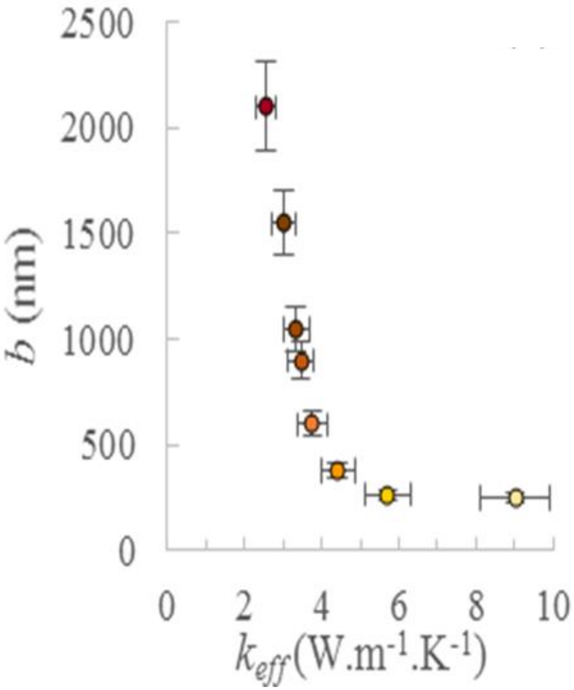


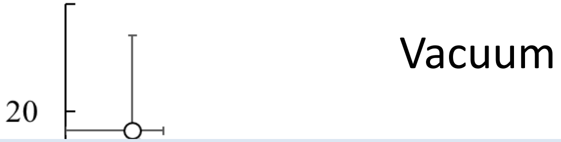




$\bar{T}_p = 80°C$







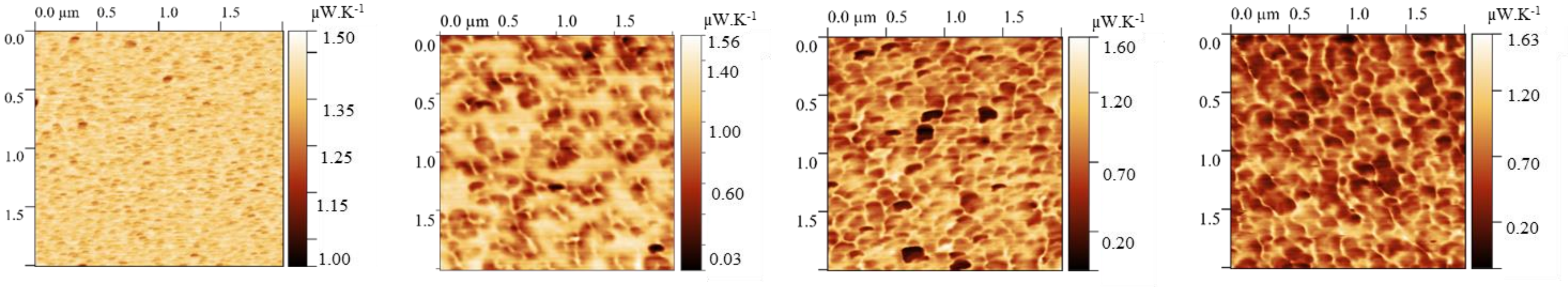
- ➢ High sensitivity to ultra-thin films
- ➢ b varying as a function of k_{eff}
- ➢ Probed depth μ_{SThM}:
  - ○ Air: $\mu_{SThM} > 1$ µm
  - ○ Vacuum: $\mu_{SThM} < 100$ nm

Roughness effect

APL, *119*(16), 161602 2021

Analysis of Si samples with roughness differing

$\delta Z = 0.35$ nm　$\delta Z = 3.96$ nm　$\delta Z = 6.56$ nm　$\delta Z = 10.94$ nm



$G_{th\ contact} = 1.37\ \mu W.K^{-1}$　$G_{th\ contact} = 0.97\ \mu W.K^{-1}$　$G_{th\ contact} = 0.85\ \mu W.K^{-1}$　$G_{th\ contact} = 0.68\ \mu W.K^{-1}$

$\delta G = 0.036\ \mu W.K^{-1}$　$\delta G = 0.225\ \mu W.K^{-1}$　$\delta G = 0.239\ \mu W.K^{-1}$　$\delta G = 0.270\ \mu W.K^{-1}$

→ The roughness induces:

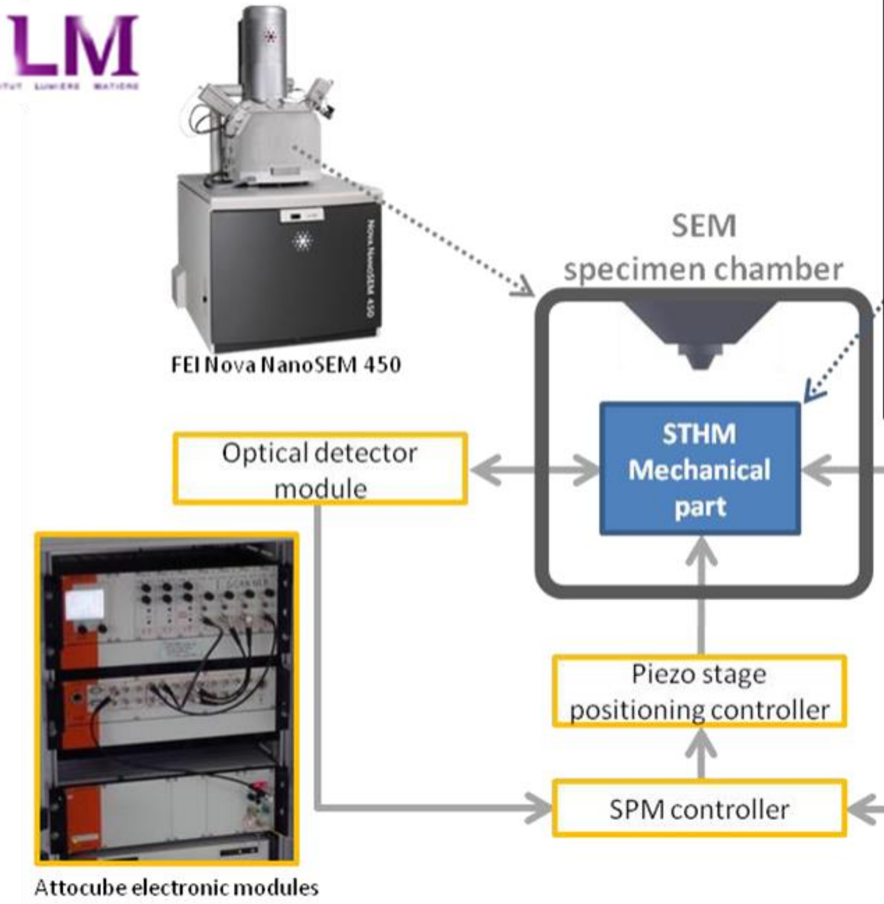
- a thermal conductance decrease between 20 % and 50%
- an increase of standard deviation from 2% for a flat surface to 40% for the roughest samples

Scanning Thermal Microscopy (SThM): Nanoscale thermal measurements

1. **Instrumentation and modes**
2. **Influence factors on measurements** (simple model) & **methodologies used to calibrate the technique**
3. **SThM analysis of a nanostructure**: main measurement steps

   Small (2023): 2305831.
4. **Conclusion and main challenges**

In-situ SEM SThM



SEM specimen chamber

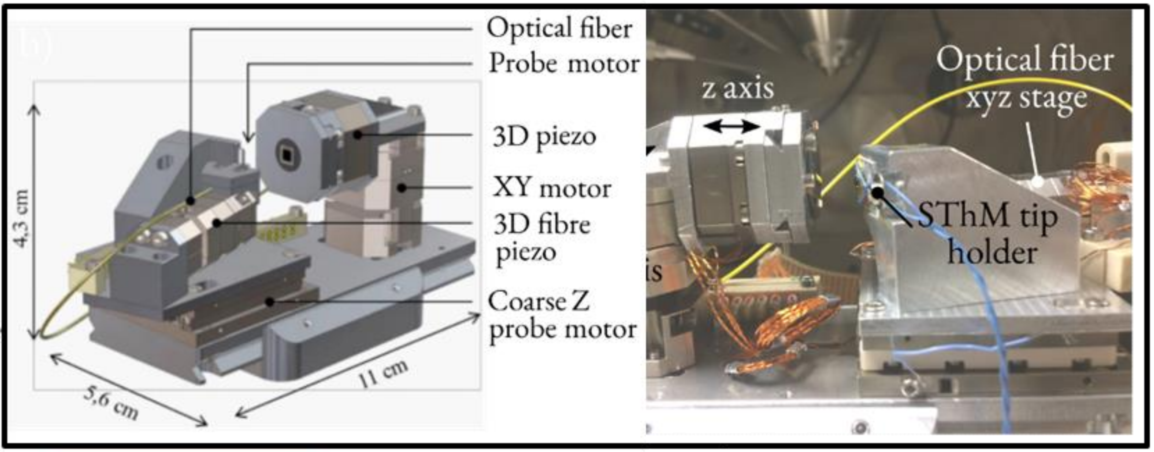
STHM Mechanical part

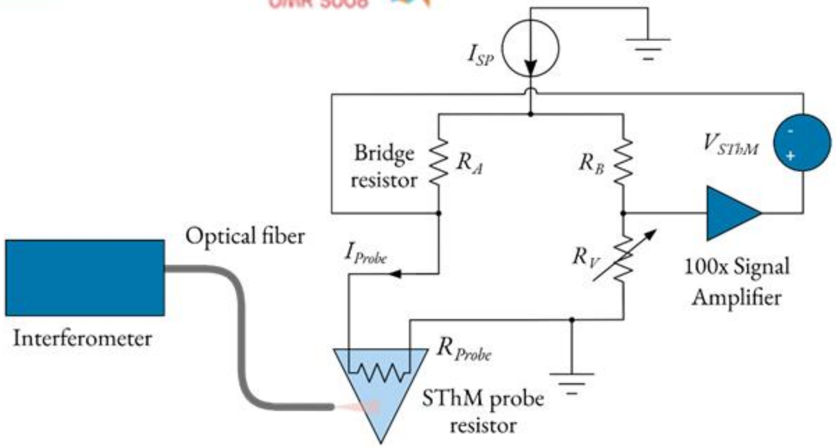
Optical detector module

Thermal control unit

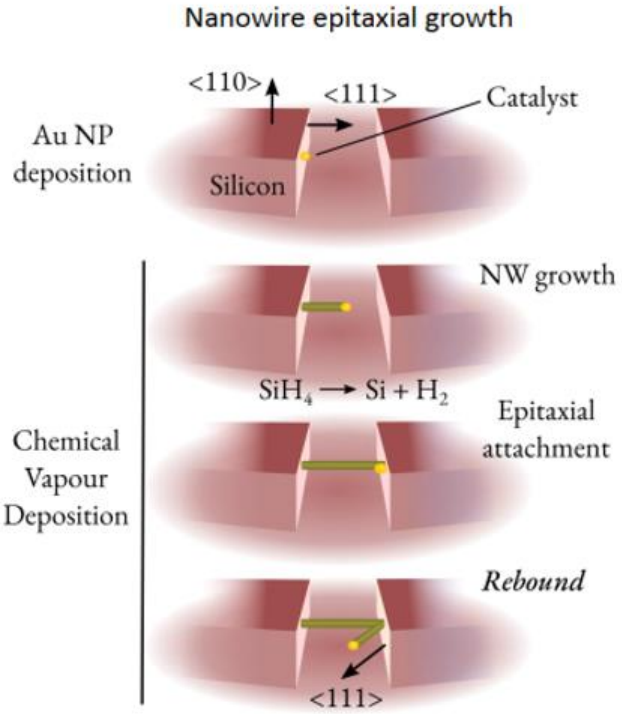
Piezo stage positioning controller

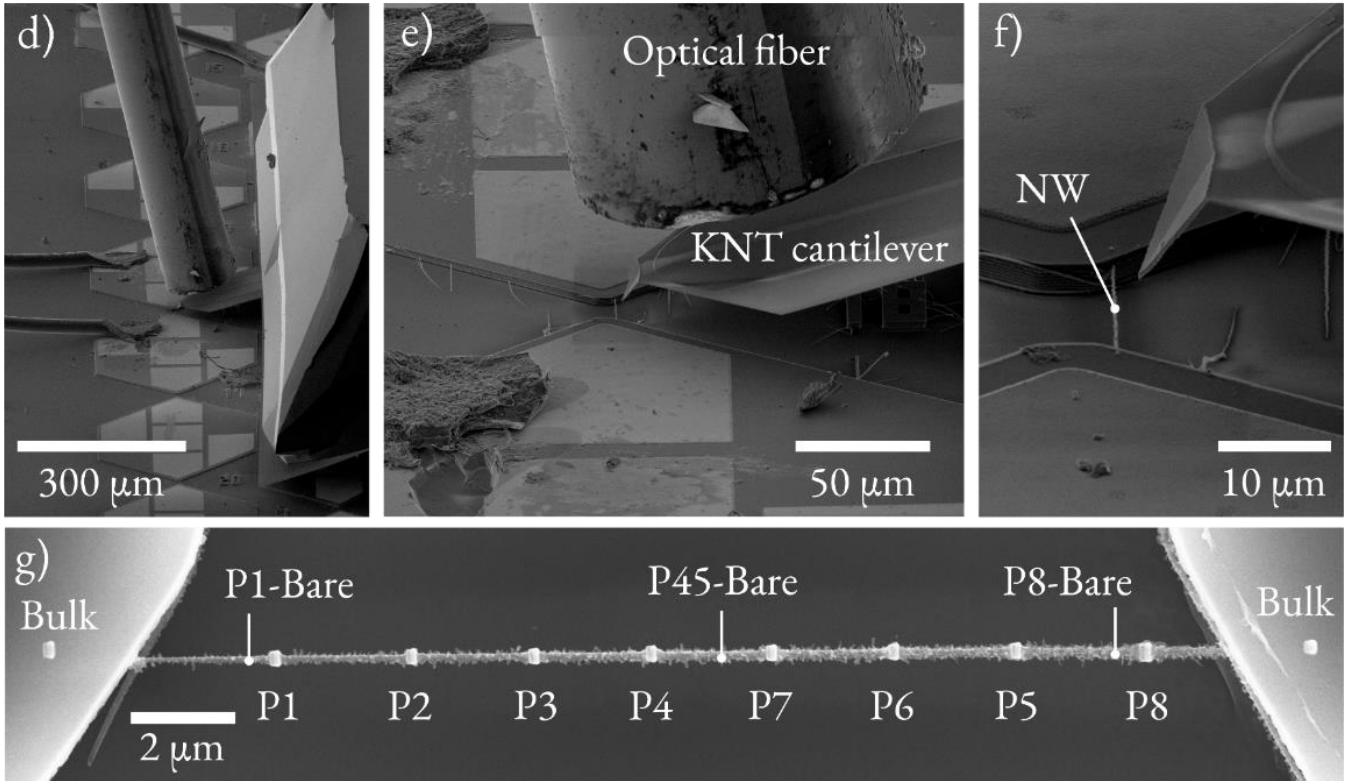
SPM controller



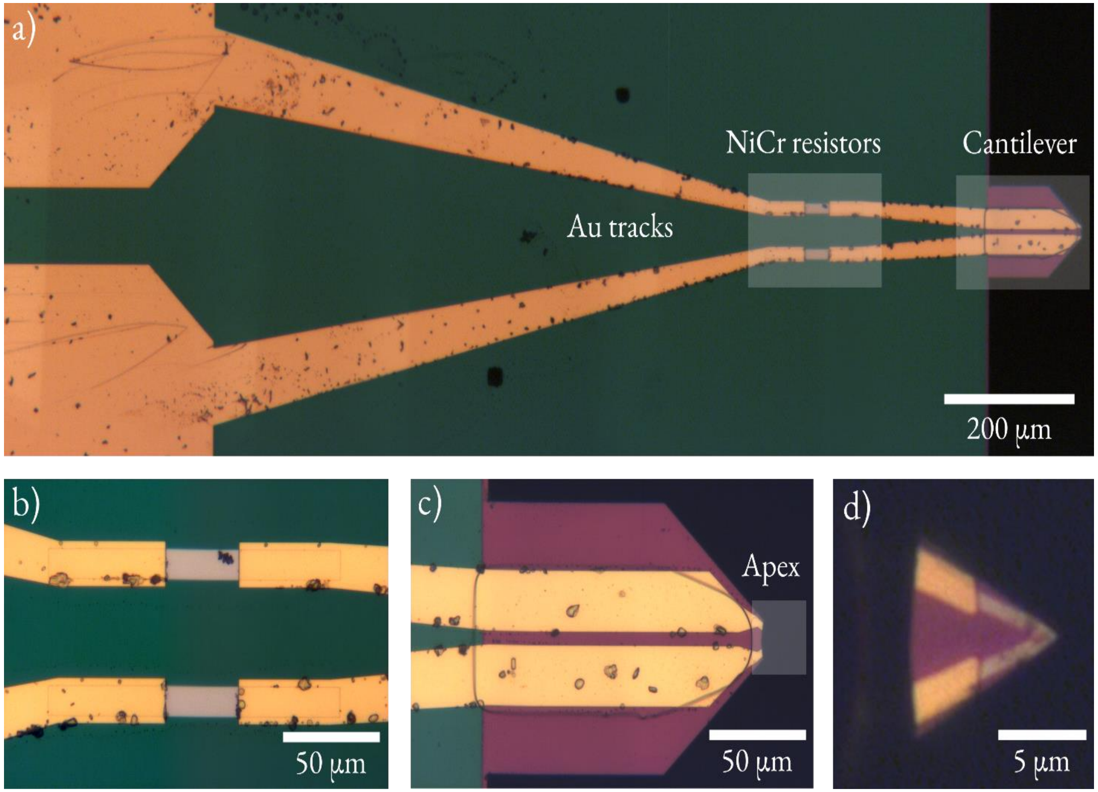


Measurement configuration





KNT SThM probe used

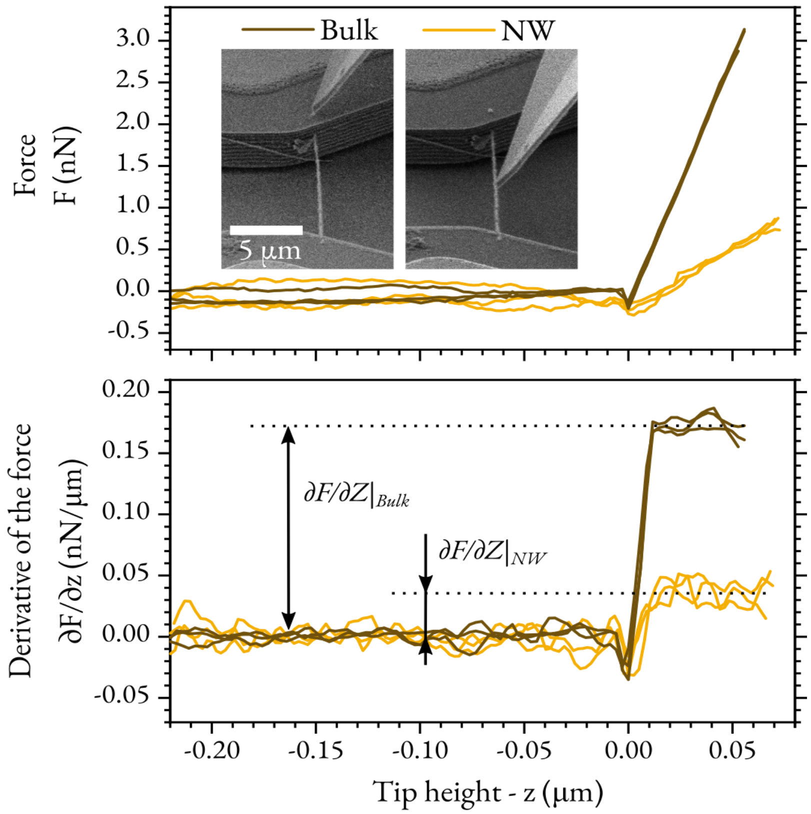


- Optical and SEM observations for **identification of probe components' geometry and dimensions**

- Electrical measurements **to estimate electrical resistance of metallic components**

Nanowire elastic constant

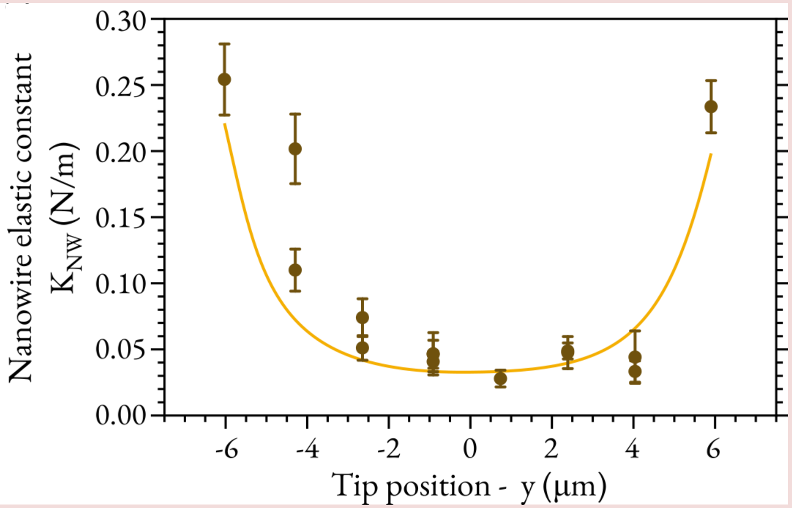
Force and force first derivative
vs.
tip height - z



Step fits over these curves used to calculate the K_{eq} at each point along NW

$$K_{eq} = \frac{1}{K_{NW}^{-1} + K_{probe}^{-1}} \quad \text{with} \quad K_{NW}(y) = \frac{3EIL^3}{\left(L/2+y\right)^3 \left(L/2-y\right)^3}$$

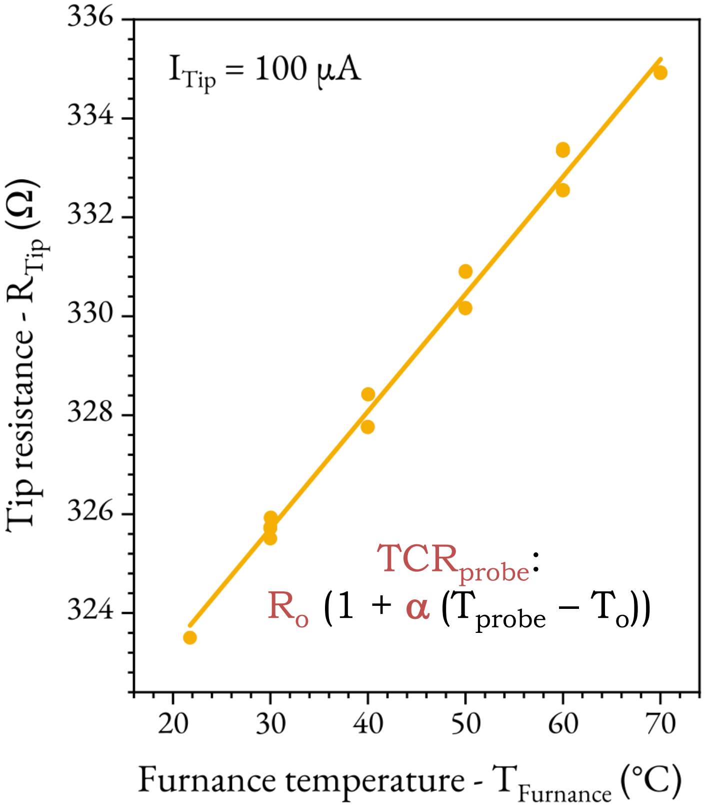
K_{NW} vs. tip position along the nanowire



→ *I* NW effective moment of inertia: ≅ 6.1 ±1.0×10^{-30} m^4

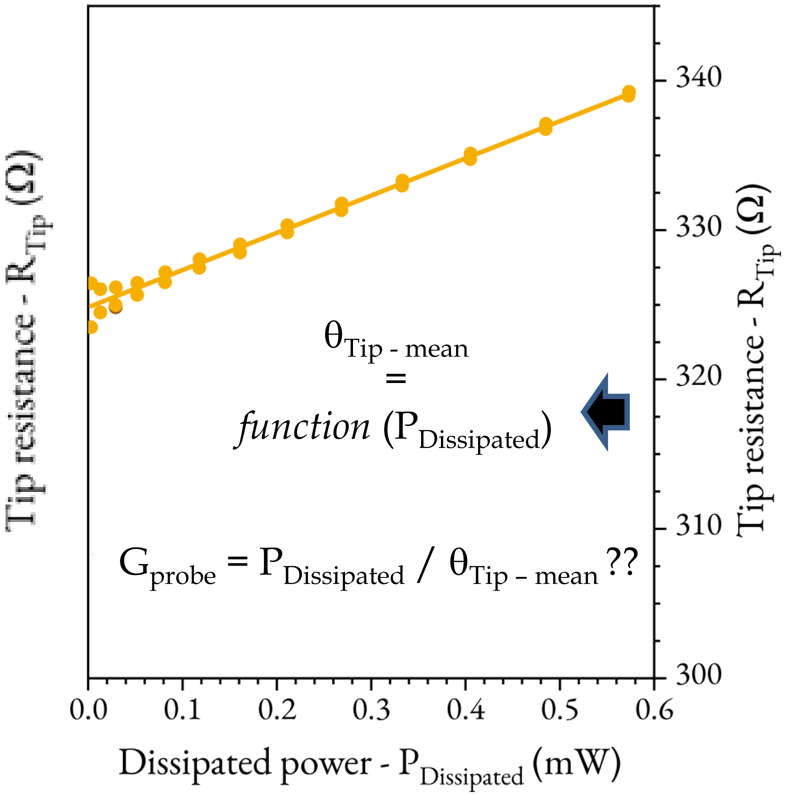
Probe thermal calibration in 3 steps

Step 1:
Temperature calibration in an oven



TCR_{probe}:
$R_o\,(1 + \alpha\,(T_{probe} - T_o))$

Step 2:
Electrical calibration

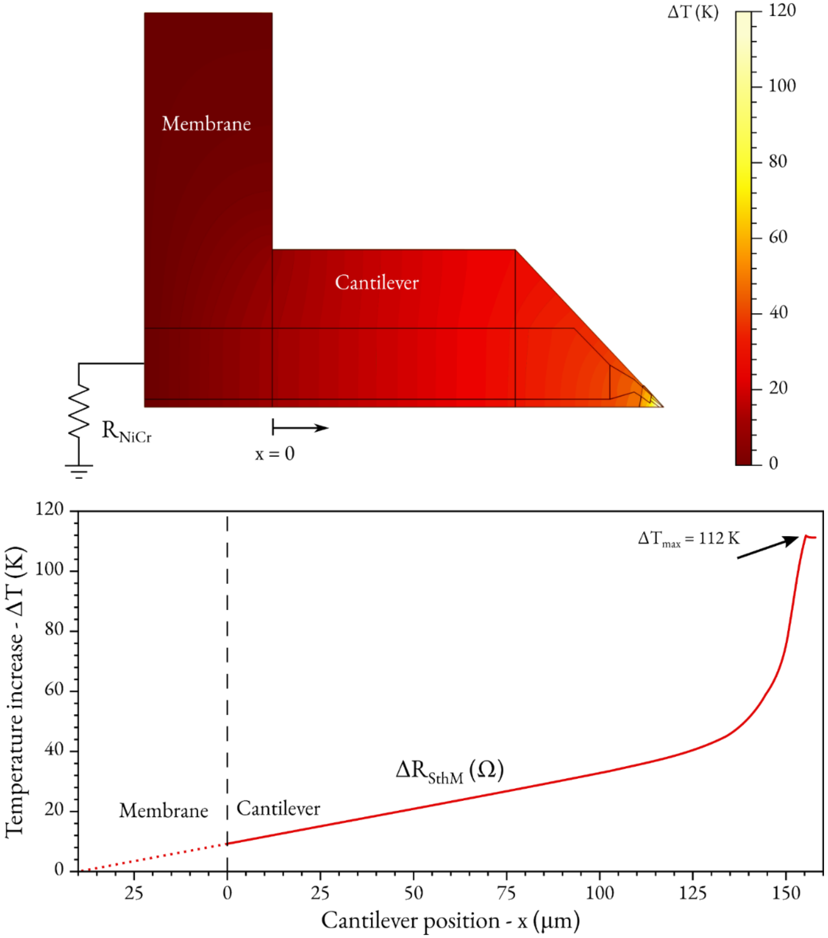


$\theta_{Tip\text{ - }mean}$
=
function ($P_{Dissipated}$)

$G_{probe} = P_{Dissipated} / \theta_{Tip\text{ – }mean}$??

Probe thermal calibration in 3 steps

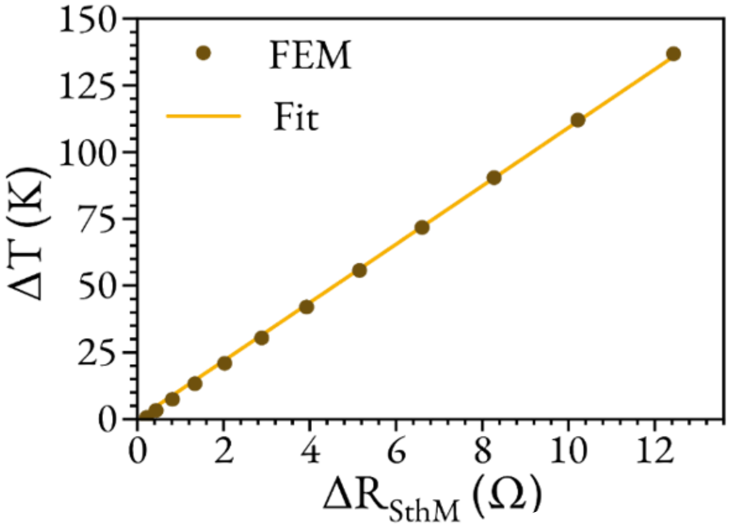
FEM solution of the temperature profile of the calibrated KNT probe in operation



Step 3:

Fitting experimental data with modeling

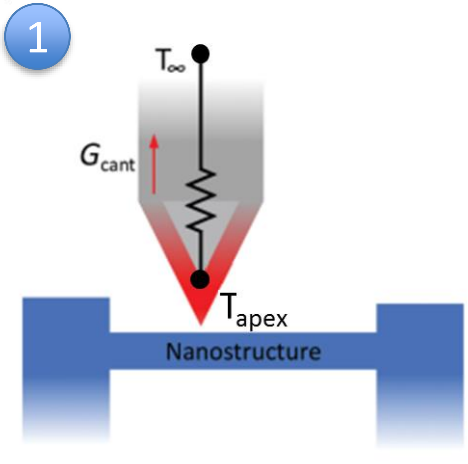
Temperature increase at tip apex as a function of overall tip electrical resistance variation ΔR_{SThM}

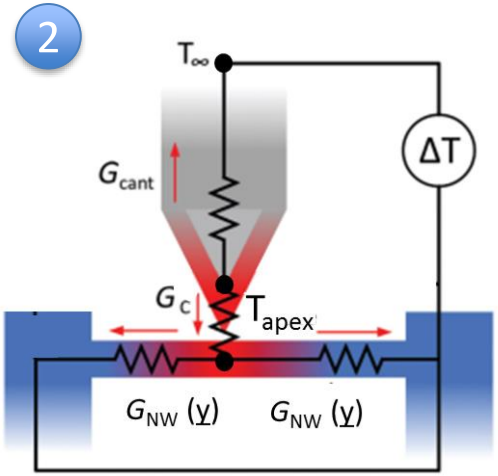


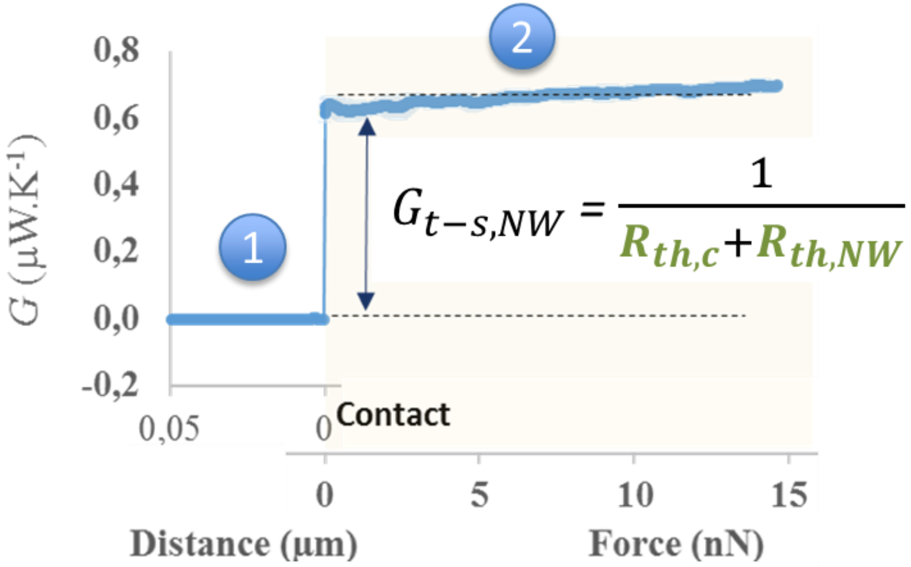
Thermal conductance measured

In each point along the wire

- **two steps with**:







- **NW**: 1D nanostructure:

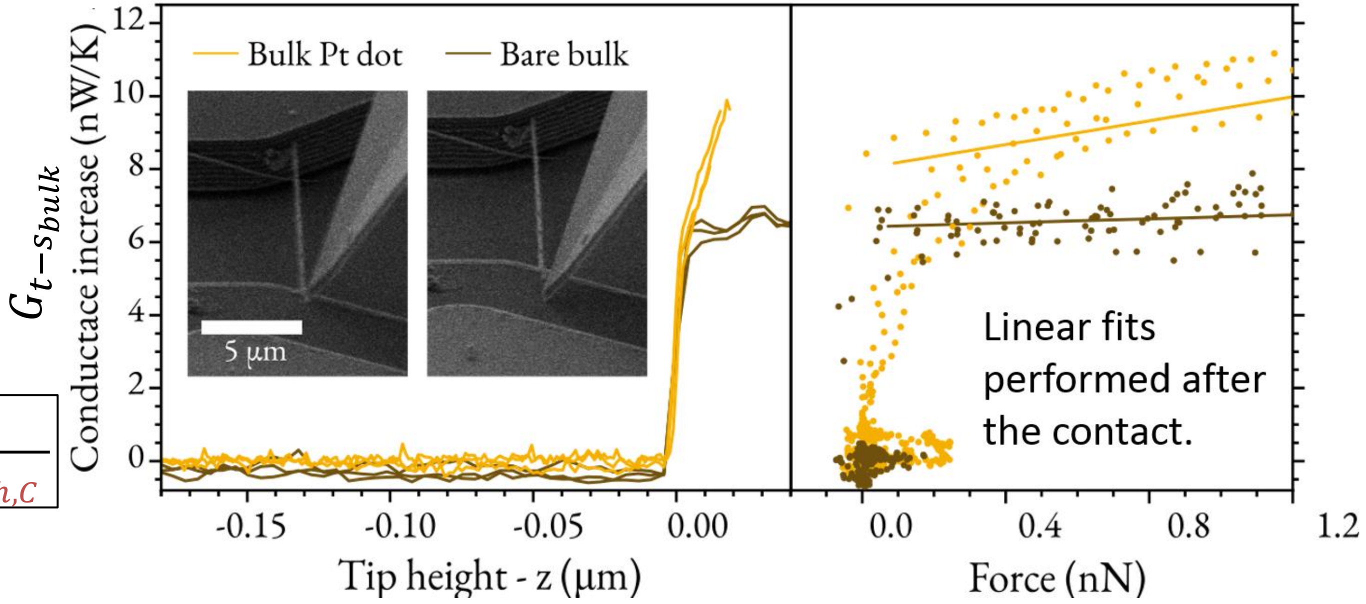
$$R_{th,NW\,(y)} = \frac{L}{A\kappa}\left[\frac{1}{4} - \left(\frac{y}{L}\right)^2\right]$$

Contact thermal resistance $R_{th,c}$

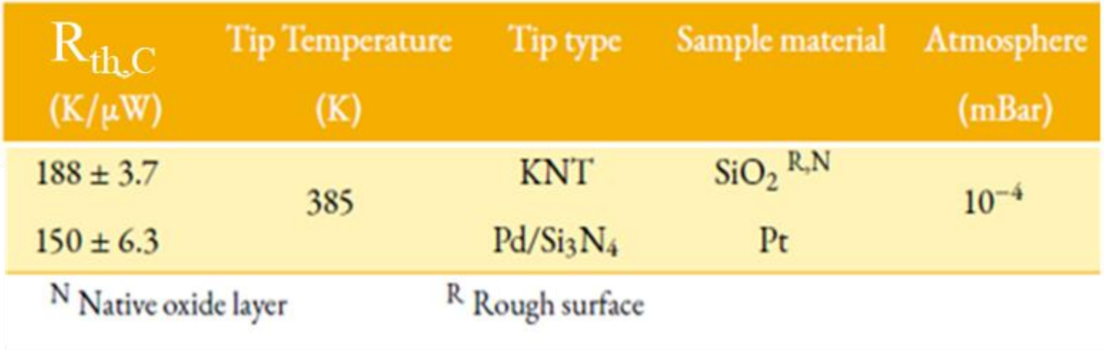
From approaches on

- ○ **bare silicon bulk**
- ○ **Pt nanodot on bulk silicon**

$$G_{t-s_{bulk}} = \frac{4k_{bulk}b}{1+4k_{bulk}b\,R_{th,C}}$$



Linear fits performed after the contact.

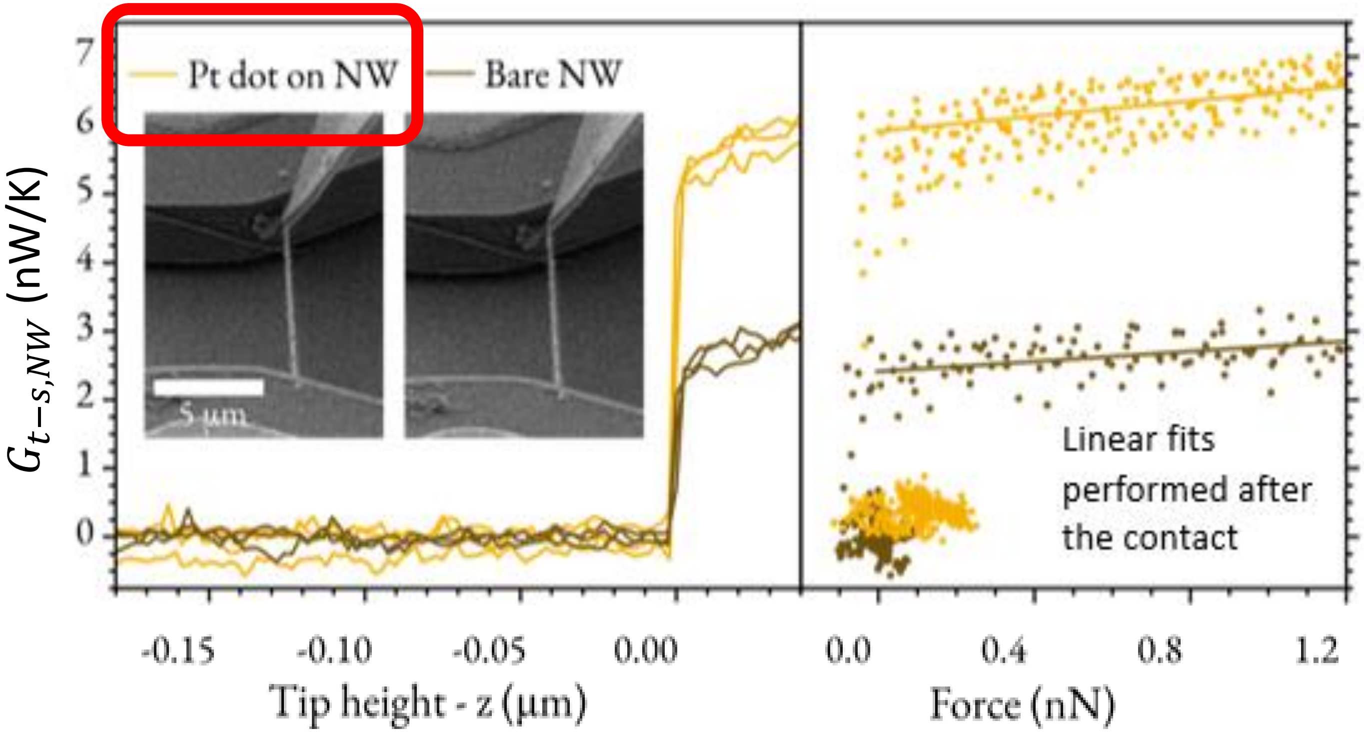


$R_{th,C}$ (K/µW)	Tip Temperature (K)	Tip type	Sample material	Atmosphere (mBar)
188 ± 3.7	385	KNT	SiO_2 ^{R,N}	10^{-4}
150 ± 6.3		Pd/Si_3N_4	Pt	

^{N} Native oxide layer ^{R} Rough surface

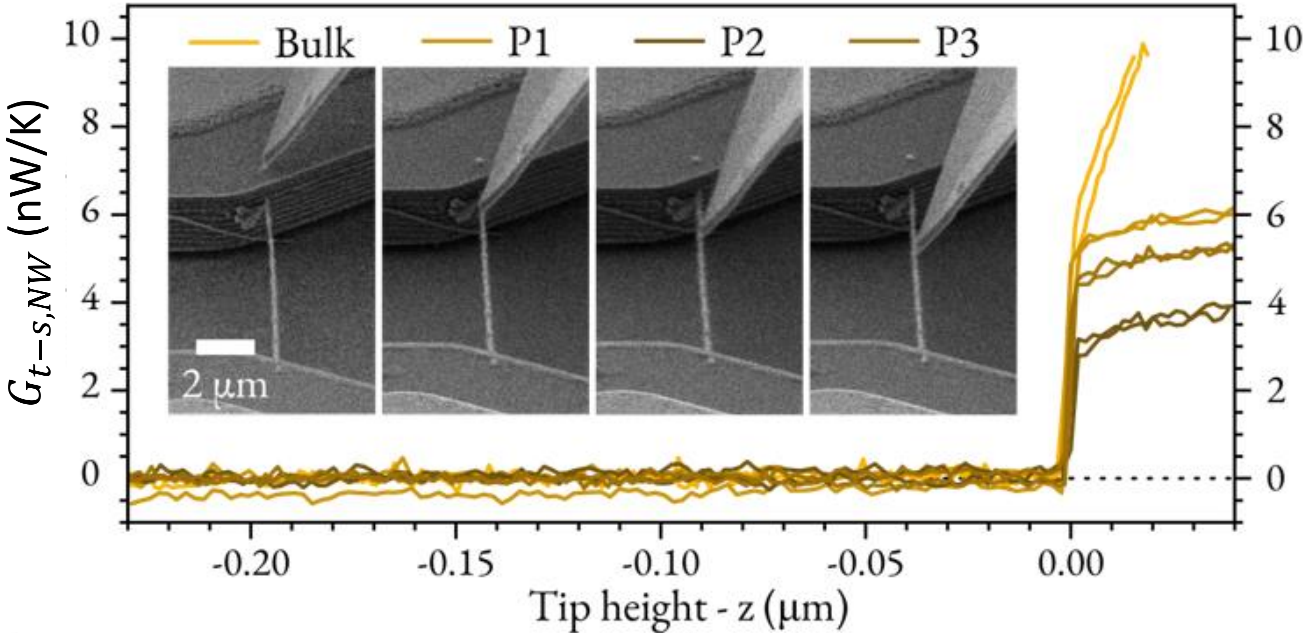
Thermal conductance $G_{t\text{-}s,NW}$ in a given y

- **Approaches over a bare section of the NW compared to those performed over a Pt nanodot deposited over the NW**

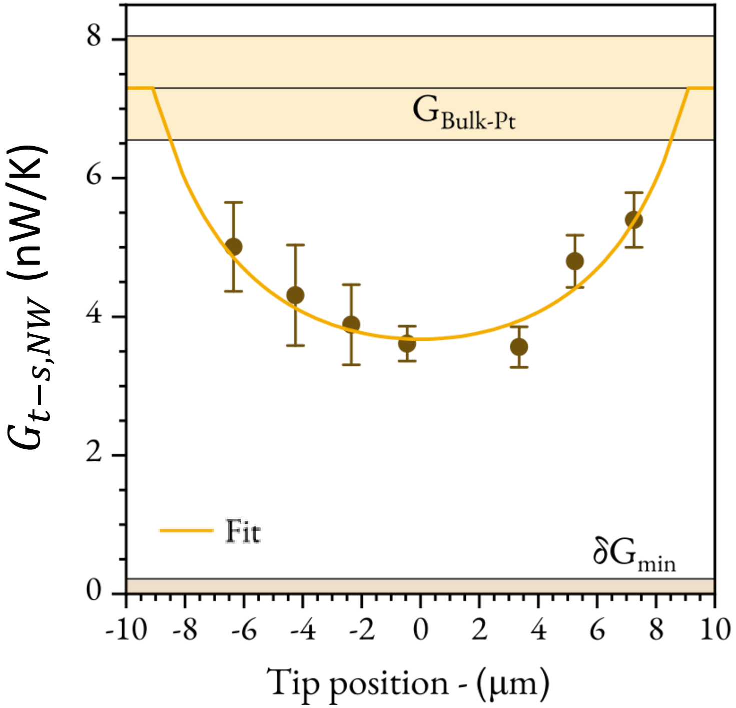


Thermal conductance $G_{t\text{-}s,NW}$ along NW

- **Conductance increase vs. tip height *z* for approaches over different deposited Pt nanodots along NW**



NW equivalent thermal conductivity k



$$G_{t-s,NW} = \frac{1}{R_{th,c} + R_{th,NW}}$$

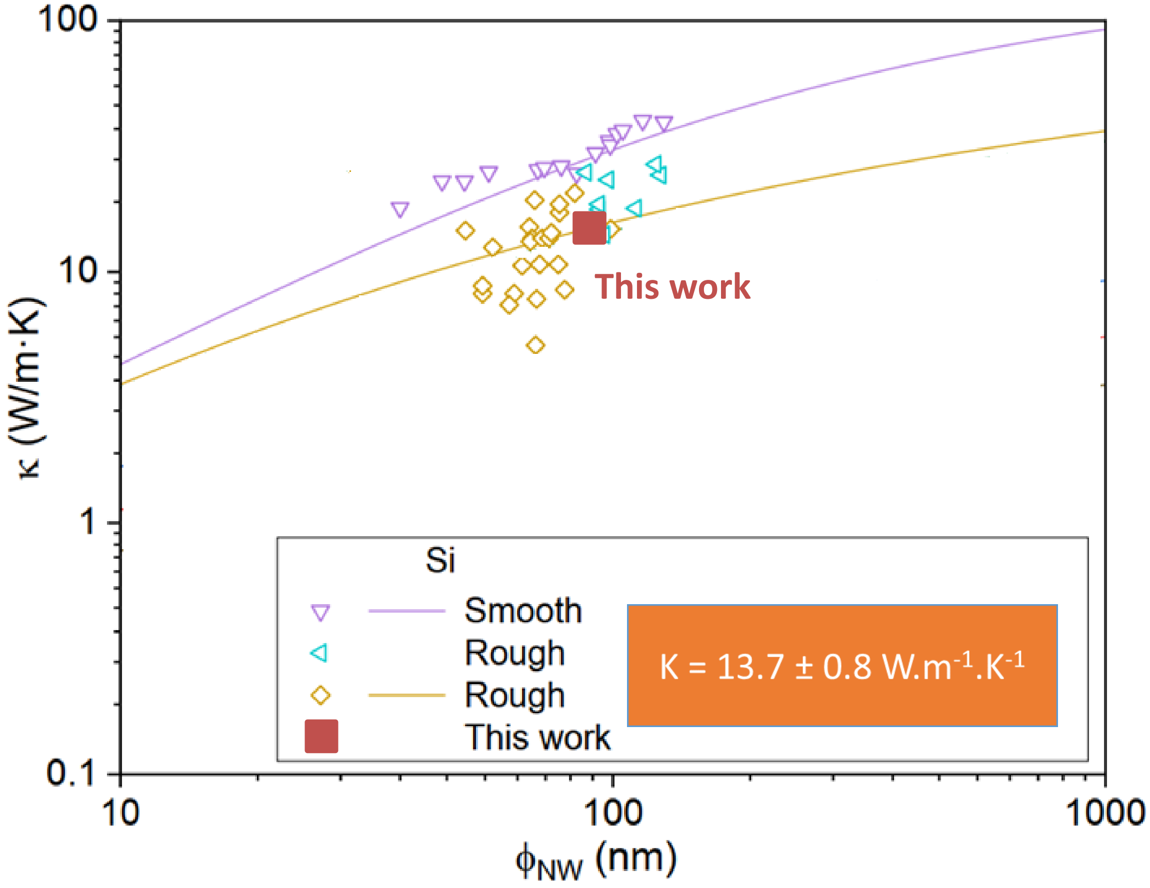
$$G_{t-s,NW}(y) = \frac{1}{R_{th,c} + \frac{L}{A\kappa}\left[\frac{1}{4} - \left(\frac{y}{L}\right)^2\right]}$$

Fit

K = 13.7 ± 0.8 $W.m^{-1}.K^{-1}$

NW equivalent thermal conductivity k

Good agreement with previous works



K = 13.7 ± 0.8 W.m^{-1}.K^{-1}

Theoretical values from

1. Wang et al. APL 2020
2. Ohishi et al. Jpn. J. Appl. Phys 2015
3. Yang et al. Chinese Phys. B. 2020

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Small (2023): 2305831.

Conclusion & challenges

- ☐ **Instruments**
  - Highly sensitive and low-noise electronics required
  - Highly controlled environment

Highly sensitive probe required

- ☐ **SThM allows thermal conductance measurements**
  - Relevance of the determination of the thermal properties depending on model used

Thermal contact radius and G_c are crutial parameters

Physics of G_c still not well-known

- ☐ **Conventional SThM calibration (using bulk materials) mainly adapted to the analysis of low thermal conductivity materials**
  - polymeric materials
  - optimized TE materials
  - low thermal effective thermal conductivity materials (thin films on substrate)

- ☐ **SThM allows measuring low thermal conductance objects (nanowires, suspended membranes…)** whatever their thermal conductivity

Whatever the sample studied,
complementary analyses (roughness, microsctructure, geometry …) needed

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Thank you for your attention!

Thermal methods to acquire new knowledge

Optical methods

Spatial resolution

~ Lateral: 30 µm (PTR), 1 µm (FD-TR)

~ In depth: 50 nm (PTR, Up to 50 MHz), 20 nm (FDTR: 1 kHZ-300 MHz; **TDTR: 100 MHz-1 THz**)

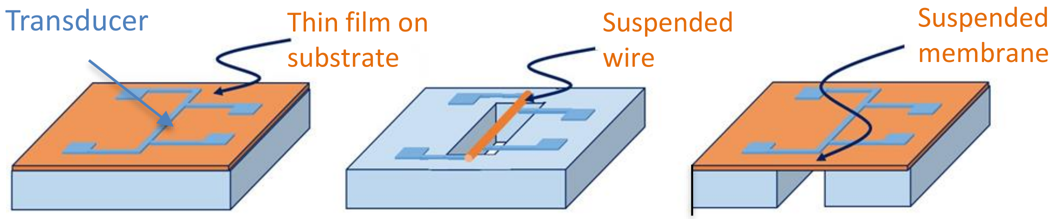
Particularly well adapted for ultrathin film and interfacial resistance measurement

Electrothermal methods

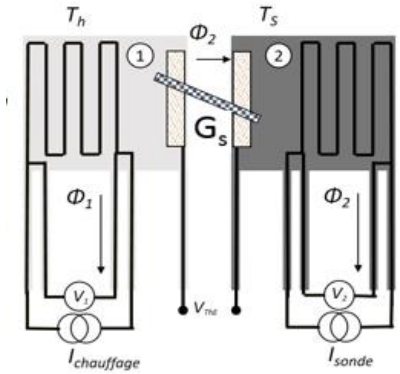
2ω and 3ω methods

Cryogenic methods

Thermal bridge method



Paterson (J.) Thesis Université de Grenoble, France (2020)



Nature Commun., 9, 4287 (2018).

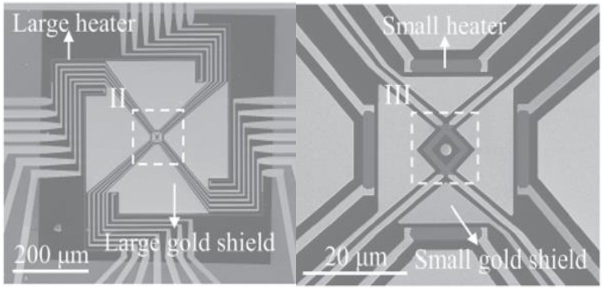
SPM - techniques

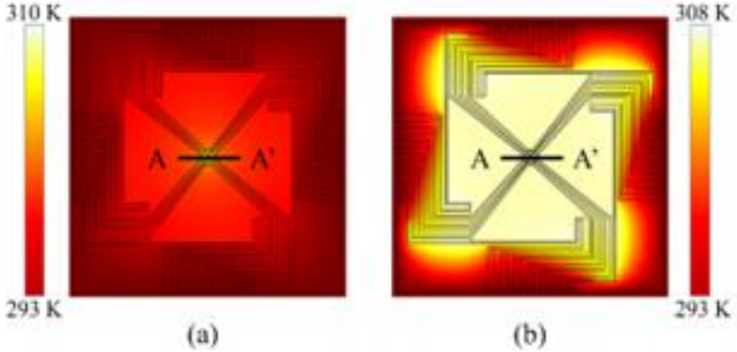
Scanning thermal microscopy - SThM

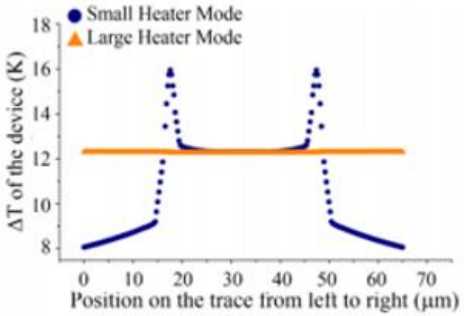
Calibration, passive mode

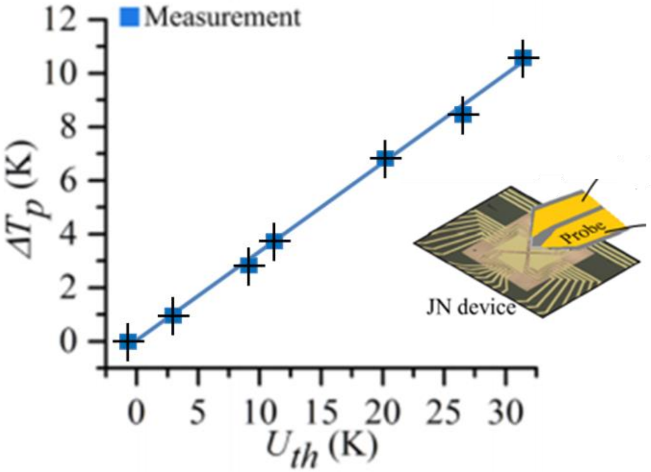
Surface temperature measurement

Quantiheat Appl. Note 2017







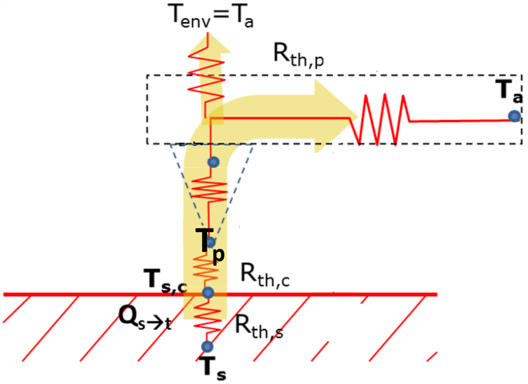


However,

- **error not easily measurable**

$$\delta T_p = T_s - T_p = \frac{(T_p - T_a)(R_{th,s} + R_c)}{R_{th,p}}$$

$R_{th,s}$ sample thermal resistance
$R_{th,c}$ contact thermal resistance
$R_{th,p}$ cantilever – environment thermal resistance

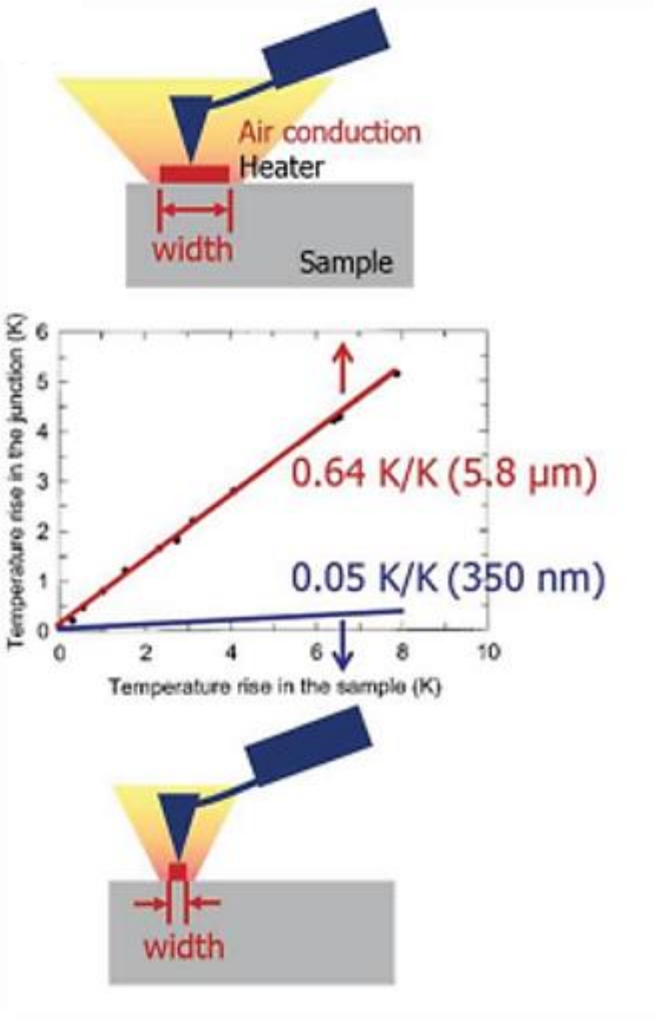


- **not applicable to nanodevices** because sample heating by the sample completly differing mainly due to the air conduction in this case.

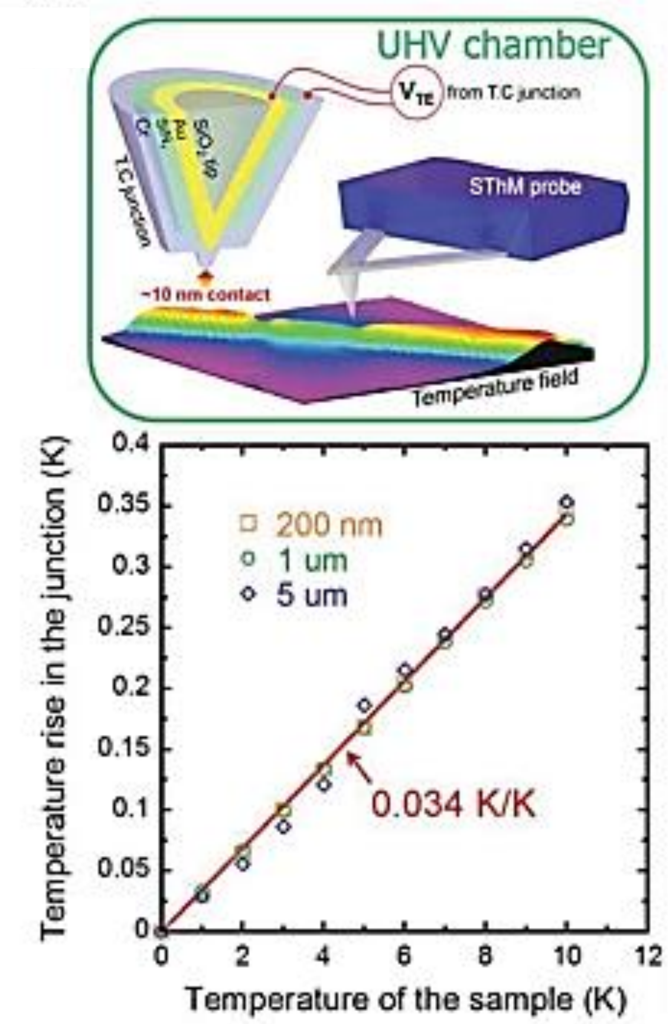
Requirement: reference active **nanodevice** with ultra localized heating source

Calibration, passive mode

Air conditions



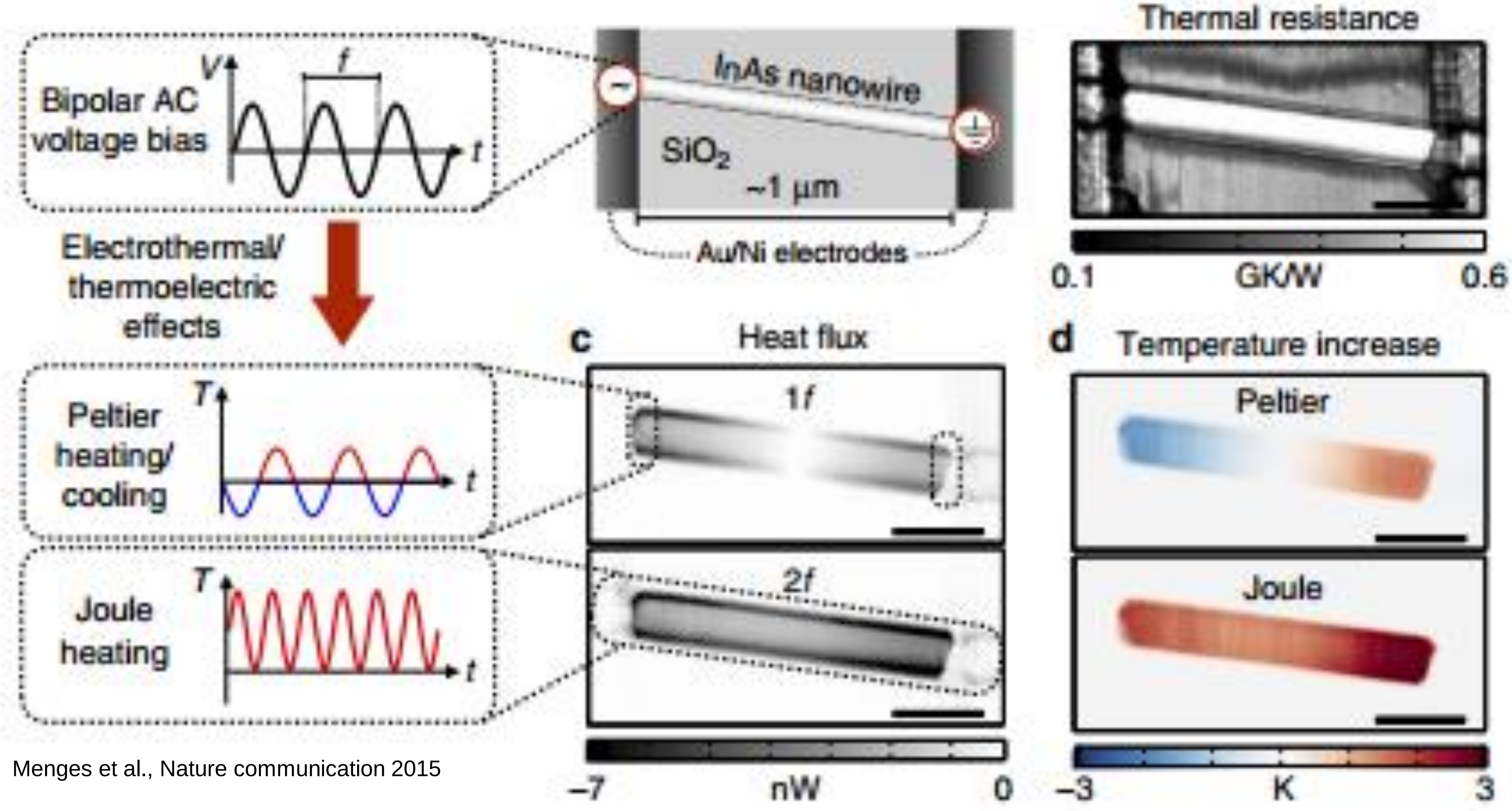
UHV conditions



ACS Nano 2012, 6, 4248.

Active mode – temperature measurement

Measurement of the local Joule and Peltier effects of a self-heated nanowire



Menges et al., Nature communication 2015

Temperature measurement uncertainty estimated at 20%