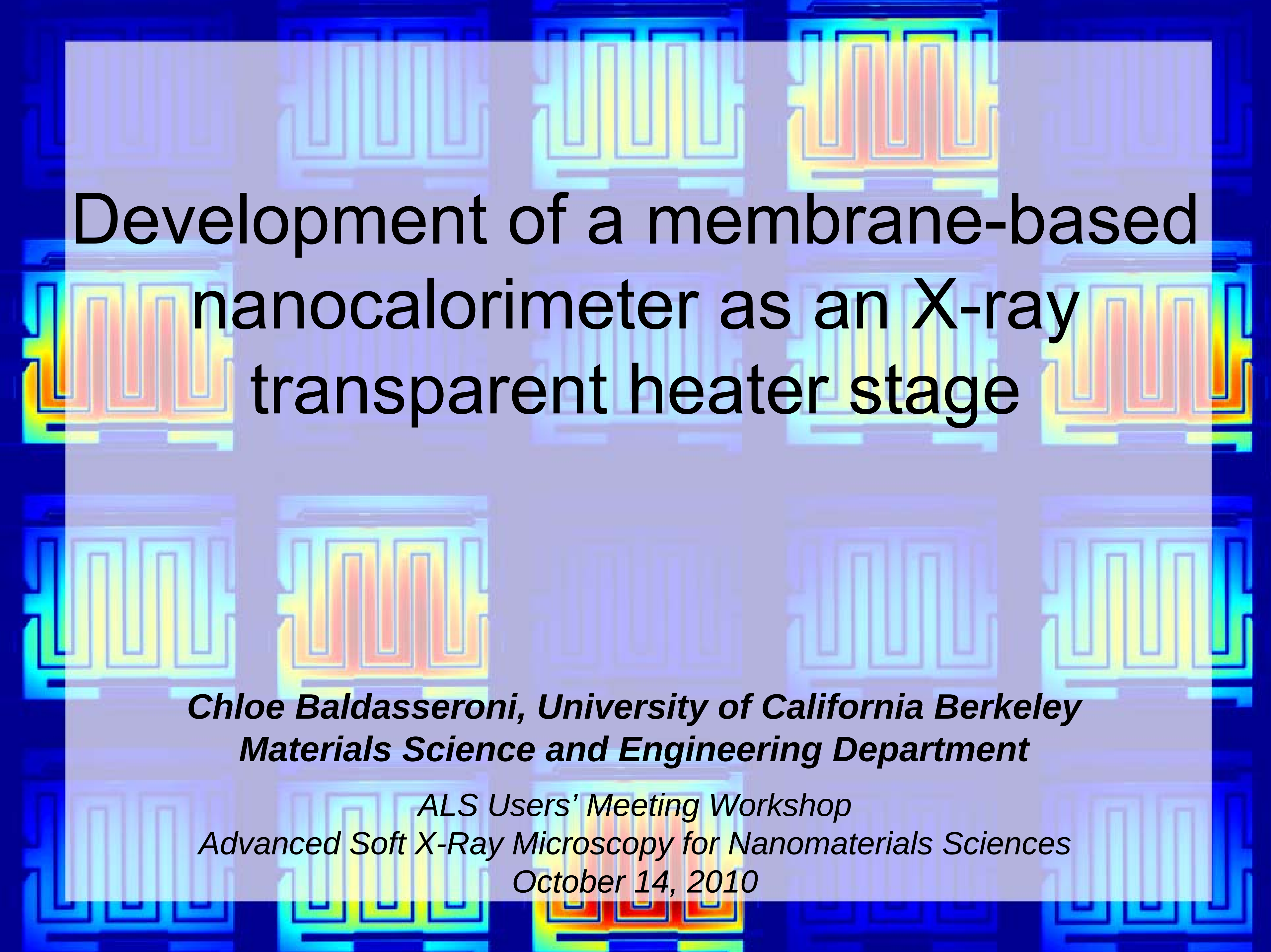

# Development of a membrane-based nanocalorimeter as an X-ray transparent heater stage

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**Materials Science and Engineering Department**

*ALS Users' Meeting Workshop*
*Advanced Soft X-Ray Microscopy for Nanomaterials Sciences*
*October 14, 2010*

# Outline

- Motivation
- Microfabricated heaters
- Our current design: nanocalorimeter
- Understanding temperature distribution
  - Experimental thermal characterization
  - Steady-state simulation
- New heater-stage design
- Outlook

# Temperature dependent X-ray microscopy
# Motivation

- Probing of atomic and molecular properties during phase changes of materials
- Study of temperature activated phenomena
- Quench dynamics and metastable structures

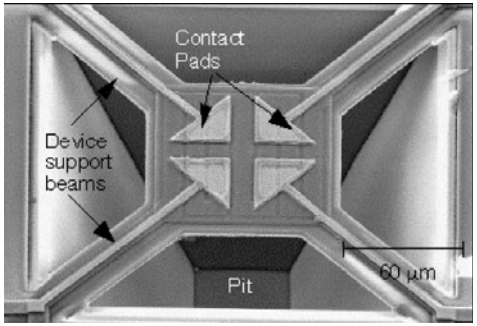
- Heating constraints in microscopes:
  - space limited
  - geometry restrictions
  - heat confinement

- Advantages of microfabrication:
  - Small size: “heater on a chip“
  - Localized heating
  - Fast heating and cooling
  - Small power

# Microfabricated heater devices

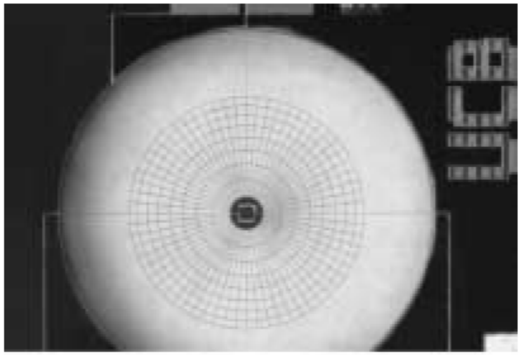
- MEMS sensor

  Suspended polysilicon



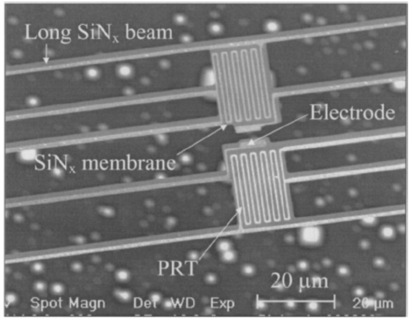
- Bolometer

  Silicon nitride spiderweb



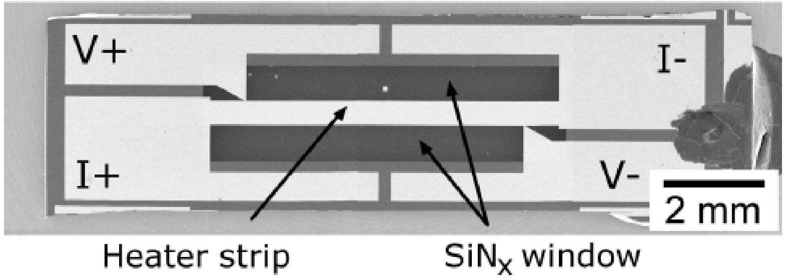
- Thermal conductivity device

  Pt heater with suspended Si-N legs



- Calorimeter

  Metal strip on Si-N window

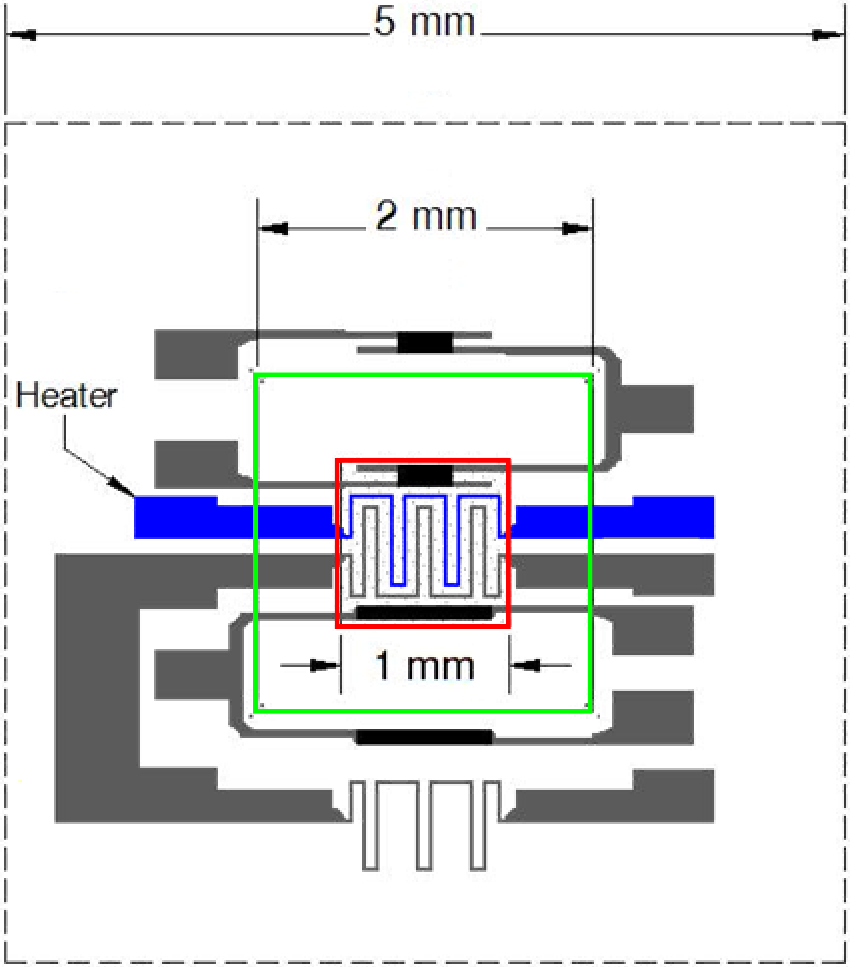


*S. Semancik et al. / Sensors and Actuators B 77 (2001) 579-591*
*L. Gildemeister et al. / Appl. Phys. Lett. Vol. 74 No. 6 (1999)*
*Sultan et al. J. Appl. Phys. 105, 043501 (2009)*
*M. Zhang et al. / J. Mater. Res., Vol. 20, No. 7, Jul 2005 1802*

# Nanocalorimeter

- Free standing aSi-N membrane on silicon frame with patterned Pt heater and thermometers
- Sample area weak thermal link to silicon frame

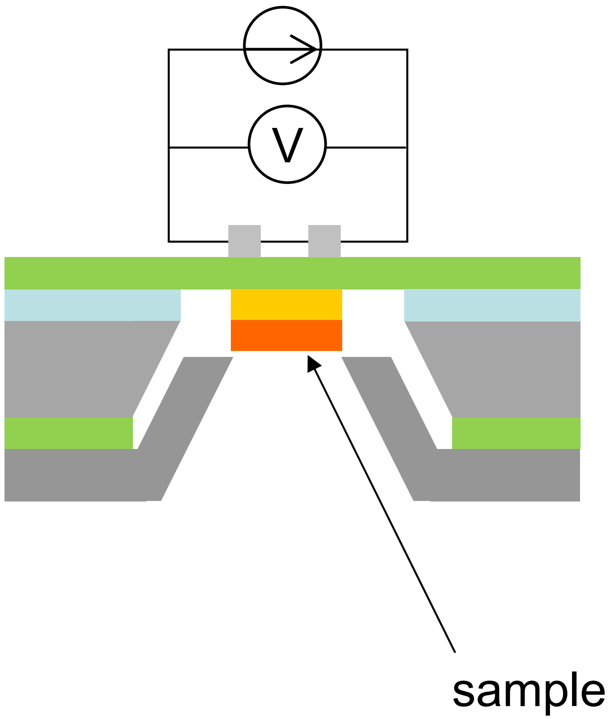
Thickness 30 to 50 nm



*D. W. Denlinger et al., Rev. Sci. Instrum. 65, 946 (1994)*
*D. R. Queen and F. Hellman, Rev. Sci. Instrum. 80, 063901 (2009)*

# Fabrication

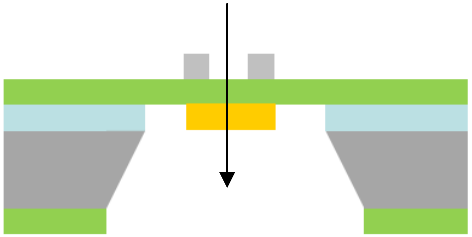
- Process Flow
  - Thermal Oxidation
  - Low stress Silicon Nitride
  - Platinum
  - Si Etch

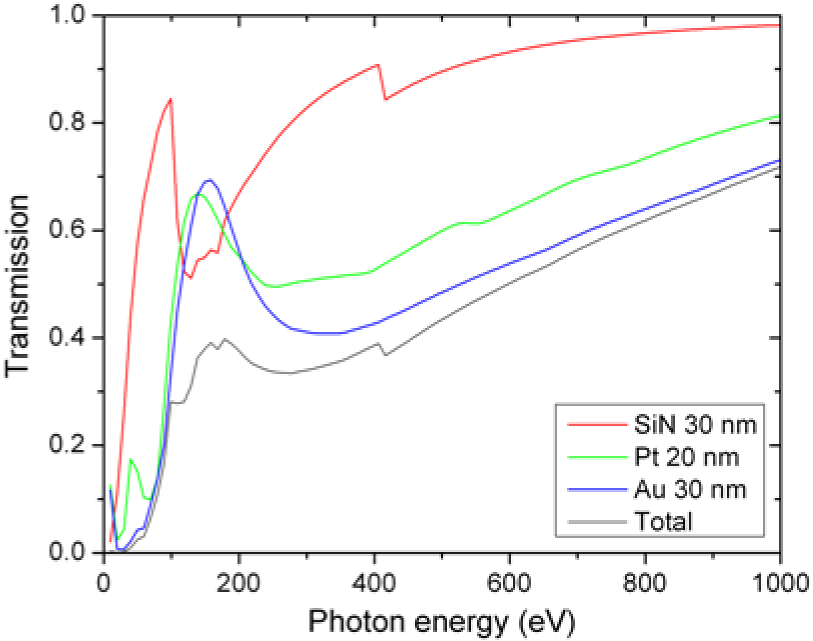


- Platinum:
  - Heater: Joule heating
  - Thermometer: R vs T calibration
- Temperature control/measurement by 4 probe configuration with current source and voltmeter in parallel

# X-ray transparent heater stage criteria

- X-ray transparency



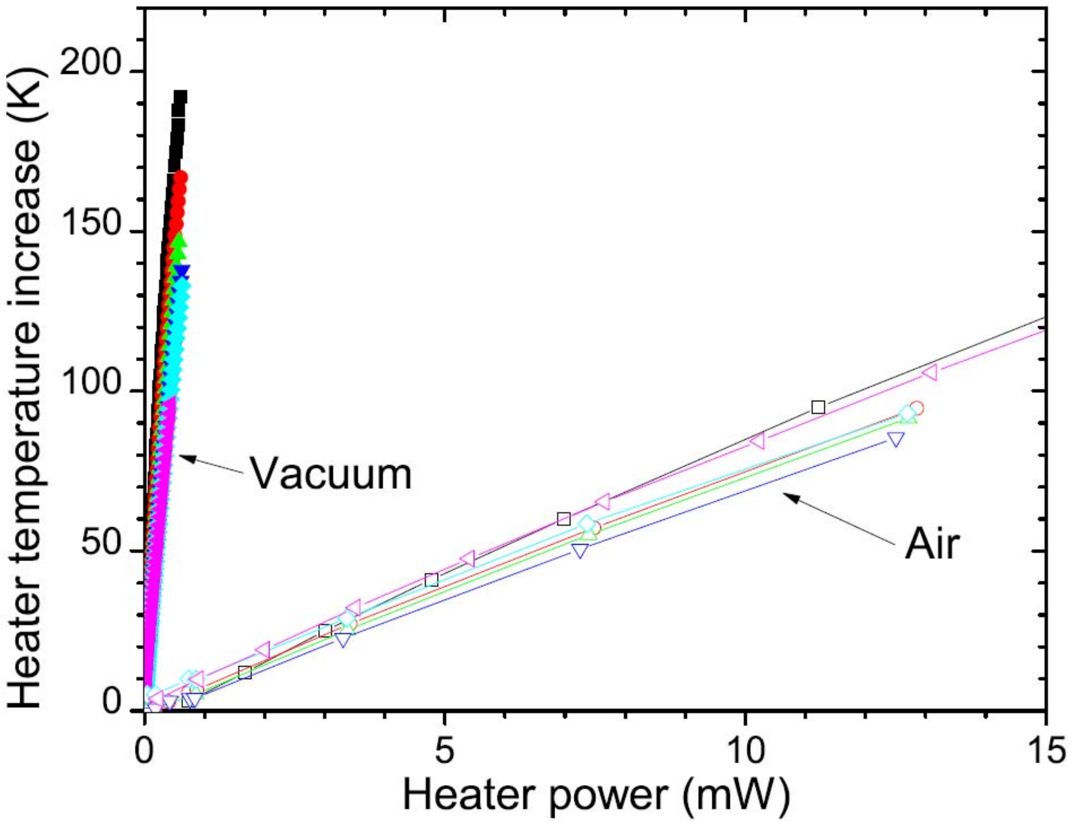


- Chemical and mechanical stability over 20 - 650°C temperature range

- Isothermal temperature distribution of sample area or control of temperature distribution

***Need to understand temperature distribution for large increases of temperature and non-vacuum***

# Power dissipation in vacuum and in air



- Between vacuum and air: heat transfer through gas
- Average heater temperature:
  no information on local temperature distribution

# Microscale thermography

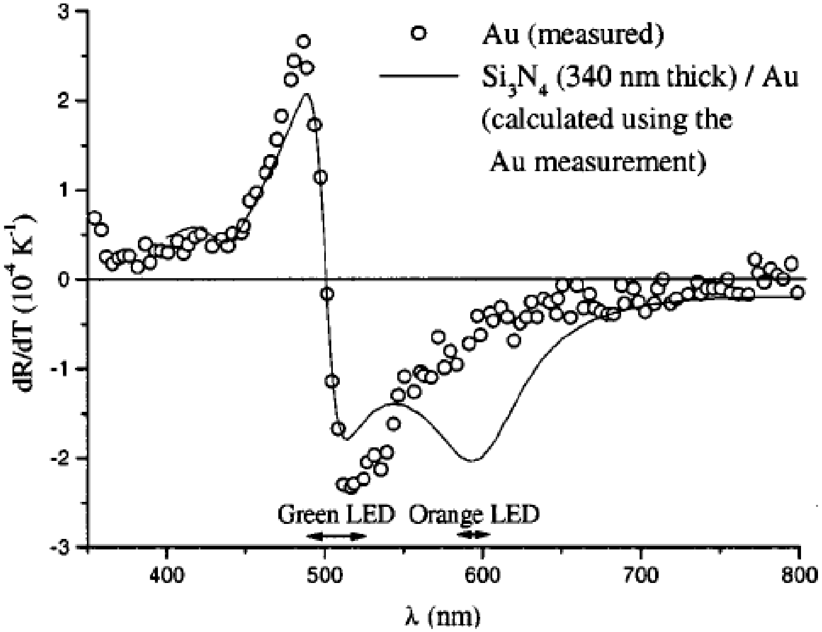
Thermoreflectance microscopy

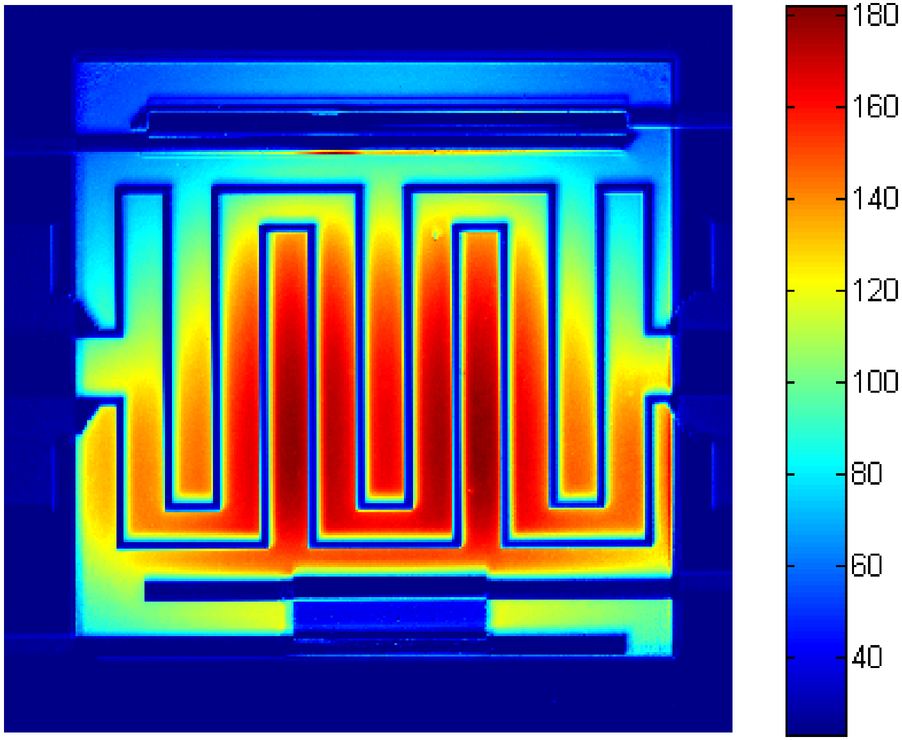
$$\Delta R = \frac{\partial R}{\partial T}\Delta T$$

Collaboration with Kerry Maize from Prof. Shakouri's group at UC Santa Cruz

Calibration: $\frac{\partial R}{\partial T}$ or $C_{th}$

submicron spatial resolution





Experimental thermal map of nanocalorimeter in air

Cth = $-2.4\times10^{-4}$ $K^{-1}$

*G. Tessier, S. Hole, and D. Fournier, Applied Physics Letter 78 16 2001*

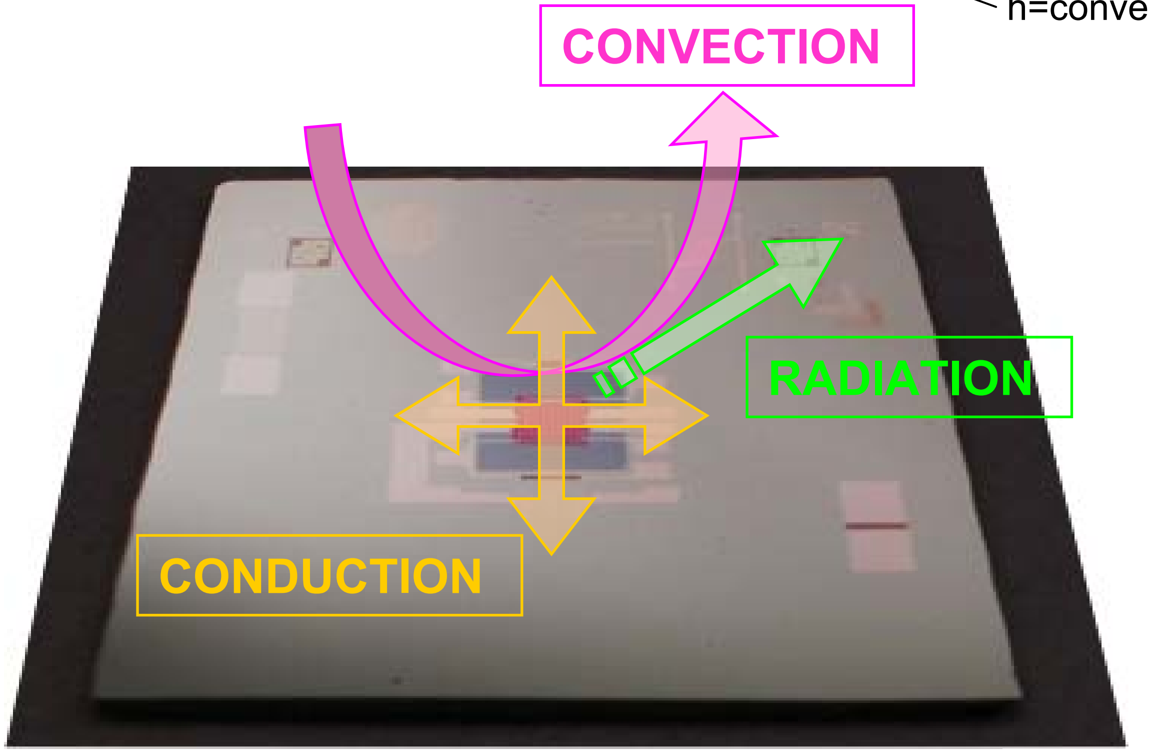
# Different modes of heat transfer

Heat transfer between a surface and a gas moving over the surface

***Free*** or ***natural convection***

$$q = hA_s\left(T_s - T_\infty\right)$$

h=convection coefficient



Stefan-Boltzmann law of blackbody emission

$$q_1 = A_1\sigma\varepsilon_1\left(T_1^{\,4} - T_2^{\,4}\right)$$

Fourier's law of heat conduction

$$q_x = -kA\frac{dT}{dx}$$

Heat rate

Surface area

Thermal conductivity

Temperature gradient

# Nonlinear steady-state heat equation

Form of the heat equation for our model:

$$-\nabla\cdot(k_{2D}\nabla u)+hu+\sigma\varepsilon\left((u+u_0)^4-u_0{}^4\right)=P_{2D}$$

← Power dissipated in heater

conduction ↑ convection ↑ radiation ↑

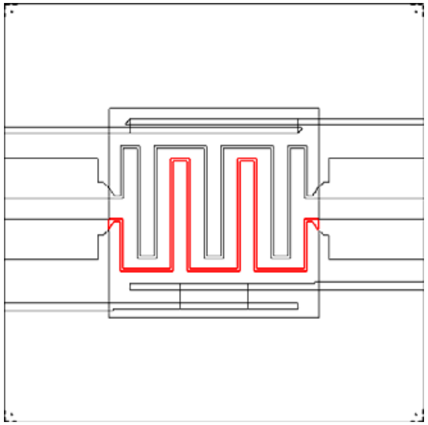
$k_{2D}$ = 2D thermal conductivity in W/K

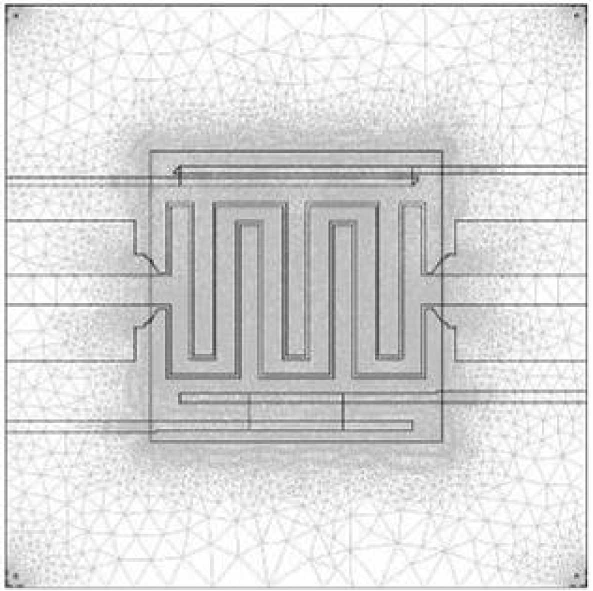
h = convection heat transfer coefficient in W/(K.cm$^2$)

$\varepsilon$ = emissivity, unitless

Use Matlab PDE toolbox (partial differential equation) to solve heat equation using a finite element method:

1. Describe geometry and boundary conditions
2. Build triangular mesh
3. Discretize PDE and solve





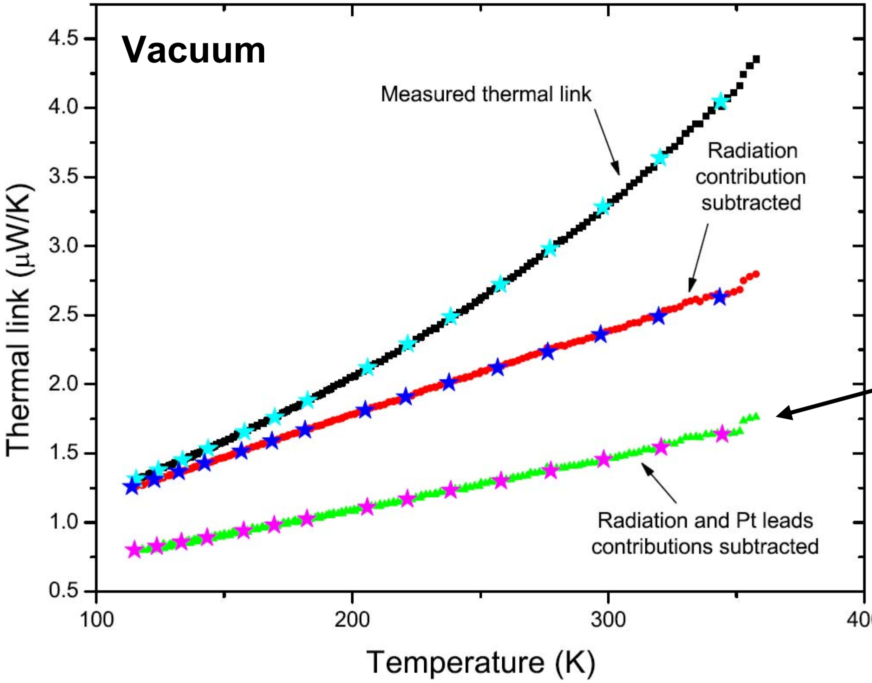
~300 000 triangles

# Model parameters

- Conduction

Thermal
link

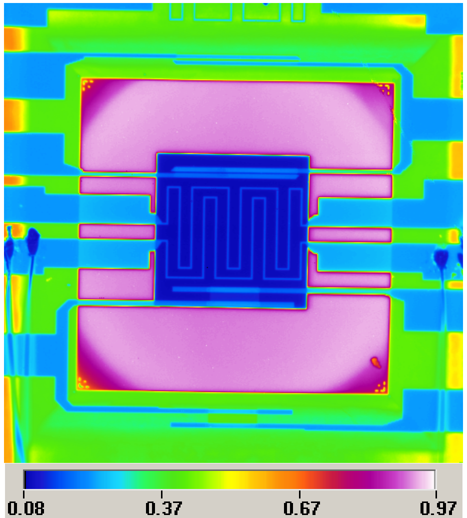
$$K = \frac{P}{\Delta T}$$



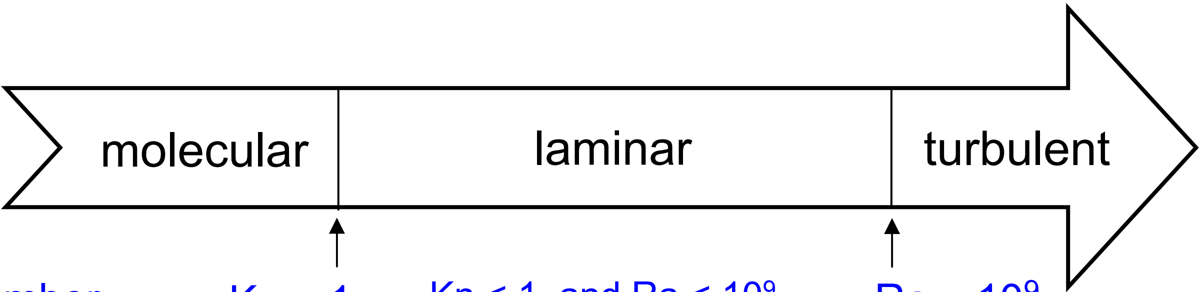
Gives Si-N
conduction
contribution

- Radiation

Infrared thermography:
emissivity of each material



# Model parameters - Convection



Dimensionless number — $Kn \approx 1$ — $Kn < 1$ and $Ra < 10^9$ — $Ra \approx 10^9$

Pressure — $p \approx 140$ mTorr — $p > 760$ Torr

Using characteristic length ~500 µm

- Laminar regime
  - Constant over the entire regime $h = \dfrac{k}{d}$

Thermal conductivity of gas

**$k(N_2) = 2.60e^{-2}$ W/m.K**
**d = 475 µm**
**$h = 0.0109$ W/K.cm$^2$**

- Molecular flow regime
  - Proportional with pressure $h = J_i \gamma' \dfrac{c_v}{N_A}$ $\quad J_i = \dfrac{N_A P}{\sqrt{2\pi MRT}}$

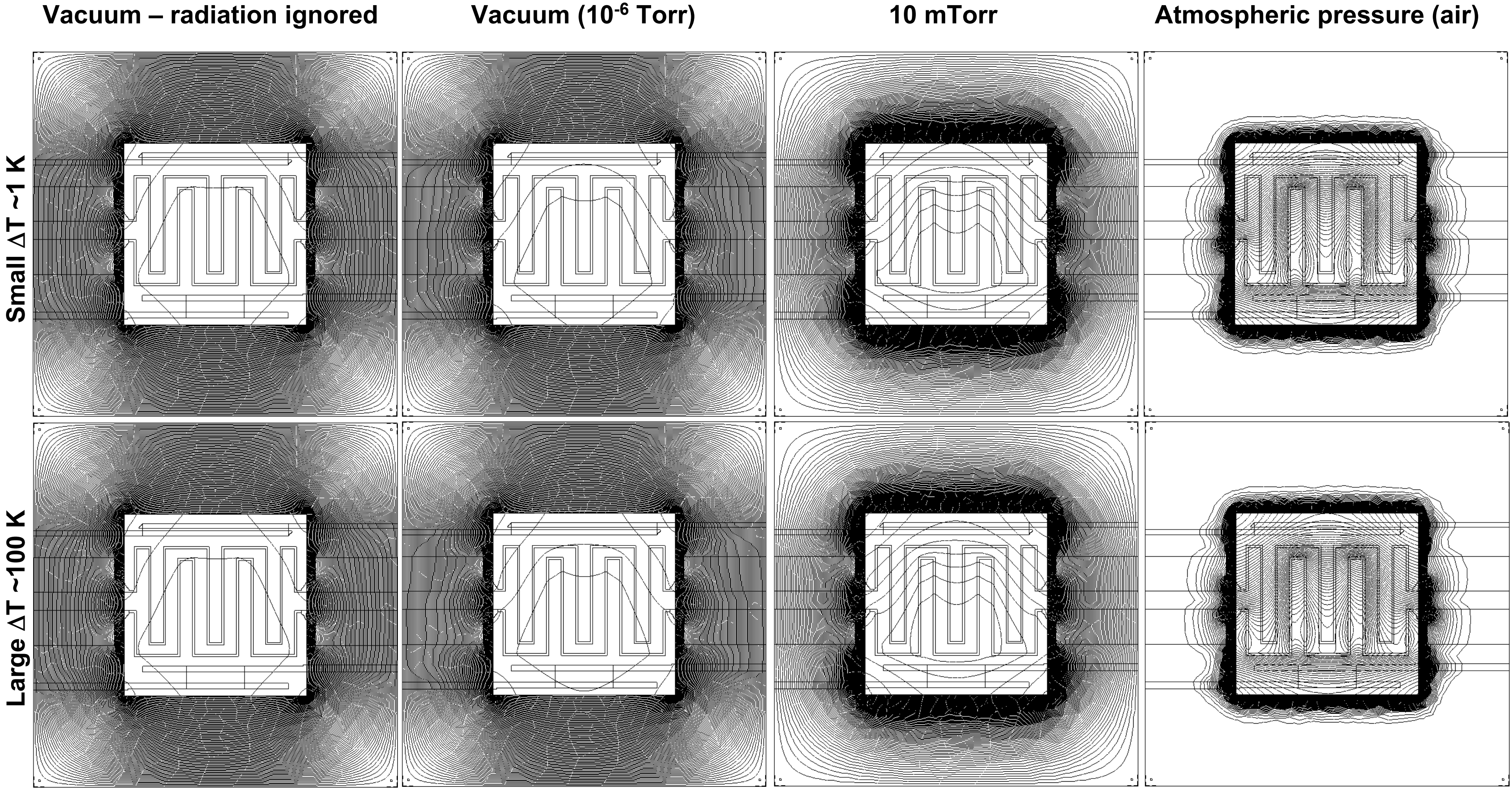
Thermal accommodation coefficient
$\gamma' = 1$ for $N_2$

**P = 10 mTorr h = 0.000374 W/K.cm$^2$**
**P = 1 mTorr h = 0.0000374 W/K.cm$^2$**

*Donald L. Smith, Thin-Film Deposition, Principles and Practice (pp 21-32) McGraw-Hill*

# Simulation results

1% isothermal contours



**Vacuum – radiation ignored**

**Vacuum ($10^{-6}$ Torr)**

**10 mTorr**

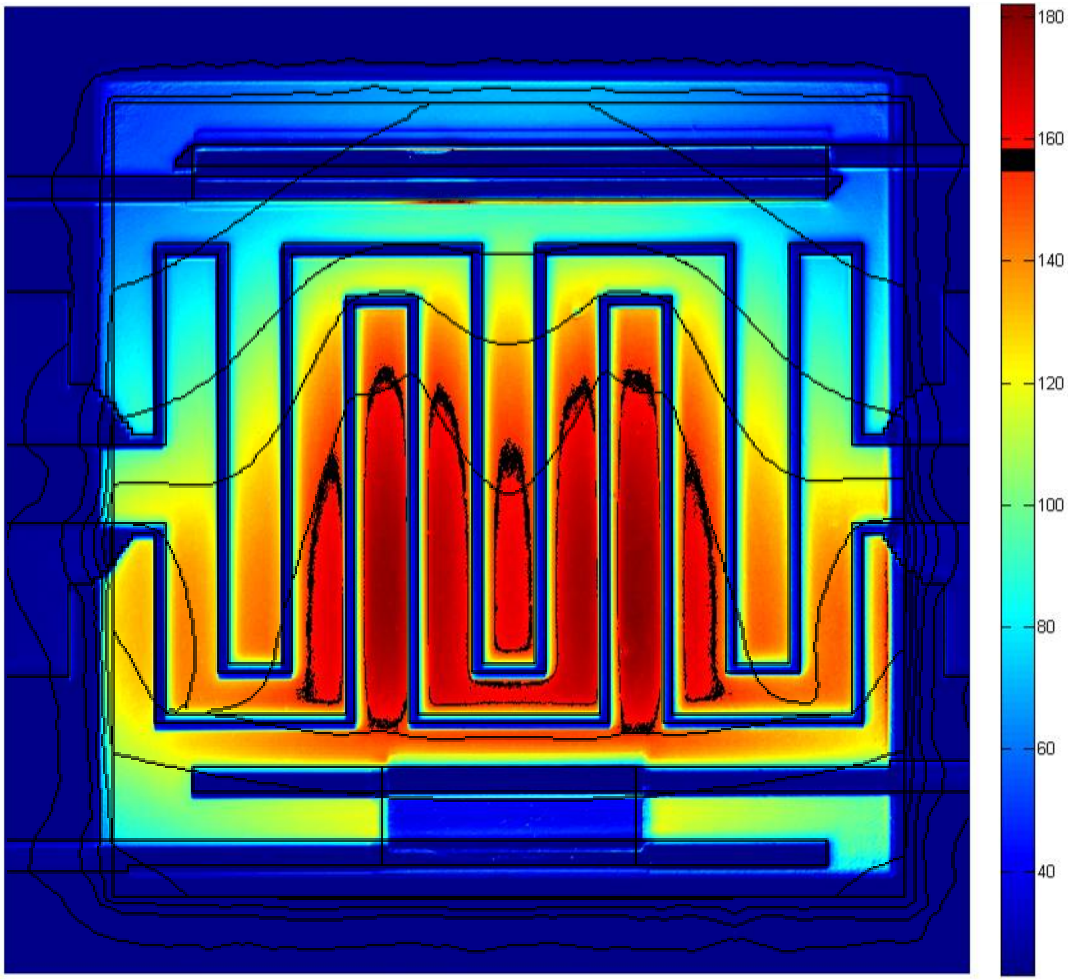
**Atmospheric pressure (air)**

**Small ΔT ~1 K**

**Large ΔT ~100 K**

| | Vacuum – radiation ignored | Vacuum ($10^{-6}$ Torr) | 10 mTorr | Atmospheric pressure (air) |
|---|---|---|---|---|
| **Sample area temperature gradient** | 3% | 4% | 8% | ~50% |

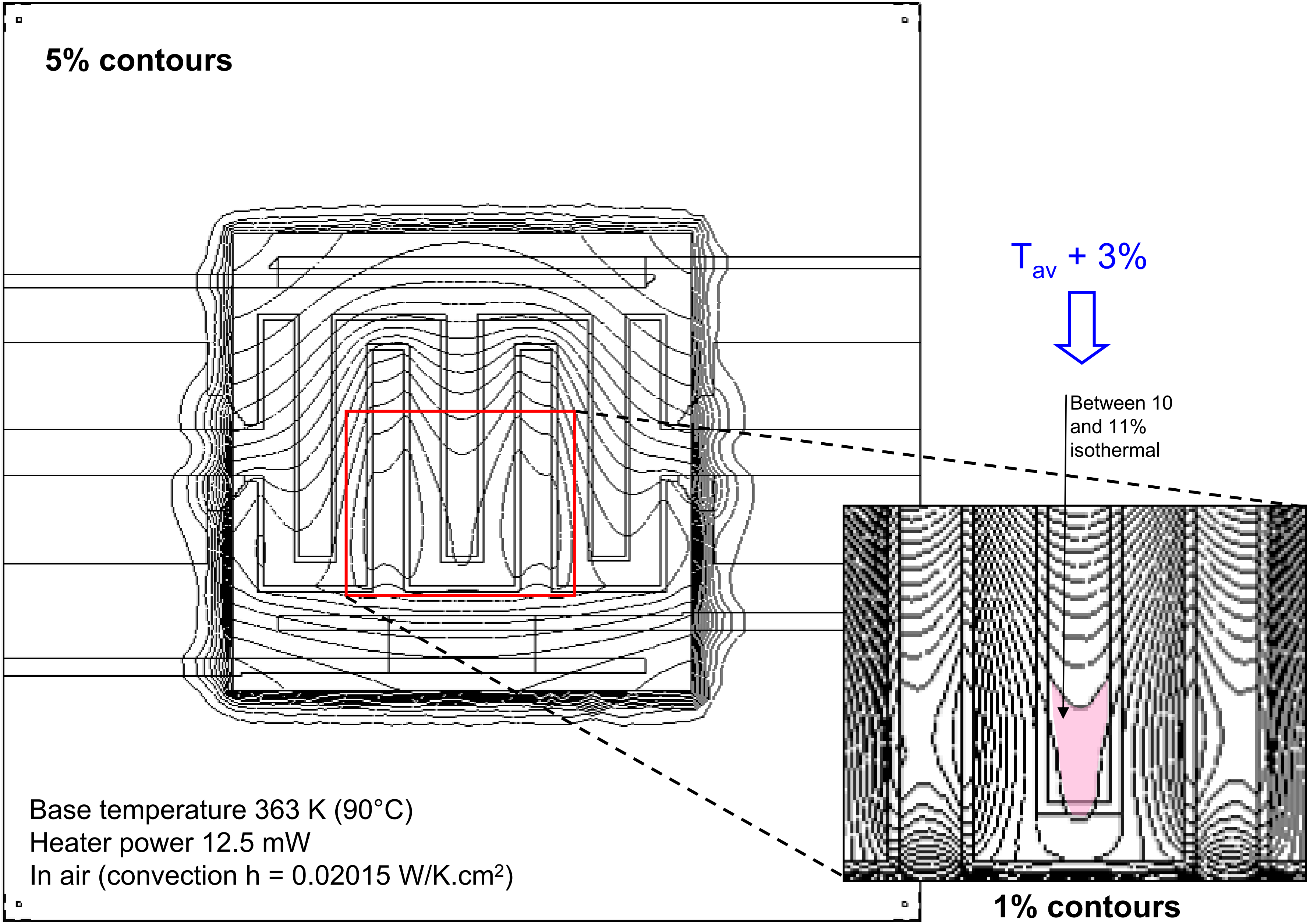
# Comparison simulation/experimental



← 156°C
16% contour

Base temperature 295 K
Convection h = 0.024 W/K.cm$^2$
16% isothermal contours

# Using the simulation to determine temperature map



**5% contours**

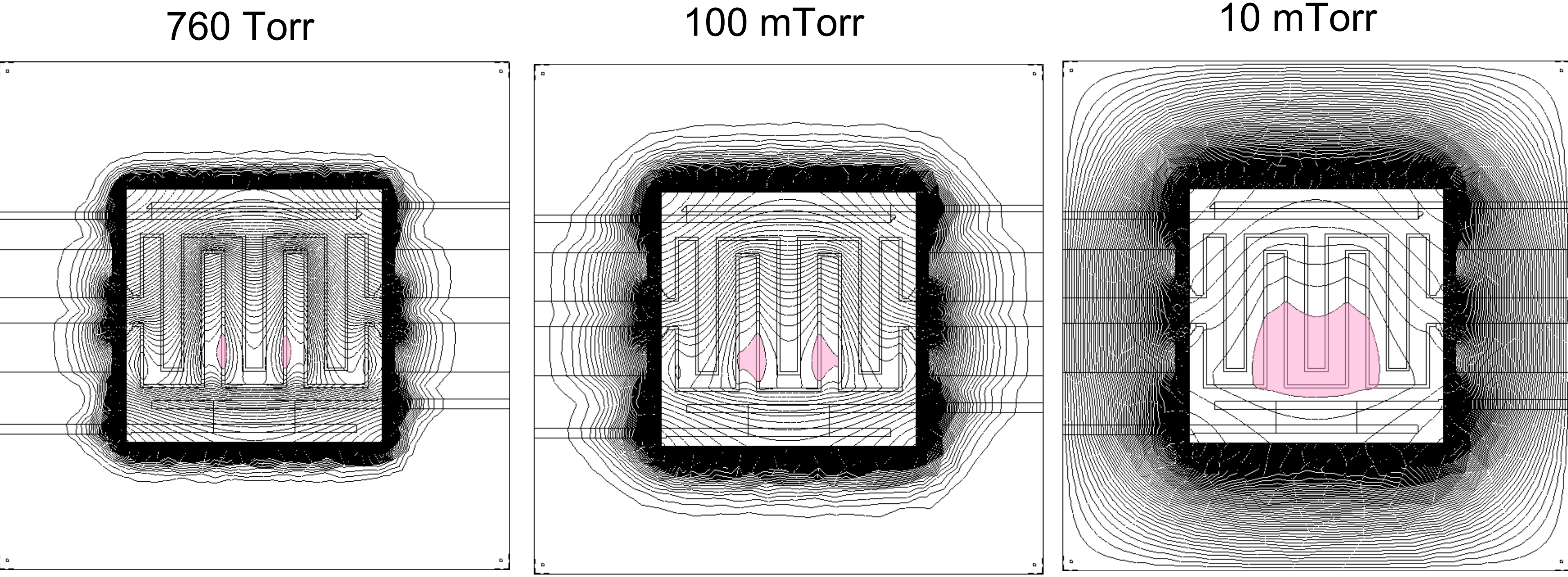
$T_{av}$ + 3%

Between 10
and 11%
isothermal

**1% contours**

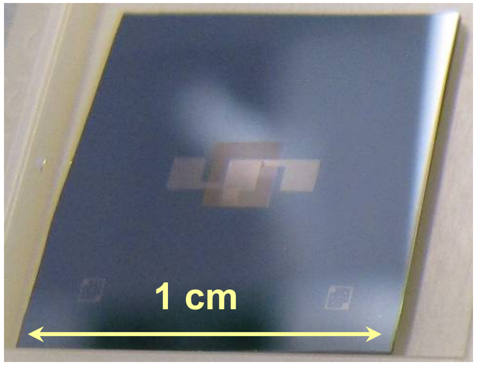
Base temperature 363 K (90°C)
Heater power 12.5 mW
In air (convection h = 0.02015 W/K.cm$^2$)

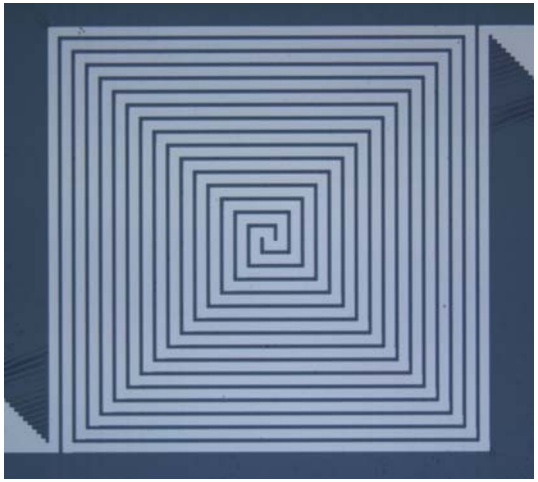
# Effect of reducing pressure

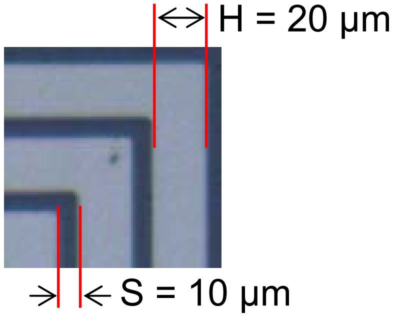


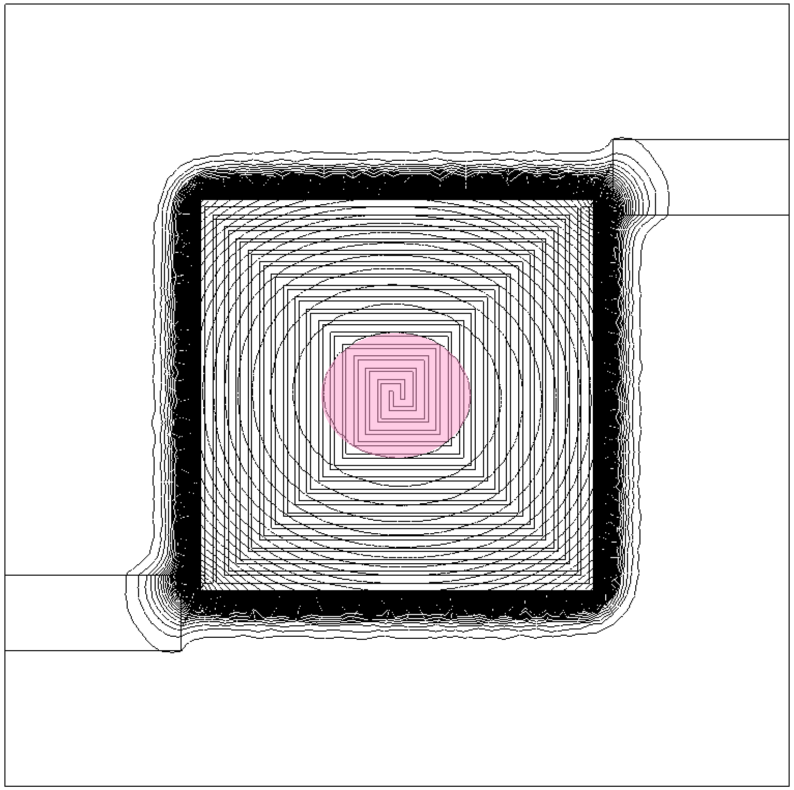
# New heater stage design

- Double Pt spiral
- Heater width H Spacing between heater S









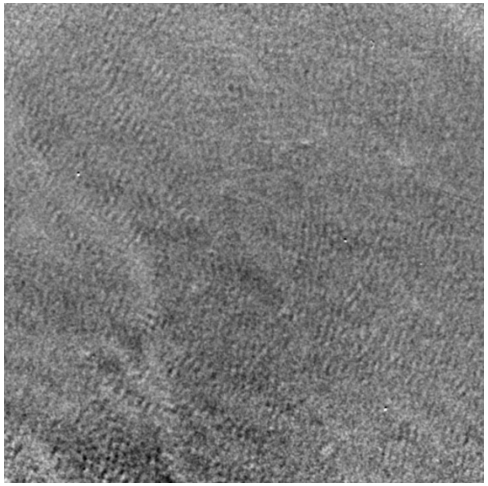
*Simulated 1% isothermal contours*

Large ΔT in air

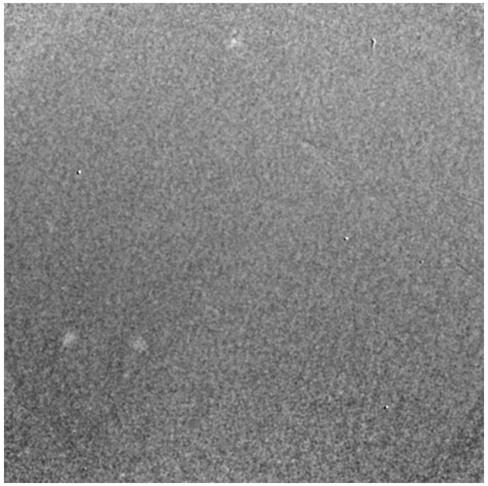
# Proof of principle at XM-1

- Appearance and disappearance of FM in-plane domains in 30 nm Ni film ($T_C$ = 358°C)

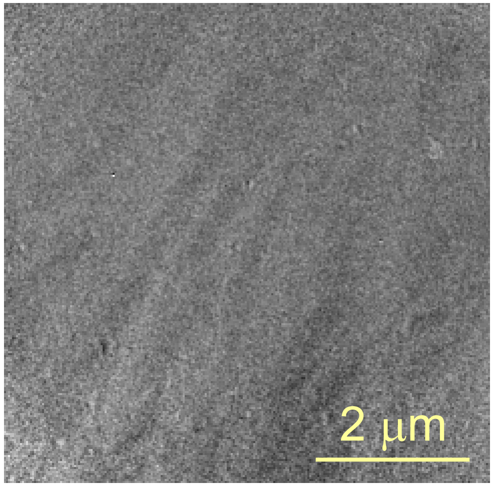
  *Using nanocalorimeter*



~320°C (below $T_c$)



~400°C (above $T_c$)



~320°C (below $T_c$)

- 400 C achieved
- Fast cooling, convenient for cycling

Collaboration with M.-Y. Im and P. Fischer

# Conclusions and future work

- Good knowledge of thermal behavior of nanocalorimeter thanks to:
  - Experimental thermography
  - Heat transfer simulation model
- New improved heater stage design

- Thermal characterization of new design
- Temperature driven magnetic transitions (AFM to FM transition in FeRh)

- Open to collaborations for temperature dependent X-ray microscopy experiments

# Acknowledgements

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  - Frances Hellman
  - Catherine Bordel, David Cooke, Daniel Queen
- Collaborators
  - K. Maize, A. Shakouri (UC Santa Cruz)
  - M.-Y. Im and P. Fischer (LBNL)
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